FINAL

CONSTRUCTION WORK PLAN
FOR
FY16 RECYCLING OF MATERIALS AT RVAAP-50 ATLAS SCRAP YARD AREA OF CONCERN AND SETUP OF TEMPORARY STORAGE FACILITY

Former Ravenna Army Ammunition Plant
Portage and Trumbull Counties, Ohio

December 20, 2016

Contract No.: W912QR-12-D-0011
Delivery Order 0017

Prepared for:

US Army Corps
of Engineers®
BUILDING STRONG®

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REPORT DOCUMENTATION PAGE

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13. SUPPLEMENTARY NOTES

None

14. ABSTRACT

This Construction Work Plan outlines the management and quality control procedures to be implemented during the removal of stockpiled debris piles from RVAAP-50. Included as appendices are the UFP-QAPP, APP/SSHP and Grading, Drainage and Erosion Control Plan.

15. SUBJECT TERMS

Recycling, RVAAP-50, Atlas Scrap Yard, concrete, rail ties, telephone poles, asphalt

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DISCLAIMER STATEMENT

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Comment Resolution Table

Installation: Camp Ravenna/Former RVAAP
Document: Draft Construction Work Plan for FY16 Recycling of Materials at RVAAP-50 Atlas Scrap Yard and Setup of Temporary Storage Facility
Date: 28 November 2016

Cmt. No.	Page or Sheet	Comment	Recommendation	Response
Reviewer(s): Katie Tait, Environmental Specialist, OHARNG, (614)336-6136 or kathryn.s.tait.nfg@mail.mil				
1	Cover Page and throughout	“Ravenna Army Ammunition Plan” - Change ‘Plan’ to ‘Plant’. Insert ‘Former’ prior to ‘Ravenna’. “Ravenna, Ohio” – Change to ‘Portage/Trumbull Counties, Ohio’.		Concur. Changes made as requested. Please note that the current version of RVAAP_SFG_Version 20 presents an example of the Cover Page using the vocabulary presented in the Draft document. A revision may be considered to rectify the discrepancy.
2	Pg 1, Line 11	RVAAP Location and History	Change to Former RVAAP/Camp Ravenna Location and History	Concur. Changes made as requested
3	Pg 1, Line 28	“The RVAAP-50, formerly known as the construction camp…”	Change to “RVAAP-50, Atlas Scrap Yard, formerly known as the construction camp,…”	Concur. Requested change has been made and the revised sentence states, “*RVAAP-50, Atlas Scrap Yard, formerly known as the construction camp, is located on approximately 73 acres in the central portion of Camp Ravenna south of Newton Falls Road and west of Paris Windham Road*”
4	Pg 1, Line 30	“There is no fence around the AOC as a perimeter boundary but the AOC is bounded by…” The site boundary is marked with siebert stakes. Please revise.		Concur. The text has been revised to state, “*The site boundary is marked with siebert stakes, and is bounded by Newton Falls Road to the north and Paris Windham Road to the east.*”
5	Pg 1, Line 41	“The Administrative Record contains the former RVAAP restoration program records….” Delete the word former as the restoration program and the records still exist.	Change to “The Administrative Record contains the RVAAP restoration program records….”	Concur. The text has been revised to state, “*The Administrative Record (AR) contains the RVAAP restoration program records and historical maps for the former RVAAP/Camp Ravenna*.”
6	Pg 2, Line 11	“Mobilize site management, field crews, and heavy equipment to RVAAP on a mutually agreeable date with the ARNG, OHARNG, CELRL, and RVAAP personnel;” RVAAP does not exist as a facility and there are no RVAAP personnel. Please revise.	Change to “Mobilize site management, field crews, and heavy equipment to the former RVAAP on a mutually agreeable date with the ARNG, OHARNG, and CELRL;”	Concur. The text has been revised to state, *“Mobilize site management, field crews, and heavy equipment to the former RVAAP on a mutually agreeable date with the ARNG, OHARNG, and CELRL;”*

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7	Pg 11, Line 1 and 42	Change UFP-QAAP to UFP-QAPP.		Concur. Error has been corrected in both instances.
8	Pg 11, Line 5	"… at the RVAAP."	Change to "… at the former RVAAP."	Concur. Text has been revised to state, *"Mobilization involves the movement of personnel, equipment, and materials to RVAAP-50 at the former RVAAP."*
9	Pg 11, Line 21	"The ERT team will set up a staging area and portable toilet at a location agreed upon by the Contracting Officer's Representative (COR) and RVAAP personnel. Site controls and staging areas will be established to support the project activities. Security access for field crews will be coordinated with RVAAP personnel as the work site is located within a restricted area of RVAAP."	Change to "The ERT team will set up a staging area and portable toilet at a location agreed upon by the Contracting Officer's Representative (COR) and ARNG/OHARNG personnel. Site controls and staging areas will be established to support the project activities. Access for field crews and deliveries will be coordinated with the OHARNG at least 48 hours in advance."	Concur. Text has been revised as suggested.
10	Section 3.6.1 Site Preparation and Support Activities	Will you being conducting any vegetation removal activities? If yes, please detail this in the Work Plan as well.		Clarification. We do not intend to perform any removal of vegetation or falling of tress that are adjacent to the piles, or to gain access to the piles. However, there is vegetation and sampling that have grown through the piles that will need to be removed with the piles of concrete and asphalt. As this is not maintained vegetation we do not call this out in our work plan. The following text has been included in Section 3.6.1, "*We do not intend to perform any vegetation clearance or falling of trees to access the piles of concrete and asphalt. However, vegetation and saplings that have grown through the concrete and asphalt piles will be removed during the removal of the stockpiled material. Any vegetation and/or saplings that are removed will be left in place.*"
11	Pg 11, Line 35	"… coordinated with the RVAAP point of contact Katie Tait…" I don't work for RVAAP. I works for the OHARNG. Please revise.	Change to "… coordinated with Katie Tait…"	Concur. Text has been revised to state, "*All waste characterization sampling and subsequent waste shipments will be coordinated with Katie Tait, OHARNG at (614)-336-6136.*"

12	Pg 11, Line 32	"Prior to the commencement of construction activities, and in accordance with the PWS, waste characterization sampling will be conducted for stockpiled rail tie and timber material to be disposed." Will telephone poles be sampled as well? Please include. Also, as far as I know, the concrete to be recycled is not painted. However, please field confirm. If painted, recommend pulling a sample to characterize. If not painted, no sampling is required.		Clarification. Text has been revised for clarification to state, "*Prior to the commencement of construction activities, and in accordance with the PWS, waste characterization sampling will be conducted for stockpiled rail tie and telephone/timber material to be disposed.*"
13	Section 3.6.3 Loading of Stockpiled Materials	Please also include the telephone poles and the large piece of concrete on Paris Windham Road as part of the materials that need loaded out and recycled/disposed. This was in the SOW.		Clarification. Text has been revised to state, "T*he ERT team will mark the extents of the areas of stockpiled rail ties and timbers, and the extent of stockpiled concrete and asphalt for offsite disposal. This includes the telephone poles and large piece of concrete located on Paris Windham Road. The telephone poles will be live loaded into semi-tractor dump trailers.*"
14	Pg 12, Section 3.6.4 Transportation and Off-Site Disposal/Recycling	Please justify and explain why the railroad ties are being disposed and not recycled.		We looked at various options for disposal, and no local viable sources for recycling/repurposing could be found. When the project initially was bid, there was an opportunity for repurposing, but is now not an option. Additional text has been included stating, "*The potential to repurpose the railroad ties and telephone poles was evaluated during the development of this project. However, there are currently no vendors that are repurposing railroad timbers or telephone poles in the region. Therefore, the opportunity to repurpose the materials was determined to be currently unfeasible.*"

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15	Pg 13, Section 3.6.5 Grading and Restoration	When you grade, please grade gently as we do not want to move the soils around too much due to PAHs and potentially other contaminants being in the soil that will need to be sampled and potentially removed in the future.		We have included grading as a restoration activity that is typical of stockpile loadouts. However, if the OHARNG prefers, we will not grade the areas but rather solely track back and forth on the loadout areas to stabilize any potential loose soil. Text has been revised to state, "*Upon attainment of the removal objectives, the ERT team will compact loose material prior to any re-vegetation that may be necessary. Areas will be compacted to promote future drainage of the area and eliminate potential pooling of water. Multiple passes will be made over disturbed areas to compact the material into place.*"
16	Pg 13, Line 33	"In the event that vegetated areas are disturbed, ERT will prepare a seed bed and seed the area with an appropriate native seed mix, such as annual ryegrass or winter wheat based on the activities being performed in late fall/early winter." Seeding activities need to be coordinated with and approved by the Camp Ravenna Environmental Office. If seeding in January or February we suggest seeding with a finished turf mix in accordance with the Camp Ravenna. 50% Kentucky Bluegrass (Blueridge, Argyl, BlueStar, Ken Blue, Limousine or Baron varieties), 40% Perennial Ryegrasses (Peak, Sonata, Wind Dance, Integra, Morning Star or Shining Star varieties), and 10% Creeping Red Fescue (Lustrous or Boreal varieties) This is the cheapest of our approved mixes		Clarification. Text has been revised to state, "*If vegetated areas are disturbed, ERT will coordinate activities with the Camp Ravenna Environmental Office. We will prepare a seed bed and seed the area with 50% Kentucky Bluegrass (Blueridge, Argyl, BlueStar, Ken Blue, Limousine or Baron varieties), 40% Perennial Ryegrasses (Peak, Sonata, Wind Dance, Integra, Morning Star or Shining Star varieties), and 10% Creeping Red Fescue (Lustrous or Boreal varieties) based on the activities being performed in late fall/early winter.*"

		and we think it will seed and take well for that time of year and into the Spring. Additionally, using an expensive seed mix does not make sense at the area will most likely be disturbed in the future as part of the restoration program. Please revise.		
17	General – Sections 3.6.3 and 3.6.5	In the back of the document it indicates that 1.25 acres will be disturbed. This would require you to get coverage under a general stormwater permit as it would be over an acre of disturbance. How did you calculate this disturbance acreage? I think that your disturbance will mainly be during grading and seeding and again we ask that you be gentle in those restoration activities. I don't think you will be doing much disturbance when loading trucks. We really do not want to get a stormwater permit for this project as it does not seem justified. Please recalculate and reconsider you acreage and disturbance. Please clarify.		Concur. We agree that if the entire areas will not be graded as requested, we do not anticipate any actual land disturbances over 0.25 acres. All other activities will be performed on existing gravel roadways. There is no need for a NPDES permit and ESPCP plan. Text in Section 2.0 of Appendix C has been revised to state, "*ERT estimates that approximately 0.25 acres total will be disturbed during the removal activities; therefore there is no need for a National Pollutant Discharge Elimination System permit or Erosion, Sediment, and Pollution Control Plan. However, best management practices will be utilized during the execution of the project.*"
18	Section 3.6.6 Setup of Temporary Storage Facility	1. Please include design plans or a schematic of the temporary structure (both plan and profile view). 2. OHARNG leadership would like the structure to be located adjacent to the Camp Ravenna Environmental Office Building at the east gate of the facility. Please revise the location. 3. Please show all utility hookup locations and associated ground disturbance (if required) on the plans. 4. How will the building be heated? Natural gas? Propane? Please indicate. 5. Please provide a description of what the		1. Design plans have been included in this RTC and will be included as an appendix to the Work Plan. Please see the revised Figure 3-3 attached to the RTCs depicting the new location for the temporary storage facility, the location of the power drop, and the proposed meter location for the temporary storage location. 2. Location has been revised as requested by ARNG/OHARNG. Please see attached Figure 3-3. 3. Please response to comment 18(1).

		structure will look like. 6. Will there be a meter for electric? 7. Based on the generic plan provided, we have the following comments: -Suggest having an overhead door as the map cabinets are large and will not fit through a standard door. -Suggest removing all interior walls and offices and just having 1 open room for storage. -No bathroom is required. -Please provide the height of the ceiling. -Will shelving be provided? Need for shelving was discussed at the prebid meeting. If yes, please detail?		4. Please see response to comment 18(1). The unit is heated with electricity. 5. Please see response to comment 18(1). 6. Sentence in Section 3.6.6 revised to state, "*A power drop and electrical meter will be installed from the nearest powerline and transformer (identified in **Figure 3-3**).*" 7. A) As discussed on December 9, 2016, ERT will disassemble maps cabinets if necessary to get them through the doors. Map drawers will be removed as necessary to prevent disturbance of maps. B) As discussed on December 9, 2016, based on availability of rental units, the unit will have a layout as shown in attached figures, except there will be no bathroom. C) Noted, the unit will not have a bathroom included. D) Please see response to comment 18(1). The ceiling is 8 feet tall. E) Sentence has been included in Section 3.6.6 stating, "*Metal-wire or HDX shelving units will be provided for additional storage. Shelving units will be approximately 18" deep, 48" wide, and 84" tall.*"
19	Pg 14, Line 6	"All temporary support facilities will be removed during demobilization, including temporary erosion control measures. The ERT team will remove the erosion and sediment controls in place when the vegetative cover is established or the soil has been stabilized."		Concur. Text has been revised to state, "*The ERT team will remove the erosion and sediment controls in place when the vegetative cover is 75% established.*"

		Erosion control measures must be left in place and inspected until a 75% vegetative coverage is established. Please revise.		
20	Pg 15, Figure 3-1	1. Change Ravenna Army Ammunition Plant for Former Ravenna Army Ammunition Plant. 2. Please include location of telephone poles and concrete on Paris Windham Road. 3. Recommend that the trucks use Paris Windham Road and South Service Road, not Newton Falls Road. The turn out of Atlas from the railroad ties will be much easier to the right than the left. Please revise. 4. Please verify that all of your routes are clear of debris and that no vegetation clearing is required for access.		1-3. Concur the requested changes have been made 4. During a site visit performed on 11/8, it was confirmed that all routes are free of debris and no vegetation clearance is required for access.
21	Figure 3-3	See prior comment about location and revise figure. Also show the utility hookups.		Clarification. The figure has been revised to show the newly requested location. Pending notification that the utility pole closest to the location (outside the fence along Route 5) can be used for the drop, we will show this as the utility hook-up location.
22	QAPP Worksheet 1 and 2	Delete 'Former' prior to Ravenna Army Ammunition Plant Restoration Program. Under Project Stakeholders, delete Property management under ARNG. Property management is conducted by OHARNG through the USP&FO.		Concur. Text has been revised to state, "*FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility, Ravenna Army Ammunition Plant Restoration Program*" Concur. Text has been revised to state, "*Restoration Program Management*"
23	QAPP Worksheet 11	"The former RVAAP-50 AOC was utilized as a load, assemble, and pack facility for munitions." This is incorrect. RVAAP-50 was used as a		Concur. Text has been revised to state, "*The former RVAAP-50 AOC was utilized as a construction camp and to store stockpiled materials.*"

		construction camp and was also used to stockpile materials. Please revise.		
24	QAPP Worksheet 17 Solid Waste Characterization	Will telephone poles be sampled as a composite? Recommend analyzing separately as waste stream is different.		Concur. Text has been revised to state, "*Chip samples will be collected from a minimum of 8 locations along the stockpiled rail ties pile and chip samples will be collected from a minimum of 8 locations along the telephone poles/timbers pile. Each set of chips will be composited independently into the sample containers.*"
25	QAPP Worksheet 17 Sample Container Labeling, Storage and Shipment	A chain of custody was not mentioned in the process. Please include.		Clarification. Sample custody is addressed in QAPP Worksheets #26 and #27 – Sample Handling, Custody, and Disposal. Text on page 15, line 39 has been revised to state, "*Worksheet #26 & #27 provides unique labeling guidance and chain-of-custody (CoC) procedures.*"
26	APP, Pg 2, Line 10	Change Former Camp Ravenna Army Ammunition Plant to Former Ravenna Army Ammunition Plant.		Concur. Text has been revised to state, "*This APP applies to intrusive field investigation activities occurring at the Former Ravenna Army Ammunition Plant in Portage and Trumbull Counties, OH.*"
27	APP, Pg 22, Section 2 Spill Plans	Please be advised that all spills are reported to Camp Ravenna and are called into Camp Ravenna Range Control. Please utilize the Camp Ravenna First Responder Form and reference and provide it in this document.		Concur. Text has been included stating, "*Any spill of petroleum products over 25 gallons, or a spill of any amount into a body of water, must be reported to the OHARNG Environmental Office immediately. All other spills will be reported in writing on the OHARNG Spill Report Form (included within* ***Attachment 4*** *of this APP) to the OHARNG within 24 hours.*"
28	APP, Table 2 Emergency Contact Phone Numbers	1. Do not call 911 if there is an emergency. Please contact Camp Ravenna Range Control at 614-336-6041 in the event of an emergency who will coordinate emergency services. Please revise.		1. Emergency Services has been revised to "Range Control" and the telephone number has been revised to "614-336-6041." 2. Yes. 3. Both USEPA and OHARNG have

		2. Spill Response – Chemtrec – Do you have a contract with Chemtrec so you can use them for spill response? 3. You are not to contact the USEPA Response Center or the Ohio EPA hotline or Project Coordinator. Once your spill is called into Range Control, the Camp Ravenna Environmental Office will be notified and will contact outside agencies. Please revise. 4. Pat Ryan and Gail Harris are not Emergency Contacts. Please remove them from this table. 5. Please also include Kevin Sedlak on this Contact List.		been removed Emergency Contact Phone Numbers Table 4. Pat Ryan and Gail Harris have been removed from the Emergency Contact Phone Numbers Table 5. Kevin Sedlak has been added to the Emergency Contact Phone Numbers Table
29	APP, Figure 1 Site Location Map	Please also include the concrete location on Paris Windham Road and the telephone pole location as well.		Concur. Location of concrete and telephone pole has been added to Figure 1.
30	SSHP, Table 1 Project Personnel	Please also include Kevin Sedlak.		Concur. Kevin Sedlak has been included in the Project Personnel Table.
31	SSHP, Table 6 Emergency Contacts	Please see prior comments on the APP Emergency Contact Table. Please revise.		Concur. Please see response to comment #28
32	SSHP - General	We have a map with designated medical transfer points. Please include this map and call out the closest transfer point.		This map (as presented in the Camp Ravenna Access Guidelines that were provided as part of the pre-bid package) has been included as Enclosure E, and referenced within the text of the SSHP, Section 11.2, “Within RVAAP, there are designated medical transfer points (see map provided in **Enclosure E**). The closest medical transfer point to the project site is located at the intersection of Old Newton Falls Road and Paris Windham Road.” If a higher resolution copy of this map is available, please provide, and ERT will

				incorporate. For field use, however, knowing that the medical transfer point closest to the site is at the intersection of Old Newton Falls Road and Paris Windham Road will suffice.
33	SSHP, Pg 48, Line 4	Trauma Center is mentioned in Middleburg Heights. This is incorrect. Please revise.		Concur. The closest Level I trauma center to the site is Akron General Medical Center in Akron, Ohio. Applicable text has been updated.
34	Appendix C Grading, Drainage and Erosion Control Plan	Please see prior comments about amount of ground disturbance. Please recalculate and reconsider how much acreage is being disturbed. The erosion control measures in this section should be considered Best management practices as a SWPPP and permit is hopefully not required. Erosion control measures should be inspected and stay in place until 70% coverage is reached. What inspection form for erosion control will be used? Please provide. The condition of the site must be approved by USACE, ARNG and OHARNG before erosion control measures are removed.		Concur. Please see response to comment #17. A NPDES permit will not be required to complete the project objectives. Section 3.1, bullets #4 and #5 have been revised for clarification to state, “ “*4. Plant the any disturbed areas with 50% Kentucky Bluegrass (Blueridge, Argyl, BlueStar, Ken Blue, Limousine or Baron varieties), 40% Perennial Ryegrasses (Peak, Sonata, Wind Dance, Integra, Morning Star or Shining Star varieties), and 10% Creeping Red Fescue (Lustrous or Boreal varieties) to promote the establishment of the vegetative cover.* *5. Temporary erosion control BMPs will be left in place until the vegetative cover is 75% established, at which point they will be removed upon inspection and approval by CELRL, ARNG, and OHARNG.*” The following text has been revised in Section 3.1 to state, “*The Site Superintendent will inspect the erosion and sediment control measures daily during construction activities, as well as after each runoff producing rainfall event, using the Erosion Control Inspection Form presented as* ***Enclosure***

				1," and the form as an Enclosure to the GD&E Plan and provided as part of the RTCs.
35	Appendix C Grading, Drainage and Erosion Control Plan, Figure 3-1	Where will erosion control measures be placed? How can you have silt fence all the way around each of these areas and still access the site? Please clarify.		Figure 3-1 has been revised to show open sections of silt fence.
36	Appendix C Grading, Drainage and Erosion Control Plan, Pg 9, Line 3	"Waste materials will be collected and stored by site personnel." What does this mean? Is this for municipal trash? Please clarify.		Clarification. Text has been revised to state, "*Municipal waste materials will be collected and stored by site personnel in appropriate waste containers bins.*"
Reviewer(s): Kevin Sedlak, Restoration Project Manager, ARNG, (614) 336-6000 ext 2053 or kevin.m.sedlak.ctr@mail.mil				
KMS 1	Page 12 line 3	Suggest changing from Express mail to overnight shipping.	This does not match what is in the QAPP.	Concur. Text has been revised to state, "*After sample collection, the samples will be shipped via overnight mail to:*"
KMS 2	Page 12 line 12	Was recycling of the RR ties taken into consideration as opposed to just landfilling them?	Provide the cost of recycling and the cost of disposal for comparison.	Recycling/repurposing of ties was taken into consideration. One vendor was available at the time of the bid, however there are currently no known vendors that will recycle/repurpose rail ties of this age. We did however have discussions with American Landfill Management, specifically Steve Kipler, who was interested in using the site as a pilot test for a new technology that stabilizes and/or destroys the chemical constituents so that it can be recycled. He is collaborated with Univ. of Akron, and thought that he could have the paperwork approved within 6 months. However, this would not allow for us to complete the project within the PoP, and it would also likely then provide opportunity for additional regulatory involvement. If the government is interested in this approach, we are open to discussing it further.

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KMS 3	Page 12 line 28	Remove the word “from” it is repeated.		Concur, Text revised to state, “*The ERT team intends to progress the load-out from the north to south along the stockpiles of material.*”
KMS 4	Page 13 line 33	Camp Ravenna has a specific seed mix one for shade and one for sunny areas these seed mixes will be used only.	Change text to Camp Ravenna seed mix will be used.	Concur. Please see response to comment #13
KMS 5	Page 14 line 15	There is a more recent document guide available from Vista Sciences.		Clarification. ERT was provided with the 2012 SFG document upon award of the TO. Please forward the more recent version for review and incorporation. Thank you.
KMS 6	Figure 3-1	The large piece of concrete located alongside Paris Windham Rd is not denoted on the figure.	Add this to the figure	Concur. Please see response to comment #20.
KMS 7	Page 4 line 29 – 35 APP	All utility checks need to be coordinated through the facility DPW office at Comp Ravenna along with OUPS.		Concur. Text has been revised to state, “*At least 72 hours prior to intrusive field activities, ERT will coordinate through the Camp Ravenna Department of Public Works and local utility companies (via Ohio Utilities Protection Services or 800-362-2764) to mark-out and identify all underground utilities in the investigation areas.*”
KMS 8	Page 20 line 22 APP	If there is an accident that requires emergency services of any kind Range Control will be notified first and immediately. Range control will dispatch emergency responders to your location.	Range Control numbers are 614-336-6041 and a cell number is 614-202-5783. Add this information to your APP and HASP.	Concur. Text has been revised to state, “*In the event of an accident that requires off-site treatment, or any incident that could bring adverse attention or publicity to the U.S. Army, the ERT PM will notify Range Control immediately, followed second by notifying the CELRL COR/TM and/or CELRL PM.*”
KMS 9	Page 22 line 18 APP	Any spills no matter the size must be immediately reported to Range control.	Add this to the APP and HASP.	Concur. Text has been revised starting at line 27 to state, “*There is a possibility of fuel spills during equipment/vehicle refueling. Any spills no matter the size must be reported to Range Control.*”
KMS 10	Page 23 Table 2 APP	Remove from the table 911 if you were to call it is unknown which city may get the 911 call replace with range control numbers. Remove spill response and		Concur. Please see response to comment # 28

		replace with Range Control. Remove U.S. EPA number and replace with Range Control. It is the facilities responsibility to notify the EPA of any spills. Remove OEPA contacts the contractor is not permitted to contact the OEPA this is the responsibility of the Restoration program. Replace animal control with Range Control.		
KMS 11	Page 28 line 14 APP	There is one large chunk of concrete along Paris Windham Rd this will require the road to be closed while this is removed.	Add this to the APP.	Concur. Text within Section 9.y. will be revised to state, "*Due to proximity of a limited number of stockpiles adjacent to Paris Windham Road, field personnel may also be exposed to hazards associated working near moving vehicles during the project. To control these hazards, the following safety requirements will be strictly enforced:* ▪ *The work area will be cordoned off with safety cones, and a sign will be placed, per the example (or equivalent) provided in Enclosure B of the SSHP;* ▪ *In the unlikely event that road closure is necessary, ERT will work with Fort Benning personnel;* ▪ *Class II Safety Vests (fluorescent yellow-green) will be worn by site personnel when the following applies:* o *Exposure to vehicular traffic at speeds up to 45 mph;* o *Limited visibility of workers due to mobile heavy equipment operations, vehicles, load handling, or other hazardous activities;* o *Reduced visibility conditions due to inclement weather; and/or* o *No protective barriers when*

				near vehicular traffic.”
KMS 12	Page 2 line 9 HASP	This is an incorrect statement. The RR ties may have arsenic creosote and other preservative chemicals used in the 1940s. The soil in and around the Atlas Scrap yard contains elevated levels of SVOCs, PAHs and lead.	You may want to review documents for Atlas Scrap yard that are available on RVAAP.ORG.	Concur. Text within Section 1.2 has been revised to state, “*The railroad ties are suspected to potentially contain arsenic, creosote, and/or other preservative chemicals that may have been present during the time of their manufacture (1940’s). Additionally, soil in and around the Atlas Scrap Yard may contain semi-volatile organic compounds, polycyclic aromatic hydrocarbons, and/or lead, due to historical usage of the area. As site operations, will not involve digging into the soil, there will not be any exposure to constituents of concern (COCs) present in soil during this project. As railroad tie material, will be handled via heavy machinery and handled in such a way that debris remains in-tact, any COCs present in the railroad tie material will not become friable, and as such, there will not be any exposure to COCs from handling railroad ties.*”
KMS 13	Page 4 line 23 HASP	All emergency calls go to Range Control.	Replace all 911 call information with Range Control throughout all documents.	Concur. Please see response to comment # 28 as the same changes have been made to this table.
KMS 14	Page 6 line 36 HASP	The 1000 yards number is different from other parts of the document.	Make all of the sampling consistent throughout all of the documents.	Concur. Text has been revised to state, “*However, ERT team will submit one composite soil sample of the railroad tie material and telephone pole material scheduled for offsite recycling/disposal*.”
KMS 15	Page 12 line 9 HASP	Any situation with animals you are to call Range Control. Twinsburg is 45 minutes away and has nothing to do with RVVAP.		Concur. Text has been revised to state, “*If it is necessary to use or move an object where an animal is hiding, the SSHO/Site Superintendent may have to notify the Range Control. Range Control should be contacted to aid or subdue an animal that may cause a risk to workers (i.e., a raccoon).*”

Comment Resolution Table

Installation: Camp Ravenna/Former RVAAP
Document: Draft Construction Work Plan for FY16 Recycling of Materials at RVAAP-50 Atlas Scrap Yard and Setup of Temporary Storage Facility
Date: 28 November 2016

KMS 16	Page 37 line 20 HASP	Any spill will be immediately reported to Range Control.		Concur. Text has been revised to state, “*There is a possibility of fuel spills during equipment/vehicle refueling. Any spills no matter the size must be reported to Range Control*.”
KMS 17	Page 43 line 33	Remove “local Emergency Medical Services will be notified immediately” ;	Replace with Range Control will be called.	Concur. Text has been revised to state, “*For any injury requiring care beyond First Aid, the Range Control will be notified immediately; contact numbers are provided in* ***Table 6***.”
KMS 18	Page 46 line 24	Remove calling 911 replace with calling Range Control.		Concur. Text has been revised to state, “*Emergency response services will be activated by Range Control from landline phone or from a mobile phone.*”
KMS 19	Page 48 line 2	Portage Medical Center is a level 3 trauma Center as is Southwest General Hospital. A level 1 trauma center is at Akron General which is closer than Southwest General.	Review your data to be sure this is correct.	Concur. Southwest General Hospital has been replaced globally with Akron General Medical Center.
Reviewer(s): USACE-LRL				
1	General	Greg Moore is no longer USACE PM. Please replace with Craig Coombs (502-315-6324). See page vii, page 5 of Appendix A, page 24 of Appendix B, page 17 and 48 of Attachment 2 of Appendix B, and anywhere else USACE PM appears.		Concur. “Greg Moore” has been switched out for “Craig Coombs” uniformly across the documents. Please thank Greg for his support on this TO.
2	SSHP Page 49	Step three says to turn Left onto Pa St 1 to get to OH-5 West. In other words, exit installation on Pa St. The Main Gate is on George Road. Please correct street name. Also, add a statement into this section, that turn-by-turn directions (including street names and distances) will be verified by driving the route prior to beginning field work. If necessary pen and ink changes to SSHA can be made for copies in the field.		Concur. Step 3 has been revised to state, “*3. Turn Right onto Pa St. to exit the Installation via George St. 1, 0.3 mi.*” Text has been added to the end of the third paragraph of Section 11.6 to state, “*Turn-by-turn directions (including street names and distances) will be verified by driving the route prior to beginning field work. If necessary, pen and ink changes to this SSHP can be made to copies being used in the field.*”

MODEL 2464F0-1
APPLE LEASING
2464 OFFICE
23'-6" X 60'-0" ACTUAL SIZE
BUILDIN LOCATION: OHIO

FRAME

I-BEAM:	12" JR. I BEAM (PERIMETER TYPE)
CROSS MEMBER:...	10 X 8 I-BEAM AT 48" O.C.
HITCH:.........	DEMOUNTABLE WITH 2-5/16" COUPLER & ELEVATING JACK
AXLES:.........	QUAD 6000# WITH ELECTRIC BRAKES
TIRES:.........	8:00 x 14.5 10 PLY
PAINT:.........	MINIMUM 3 MIL BLACK ASPHALT
MISCELLANEOUS:.	N/A

FLOOR

BOTTOM BOARD:..	WOVEN PLASTIC VAPOR BARRIER TYPE BOTTOM BOARD
INSULATION:....	R-11 (3 1/2") UNFACED FIBERGLASS
JOISTS:.......	2X6 #2SPF EQUAL OR BETTER @ 16" O.C. LONGITUDINAL
PERIMETER JOIST:........	2X6#2SPF EQUAL OR BETTER (DOUBLE @ MATING LINE)
DECKING:.......	SINGLE 5/8" T&G PLYWOOD W/16" O.C. SPAN INDEX
COVERING:......	1/8" VINYL TILE COLOR: #51839 FORTRESS WHITE

EXTERIOR WALLS

STUDS: ....::.	2X4 STUD GRADE SPF EQUAL OR BETTER @16" O.C.
BOTTOM PLATE: :	SINGLE 2X4 #3SPF EQUAL OR BETTER
TOP PLATE:	DOUBLE 2X4 #3SPF EQUAL OR BETTER
WALL HEIGHT:	8'-0"
WALL COVERING:	1/2" VINYL COVERED GYPSUM (CLASS A F.S.) COLOR: HAMPTON GRAY
INSULATION:	R-11 (3 1/2") KRAFT BACKED FIBERGLASS
SHEATHING:	1/8" STRUCT. THERMAL-PLY
SIDING:	.019 VERT. ALUM.
SIDING COLOR: :	COLONIAL WHITE - SMOOTH
TRIM:	.019 ALUMINUM
TRIM COLOR: ...	BRANDYWINE - RCE
FRONT DESIGN: :	SQUARE

INTERIOR WALLS

STUDS: ....::.	2X4 STUD GRADE SPF EQUAL OR BETTER @ 16" O.C.
BOTTOM PLATE: :	SINGLE 2X4 #3SPF EQUAL OR BETTER
TOP PLATE:	DOUBLE 2X4 #3SPF EQUAL OR BETTER
WALL HEIGHT: ..	8'-0" ACTUAL HEIGHT
WALL COVERING:	1/2" VINYL COVERED GYPSUM (CLASS A F.S.) WASHABLE COLOR: HAMPTON GRAY

ROOF

RAFTERS:	MONO SLOPE TRUSSES @16" O.C., 30# LIVE LOAD 24" RIDGE, 5 3/4" HEEL X 137 7/8" LONG TRUSS#J0005T0220
MATE BEAM:	1-PLY 24" HIGH 1 3/4" MICROLAM PER PRINT 2X3 ROOF RAILS PER PRINT
CEILING PANEL:	1/2" PRE FINISHED GYPSUM (CLASS A F.S.)
INSULATION: ...	R-19 (6") W/VAPOR BARRIER
SHEATHING: .:..	7/16" OSB BOARD W/ 16/24 SPAN INDEX W/1/4" DENS DECK
ROOFING:	.045 EPDM BLACK RUBBER FULLY ADHERED (CLASS C)
ROOF VENTS: ...	(2) POWER TO OPERATE AT 60% RELATIVE HUMIDITY
EAVE VENTS: ...	(4) 14"X8" END VENTS
TIE DOWNS:	(4) OVER THE ROOF TYPE EACH MOD.
MISC.:.....	STD. J-RAIL

TRIM PACKAGE	DELUXE TRIM AT DOOR & WINDOWS W/MATCHING BATTENS & 4" VINYL COVE THRU-OUT (6" COVE IN R.R.) COLOR: TOFFEE
EXTERIOR DOORS	(2) 36" X 80" STANDARD DOOR W/10 X 10 SAFETY GLASS WINDOW , LEVER L.SET, WEATHER STRIPPING & THRESHOLD
INTERIOR DOOR	(5) 36" X 80" PRE FINISHED HOLLOW CORE DOORS WITH PINE JAMBS (4) PASSAGE & (1) PRIVACY LEVER HANDLES

GENERAL NOTES

THESE PLANS SHOULD NOT BE SCALED FOR DIMENSIONAL REFERENCE. ALL DIMENSION LINES AND NOTES SUPERCEDE ANY SUCH REFERENCES.

C&B SHALL NOT RESPONSIBLE FOR LOCAL CODES OR PERMITS.

ALL NOTES PERTAINING TO "IN FIELD, ON-SITE, OR BY BUILDER" SHALL BE THE RESPONSIBILITY OF THE CONTRACTOR.

WINDOW	(10) 46" X 27" HORIZ. SLIDER WINDOW WITH MILL FINISH ALUM. FRAME SINGLE GLASS, TRIM AND INSECT SCREEN & BLINDS

ELECTRICAL

SERVICE:	120/240 VOLT SINGLE PHASE, 3 WIRE , 60Hz. WITH GROUND
LOAD CENTER: ..	(2) 125 AMP 240 VOLT SINGLE PHASE SUB PANEL (M.D.P. BY OTHERS AT SITE)
ENTRANCE: :.:..	1 1/2" EMT CONDUIT THROUGH FLOOR
RACEWAY:	MIN# 12 ROMEX
LIGHTS:	(29) TWO TUBE FLUORESCENT WITH DIFFUSER (SURFACE MOUNT) (2) 60 WATT WEATHER PROOF INCANDESCENT EXTERIOR PORCH LIGHT
RECEPTACLES: ..	(21) 125 VOLT 20 AMP IVORY DUPLEX GROUNDING TYPE (1) GFCI 125 VOLT 20 AMP IVORY DUPLEX GROUNDING TYPE (1) 125 VOLT WEATHER PROOF DUPLEX GROUNDING TYPE FOR HEAT TAPE
SWITCHES:	(9) 120 VOLT IVORY TOGGLE TYPE
EXHAUST FAN:	100 CFM CEILING EXHAUST FAN W/ROOF CAP
ROOF VENTS: ...	(2) POWER TO OPERATE AT 60% RELATIVE HUMIDITY EXTERIOR METAL TO BE GROUNDED PER N.E.C.

SITE WORK

SITE WORK

PLUMBING

SUPPLY LINES: :.:..	TYPE "L" COPPER & CPVC
WASTE LINES:	PVC
WATER HEATER: .:....	(1) 240 VOLT ELECTRIC INSTANTANEOUS (EEMAX) W/LOCK OUT AT EP
WATER CLOSET: ...:..	(1) WHITE CHINA TANK W/1.6 GAL. FLUSH, ELONGATED BOWL & OPEN SEAT
LAVATORY: .:..:.	(1) WHITE CHINA WALL HUNG W/FAUCET
GRAB BARS: .:..::	(1) SET OF 42" & 36"
ACCESSORIES:	(1) TOILET PAPER HOLDER (1) SOAP DISH (1) MIRRORS

H.V.A.C.

HEATING: :.....	(2) 15 KW 240 VOLT ELECTRIC RESISTANCE HEAT STRIP
COOLING:	(2) 36,000 BTU 240 VOLT SINGLE PHASE BARD AIR CONDITIONER
DUCTS: ...:....	6"X16" FIBERGLASS DUCT BOARD IN CEILING
DIFFUSERS:	(16) 8"X8" WHITE ADJUSTABLE
RETURN AIR:	GYP. LINED PLENUM WALL W/ (2) 30X16, (2) 30X8, & (2) 14X8 GRILLES

MISCELLANEOUS	ALL MATERIALS ARE C&B CUSTOM MODULAR STANDARD UNLESS OTHERWISE NOTED

SITE WORK

ALL DOOR ADJUSTMENTS ARE ON SITE BY OTHERS

C&B CUSTOM MODULAR IS NOT RESPONSIBLE FOR LOCAL CODES OR PERMITS

BUILDING ACCESSIBILITY PROVIDED BY OTHERS AT SITE BY MEANS OF RAMP

ADEQUATE HANDICAP TOILET FACILITES TO BE PROVIDED AT SITE ON SAME PROPERTY AND ARE CAPABLE OF WITHSTANDING THE INCREASED OCCUPANT LOAD.

BOTTLED WATER PROVIDED BY OTHERS AT SITE

SERVICE SINK PROVIDED BY OTHERS AT SITE

THIS BUILDING IS APPROVED FOR PLACEMENT IN OHIO ONLY WHEN THE FOLLOWING OCCURS:

1) BUILDING OFFICIAL HAS DETERMINED THAT PLUMBING FIXTURES TO BE INSTALLED AT SITE.
2) OR THE PROVISIONS OF SECTION 2902.1 ARE OTHERWISE MEET
3) OR A VARIANCE FROM SECTION 2902.1 HAS BEEN GRANTED

*

✳ DENOTES DRAWING CONTAINING WORK TO BE ACCOMPLISHED AT THE BUILDING SITE

DESIGN CRITERIA:

OCCUPANCY GROUP "B"
CONSTRUCTION TYPE V-B
1410 SQUARE FEET
OCCUPANT LOAD: 14

ROOF LIVE LOAD:..........30 psf
ROOF DEAD LOAD::..:..:.10 psf
FLOOR LIVE LOAD::..:..::.50 psf 100 pfs in common areas
FLOOR DEAD LOAD:;.....:7psf
WIND SPEED:.......90 MPH 3-SEC. GUSTS
WIND EXPOSURE:......:.."C"
GROUND SNOW LOAD:.......25 psf
SEISMIC ZONE:.......Ss = 30%g S1 = 8.4%g Site Class D

CODES:

2002 NATIONAL ELECTRICAL CODE
2002 OHIO BUILDING CODE-BUILDING
2002 OHIO BUILDING CODE-MECHANICAL
2002 OHIO BUILDING CODE-PLUMBING
1999 ASHRAE 90.1 ENERGY CODE
ADA HANDICAP GUIDLINES

SITE WORK

Building Notes:

The job specifications supersede the detail specifications where there is a conflict. Building to be located greater than 10' from existing property line. Buildings on the same property shall be assumed to have a property line between them. The building exterior and the facilities within the building shall be identified with the international symbol of accessibility others>

The dealer/owner shall be responsible for on site barrier free provisions, i.e. steps, ramps, parking signs, etc.

Building approach (walkway or ramp) to be 5' wide minimum and a gradient of not more than 1' in 20'.

Exit doors to remain openable during business hours from the inside without the use of a key or any special knowledge.

Corrosion resistant flashing at top and sides of doors, windows and at roof penetrations shall be provided.

	SHEET NUMBER	DRAWING TITLE
✳	1	SPECIFICATIONS
	2	FLOOR PLAN W/HVAC
✳	3	ELECTRICAL PLAN
✳	4	BLOCKING PLAN
✳	5	ELEVATIONS
	6	CROSS SECTION
✳	7	PLUMBING PLAN
	8	HANDICAP ELEVATIONS

REVISIONS/DATE

C & B CUSTOM MODULAR
2224 BLOOMINGDALE DR.
BRISTOL, INDIANA 46507
(574) 848-7300

SCALE: NONE	PAGE: 1	DRAWN BY: BB
DATE: 1-31-03		CHECKED BY:

TITLE: APPLE LEASING
2464 OFFICE (23'-6" X 60'-0")

MODEL:	SERIAL #:	DRAWING #:
2464F0-1	03-8075-76	03-8075C

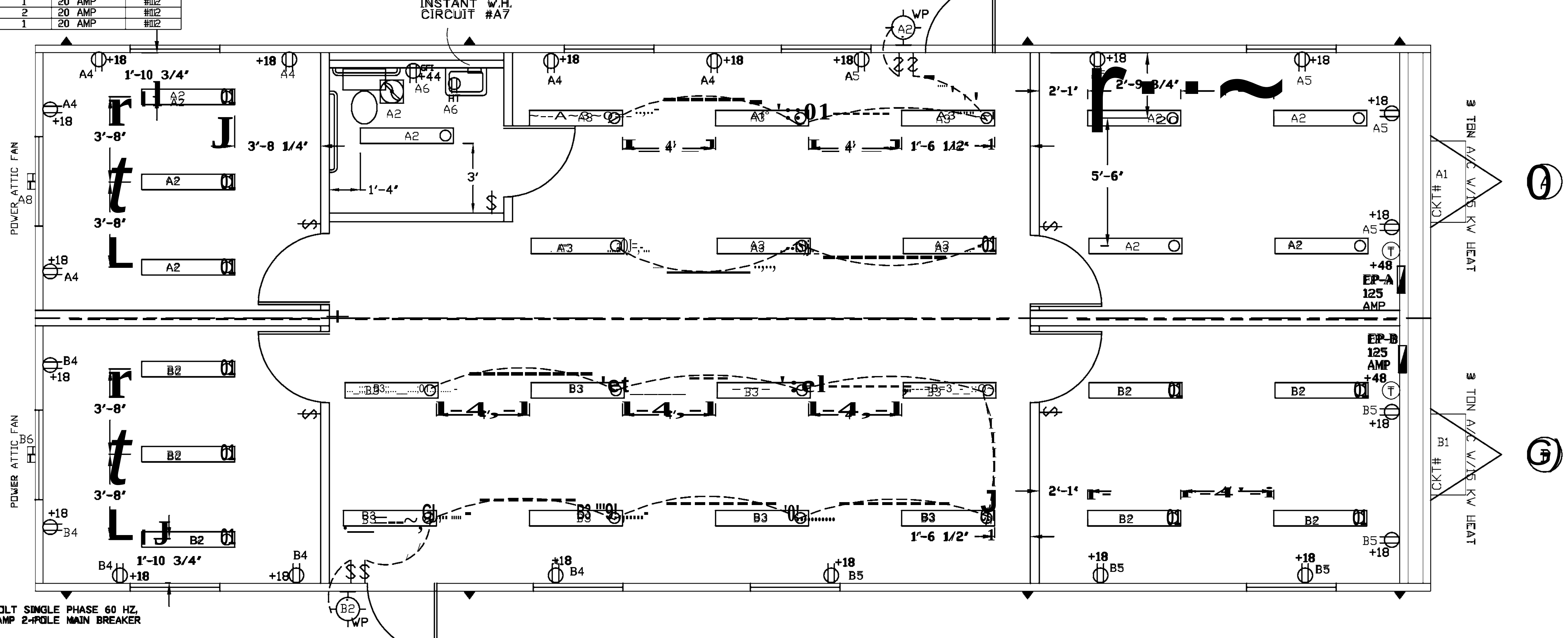
ELECTRICAL PANEL SCHEDULE (EP-A)

CKT. ID #	ITEM	POLES	BREAKER	WIRE
1	HVAC	2	90 AMP	#3
2	LIGHTING/FAN	1	20 AMP	#12
3	LIGHTING	1	20 AMP	#12
4	RECEPTS	1	20 AMP	#12
5	RECEPTS	1	20 AMP	#12
6	GFI/HEAT TAPE	1	20 AMP	#12
7	WTR. HTR.	2	20 AMP	#12
8	ATTIC FAN	1	20 AMP	#12

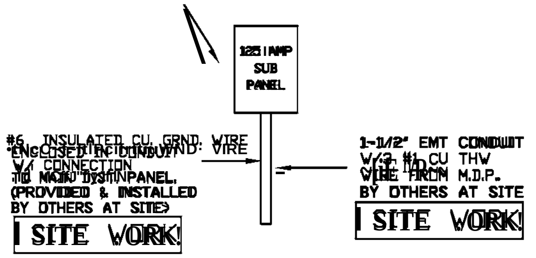
ELECTRICAL PANEL SCHEDULE (EP-B)

CKT. ID #	ITEM	POLES	BREAKER	WIRE
1	HVAC	2	90 AMP	#3
2	LIGHTING	1	20 AMP	#12
3	LIGHTING	1	20 AMP	#12
4	RECEPTS	1	20 AMP	#12
5	RECEPTS	1	20 AMP	#12
6	ATTIC FAN	1	20 AMP	#12

PROVIDE LOCKOUT
AT EP FOR
INSTANT W.H.
CIRCUIT #A7



125 AMP 120/240 VOLT SINGLE PHASE 60 HZ,
SUB PANEL W/125 AMP 2-POLE MAIN BREAKER



TYPICAL RISER DIAGRAM

ELECTRICAL PLAN

ELECTRICAL NOTES

1. ALL RECEPTACLES TO BE GROUNDING TYPE.
2. ALL WIRING TO BE PER THE CURRENT CODE IN EFFECT.
3. MAIN PANEL TO BE MARKED "SUITABLE FOR USE AS SERVICE EQUIPMENT" AND BE EQUIPPED WITH BREAKER/FUSE TYPE OVERCURRENT PROTECTION.
4. PROPER THERMAL OVERLOAD PROTECTION TO BE PROVIDED FOR ALL MOTORS.
5. DISCONNECTING MEANS WITHIN SIGHT REQUIRED FOR ALL MOTORS.
6. WEATHER PROOF PROTECTION REQUIRED FOR ALL OUTDOOR LIGHTS, RECEPTACLES, AND DISCONNECTS.
7. PROPER WORKING CLEARANCES TO BE PROVIDED AND MAINTAINED ABOUT ALL ELECTRICAL EQUIPMENT.
8. ALL FLUORESCENT FIXTURES REQUIRE THERMAL PROTECTION AND PROPER CLEARANCES FROM INSULATION ALSO APPLICABLE FOR INCANDESCENT FIXTURES.
9. COMBINATION EXHAUST FAN/LIGHT AND ALL RECESSED INCANDESCENT FIXTURES TO BE THERMAL PROTECTION.
10. EXIT LIGHTS "IF ELECTRIC" MUST BE FED FROM AND APPROVED EMERGENCY SERVICE CONNECTED AHEAD OF BUT NOT WITHIN MAIN SERVICE DISCONNECTING MEANS ENCLOSURE AND INSTALLED AS PER SERVICE REQUIREMENTS OR BE BATTERY BACKUP TYPE UNITS.
11. EXTERIOR METAL GROUNDING PER N.E.C. SITE WORK!

DRAWING LEGEND

SYMBOL	QTY.	DESCRIPTION
▬	2	120/240 VOLT ELECTRICAL PANEL
▭	29	4' DOUBLE TUBE FLUOR. LIGHT W/DIFFUSER
⊙	2	EXTERIOR 60 WATT W.P. INCANDESCANT LIGHT
⦶	21	120 V. 20 AMP DUPLEX RECEPT
⦶GFI	1	120 V. DUPLEX GFI RECEPT
⦶HT	1	120 V. DUPLEX HEAT TAPE RECEPT
$	6	120 V. SWITCH
Ⓣ	2	THERMOSTAT
⊠	1	100 CFM CEILING EXHAUST FAN

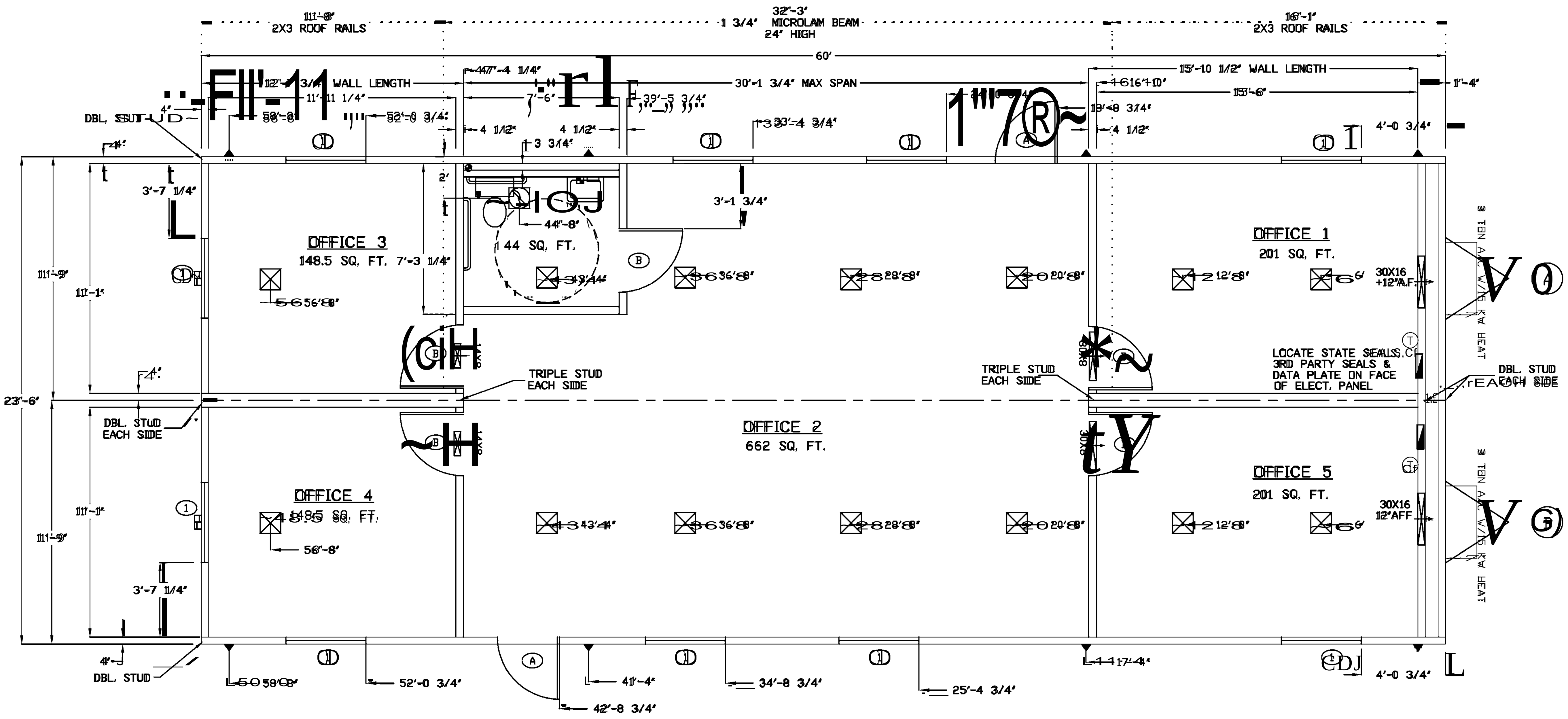
REVISIONS/DATE

C & B CUSTOM MODULAR
2224 BLOOMINGDALE DRIVE
BRISTOL, INDIANA 46507
(574) 848-7300

SCALE: 3/16"=1'-0"	PAGE: 3	DRAWN BY: BB
DATE: 1-31-03		CHECKED BY:

TITLE: APPLE MOBILE
2464 OFFICE (23'-6" X 60'-0")

MODEL:	SERIAL #:	DRAWING #:
24640-1	03-8075-76	03-8075E



FLOOR PLAN

PROVIDE BLOCKING IN FLOOR AND ROOF
AT COLUMN LOCATIONS

HEADER NOTES

1) ALL STRUCTURAL HEADERS SHALL BE GLUED AND FASTENED & BEAR ON 1-2X4 JACK STUD #2 SPF EACH SIDE.

2) DOORS, WINDOWS, OPENINGS ON END WALLS DO NOT REQUIRE STRUCT. HEADER BECAUSE ROOF TRUSS CARRIES THE LOAD.

DRAWING LEGEND

SYMBOL	QTY.	DESCRIPTION
▬	2	120/240 VOLT ELECTRICAL PANEL
Ⓣ	2	THERMOSTAT
☒	14	8 X 8 CEILING SUPPLY AIR DIFFUSER
●	6	THRU WALL RETURN AIR GRILLE SIZE PER FLOOR PLAN
☒	1	100 CFM CEILING EXHAUST FAN
*	8	TIE DOWN STRAP LOCATION
■	1	WATER SUPPLY INLET LOCATION
●	1	DWV-MAIN DRAIN LOCATION

DOOR & WINDOW SCHEDULE

SYMBOL	QTY.	DESCRIPTION	ROUGH OPENING	HEADER
Ⓐ	2	3680 STANDARD W/ 10" LITE, STAY CHAIN & LEVER L.SET	36 X 80	2-2X4 #2 SPF FLAT
Ⓑ	5	3680 COL. BIRCH H.C. W/PINE JAMBS & LEVER L. SET	38 X 82	NON-STRUCT.
①	10	4627 HORZ. SLDR. W/ MILL FIN. & BLINDS SILL HT. 41 1/4"	46 1/4 X 27 1/4	2-2X4 #2 SPF FLAT

REVISIONS/DATE

C & B CUSTOM MODULAR
2224 BLOOMINGDALE DRIVE
BRISTOL, INDIANA 46507
(574) 848-7300

SCALE: 3/16"=1'-0"
DATE: 1-31-03
PAGE: 2
DRAWN BY: BB
CHECKED BY:
TITLE: APPLE MOBILE
2464 OFFICE (23'-6" X 60'-0")
MODEL: 24640-1
SERIAL #: 03-8075-76
DRAWING #: 03-8075F

Legend

- 🟪 Temporary Storage Facility
- 🔴 Power Source/Meter Pole
- 🟢 Transformer for Power Drop
- ▢ Camp Ravenna Boundary

N
W E
S

0 75 150
Feet

Figure 3-3
Temporary Storage Facility Setup Location

Atlas Scrap Yard Area of Concern
Former Ravenna Army Ammunition Plant
Ravenna, Ohio

US Army Corps of Engineers®
BUILDING STRONG®

ERT

Revision Date: December 2016

Data Source: RVAPP, ESRI Street Map

Document Path: N:\GIS\Midwest\Ohio\RVAAP\MXD\Construction Routes\Storage Facility Setup Location.mxd

COMPLETION OF SENIOR TECHNICAL REVIEW

This document has been produced within the framework of the ERT, Inc. (ERT) quality management system. As such, a senior technical review has been conducted. This included review of all elements addressed within the document, proposed or utilized technologies and alternatives and their applications with respect to project objectives and framework of U.S. Army Corps of Engineers regulatory constraints under the current project, within which this work has been completed.

ELECTRONIC SIGNATURE

18 September 2016

Thomas Bachovchin, Professional Geologist Date
Senior Technical Reviewer (ERT)

COMPLETION OF INDEPENDENT TECHNICAL REVIEW

This document has been produced within the framework of ERT, Inc. total quality management system. As such, an independent technical review, appropriate to the level of risk and complexity inherent in the project, has been conducted. This included review of assumptions (methods, procedures, and material used in analyses), alternatives evaluated; the appropriateness of data used and level of data obtained; and reasonableness of the results, including whether the product meets the project objectives. Comments and concerns resulting from review of the document have been addressed and corrected as necessary.

Electronic Signature

17 September 2016

Michelle Chesnut Date
Independent Technical Reviewer (ERT)

Draft
Construction Work Plan

for the

FY16 Recycling of Materials at RVAAP-50 Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility

Former Ravenna Army Ammunition Plant
Ravenna, Ohio

November 11, 2016

Contract W912QR-12-D-0011
Delivery Order 0017

Prepared for:

U.S. Army Corps of Engineers
Louisville District
United Stated Army Corps of Engineers
Louisville District
600 Martin Luther King, Jr. Place
Louisville, Kentucky 40202

Prepared by:

ERT, Inc.
14401 Sweitzer Lane, Suite 300
Laurel, Maryland 20707
(301) 361-0620

Approvers:

20 December 2016

Sean Carney, Project Management Professional (PMP) Date
Project Manager

22 September 2016

Jennifer Harlan, PMP Date
Division Manager

DOCUMENT DISTRIBUTION

for the

Final Construction Work Plan

for

FY16 Recycling of Materials at RVAAP-50 Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility

Former Ravenna Army Ammunition Plant
Ravenna, Ohio

Name/Organization	Number of Printed Copies	Number of Electronic Copies
Mark Leeper, ARNG	0	1
Kevin Sedlak, ARNG	0	1
Katie Tait, OHARNG	1	1
Gail Harris, Vista Sciences Corporation, RVAAP Administrative Record	2	2
Pat Ryan, Leidos – REIMS	Transmittal Letter Only	
Nathaniel Peters, CELRL, COR	Transmittal Letter Only	
Craig Combs, CELRL PM	Transmittal Letter Only	
Kevin Mieczkowski, CELRL TM	2	1

Legend:
ARNG – U.S. Army National Guard
CELRL – U.S. Army Corps of Engineers, Louisville District
COR – Contracting Officer's Representative
OHARNG – Ohio Army National Guard
PM – Project Manager
REIMS – Ravenna Environmental Information Management System
RVAAP – Former Ravenna Army Ammunition Plant
TM – Technical Manager

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF APPENDICES

Appendix A	Uniform Federal Policy for Quality Assurance Project Plan
Appendix B	Accident Prevention Plan/Site Safety and Health Plan
Appendix C	Grading, Drainage, & Erosion Control Plan

LIST OF ACRONYMS AND ABBREVIATIONS

AOC	Area of Concern
APP/SSHP	Accident Prevention Plan/Site Safety and Health Plan
AR	Administrative Record
ARNG	U.S. Army National Guard
CELRL	U.S. Army Corp of Engineers, Louisville District
COR	Contracting Officer's Representative
CWP	Construction Work Plan
DO	delivery order
ERT	ERT, Inc.
ITR	Independent Technical Review
OHARNG	Ohio Army National Guard
PM	Project Manager
PMP	Project Management Plan
POC	point of contact
PPE	personal protective equipment
PWS	Performance Work Statement
QA	Quality Assurance
QC	Quality Control
QSM	Quality Services Manual
RVAAP	Former Ravenna Army Ammunition Plant
RVAAP-50	Atlas Scrap Yard
SSHO	Site Safety and Health Officer
STR	Senior Technical Review
UFP-QAPP	Uniform Federal Policy for Quality Assurance Project Plan
USACE	U.S. Army Corp of Engineers
USP&FO	U.S. Property and Fiscal Officer
WWII	World War II

1.0 INTRODUCTION

1.1 General

ERT, Inc., (ERT) has been contracted to perform recycling/disposal of stockpiled material at the Atlas Scrap Yard (RVAAP-50) Area of Concern (AOC), and setup a temporary storage facility for the restoration document repository at the Former Ravenna Army Ammunition Plant (RVAAP), now known as the Camp Ravenna Joint Military Training Center (Camp Ravenna), Portage and Trumbull Counties, Ohio. The activities will be performed under a Firm Fixed Price Delivery Order (DO), as outlined in the Performance Work Statement (PWS), under United States Army Corps of Engineers (USACE), Louisville District (CELRL) contract W912QR-12-D-0011, DO 0017.

1.2 Former RVAAP/Camp Ravenna Location and History

The former RVAAP was utilized as a load, assemble, and pack facility for munitions. At the onset of World War II (WWII), the RVAAP was built to produce large-caliber artillery projectiles and bombs. Although RVAAP downsized after WWII, plant production lines were reactivated during the Korean War and the Vietnam conflicts. Additionally, the plant conducted nearly continuous demilitarization of war stocks, refurbishment of inventoried ammunition, and minor research and development projects through 1992.

In 1992, the installation's status changed from "inactive but maintained" to that of "caretaker." Administrative control of the facility acreage was transferred from the Base Realignment and Closure Division to the U.S. Property and Fiscal Officer (USP&FO) for Ohio in a series of transfers from 1999 to 2013. As of September 2013, administrative accountability for the entire 21,683-acre facility has been transferred to the USP&FO for Ohio and subsequently licensed to the Ohio Army National Guard (OHARNG) for use as a military training site. The RVAAP restoration program involves cleanup of former production/operational areas throughout the facility related to former activities conducted under the RVAAP. The RVAAP Restoration Program, managed by the Army National Guard (ARNG) and the OHARNG, encompasses investigation and cleanup of past activities over the entire 21,683 acres of the former RVAAP (USACE, 2016).

RVAAP-50, Atlas Scrap Yard, formerly known as the construction camp, is located on approximately 73 acres in the central portion of Camp Ravenna south of Newton Falls Road and west of Paris Windham Road. The site boundary is marked with siebert stakes, and is bounded by Newton Falls Road to the north and Paris Windham Road to the east. Load Line 4 is located to the south of the AOC. The Atlas Scrap Yard has served many operational functions over the history of the former RVAAP, but the AOC was never used for munitions productions. From 1940-1945 the AOC operated as a construction camp to house workers and their families while the facility was being constructed. By the end of WWII the majority of buildings and structures at the AOC had been demolished or relocated to other areas. The facilities that remained were used to support roads and grounds maintenance activities. The remaining structures were demolished after the Vietnam War, and the AOC became a stockpile storage area for bulk materials including gravel, railroad ballasts, sand and culverts (Leidos, 2015). The AOC contains several piles of debris consisting of railroad ties, telephone poles, and concrete (USACE, 2016).

The Administrative Record (AR) contains the RVAAP restoration program records and historical maps for the former RVAAP/Camp Ravenna. It is currently stored at multiple locations at Camp Ravenna, including the Camp Ravenna Environmental Office (1438 State Route 534 SW, Newton Falls, Ohio 44444) and Buildings 1038 and 1047 (closest address 8451 State Route 5, Ravenna

Ohio 44266). The AR is to be moved from its current locations and consolidated to a temporary storage facility within the Camp Ravenna property boundary (USACE, 2016).

1.3 Scope

ERT intends to complete the following construction field activities in compliance with the successful execution of the PWS:

1. Prepare, submit for approval, and respond to comments on the project planning documents to detail the construction and restoration activities at RVAAP-50;
2. Collect a representative sample and perform the waste stream characterization of rail ties and timbers necessary for the waste disposal profile requirements of the contracted disposal facility;
3. Mobilize site management, field crews, and heavy equipment to the former RVAAP on a mutually agreeable date with the ARNG, OHARNG, and CELRL;;
4. Perform the site preparation as necessary including placement of road aprons, performing initial site surveys, installation of temporary erosion control measures, and temporary facilities (i.e., portable toilet);
5. Remove and load-out the stockpiled material at RVAAP-50, including rail ties and timbers, and concrete and asphalt stockpiles;
6. Transport the stockpiled material to the designated local disposal facility and recycling facilities, respectively;
7. Perform the grading and stabilization via tracking necessary to flatten the areas of disturbance and restore the area with a gradual topography;
8. Remove the temporary facilities and erosion control measures;
9. Mobilize and setup a temporary storage facility for restoration document repository, and move specified documents from current storage into the temporary storage facility,
10. Demobilize the field crew and equipment from the site

2.0 PROJECT ORGANIZATION

Based on requirements and our understanding of the project, ERT has developed a team with appropriate qualifications and demonstrated capabilities in design, construction, and field services to execute this DO. The Organization Chart (**Figure 2-1)** identifies project personnel required to manage and successfully complete the identified project elements outlined in the PWS; the names of the individuals filling the key positions have been identified. Roles and responsibilities of the project and key personnel are detailed in **Table 2-1.** If any of these individuals become unavailable before the project is completed, they will be replaced with other ERT personnel possessing similar qualifications.

ERT's Division Manager is Jennifer Harlan. Ms. Harlan is responsible for the overall successful execution of ERT contracts. Mr. Sean Carney is ERT's Project Manager (PM) for the DO. The ERT PM is supported by key personnel who are responsible for completing all of the required elements of the PWS that are related to their respective technical areas. ERT's PM will rely on staff resources that are available to the project team. Additional Key Personnel include Thomas Bachovchin, PG as the Senior Technical Reviewer, Ms. Michelle Chesnut as the Independent Technical Reviewer and Quality Assurance/Quality Control Manager, and Michael Barsa, CSP as the Safety and Health Manager.

2.1 A-Zone Environmental Field Crew and Resources

ERT has subcontracted with A-Zone Environmental to support construction activities including; excavation, load-out, and re-grading activities. Mr. Jesse Morgan will serve as the A-Zone Environmental Resource Manager.

A-Zone Environmental will support the field scope of work with a small field team consisting of:

Labor:

- (1) Site Superintendent
- (1) Equipment operators
- (1-2) Laborer

Equipment:

- (1) Excavator
- (1) Dozer
- (1) Water Truck/Wagon (as necessary)
- (1-2) Service/Pick-up Trucks
- (3-6) Semi-tractors with end-dumps

The construction field team will be trained in accordance with 29 Code of Federal Regulations 1910.120. The ERT team will provide miscellaneous support equipment including a 3-inch trash pump and trencher as needed.

2.2 Additional Subcontractors

The following additional services will be subcontracted by the ERT team to complete the PWS for construction activities:

Analytical Services:

CT Laboratories, LLC

Transportation and Disposal of Railroad Ties, Telephone Poles, and Asphalt

American Waste Management Services

1 American Way

Warren, Ohio 44484

Transportation and Recycling of Concrete

Patrick Incorporated
5839 State Route 5
Ravenna, Ohio 44266

Temporary Storage Services

Apple Mobile Leasing

The ERT team intends to utilize key materials as necessary during the removal and re-grading phases of work including:

- Filtrexx; and
- CA-6 rock and 2-3-inch rock, for road access/curtain and anti-scour pad.

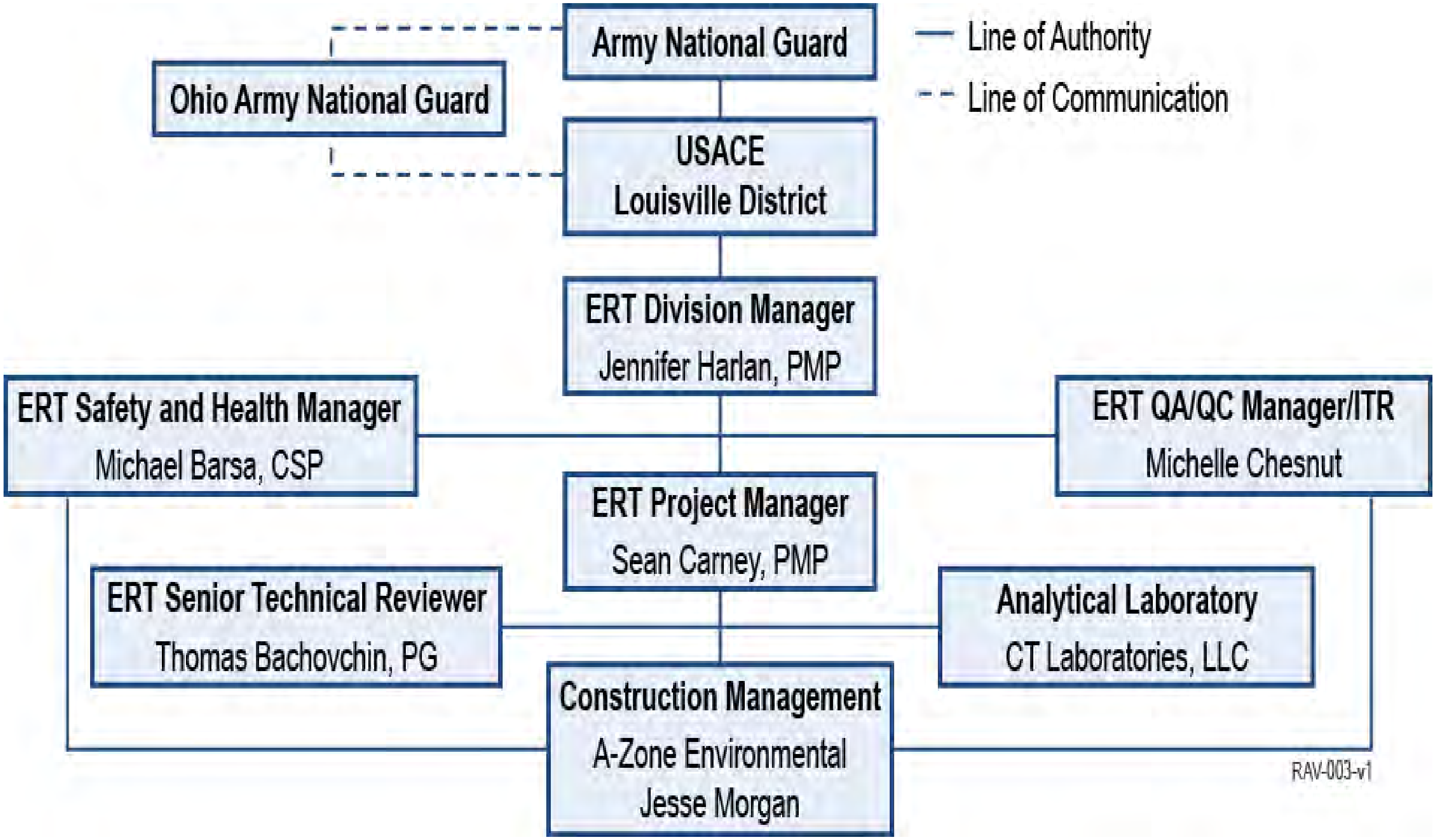


Figure 2-1. Organization Chart

Table 2-1. Roles and Responsibilities of Project Personnel

Position/Contact Information	Role & Responsibilities	Authorities	Communication with USACE
ERT Division Manager Jennifer Harlan 301-323-1394 jennifer.harlan@ertcorp.com	▪ Oversees the contract to ensure ERT's commitment ▪ Responsible for all aspects of contract execution ▪ Assigns corporate resources to tasks ▪ Assigns and oversees work element QC manager(s)	▪ Assigns task order PM and Team ▪ Commits resources	▪ Principal POC for USACE on all aspects of contract performance ▪ POC for USACE on quality of deliverables ▪ Principal POC for USACE on processing of contract modifications
ERT PM Sean Carney 607-259-7017 sean.carney@ertcorp.com	▪ Develops Task Order and assigns corporate resources to tasks ▪ Maintains close communication with USACE ▪ Coordinates work and ensures technical excellence of all activities ▪ Controls program cost and schedule ▪ Negotiates and develops subcontract agreements ▪ Oversees preparation of monthly financial, invoicing, and progress reports	▪ Evaluates and approves task order staffing and subcontractor assignments ▪ Approves task plans ▪ Approves task budgets, change orders, and expenditures ▪ Reviews/approves deliverables for release to USACE ▪ Stop work authority ▪ Issues subcontract agreements ▪ Reviews and approves invoices for final submittal to USACE	▪ Principal POC for USACE on DO assignment ▪ Frequent contact with USACE and staff on DO progress and Team performance
ERT Senior Technical Reviewer Thomas Bachovchin, PG 301-323-1442 thomas.bachovchin@ertcorp.com	▪ Performs STR on all project documents ▪ Assists with work plan development, performs data analyses and evaluations, develops plans and reports	▪ Ability to stop work, request corrective actions or redo the work associated with deviations from the work plan	▪ Not applicable

Table 2-1. Roles and Responsibilities of Project Personnel

Position/Contact Information	Role & Responsibilities	Authorities	Communication with USACE
ERT Independent Technical Reviewer/QA/QC Michelle Chesnut 301-323-1396 michele.chesnut@ertcorp.com	▪ Assists with DO Quality Control Plan development, ensures compliance with work plans and Standard Operating Procedures ▪ Performs QC on field activities, sampling, laboratory analysis, remedial activities, and ITR of deliverables	▪ Authority to stop work or require re-analysis due to quality issues that require corrective actions.	▪ Not applicable
Safety and Health Manager Michael Barsa, CSP 301-323-1447 michael.barsa@ertcorp.com	▪ Oversees compliance with site safety and health ▪ Assists PM in coordinating site activities	▪ Stop work authority for all safety related issues, work deviations from the work plan, and quality issues identified in the field	▪ Per direction of PM
A-Zone Environmental Resource Manager Jesse Morgan 304-724-6458 jmorgan@a-zoneenvironmental	▪ Oversees compliance with work specification, quality of field work, site safety and health, and schedule ▪ Assists PM in coordinating site activities	▪ Stop work authority for all safety related issues, work deviations from the work plan, and quality issues identified in the field	▪ Per direction of PM

Legend:
ITR = independent technical review
POC = point of contact
QA = quality assurance
QC = quality control
STR = senior technical review

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3.0 WORK DESCRIPTIONS

The activities to be conducted during the execution of the DO are identified in Section 1.3 of this Construction Work Plan (CWP) and are further described below.

3.1 Planning

Planning includes the preparation of the various project plans required for execution of the PWS. Project plans include:

- Project Management Plan (PMP);
- CWP containing as **Appendices A, B and C**, respectively:
  - Accident Prevention Plan/Site Safety and Health Plan (APP/SSHP);
  - Uniform Federal Policy – Quality Assurance Project Plan (UFP-QAPP); and
  - Grading, Drainage, and Erosion Control Plan.

3.2 Stakeholder Coordination

All project plans will be prepared by ERT and/or the relevant subcontractors, and submitted to CELRL for review and acceptance. ERT will distribute documents for review and acceptance to the appropriate stakeholder as outlined in the Final PMP (ERT, 2016a). ERT will be responsible for responding to comments with appropriate concurrence on project deliverables from stakeholders. Specific stakeholders for this DO include the following:

- CELRL;
- ARNG; and
- OHARNG

Any necessary revisions will be made expeditiously, and the revised plan(s) will be resubmitted for subsequent review and acceptance. Copies of all accepted plans will be available on-site and will be reviewed and signed by project team personnel prior to the initiation of field work.

Additionally, a pre-construction meeting will be held on-site prior to the start of the work and will involve the ERT team; USACE, ARNG, and OHARNG will be invited to attend. At such time the final established sequence of activities and final coordination items will be communicated to all stakeholders. ERT will provide meeting minutes to all stakeholders.

3.3 Site Management

Site management will be a team effort performed by a combination of personnel. Site management will commence upon mobilization and includes, but is not limited to, oversight of site staff, subcontractors, and visitors. Site management will also involve the preparation and maintenance of the various forms of photographic and written documentation that is required to adequately depict the activities being performed at the site. Daily activities will be documented by field personnel and the Site Superintendent in accordance with the various plans developed for this project. Site management will also involve required administrative functions such as purchasing, directing, discussing progress with CELRL personnel, as well as the management and support of ERT team field staff.

Site security is also a site management task. Access to the site will be limited to persons immediately involved with the project. Heavy duty safety orange construction fencing and/or construction barrier tapes will be used to demarcate the exclusion and support zones of each work site. A site visitors log will be kept to assist in maintaining proper site security.

The Site Superintendent will be responsible for the daily coordination and scheduling of trucks with the transportation subcontractor during the excavation phase. The Site Superintendent will plan the fieldwork in accordance with available resources and will be cognizant of current and forecasted weather conditions.

The Site Safety and Health Officer (SSHO) is responsible for providing the initial and daily health and safety briefings for all project personnel performing work at the site. An initial health and safety briefing will also be provided by the SSHO for visitors when accessing the site; visitors will be escorted always during their visit, even after the initial health and safety brief.

3.4 Quality Control

Effective QC is paramount to the successful completion of any project. QC not only addresses the quality of the finished product, but also influences the degree to which work is considered complete, thereby influencing the accuracy, timing, and safety of what is being accomplished. QC is everyone’s responsibility, just as safety is everyone’s responsibility. As with safety, overall QC is assigned to one or more individuals. Ms. Michele Chesnut is ERT’s overall QC Manager for this project.

During the construction phases (e.g., removal, load out and re-grading of RVAAP-50, the setup of the temporary storage facility, and moving of applicable restoration document repository documents) of work for the project, the Site Superintendent will have direct responsibility for establishing and maintaining appropriate field quality control.

The three-phase inspection system, established by USACE, will be employed by the ERT team during the construction operations at RVAAP. The three-phase inspection system utilizes a system of preparatory meetings, initial inspections, and follow-up inspections.

- Preparatory meetings are conducted at the work site prior to the initiation of each definable stage of work. They are designed to review the safety aspects of each task, the required materials and equipment, and details of the task being initiated. Representatives of all relevant parties attend the meetings, such as the resident officer in charge of construction, the SSHO, and Site Superintendent of the field crews. Crew members are also invited and attendance is suggested, if other ongoing work is not interrupted.
- The initial inspection is conducted during the initial phases of work to establish the level of workmanship and to ensure that all crew members are clear on the particulars of the activities they are assigned, including, but not limited to, required safety procedures, personal protective equipment (PPE), and materials. If adjustments in procedures or PPE are required, they are identified during the initial inspection.
- Follow-up inspections are performed daily on all ongoing tasks. They are performed to ensure that the level of workmanship established in the initial phase inspection is being maintained and that no deviations from the requirements set forth in the contract specifications have been made. The UFP-QAPP (**Appendix A**) provides additional quality control activities to be performed during the project.

3.5 Mobilization and Site Preparation

Mobilization involves the movement of personnel, equipment, and materials to RVAAP-50 at the former RVAAP. Heavy equipment to be mobilized includes an excavator, site trucks and other miscellaneous hand and power tools. Site preparation involves preparing the site for execution of the work such as staging portable sanitation units at a convenient location on-site for use by site workers.

A detailed and thorough inspection of the work site will be made by the Site Superintendent during the mobilization and site preparation phase of the project. The inspections will be made to determine if any relevant changes to the work areas, such as site access, etc., have occurred that would impact field activities. Changes will be addressed and corrected before initiating field work.

3.6 Field Work

The ERT team will mobilize all resources necessary (personnel, equipment, and materials) to successfully perform the work tasks identified in this CWP in order to fulfill the PWS. Site personnel will work with and under the direction and supervision of the Site Superintendent to complete the construction activities. Individual tasks to be conducted as a part of the overall field work are described below.

3.6.1 Site Preparation and Support Activities

The ERT team will set up a staging area and portable toilet at a location agreed upon by the Contracting Officer's Representative (COR) and ARNG/OHARNG personnel. Site controls and staging areas will be established to support the project activities. Access for field crews and deliveries will be coordinated with the OHARNG at least 48 hours in advance.

Additional details on the site preparation and support activities are available in the Grading, Drainage, and Erosion Control Plan (**Appendix C**). **Figure 3-1** illustrates the proposed location of the temporary access points (i.e., rock apron from Old Newton Falls Road to the dirt road and Paris Windham Road to the dirt road), loading zone, and erosion control measures incorporated into the Grading, Drainage, and Erosion Control Plan.

We do not intend to perform any vegetation clearance or falling of trees to access the piles of concrete and asphalt. However, vegetation and samplings that have grown through the concrete and asphalt piles will be removed during the removal of the stockpiled material. Any vegetation and/or saplings that are removed will be left in place.

3.6.2 Rail Ties and Timber Waste Characterization Sampling

Prior to the commencement of construction activities, and in accordance with the PWS, waste characterization sampling will be conducted for stockpiled rail tie and telephone/timber material to be disposed. All waste characterization sampling and subsequent waste shipments will be coordinated with Katie Tait, OHARNG at (614)-336-6136. Upon receipt of the waste profile from the disposal facility, ERT will report the profile to Ms. Tait or her designated appointee for review and approval prior to commencing with the load-out of material.

Based on the requirements of the disposal facility, one representative sample will be collected for waste characterization. Further details of the constituent of concern waste characterization sampling approach, rationale, and design are provided in Worksheet #17 of the Draft UFP-QAPP (ERT, 2016b).

All waste characterization samples will be analyzed by CT Laboratories, LLC who is approved to conduct sampling analysis under the DoD Quality System Manual System (QSM) and the Louisville QSM Supplement. After sample collection, the samples will be shipped via overnight mail to:

CT Laboratories LLC
1230 Lange Court
Baraboo, Wisconsin 53913
Phone: 608-356-2760

3.6.3 Loading of Stockpiled Materials

The ERT team will mark the extents of the areas of stockpiled rail ties and timbers, and the extent of stockpiled concrete and asphalt for offsite disposal. This includes the telephone poles and large piece of concrete located on Paris Windham Road. The telephone poles will be live loaded into semi-tractor dump trailers. We anticipate live-loading rail ties and telephone/timbers stockpiles into semi-tractor dump trailers for disposal at American Waste Management, Warren, Ohio. We anticipate live-loading concrete and asphalt stockpiles into semi-tractor dump trailers for recycling at Patrick Incorporated, Ravenna, Ohio. The total volume of rail ties and timbers to be removed is estimated to be 3,200 cubic yards. The total volume of concrete and asphalt to be removed is estimated to be 4,000 cubic yards. The actual volumes to be removed are subject to revision based on the actual volume of stockpiled material.

The ERT team intends to load the rail ties and timbers using a hydraulic excavator, CAT 320 type, or equivalent. The excavator will be equipped with a hydraulic thumb to provide better control of the loading of timbers into the semi-tractor dump trailers. Rail ties and timbers will be directly loaded into semi-tractors equipped with dump trailers and capable of hauling a typical 22-ton payload. The ERT team intends to progress the load-out from the south to the north. The ERT team will establish a load-out zone that will move as the material stockpile is removed.

Similarly, the ERT team intends to load the concrete and asphalt using a hydraulic excavator, CAT 320 type or equivalent. The excavator will be equipped with a hydraulic thumb to provide better control while loading of concrete into the semi-tractor dump trailers. Concrete and asphalt will be directly loaded into semi-tractors equipped with dump trailers and capable of hauling a typical 22-ton payload. The ERT team intends to progress the load-out from the north to south along the stockpiles of material. The ERT team will establish a load-out zone that will move as the material stockpiles are removed.

The ERT team will remove accumulated dirt from truck tires and bodies prior to each truck leaving the site. The ERT team will manually remove soils adhered to excavator bucket and undercarriage of the excavator to a visually clean standard. All soils removed, brushed, and scraped from the machine will be collected and used during grading activities after the final stockpiled material has been removed. The ERT team does not anticipate having to power wash the machine; thereby not creating liquid waste.

The proposed Construction Site Layout and Truck Access Routes are provided in **Figure 3-1** and **Figure 3-2**, respectively.

3.6.4 Transportation and Off-Site Disposal/Recycling

ERT has selected the following facilities and transporter for disposal and recycling:

Transportation and Disposal of Railroad Ties, Telephone Poles, and Asphalt

American Waste Management Services

1 American Way

Warren, Ohio 44484

Transportation and Recycling of Concrete

Patrick Incorporated
5839 State Route 5
Ravenna, Ohio 44266

The potential to repurpose the railroad ties and telephone poles was evaluated during the development of this project. However, there are currently no vendors that are repurposing railroad timbers or telephone poles in the region. Therefore, the opportunity to repurpose the materials was determined to be currently unfeasible.

Coordination of all waste generated and shipments will be made with Ms. Katie Tait, OHARNG. ERT does not intend to stage any materials onsite; however, we will ensure that Ms. Tait or her appointed designee is available to sign all disposal and recycling bills of laden during the offsite shipment of materials for recycling and disposal.

Each truck will undergo a dry decontamination via brushing to remove soil from the tailgate and side-boards of the trailer that may have dropped during load-out. This will occur in the designated loading zone. The ERT team's laborer will brush the soil away from the tires of the trailer so that the excavated material does not come into contact with the tires as the truck pulls away. All spilled residues will be collected so that the loading zone is clean to a visual standard before being occupied by the next trailer. The ERT team does not anticipate having to power wash the trucks; thereby not creating liquid waste.

The ERT team will track the disposal quantities and will collect and report the tonnages as received by the disposal and recycling facilities daily. Each load will be manifested and weighed at the disposal facility.

3.6.5 Compaction and Restoration

Upon attainment of the removal objectives, the ERT team will compact loose material prior to any re-vegetation that may be necessary. Areas will be compacted to promote future drainage of the area and eliminate potential pooling of water. Multiple passes will be made over disturbed areas to compact the material into place. No compaction testing will be performed since no compaction standards are specified. Tracking in place will provide sufficient compaction to stabilize the soils in advance of any necessary re-vegetation.

f vegetated areas are disturbed, ERT will coordinate activities with the Camp Ravenna Environmental Office. We will prepare a seed bed and seed the area with 50% Kentucky Bluegrass (Blueridge, Argyl, BlueStar, Ken Blue, Limousine, or Baron varieties), 40% Perennial Ryegrasses (Peak, Sonata, Wind Dance, Integra, Morning Star or Shining Star varieties), and 10% Creeping Red Fescue (Lustrous or Boreal varieties) based on the activities being performed in late fall/early winter.

3.6.6 Setup of Temporary Storage Facility and Relocation of the RVAAP Restoration Document Repository

Upon completion of the load-out of stockpiled material, ERT will mobilize and setup a 24 foot by 60-foot temporary storage facility between Buildings 1034 and 1036 as depicted in **Figure 3-3**. ERT will establish the temporary storage facility utilities through the City of Newton Falls. A power drop and electrical meter will be installed from the nearest powerline and transformer (identified in **Figure 3-3**).

Upon establishment of the temporary storage facility, ERT will move restoration document repository records, documentations, maps, shelving, and map cases from three current locations to the temporary storage facility. Metal-wire or HDX shelving units will be provided for additional storage. Shelving units will be approximately 18” deep, 48” wide, and 84” tall. ERT will maintain a contract for the temporary storage facility for a period of 12-months.

3.6.7 Demobilization

All temporary support facilities will be removed during demobilization, including temporary erosion control measures. The ERT team will remove the erosion and sediment controls in place when the vegetative cover is 75% established.

3.7 Completion Report and Data Management

Following the completion of the field activities, ERT will develop a Completion Report for the FY16 Recycling/Disposal Activities at RVAAP-50. After approval of the Completion Report, ERT will support RVAAP and provide all necessary information and documentation required to update the project repository.

ERT will ensure that final documents are presented in accordance with the RVAAP standard format guidelines (Vista Sciences Corporation and Sciences Applications International Corporation, 2012) and accompanied by defined metadata for upload into the U.S. Army Repository of Environmental Documents.

Geographical Information System data and associated metadata will be provided to the USACE upon completion of each project deliverable, or as requested by USACE. The spatial data will be compliant with the Spatial Data Standards for Facilities, Infrastructure, and Environment v2.6 and Army geospatial data standards.

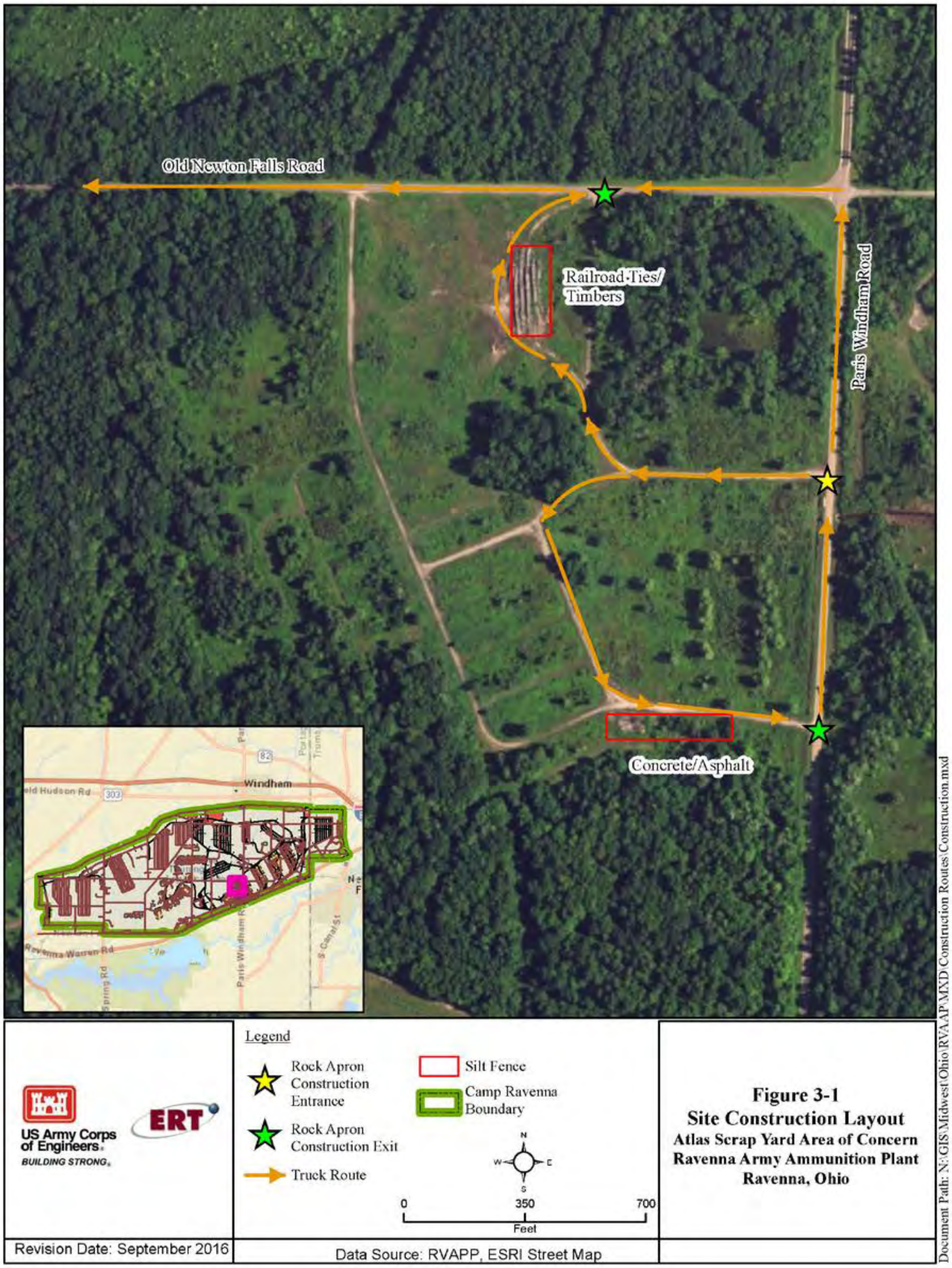

Figure 3-1. Site Construction Layout

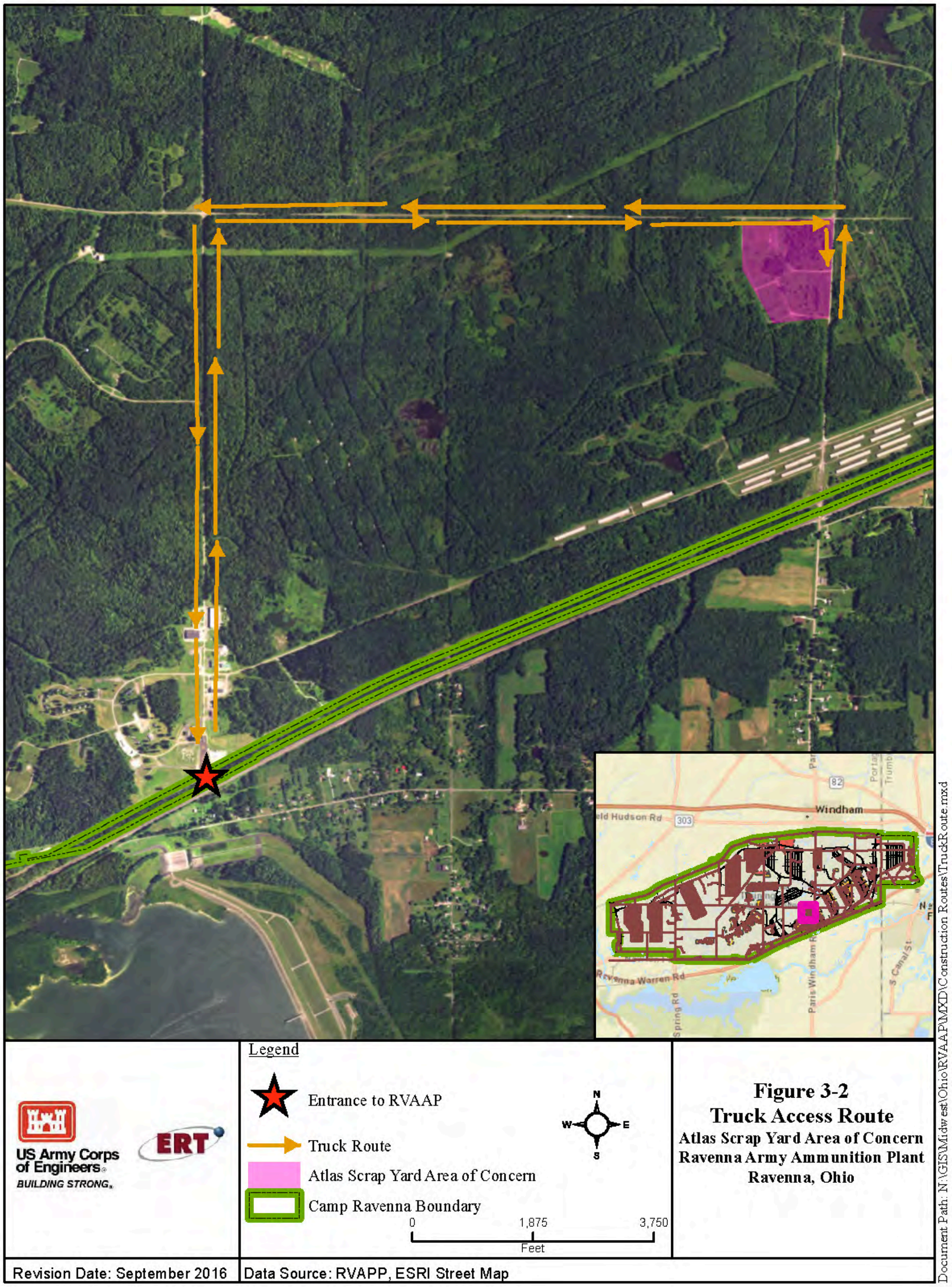

Figure 3-2. Truck Access Route

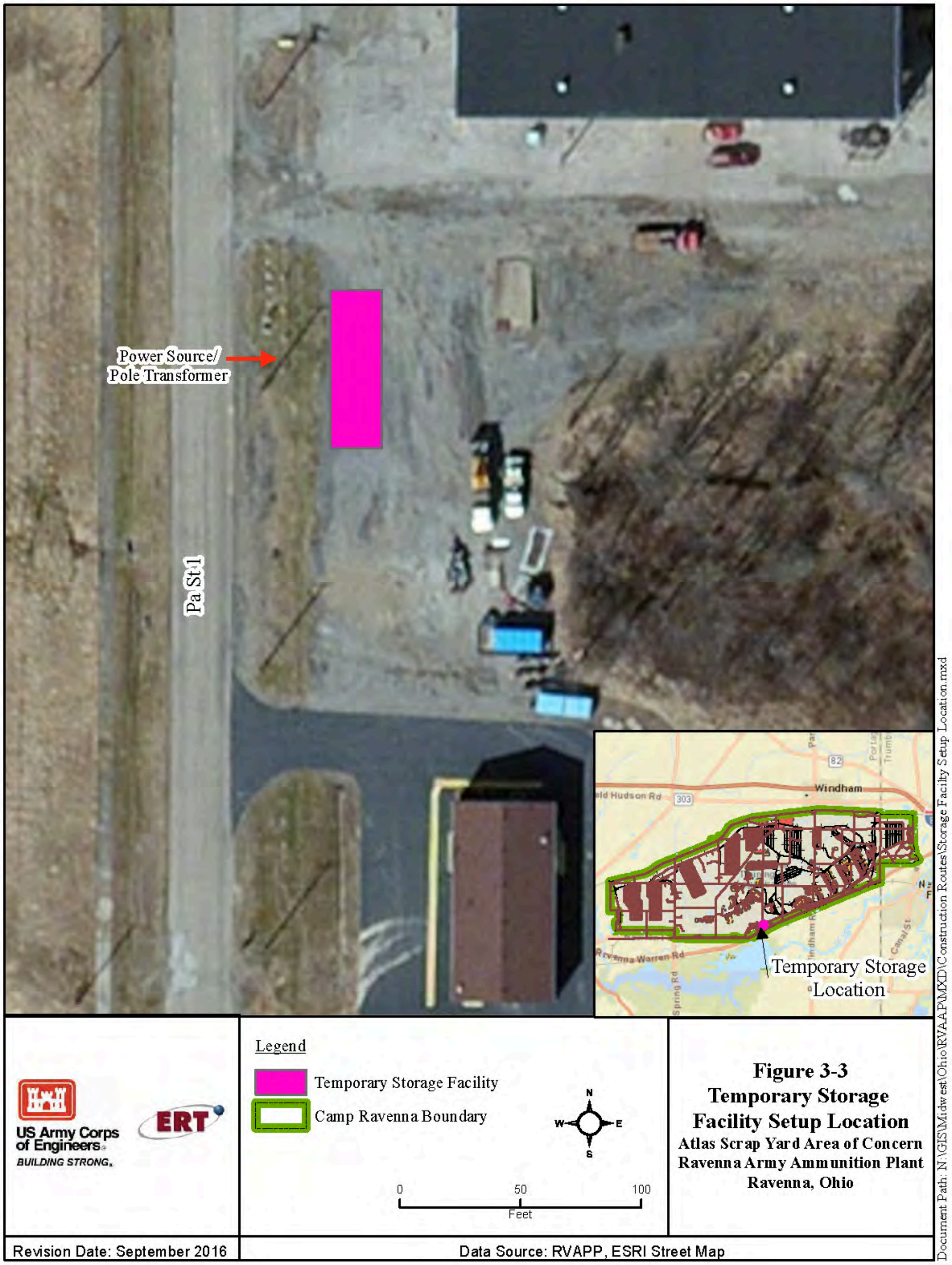

Figure 3-3. Temporary Storage Facility Setup Location

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4.0 SCHEDULE

4.1 Project Schedule

The general schedule for preparing the deliverables and performing the field work required by the PWS is presented in **Figure 4-1,** as well as detailed in the Final PMP (ERT, 2016a). The ERT team will coordinate activities with the COR to ensure that the proposed project schedule does not conflict with other activities on site. The schedule will be updated, as needed, during the course of the project.

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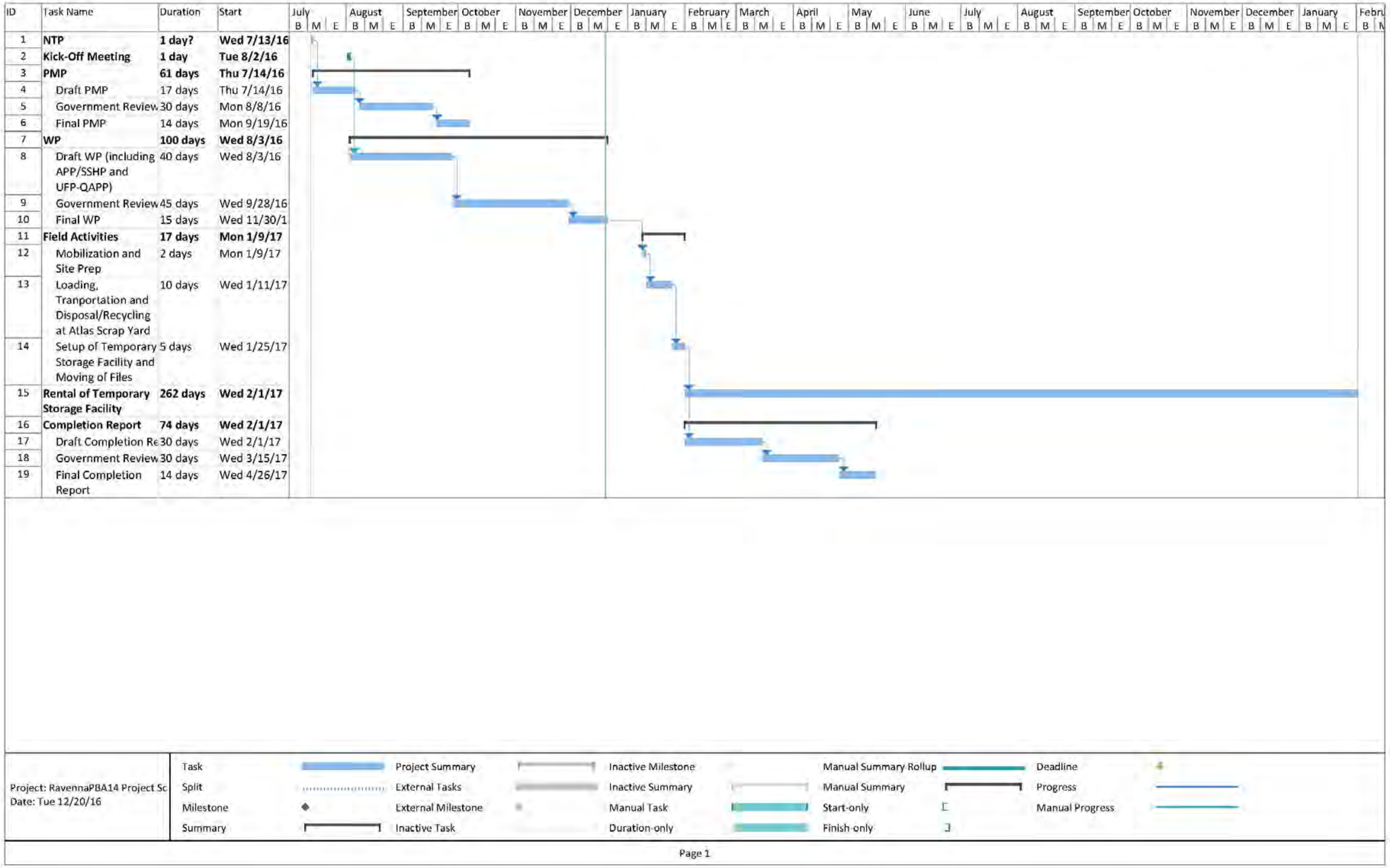

ID	Task Name	Duration	Start
1	**NTP**	**1 day?**	**Wed 7/13/16**
2	**Kick-Off Meeting**	**1 day**	**Tue 8/2/16**
3	**PMP**	**61 days**	**Thu 7/14/16**
4	Draft PMP	17 days	Thu 7/14/16
5	Government Review	30 days	Mon 8/8/16
6	Final PMP	14 days	Mon 9/19/16
7	**WP**	**100 days**	**Wed 8/3/16**
8	Draft WP (including APP/SSHP and UFP-QAPP)	40 days	Wed 8/3/16
9	Government Review	45 days	Wed 9/28/16
10	Final WP	15 days	Wed 11/30/1
11	**Field Activities**	**17 days**	**Mon 1/9/17**
12	Mobilization and Site Prep	2 days	Mon 1/9/17
13	Loading, Tranportation and Disposal/Recycling at Atlas Scrap Yard	10 days	Wed 1/11/17
14	Setup of Temporary Storage Facility and Moving of Files	5 days	Wed 1/25/17
15	**Rental of Temporary Storage Facility**	**262 days**	**Wed 2/1/17**
16	**Completion Report**	**74 days**	**Wed 2/1/17**
17	Draft Completion Re	30 days	Wed 2/1/17
18	Government Review	30 days	Wed 3/15/17
19	Final Completion Report	14 days	Wed 4/26/17

Project: RavennaPBA14 Project Sc
Date: Tue 12/20/16

Page 1

Figure 4-1. Project Schedule

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5.0 REFERENCES

ERT, Inc. (ERT). 2016a. *Final Project Management Plan for FY16 Recycling of Materials at RVAAP-20 Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility, Former Ravenna Army Ammunition Plant Restoration Program*. September.

ERT. 2016b. *Draft Uniform Federal Policy for Quality Assurance Project Plan for FY16 Recycling of Materials at RVAAP-20 Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility, Former Ravenna Army Ammunition Plant Restoration Program*. September

Leidos, 2015. *Remedial Investigation Report for Soil, Sediment, and Surface Water at RVAAP-50 Atlas Scrap Yard, Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio*. June.

USACE, 2016. *Performance Work Statement, FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility, Former Ravenna Army Ammunition Plant Restoration Program, Camp Ravenna, Portage and Trumbull Counties, Ohio*. May.

Vista Sciences Corporation and Sciences Applications International Corporation, 2012. *Submission Format Guidelines, Ravenna Army Ammunition Plant, Version 20*. March

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Appendix A
Uniform Federal Policy for Quality Assurance Project Plan

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FINAL

UNIFORM FEDERAL POLICY –
QUALITY ASSURANCE PROJECT PLAN
FOR
FY16 RECYCLING OF MATERIALS AT RVAAP-50 ATLAS SCRAP YARD AREA OF CONCERN AND SETUP OF TEMPORARY STORAGE FACILITY

Former Ravenna Army Ammunition Plant Restoration Program
Portage and Trumbull Counties, Ohio

Contract No.: W912QR-12-D-0011
Delivery Order 0017

Prepared for:

US Army Corps
of Engineers®
BUILDING STRONG®

Prepared by:

ERT, Inc.
14401 Sweitzer Lane, Suite 300
Laurel, Maryland 20707
(301) 361-0620

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Final

Uniform Federal Policy - Quality Assurance Project Plan

For

FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility
Former Ravenna Army Ammunition Plant Restoration Program
Camp Ravenna, Portage and Trumbull Counties, Ohio

Prepared for:

U.S. Army Corps of Engineers
Louisville District

Contract No: W912QR-12-D-0011
Delivery Order: 0017

Prepared by:

ERT, Inc.
14401 Sweitzer Lane, Suite 300
Laurel, Maryland 20707
(301) 361-0620

Approvers:

20 December 2016
Sean Carney, Project Management Professional (PMP) Date
Project Manager

26 September 2016
Jennifer Harlan, PMP Date
Division Manager

TABLE OF CONTENTS

LIST OF TABLES

LIST OF ATTACHMENTS

Attachment 1	ERT Field Standard Operating Procedures
Attachment 2	CT Laboratories, LLC. Analytical Standard Operating Procedures, Quality Assurance Plan, and DoD Accreditation

LIST OF ACRONYMS AND ABBREVIATIONS

A	analytical
ANSI	American National Standards Institute
AOC	Area of Concern
ARNG	U.S. Army National Guard
bgs	below ground surface
CAS	chemical abstract service
CELRL	U.S. Army Corp of Engineers, Louisville District
CFR	Code of Federal Regulations
COC	chain-of-custody
DoD	Department of Defense
DQI	data quality indicator
DQO	data quality objective
EDD	electronic data deliverable
ERT	Earth Resources Technology, Inc.
ft	foot or feet
ITR	Independent Technical Review
LCS	laboratory control sample
LOD	limit of detection
LOQ	limit of quantitation
mg	milligram
mg/L	milligrams per liter
MDL	minimum detection limit
MPC	measurement performance criteria
N/A	not applicable
OHARNG	Ohio Army National Guard
PM	Project Manager
PMP	Project Management Plan
PQO	project quality objective
PT	proficiency testing
PWS	Performance Work Statment
QA	quality assurance
QAPP	Quality Assurance Project Plan
QC	quality control
QSM	Quality Systems Manual
RVAAP	Former Ravenna Army Ammunition Plant
RVAAP -50	Atlas Scrap Yard
SOP	standard operating procedure
SVOC	semi-volatile organic compound
TCLP	toxicity characteristic leaching procedure
UFP	Uniform Federal Policy
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
VOC	volatile organic compound
ºC	degrees Celsius

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INTRODUCTION

Earth Resources Technology, Inc., (ERT) has been contracted to perform a removal action for the disposal and recycling of stockpiled material at the Atlas Scrap Yard (RVAAP-50) Area of Concern (AOC) at the Former Ravenna Army Ammunition Plant (RVAAP), Camp Ravenna Joint Military Training Center (Camp Ravenna), Portage and Trumbull Counties, Ohio. This Uniform Federal Policy for Quality Assurance Project Plan (UFP-QAPP) prepared in accordance with United States Environmental Protection Agency (USEPA) guidance (USEPA, 2005), is site-specific and presents specific quality assurance/quality control (QA/QC) procedures associated with the waste characterization of telephone poles and railroad ties at Camp Ravenna. The activities will be performed under U.S. Army Corps of Engineers (USACE), Louisville District (CELRL) contract W912QR-12-D-0011, Delivery Order 0017.

It details the planned activities, project and data quality objectives, applicable measurement and data acquisition elements, and data review procedures. Revised QAPP worksheets developed by the Intergovernmental Data Quality Task Force in 2012 are used and completed with project-specific data (USEPA, 2012). The optimized worksheets address all requirements of the American National Standards Institute (ANSI)/American Society for Quality *E4-2004: Quality Systems for Environmental Data and Technology Programs: Requirements with Guidance for Use* (ANSI, 2004) and *EPA Chief Information Officer 2106-G-05: Guidance on Quality Assurance Project Plans* (USEPA, 2012). Elements that discuss project management, objectives, and background information are not provided in this QAPP, as these elements are provided in supplemental project planning documents. A cross reference to all UFP-QAPP elements within project planning documents is provided in **Table 1**.

Table 1. UFP-QAPP Elements Cross Reference

QAPP Worksheet Information	Location of Information	Comments
Title and Approval Page	QAPP Worksheet #1 & #2	
Project Organization QAPP Distribution (QAPP Worksheet #3 & #5)	PMP, 2016[1] Section 3.1, Section 3.5	
Personnel Qualifications Sign-off Sheet (QAPP Worksheet #4, #7 & #8)	Not included. Personnel qualifications and resumes included in the QCPP	
Communication Pathways (QAPP Worksheet #6)	PMP, 2016[1] Section 3.1, Table 3-1	
Project Planning Sessions Summary (QAPP Worksheet #9)	PMP, 2016[1] Appendix A: PWS	
Conceptual Site Model (QAPP Worksheet #10)	N/A	
Project/Data Quality Objectives	QAPP Worksheet #11	

Table 1. UFP-QAPP Elements Cross Reference

QAPP Worksheet Information	**Location of Information**	**Comments**
Measurement Performance Criteria	QAPP Worksheet #12	
Secondary Data Uses and Limitations (QAPP Worksheet #13)	PMP, 2016[1] Section 1.2	
Project Tasks Schedule (QAPP Worksheet #14 & #16)	PMP, 2016[1] Section 3.2	
Project Action Limits and Laboratory-Specific Detection/Quantitation Limits	QAPP Worksheet #15	Field SOPs included in **Attachment 1**
Sample Design and Rationale	QAPP Worksheet #17	Field SOPs included in **Attachment 1**
Sampling Locations and Methods/SOP Requirements	QAPP Worksheet #18	
Sample Containers, Preservation, and Hold Times	QAPP Worksheet #19 & #30	
Field Quality Control Sample Summary	QAPP Worksheet #20	
Project Sampling SOP References	QAPP Worksheet #21	Also included in **Attachment 1**
Field Equipment Calibration, Maintenance, Testing, and Inspection (QAPP Worksheet #22)	N/A	
Analytical SOP References	QAPP Worksheet #23	Analytical SOPs included in **Attachment 2**
Analytical Instrument Calibrations	QAPP Worksheet #24	Analytical SOPs included in **Attachment 2**
Analytical Instrument and Equipment Maintenance, Testing, and Inspections	QAPP Worksheet #25	Analytical SOPs included in **Attachment 2**
Sample Handling, Custody, and Disposal	QAPP Worksheet #26 & #27	
Analytical Quality Control and Corrective Action	QAPP Worksheet #28	

Table 1. UFP-QAPP Elements Cross Reference

QAPP Worksheet Information	Location of Information	Comments
Project Documents and Records (QAPP Worksheet #29)	PMP, 2016[1] Section 3.4, Section 3.5	
Assessments and Corrective Action (QAPP Worksheet #31, #32 & #33)	PMP, 2016[1] Section 5.0	
Data Verification Inputs	QAPP Worksheet #34	
Data Verification Procedures	QAPP Worksheet #35	
Data Validation Procedures	QAPP Worksheet #36	
Data Usability Assessment	QAPP Worksheet #37	

Legend:
QCPP = Quality Control Project Plan
N/A = Not Applicable
PMP = Project Management Plan
PWS = Performance Work Statement
QAPP = Quality Assurance Project Plan
SOP = Standard Operating Procedure
1 = Final Project Management Plan for FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility Former Ravenna Army Ammunition Plant Restoration Program, Camp Ravenna, Portage and Trumbull Counties, Ohio (ERT, 2016a).

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QAPP WORKSHEET #1 & 2 – TITLE AND APPROVAL PAGE

FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility, Ravenna Army Ammunition Plant Restoration Program
Project Name

Camp Ravenna, Portage and Trumbull Counties, Ohio
Project Location

W912QR-12-D-0011, Delivery Order: 0017
Contract Number/Delivery/Task Order

Lead Organization's Project Manager (PM):

Craig Combs/CELRL/DATE
Signature *Printed Name/Organization/Date*

Lead Organization's Quality Manager:

Kevin Mieczkowski/CELRL/DATE
Signature *Printed Name/Organization/Date*

Project Stakeholders:

Agency	**Connection**
CELRL	Contracting Organization
ARNG	Restoration Program Management
OHARNG	Licensed User for Military Training Exercises
Legend: *ARNG = U.S. Army National Guard OHARNG = Ohio Army National Guard*	

QAPP Revision History:

Version	**Revision History**
Original	N/A

Plans and reports from previous investigations relevant to this project:

Title	**Date**
N/A	N/A

QAPP WORKSHEET #11 – PROJECT/DATA QUALITY OBJECTIVES

This worksheet identifies the project quality objectives (PQOs) or data quality objectives (DQOs) that have been developed through a systematic planning process for this RA. This information documents the environmental decisions that have been made and the level of data quality required to ensure that those decisions are based on sound scientific data.

Data Quality Objectives

The DQO process flow for this project follows the requirements of EPA QA/G-4 (USEPA, 2006). The overview of DQOs for chemical sampling and analysis are presented below.

Step 1. State the Problem

The former RVAAP-50 AOC was utilized as a construction camp and to store stockpiled materials. There are several piles of debris within the RVAAP-50 AOC consisting of railroad ties, telephone poles, and concrete. The estimated volume of the debris piles totals approximately 300,000 cubic yards.

Step 2. Identify the Goals of the Study

- Collection of representative samples of railroad ties and timbers for Toxicity Characteristic Leaching Procedure (TCLP) volatiles organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP metals (eight RCRA metals), and flashpoint analysis prior to recycling/disposal activities to meet disposal permit requirements.
- Remove approximately 3,200 cubic yards of stockpiled rail ties and timbers materials and dispose in accordance with disposal permit requirements.
- Remove approximately 4,000 cubic yards of stockpiled concrete and asphalt materials and recycle in accordance with permitted recycling facility.

Step 3. Identify Information Inputs

- Samples collected from railroad ties and timbers;
- Analytical results;
- Project plans developed for this project.

Step 4. Define the Boundaries of the Study

The RVAAP-50 AOC is located on approximately 73 acres in the central portion of Camp Ravenna south of Newton Falls Road and west of Paris Windham Road. There is no fence around the AOC as a perimeter boundary, but the AOC is bounded by Newton Falls Road to the north and Paris Windham Road to the east. Load Line 4 is located to the south of the AOC. The AOC contains several piles of debris consisting of railroad ties, timbers, concrete, and asphalt. The estimated volume of the debris piles totals approximately 7,200 cubic yards.

Step 5. Develop the Analytic Approach

- All parameters will be analyzed according to the USEPA Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846) (USEPA, 2014).
- Waste characterization to meet the permit requirements of the contracted disposal facility includes the following analysis:

    - TCLP VOCs,
    - TCLP SVOCs,
    - TCLP metals, and
    - Flashpoint.
- Waste characterization results will dictate the final disposition of the debris.

Step 6. Specify Performance or Acceptance Criteria

Analytical data will be obtained in compliance with the Quality System Management for Environmental Laboratories (Department of Defense [DoD], 2010). In addition, all data deliverables will be of sufficient quality to meet the requirements of 40 Code of Federal Regulations (CFR) 300.430.

However, project-specific objectives for the data quality indicators of precision, accuracy/bias, representativeness, data comparability, completeness, and sensitivity have been not been developed for this project as waste characterization sampling is unvalidated data collected for the substantive requirements of the disposal facilities permit requirements.

Step 7. Develop the Detailed Plan for Obtaining Data

This QAPP was developed based on the needs of the project and obtaining sufficient quality data to address the project objective. Input from stakeholders through review of this QAPP and other planning documents related to this removal action will optimize the overall project design.

QAPP WORKSHEET #12 – MEASUREMENT PERFORMANCE CRITERIA

This worksheet documents the quantitative measurement performance criteria (MPC) for both field and laboratory measurements. The MPC provided were developed to ensure collected data will satisfy the PQOs or DQOs documented in Worksheet #11. Only those MPC for waste characterization are provided.

Matrix	**Debris (railroad ties, telephone poles, concrete)**				
Analytical Group	**TCLP VOCs, TCLP SVOCs, TCLP metals**				
Concentration Level	**Standard**				
Sampling Procedure	**Analytical Methods/SOPs****	**Data Quality Indicators**	**Measurement Performance Criteria**	**QC Sample and / or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling Analytical**
Composite	SW-846 8260B,8270C,6010B,1010A / VO004, SV007, MT009	Accuracy/Bias	No false positives or negatives	PT sample	A

Matrix	**Debris (railroad ties, telephone poles, concrete)**
Analytical Group	**TCLP VOCs, TCLP SVOCs, TCLP metals**
Concentration Level	**Standard**

Sampling Procedure	**Analytical Methods/SOPs****	**Data Quality Indicators**	**Measurement Performance Criteria**	**QC Sample and / or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling Analytical**
		Sensitivity	Project-specific reporting limits	MDL studies	A

Legend:
A = analytical
MDL = method detection limit
PT = proficiency testing
[*]Reference number from QAPP Worksheet #21
[**]Reference number from QAPP Worksheet #23

QAPP WORKSHEET #15 – PROJECT ACTION LIMITS AND LABORATORY-SPECIFIC DETECTION/ QUANTITATION LIMITS

This worksheet identifies the project action limits and laboratory-specific detection/quantitation limits for waste characterization and confirmation analyses only. Limits for additional waste characterization analyses are available upon request.

Matrix	**Debris (railroad ties and timbers)**
Analytical Group	**TCLP VOCs, TCLP SVOCs, TCLP metals, Flashpoint**
Concentration Level	**Standard**

Parameter	**CAS Number**	**USEPA Hazardous Material Threshold (mg/L)**	**Achievable Laboratory Limits**	
			LOD (mg/L)	**LOQ** (mg/l)
TCLP VOCs				
Benzene	71-43-2	0.5	0.0005	0.001
Carbon tetrachloride	56-23-5	0.5	0.0005	0.001
Chlorobenzene	108-90-7	100	0.0005	0.001
Chloroform	67-66-3	6.0	0.00025	0.0005
1,2-Dichloroethane	107-06-2	0.5	0.0005	0.001
1-1 Dichloroethylene	75-35-4	0.7	0.0005	0.001
Methyl ethyl ketone	78-93-3	200	0.005	0.001
Tetrachloroethylene	127-18-4	0.7	0.0005	0.001
Trichloroethylene	79-01-6	0.5	0.0005	0.001
Vinyl chloride	75-01-4	0.2	0.0005	0.001
TCLP Metals				
Arsenic	7440-38-2	5.0	0.012	0.024
Barium	7440-39-3	100	0.0009	0.0018
Cadmium	7440-43-9	1.0	0.001	0.002
Chromium	7440-47-3	5.0	0.002	0.004

Matrix	**Debris (railroad ties and timbers)**
Analytical Group	**TCLP VOCs, TCLP SVOCs, TCLP metals, Flashpoint**
Concentration Level	**Standard**

Parameter	**CAS Number**	**USEPA Hazardous Material Threshold (mg/L)**	**Achievable Laboratory Limits**	
			LOD (mg/L)	**LOQ** (mg/l)
Lead	7439-92-1	5.0	0.002	0.004
Mercury	7439-97-6	0.2	0.00006	0.00012
Selenium	7782-49-2	1.0	0.0065	0.013
Silver	7440-22-4	5.0	0.002	0.004
TCLP SVOCs				
Total Cresol	95-48-7	200	0.002	0.005
Pentachlorophenol	87-86-5	100	0.002	0.005
2,4,5-Trichlorophenol	98-06-2	400	0.002	0.005
2,4,6-Trichlorophenol	88-06-2	2.0	0.002	0.005
1,4-Dichlorobenzene	106-46-7	7.5	0.0004	0.001
2,4-Dinitrotoluene	121-14-2	0.13		
Hexachlorobenzene	118-74-1	0.13	0.0004	0.001
Hexachlorobutadiene	87-68-3	0.5	0.0004	0.001
Hexachloroethane	67-72-1	3.0	0.0004	0.001
Nitrobenzene	98-95-3	2.0	0.0004	0.001
Pyridine	110-86-1	5.0	0.001	0.003
Flashpoint				
Flashpoint	N/A	200°F	20°F	230°F

Legend:
CAS = Chemical Abstract Service

Matrix	**Debris (railroad ties and timbers)**			
Analytical Group	**TCLP VOCs, TCLP SVOCs, TCLP metals, Flashpoint**			
Concentration Level	**Standard**			
Parameter	**CAS Number**	**USEPA Hazardous Material Threshold (mg/L)**	**Achievable Laboratory Limits**	
			LOD (mg/L)	**LOQ** (mg/l)

LOD = Limit of Detection
LOQ = Limit of Quantitation
mg/L = milligrams per liter

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QAPP WORKSHEET #17 – SAMPLE DESIGN AND RATIONALE

This worksheet describes the sampling design and the basis for its selection.

Rationale for choosing the sampling approach

1. If possible, stockpiled materials will be recycled or reused.
2. If stockpiled materials are not able to be recycled or reused than materials will be disposed of with approval by the ARNG.
3. Per PWS and disposal facility waste permit requirements, stockpile materials will be tested prior to disposal for TCLP VOCs, TCLP SVOCs, TCLP metals, and flashpoint to ensure waste stream is within permit parameters.
4. A composite sample will be taken to represent the waste characteristics for the railroad ties and timbers.

Sampling Design

The discussions below outline the procedures related to waste characterization of stockpiled materials at Camp Ravenna. Related waste characterization and decontamination procedures are also provided. ERT maintains SOPs for all field sampling procedures to be used for this RA. ERT Field SOPs are listed in Worksheet #21 and are presented in **Attachment 1**.

Solid Waste Characterization

Stockpile rail ties and telephone poles/timber materials to be disposed of from Camp Ravenna will be sampled with the sampler wearing a disposable nitrile glove, composited into a single sample, and characterized for TCLP VOCs, TCLP SVOCs, TCLP metals, and flashpoint prior to final disposition at the contracted disposal facility. Chip samples will be collected from a minimum of 8 locations along the stock[piled rail ties pile and chip samples will be collected from a minimum of 8 locations along the telephone poles/timbers pile. Each set of chips will be composited independently into the sample containers. Solid waste characterizations sample(s) will be deposited into two 250 milliliter (mg) glass jars for submittal to CT Laboratories. Based on the presumed volume of material requiring disposal and the permit requirements of the disposal facility, a single composite solid waste characterization sample is assumed to be sufficient. If conditions warrant the collection of additional solid waste characterization samples, a decision will be made in the field and documented in the daily progress reports.

Sample Preservation

Dedicated and sterilized sample containers will be supplied by CT Laboratories. Samples will be maintained between 2 to 6 degrees Celsius (ºC) as a sample preservation technique. Worksheet #19 details the specific containers and preservatives required.

Sample Container Labeling, Storage, and Shipment

Samples will be labeled, stored, and shipped as follows:

1. Labels will be affixed to each sample container and verified for completeness. Information on the sample labels will include the sampler's initials, installation name, analytical method, unique sample identification number, preservative (if applicable), and date/time of collection. Worksheet #26 & #27 provides unique labeling guidance and chain-of-custody (CoC) procedures.

2. ERT personnel performing sample collection will verify that sample container lids are secure.
3. The shipping container (i.e., commercial cooler) will have its drain plug taped inside and out, and the container will be lined with a large plastic garbage bag containing approximately 3 inches of inert packing material (e.g., vermiculite or bubble wrap) in the bottom of the liner.
4. Samples will be placed upright in the lined container so that samples do not touch each other during shipment.
5. Samples will be shipped on ice at 2 to 6ºC to:

CT Laboratories, LLC
1230 Lange Court
Baraboo, WI, 53913
Phone: 608-356-2760

Decontamination Procedures

It is anticipated that decontamination procedures of sampling equipment will not be required as dedicated and disposable sampling equipment will be used.

QAPP WORKSHEET #18 – SAMPLING LOCATIONS AND METHODS/SOP REQUIREMENTS

This worksheet is provided to allow for a completeness check for field personnel and auditors/assessors. It serves to facilitate checks to make sure all planned samples have been collected and appropriate methods have been used. As practical, this worksheet lists each individual sample that is planned to be collected, including field QC samples.

Sample Location	Matrix	Depth (ft bgs)	Type	Analytical Group	Sampling Procedure
Rail Ties and Timber Stockpiles	Wood	N/A	waste characterization	TCLP VOCs, TCLP SVOCs, TCLP metals, and flashpoint	Composite

Legend:
ft bgs = feet below ground surface
**Reference QAPP Worksheet #21*

QAPP WORKSHEET #19 & 30 – SAMPLE CONTAINERS, PRESERVATION, AND HOLD TIMES

This worksheet serves as a reference guide for field personnel. It is also an aid to completing the COC form and shipping documents.

Laboratory: CT Laboratories, LLC

Sample Delivery Method: FedEx Overnight

Applicable Laboratory Accreditations: DoD Environmental Laboratory Accreditation Program (see **Attachment 2**)

Sample Location	Analyte/ Analyte Group	Matrix	Analytical Method/ SOP	Accreditation Expiration Date	Container(s)	Preservation	Preparation Holding Time	Analytical Holding Time	Data Package Turnaround
Rail ties and timber stockpiles	TCLP VOCs, TCLP SVOCs, TCLP metals, Flashpoint	Wood	SW-846 1311/ 8260B/8270C/6010B/1010A	26 June 2017	(2) 250 mg glass Jars	N/A	14 days	7 days	15 days
Legend: *mg = milligram*									

QAPP WORKSHEET #20 – FIELD QUALITY CONTROL SAMPLE SUMMARY

This worksheet provides a summary of the types of samples to be collected and analyzed for the project. It illustrates the relationship between the number of field samples and associated QC samples. As this project only contains waste characterization sampling there will be no duplicate or matrix spike/matrix spike duplicates collected.

Matrix	Parameter	Field Samples	Field Duplicates	Matrix Spikes	Matrix Spike Duplicates	Total # Analyses
Wood	TCLP compounds and Flashpoint	1	0	0	0	1

QAPP WORKSHEET #21 – PROJECT SAMPLING SOP REFERENCES

This worksheet documents the specific field procedures being implemented, which is important for measurement traceability. All project sampling SOPs are provided in **Attachment 1**.

ERT SOP Reference Number	Title/Version	Originating Organization	Equipment Type	Modified for Project Work	Comments
General					
ERT SOP01-1	Preparing for and Concluding Field Activities	ERT	N/A	No	
ERT SOP06-1	Packaging and Shipment of Field Samples	ERT	Various	No	
ERT SOP01-2	Use of Field Logbooks	ERT	N/A	No	

QAPP WORKSHEET #23 – ANALYTICAL SOP REFERENCES

This worksheet documents information about the specific sample preparation and analytical procedures to be used, which is important for measurement traceability. SOPs for all sample preparation and analytical procedures are provided in **Attachment 2**; only those SOPs for waste characterization and confirmation analyses are provided. Additional SOPs for waste characterization analyses are available upon request.

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work
VO004	Analysis of volatiles by SW-846 Method 8260B	Definitive	TCLP VOCs	GCMS	CT Laboratories, LLC	No
SV007	Analysis of Semi-volatiles by SW-846 Method 8270D	Definitive	TCLP SVOCs	GCMS	CT Laboratories, LLC	No
MT009	Analysis of Metals by Inductively Coupled Plasma-Atomic Emission spectroscopy, spectrometric method for Trace Element Analyses Rev 19 5/4/11	Definitive	TCLP Metals	Inductively coupled plasma mass spectrometry	CT Laboratories, LLC	No

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QAPP WORKSHEET #24 – ANALYTICAL INSTRUMENT CALIBRATIONS

No analytical instruments will be used in the field. Analytical instrument calibration information for laboratory analyses of TCLP VOCs, TCLP SVOCs, TCLP metals, and flashpoint is provided within the analytical SOPs in **Attachment 2**.

QAPP WORKSHEET #25 – ANALYTICAL INSTRUMENT AND EQUIPMENT MAINTENANCE, TESTING, AND INSPECTIONS

Analytical instrument and equipment maintenance, testing, and inspection information for laboratory analyses of waste characterization is provided within the analytical SOPs in **Attachment 2**.

QAPP WORKSHEET #26 & 27 – SAMPLE HANDLING, CUSTODY, AND DISPOSAL

This worksheet documents responsibilities for maintaining custody of samples from sample collection through disposal.

Sampling Organization: ERT (waste characterization sampling).

Laboratory: CT Laboratories, LLC

Method of sample delivery (shipper/carrier): FedEx overnight

Number of days from reporting until sample disposal: 180

Activity	Organization and Title of Person Responsible for the Activity	SOP Reference*
Sample collection	ERT, Field Team	N/A
Sample labeling	ERT, Field Team	Procedure below
COC form completion	ERT, Field Team	ERTSOP06-1
Packaging	ERT, Field Team	ERTSOP06-1 and procedure below
Shipping coordination	ERT, Team Leader	ERTSOP06-1
Sample receipt, inspection, & log-in	CT Laboratory Personnel	See procedure below and Laboratory Quality Assurance Plan in **Attachment 2**
Sample custody and storage	CT Laboratory Personnel	**Attachment 2**
Sample disposal	CT Laboratory Personnel	**Attachment 2**

Legend:
**Reference number from QAPP Worksheet #21 or #23*

Sample identification procedures

Immediately upon collection, each sample for laboratory analysis will be labeled with an adhesive label, which includes the date and time of collection, sampler's initials, installation name, analysis to be performed, a unique sample identifier, and preservative, as applicable. The following sample identification scheme will be used:

- The first four letters and digits represent the Site:
  RVAAP-50 Atlas Scrap Yard Area of Concern
- The next two letters represent the medium:
  WD Wood
- The next digits represent the sample number:
  01 Sample 1

Field sample custody procedures

Samples will be containerized, labeled, and immediately placed in a cooler containing ice. Sample collection information (i.e., sample identification, time of collection, sampler's initials, type of container, analytes to be tested, etc.) will be included on the COC form. Prior to shipment to the analytical laboratory, sample containers will be bubble-wrapped and the COC forms will be inserted into a plastic bag and taped on the inside lid of the cooler. Each cooler will then be taped closed and custody seals will be initialed and dated by the field personnel. Custody seals will be affixed on the lid of the cooler in manner such that if the cooler is opened, the custody seal will break. The coolers will then be shipped overnight priority via FedEx to CT Laboratories in Baraboo, Wisconsin (Worksheet #17).

Laboratory sample custody procedures

Samples will be received and logged in by a designated sample custodian or his/her designee. Upon sample receipt, the sample custodian will:

- Examine the shipping containers to verify that the custody tape is intact.
- Determine if the temperature required for the requested testing program has been maintained during shipment and document the temperature on the COC records.
- Compare samples received against those listed on the COC.
- Verify that sample holding times have not been exceeded.
- Sign and date the COC immediately (if shipment is accepted) and attach the air bill.
- Note any problems associated with the coolers and/or samples on the cooler receipt form and notify the Laboratory PM, who will be responsible for contacting the ERT Project Chemist.
- Attach laboratory sample container labels with unique laboratory identification and test.
- Place the samples in the proper laboratory storage.

Following receipt, samples will be logged in according to the following procedure:

- The samples will be entered into the laboratory tracking system. The following information will be entered: project name or identification, unique sample numbers (both client and internal laboratory), type of sample, required tests, date and time of laboratory receipt of samples, and field identification provided by field personnel.

- The Laboratory PM and the ERT Project Chemist will be notified of sample arrival.

QAPP WORKSHEET #28 – ANALYTICAL QUALITY CONTROL AND CORRECTIVE ACTION

The purpose of this worksheet is to ensure that the selected analytical methods are capable of meeting project-specific MPC, which are based on PQOs/DQOs. The information relevant to this project is presented in **Attachment 2**.

QAPP WORKSHEET #34 – DATA VERIFICATION AND VALIDATION INPUTS

This worksheet lists the inputs that will be used during data verification and validation. Inputs include planning documents, field records, and laboratory records. Data verification is a check that all specified activities involved in collecting and analyzing samples have been completed and documented and that the necessary records (objective evidence) are available to proceed to data validation. Data validation is the evaluation of conformance to stated requirements, including those in the contract, methods, SOPs, and this QAPP.

Description	Verification (completeness)	Validation (conformance to specifications)
Planning Documents/Records		
Approved QAPP	X	
Contract	X	
Field SOPs	X	
Laboratory SOPs	X	
Field Records		
Field logbooks	X	X
COC forms	X	X
Sampling diagrams	X	X
Relevant correspondence	X	X
Change orders/deviations	X	X
Field corrective action reports	X	X
Analytical Data Package		
Cover sheet (laboratory identifying information)	X	X
Case narrative	X	X
Internal laboratory COC	X	X
Sample receipt records	X	X
Sample chronology	X	X
Communication records	X	X
MDL establishment and verification	X	X
Standards traceability	X	X
Instrument calibration records	X	X
Definition of laboratory qualifiers	X	X
Results reporting forms	X	X
Corrective action reports	X	X
Raw data	X	X
Electronic data deliverables	X	X

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QAPP WORKSHEET #35 – DATA VERIFICATION PROCEDURES

This worksheet documents procedures that will be used to verify project data. It applies to both field and laboratory records. Data verification is a completeness check to confirm that all required activities were conducted, all specified records are present, and the contents of the records are complete.

Records Reviewed	**Requirement Documents**	**Process Description**	**Responsible Person, Organization**
Field forms and notes	QAPP ERT Field SOPs	▪ Verify that records are present and complete for each day of field activities. ▪ Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. ▪ Verify that required data were provided for each day of field activities. ▪ Verify that changes/exceptions are documented and were reported in accordance with requirements. ▪ Verify that any required field monitoring was performed and results are documented.	*Daily* – ERT Site Superintendent *At conclusion of field activities* – ERT PM
COC forms	QAPP	▪ Verify the completeness of COC records. ▪ Examine entries for consistency with the field logbook. ▪ Check that appropriate methods and sample preservation have been recorded. ▪ Verify that the required volume of sample has been collected and that sufficient sample volume is available for QC samples. ▪ Verify that all required signatures and dates are present. Check for transcription errors.	*Daily* – ERT Site Superintendent *At conclusion of each sampling event* –ERT QA/QC Manager

Records Reviewed	**Requirement Documents**	**Process Description**	**Responsible Person, Organization**
Laboratory Deliverables	QAPP DoD QSM Laboratory Methods	▪ Verify that the laboratory deliverable contains all records specified in the QAPP. ▪ Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported. ▪ Compare the data package with the COC and EDDs to verify that results were provided for all collected samples. ▪ Review the narrative to ensure all QC exceptions are described. ▪ Check for evidence that any required notifications were provided to project personnel. ▪ Verify that necessary signatures and dates are present.	*Before release* – CT Laboratories PM *Upon receipt* – QA/QC Manager *At conclusion of analytical activities* – ERT PM

Legend:
EDD = electronic data deliverable
PM = Project Manager
QSM = Quality Systems Manual

QAPP WORKSHEET #36 – DATA VALIDATION PROCEDURES

This worksheet documents procedures used by the data validator; Meridian Consultant Group, Inc., to validate project analytical data. Data validation is an analyte and sample-specific process for evaluating compliance with contract requirements, methods, SOPs, and MPC. Data validation is unnecessary as the analytical data are being used to satisfy the disposal requirements of the contracted permitted disposal facility.

QAPP WORKSHEET #37 – DATA USABILITY ASSESSMENT

This worksheet documents procedures that will be used to perform the data usability assessment. The data usability assessment is performed at the conclusion of data collection activities, using the outputs from data verification and data validation. It is the data interpretation phase, which involves a qualitative and quantitative evaluation of environmental data to determine if the project data are of the right type, quality, and quantity to support the decisions that need to be made. It involves a retrospective evaluation of the systematic planning process, and, like the systematic planning process, involves participation by key members of the project team. The data usability assessment evaluates whether underlying assumptions used during systematic planning are supported, sources of uncertainty have been accounted for and are acceptable, data are representative of the population of interest, and the results can be used as intended, with the acceptable level of confidence.

Personnel responsible for participating in the data usability assessment:

Name	**Organization**	**Position**
Sean Carney	ERT	Project Manager
Michelle Chesnut	ERT	Independent Technical Review/QA/QC Manager

Data usability summary:

Step	Summary
Step 1	**Review the project's objectives and sampling design** Review the key outputs defined during systematic planning (i.e., PQOs or DQOs and MPCs) to make sure they are still applicable. Review the sampling design for consistency with stated objectives. This provides the context for interpreting the data in subsequent steps.
Step 2	**Review the data verification outputs** Review data verification reports. Perform basic calculations and summarize the data (using graphs, maps, tables, etc.). Look for patterns, trends, and anomalies (i.e., unexpected results). Review deviations from planned activities (i.e., number and locations of samples, exceeding holding times, damaged samples, non-compliant QC sample results, and SOP deviations) and determine their impacts on the data usability. Evaluate implications of unacceptable QC sample results.
Step 3	**Document data usability and draw conclusions** Determine if the data can be used as intended, considering implications of deviations and corrective actions. Discuss DQIs. Assess the performance of the sampling design and identify limitations on data use. Document conclusions.
Legend: *DQI = data quality indicator* *MPC = measurement performance criteria*	

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REFERENCES

Department of Defense, 2013. *DoD Quality Systems Manual for Environmental Laboratories, Version 5.0*. July.

ERT, Inc., 2016. *Draft Project Management Plan for FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility Former Ravenna Army Ammunition Plant Restoration Program, Camp Ravenna, Portage and Trumbull Counties, Ohio.* August.

U.S. Environmental Protection Agency (USEPA), 1999. *U.S. Environmental Protection Agency Contract Laboratory Program, National Functional Guidelines for Organic Data Review*. October.

USEPA, 2005. *Uniform Federal Policy for Quality Assurance Project Plans*. Department of Defense, Environmental Protection Agency, Department of Energy; Final, Version 1. March.

USEPA, 2006. *Guidance for the Data Quality Objective Process (USEPA QA/G-4)*. February.

USEPA. 2012. *EPA CIO 2106-G-05: Final Draft Guidance on Quality Assurance Project Plans*. January.

USEPA, 2014. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846)*. Office of Solid Waste and Emergency Response. Update IV. Third Edition. February.

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ATTACHMENT 1

ERT Field SOPs

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ERT, Inc.
Standard Operating Procedure
PREPARING FOR AND CONCLUDING FIELD ACTIVITY

Effective Date: **05/12/2016** Version: **003** SOP#: **ERT SOP-01-1**

Approvals

Jennifer Harlan, PMP Division Manager		Michael Barsa, CSP Field QA Manager	
Signature	5/12/16 Date	Signature	5/12/16 Date

1.0 PURPOSE

The purpose of this procedure is to outline requirements associated with preparing for and concluding environmental field activities.

2.0 SCOPE

This procedure applies to all field activities commencing with the Notice to Proceed and ending with the completion of all project field activities.

It is neither the intent of this procedure to fully detail all actions required for preparing and concluding field activities nor define specific methodology, but rather offer general points to be considered in the preparation and conclusion of field activities. Preparation and conclusion activities are specific to the planned field activities and will be detailed in the project-specific Sample and Analysis Plan.

3.0 REQUIREMENTS

In order to efficiently complete field work, specific tasks must be accomplished in an orderly fashion prior to actual field work (preparation) and after field work has been completed (conclusion).

4.0 RESPONSIBILITIES

4.1 Project Manager

For the purpose of this procedure, the Project Manager is responsible for providing the Field Team access to applicable project-specific information, subcontractor support and equipment necessary to complete stated Project Quality Objectives. Project Manager responsibilities include, but are not limited to, locating resources and manufacturers of the proposed equipment, initiating purchaser lease agreements, coordinating site access, coordinating field tasks with associated subcontractors/prime contractors and perform overall task management.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring the Field Team understands all associated field activities and applicable procedures, adheres to the project-specific work plans and successfully completes the field activities. The Field Team Leader is responsible for identifying and obtaining project specific field equipment and tools, appropriate field recording forms and ensuring adequate quantities of supplies. If appropriate, the Field Team Leader is responsible for coordinating necessary subcontractor support. This may include scheduling the receipt of appropriate environmental sample containers, scheduling courier services for analytical samples to the laboratory, etc, as directed by the Project Manager.

The Field Team Leader is responsible for the completion of the Field Activity Preparation Checklist and the Work Order Form. Upon completion, these forms will be reviewed with the Project Manager to ensure accuracy and completeness.

During field activities and at the conclusion of field activities, the Field Team Leader is responsible for performing periodic quality control and quality assurance checks of all environmental sampling procedures and samples obtained, arrange sample shipment to the analytical laboratory, ensure all equipment (both rented and ERT owned) and supplies are accounted for and aptly returned.

4.3 Field Team Members

The Field Team is responsible for the successful completion of all field tasks as assigned by the Field Team Leader. This will be ensured by the adherence to the project-specific operating procedure and project-specific work plan(s). For the purpose of this procedure, the Field Team will be required to fully understand tasks associated with the project-specific work plan(s). During preparation for field activities, field team members may be directed by the Field Team Leader to aid in the identification and arrangement of necessary activity specific equipment, subcontractor support and information.

5.0 EQUIPMENT

None Specified

6.0 PROCEDURE

6.1 Preparing Field Activities

Preparation is a process that begins with the Notice to Proceed and ends with the initiation of field activities. Adequate time and effort involved in preparation ensures efficient and effective execution of the work plan and completion of field activities.

The many preparation tasks to be considered include:

- Ordering and procuring items of a specialized nature, including environmental sampling equipment, environmental sample containers, etc.
- Performing a thorough review of the cost proposal to determine if additional items may be needed. This should be discussed with the personnel assigned to field activities.
- Informing personnel of the date, location and activity required to be performed. Instruct personnel as to travel arrangements.
- Locating sources for field purchased items and supplies.
- Establishing an inventory system of disposable and non-disposable items.
- Detailing specific requirements for mobilization of subcontractors to include drilling contractors, analytical laboratories. Activities to be considered include transportation, decontamination, orientation and badging, and initial setup.
- Testing and calibrating all equipment to ensure operational readiness.
- Establishing a field office or field staging areas for materials and IDW.
- Ensuring reliable communications for field personnel during field activities.

6.2 Concluding Field Activities

Terminating field activity includes activities necessary to transfer custody of materials and supplies after completion of field activities. Activities to be considered include:

- Planning for the thorough completion of field activities before conclusion of field activities. Preliminary conclusion efforts may be undertaken, but all materials and supplies necessary for field activities shall be retained until field activities are complete.
- Reviewing records and thoroughly inspecting equipment to ensure all equipment has been decontaminated.
- Adequately packing special equipment, electronic equipment and other non-disposable items for shipment.
- Completing a review of all environmental samples to be delivered to the analytical laboratory. All sample volumes, sample nomenclature, sample labels, specified sample analytics, number of samples, Chain-of Custody forms, packaging of samples and custody seal will be thoroughly reviewed by the Field Team Leader prior to relinquishing environmental samples.
- Completing an inventory review of packaged equipment for shipment.
- Contact applicable rental equipment suppliers, off-rent equipment and document confirmation off-rent number when applicable. Transfer custody of the rental equipment to the rental company or contracted courier.
- Stage and or dispose of all IDW in accordance with federal and state regulations

7.0 REFERENCES

The following documents were referenced during the development of this Standard Operation Procedure:

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York

8.0 RECORD OF CHANGES

- 22 September 2009 – Changed SOP number
- 17 March 2015 – Updated Program Manager, Field QA Manager, Effective Date, Version Number, Company Logo

9.0 ATTACHMENTS

9.1 Attachment 1 – ERT Field Activity Preparation Checklist

Prior to commencing field activities, the field team is required to amass the necessary information and equipment necessary to successfully complete the field activity objectives. To ensure the appropriate equipment has been obtained, the Field Activity Preparation Checklist will be completed and reviewed with the Field Team

Attachment 1 - ERT Field Activity Preparation Checklist

General:

- ☐ Relevant project file documents
- ☐ Field book
- ☐ Clip board / desk top
- ☐ Cell phone and charger
- ☐ Safety glasses
- ☐ Steel toe boots
- ☐ Hard hat
- ☐ Work plan
- ☐ Work gloves
- ☐ Hand tools
- ☐ HASP
- ☐ Paper towels
- ☐ Wide brimmed hat
- ☐ Caution tape
- ☐ Nitrile gloves
- ☐ Trash bags
- ☐ Chair and table
- ☐ Lunch
- ☐ Drinking water
- ☐ Rugged work ware
- ☐ Rain Gear
- ☐ Site access key
- ☐ Subcontractor contact #
- ☐ Power converter

Drilling

- ☐ PID w/ cal gas
- ☐ Water level meter
- ☐ GPS
- ☐ Decon bucket, soap and brush
- ☐ Well log forms
- ☐ Work plan
- ☐ Caution tape
- ☐ Measuring wheel
- ☐ USACE manual
- ☐ Macro core holder
- ☐ Non-HAZ stickers
- ☐ Hand auger

Ground water Sampling

- ☐ Pump
- ☐ Battery / Generator
- ☐ Tubing
- ☐ Multi-meter
- ☐ Bailers
- ☐ Measuring cup
- ☐ Stopwatch
- ☐ PID w/ cal gas
- ☐ Buckets (3) w/ lids
- ☐ Decon station
- ☐ Soap and brush
- ☐ Distilled water
- ☐ DI water
- ☐ Water level meter
- ☐ String
- ☐ Well keys
- ☐ Carbon bucket
- ☐ Bottle ware + extras
- ☐ Labels + extras
- ☐ COC
- ☐ Cooler with bubble wrap
- ☐ Ice
- ☐ Ziplock bags
- ☐ Fishing gear
- ☐ Work plan
- ☐ Work order
- ☐ Purge data sheets
- ☐ Non-HAZ stickers

Soil Sampling

- ☐ PID w/ cal gas
- ☐ zip lock bags
- ☐ Jars
- ☐ Cooler and bubble wrap
- ☐ Ice
- ☐ Chain of custody
- ☐ Labels
- ☐ Soil sample equipment
- ☐ Hand auger
- ☐ Digging Bar
- ☐ Paper towels
- ☐ Trowel

**Highlight as applicable*

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ERT, Inc. Standard Operating Procedure **USE OF FIELD LOGBOOKS**			
Effective Date: **05/12/16**	Version: **003**	SOP#: **ERT SOP-01-2**	
Approvals			
Jennifer Harlan, PMP Division Manager		Michael Barsa, CSP Field QA Manager	
Signature	5/12/16 Date	Signature	5/12/16 Date

1.0 PURPOSE

The purpose of this procedure is to detail minimum requirements related to maintaining Field Logbooks and recording all field activities within Field Logbooks.

2.0 SCOPE

This procedure applies to all Field Logbooks which are required to be maintained onsite during field activities and for the duration of a project.

3.0 REQUIREMENTS

Field Logbooks will be initiated at the start of the first field activity. Entries will be made each day that field activities occur related to Earth Resources Technology, Inc. (ERT) or ERT subcontractor activities. A current field logbook will be maintained during the duration of the project. Site activities which are nonconcurring will be recorded in the same dedicated Field Logbook.

The Field Logbook will become part of the permanent project file. Field Logbooks must be maintained properly and to the standard set forth in this procedure because information contained in the Field Logbook may be admitted as evidence in mitigation, as an accurate record of field procedures and/or representative site conditions during the time of field activities. All logbooks will be secured in a reasonable fashion for the duration of the project and filed with related project documents after the completion of the project. Field Logbooks are considered company property, as such will be retained and utilized by ERT personnel exclusively to record the events of field activities associated with ERT projects.

4.0 RESPONSIBILITIES

4.1 Project Managers

The Field Logbook is issued by the Project Manager to the Field Team Leader or other person responsible for the direction of field activities (i.e., Field Geologist, Sampling Team Leader). Upon completing field activities, the Field Logbook will be returned to the Project Manager's custody that is responsible for reviewing the daily entries and filing the logbook within the permanent project file.

4.2 Field Team Leader

Field Logbooks are issued to the Field Team Leader, or other person responsible for conducting field activities. It is the responsibility of this person to keep the logbook current, detailing all field activities and pertinent information. It is this person's responsibility to properly secure the logbook and return the logbook to the custody of the Project Manager after concluding field efforts.

5.0 EQUIPMENT

The Field Logbook shall have pre-printed numbered pages, be bound in such a way that pages cannot be readily removed, and be constructed of robust and weather resistant material.

6.0 PROCEDURE

6.1 General

The cover and inside cover of each logbook will contain at a minimum the following information:

- Project Identification
- Project number
- Project Manager's name
- Project Manager contact info
- Sequential book number
- Start date
- End date

The spine of the log book shall contain the following information

- Project Identification
- Sequential book number

As appropriate, a table of contents will be compiled on the Table of Contents page with descriptions of field activities and their respective page numbers in chronological order. The Tables of Contents page will be completed after a log book has been filled to capacity and review of the daily entries has been performed by the Project Manager.

Unless prohibited by weather, pens with permanent black ink should be used to record all activities and datum. When weather conditions do not make it conducive to use permanent ink, entries should be made using a non-smear lead pencil. Once removed from wet elements, penciled entries should be repeated with a permanent ink pen to ensure permanency of the entry. No erasures are permitted. Data or other information that has been entered incorrectly will be corrected by drawing a single line through the incorrect entry, initialing (or signing) and dating the lined-through entry. Under no circumstances will the incorrect material be erased, made illegible or obscured so that it cannot be read. The Field Team Leader or his designee will draw a diagonal line and initial at 1) the end of unfilled pages and 2) the end of all entries for each day of field activity. The final recorded information for each daily entry will be the time field personnel exited the site.

6.2 Information Required

The initial entry for each day of field activities at a minimum should include the following information:

- Date
- Day of week
- Purpose of site visit
- Time of arrival onsite
- Weather conditions
- Names and/or initials of all ERT field personnel present
- Names and/or initials of all subcontractors present
- Names of any visitors present and their affiliation

Entries will be recorded in the Field Logbook in real-time chronological order and summarize all site activities. Logbook entries will be recorded in clear concise and legible hand script, should be objective, factual, and free of personal feelings or other terminology which might prove inappropriate.

6.2.1 Information Required for Sampling Activities

- Makes and models of equipment, and identifying numbers
- Equipment calibration procedures performed including concentrations of calibration media
- Equipment decontamination procedures performed
- Equipment nonconformance
- Sampling methodology utilized
- Matrix sampled
- Sample location, when applicable
- Sample ID's
- Sample collection times
- Sample ware, i.e. number of, size, type, and preservative
- Analytical parameters requested to be performed by the contracted laboratory
- Sample custody procedure conducted
- Any deviations from the work plan that occur while conducting field activities
- Relevant Health and Safety conditions
- Notation of use of any activity specific forms utilized

6.2.2 Information Required for Soil Boring and Well Installation

- Name of subcontractor(s)
- Names of the subcontractor(s) personnel
- Drilling methodology utilized
- Location of drilling activities and the duration of drilling (start and completion times).

- Observations made during the recovery of boring cuttings, including visual, olfactory, photoioinzation detector (PID) readings, volume and geologic descriptors, if necessary.
- Well construction information, including the length of screen and riser, depth interval of filter pack, bentonite, and sealing media, depth of screened interval, and well head protection measures.
- Quantity, type, and name of manufacture of well construction material (i.e. filter sand, bentonite, grout, and well material)
- Quantity and frequency that water was added to the borehole in order to assist boring and well construction
- Depth groundwater was encountered, as applicable.
- Depth to water in completed well.
- Any complications impeding the progress of drilling activities
- Abandonment method if no well is installed in borehole
- Site sketch depicting relevant surface features, project related investigative features, and scale and North arrow if possible.
- Any deviations from the work plan that occur while conducting field activities
- Relevant Health and Safety conditions
- Notation of use of any activity specific forms utilized

Field Logbook entries are not intended to replace data recorded on activity specific data forms such as Well Log Forms, Well Construction Diagram Forms, and Purge Data Sheets. Use of such forms must be noted in the Field Logbook.

7.0 REFERENCES

The following documents were referenced in the development of this Standard Operation Procedure:

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 2001. *Requirements for the Preparation of Sampling and Analysis Plans*. EM 200-1-3. February.

United States Environmental Protection Agency (USEPA), 2007. *Field Branches Quality System and Technical Procedures*. Region 4 Science and Ecosystem Support Division. SESDPROC-010-R3. November

Weight, W. D., Sonderegger, J. L., 2001. *Manual of Applied Field Hydrogeology*, McGraw-Hill, New York.

8.0 DEFINITIONS

None

9.0 RECORD OF CHANGES

- 17 March 2015 – Updated Program Manager, Field QA Manager, Date, Version Number, Company Logo, and added Section 9.0

10.0 ATTACHMENTS

None

ERT, Inc. Standard Operating Procedure **PACKAGING AND SHIPMENT OF FIELD SAMPLES**			
Effective Date: **05/12/16**	Version: **003**	SOP#: **ERT SOP-06-1**	
Approvals			
Jennifer Harlan, PMP Division Manager		Michael Barsa, CSP Field QA Manager	
Signature	5/12/16 Date	Signature	5/12/16 Date

1.0 PURPOSE

The purpose of this procedure is to define the requirements necessary for sample packaging and information to be included on sample labels and chain-of-custody (COC) records used in sample transfer from field personnel to the analytical laboratory.

2.0 SCOPE

This procedure applies to the packaging, shipping and documentation of samples being collected during field activities and transferred contracted laboratory for analysis. Specifically, this document outlines shipping and sample documentation procedures in accordance with the U.S. Department of Transportation (DOT) guidance and regulation. This procedure is applicable to all environmental samples collected for delivery to analytical laboratories; however, this procedure does not take precedence over federal, state, or project-specific requirements for sample management and delivery to the analytical laboratory.

3.0 REQUIREMENTS

Careful packaging, shipping, and documentation are essential to insure that collected samples are received undamaged and authenticated by the contract analytical laboratory.

4.0 RESPONSIBILITIES

4.1 Project Manager

The Project Manager is responsible for providing project-specific guidelines within the Quality Assurance Project Plan (QAPP) for the proper handling and management of environmental samples, and ensuring that the field team is properly trained in the appropriate procedures.

4.2 Field Team Leader

The Field Team Leader is responsible for ensuring that samples are properly packaged, labeled, documented on the COC, and shipped to the contracted analytical laboratory in accordance with this procedure and within the project-specific QAPP. The Field Manager will also ensure that proper sample management techniques are followed for the entire duration that samples are in the custody of ERT personnel.

4.3 Field Team

The Field Team is responsible for implementing packaging, labeling, documenting, and shipping requirements detailed in this procedure and the project-specific QAPP.

5.0 EQUIPMENT

- Cooler
- Packing Tape
- Bubble Pack
- Sampling Gloves
- Heavy Duty Plastic Trash Bags
- Inert absorbent material/matting
- COC
- Pen, Black Ink
- Ziploc-type Plastic Bags
- Permanent Felt Tip Marker
- Shipping documents
- Right-side-up and Fragile Placarding
- Custody Seals
- Natural Ice
- Temperature Blanks

6.0 PROCEDURE

6.1 Sample Packaging and Shipping

Typically, samples collected for shipment to analytical laboratories will classified as non-hazardous environmental samples. ERT will not ship any material known to be hazardous. If hazardous material is suspected, the Project Manger will be notified and appropriate accommodation will be made to transfer the sample media from the site. If hazardous material is expected to be encountered during filed activities, the project-specific QAPP will details the proper procedures for handling the hazardous media.

6.2 Packaging

Environmental samples will be packaged following the procedure outlined below:

- Conduct a “bottle count” to ensure correct sample volumes for the analytical procedures requested, correct number of samples, and that information on each sample label matches the COC and complies with the project-specific QAPP.
- Line shipping cooler with plastic trash bag
- Line bottom of cooler with inert absorbent material/matting.
- Tape drain plug closed (if applicable).
- Place doubled-bagged wet ice on the bottom of the lined shipping cooler. Enclose temperature blank in bagged ice, sufficiently protected from breakage.
- Place inert cushioning material on top of ice.
- Check to ensure that sample lids are tightened to prevent leakage and with enough headspace (except VOC containers with a septum lid) to compensate for any pressure and temperature changes during shipment (approximately 10 percent of the volume of the container).
- Segregate samples from sample points into separate Ziploc-type plastic bags to minimize the potential for cross contamination and contact with any liquid generated from melting ice.
- Arrange samples within the cooler as to minimize contact between breakable sample containers.
- Fill any void spaces between samples with inert cushioning material.
- Place double-bagged ice on top of the samples.
- Close plastic bag lining cooler and tape shut.

- Place signed Chain-of-Custody form into Ziploc-type plastic bag and affix to inside of lid of the shipping cooler with packing tape. In the instances of multiple coolers for a single Chain-of-Custody place photo copies of original COC in Ziploc-type bag in each additional cooler.
- Tape the lid of the shipping cooler shut including around the edge where the lid contacts the body.
- Place COC seals across the threshold where the cooler lid contact the body of the shipping cooler, or as detailed in the project specific QAPP.
- Tape down any movable external handles.
- Affix shipping documents to the lid.
- When multiple coolers are shipped during a single event they should be numbered sequentially on the exterior (1 of 3 etc.) so that the receiving facility would be aware of any irregularities in shipment.
- "THIS SIDE UP" or "THIS END UP" and “FRAGILE” placards and upward-pointing arrows should also be affixed on the outside of the cooler.

6.2.1 <u>Shipping Papers</u>

No DOT shipping papers are required for the transport of non-hazardous environmental samples. However, appropriate COC forms must be included with the shipment.

6.2.2 <u>Transportation</u>

There are no DOT restrictions on mode of transportation for non-hazardous environmental samples.

6.3 Chain-of-Custody Guidelines

A properly completed COC ensures that handling and shipment of environmental samples has been conducted in a defensible and scientific manner. COC procedures track environmental samples from the time and place it is first obtained to the analytical laboratory. These procedures also provide an auditable trail of evidence the samples pass from the custody of one individual to another. In addition, procedures for consistent and detailed records facilitate the admission of evidence under Rule 803(b) of the Federal Rules of Evidence (P.L. 93-575).

COC procedures, record keeping, and documentation are an important in ensuring the quality of sample analytical results. Written procedures must be available and followed whenever evidence samples are collected, transferred, stored, analyzed, or destroyed.

6.3.1 <u>Sample Identification</u>

The following information shall be recorded in the Field Logbook when samples for laboratory analysis are collected:

- unique sample number
- source of sample (including name, location, and sample type)
- time of collection
- number of samples collected
- types of sample container
- preservative used

- analysis required
- field observations (include pH, temp, depth to water) when applicable
- equipment used to make physical measurements and collect samples
- calibration data for equipment used

6.3.2 Sample Labels

Samples, other than in-situ measurements, are removed and transported from the sample location to a contracted analytical laboratory or other location for analysis. Before transport, however, the collected sample volume is often divided into various containers for particular analytical procedures. Each subsample will be containerized and preserved in accordance with the project-specific QAPP.

Each sample container will have a dedicated sample label identifying the sample. The following table describes the minimum sample label requirements.

Minimum Sample Label Requirements	
Field Sample No.	The unique sample number identifying this sample as prescribed by the Work Plan and/or QAPP
Project Name	Name
Project No.	ERT Project number
Date	A six-digit number indicating the month, day, and year of sample collection; e.g., 12/21/85
Time	A four digit number indicating the 24-hour time of collection (for example: 0954 of9:54 a.m., and 1625 is 4:25 p.m.)
Media Type	Water, Soil, etc.
Method Type	Grab or Composite
Analysis	Method of analysis for the laboratory
Preservation	Type of preservative
Collector's Initials	Initials of the sampler

Once the required information is entered onto the label and affixed to the sample container, clear packing tape should be placed over the label to maintain the integrity of the label, else use adhesive labels.

6.3.3 Chain-of-Custody Procedures

After collection, separation, and identification, the sample is maintained under COC procedures until it is in the custody of the analytical laboratory and has been stored or disposed.

6.3.4 Field Custody Procedures

1. Samples are collected and labeled as described in the project-specific QAPP. Care must be taken to ensure that the sample identification on the label exactly matches sample identification on the COC.
2. Field personnel collecting the environmental samples are responsible for the care and custody of the samples collected until they are properly transferred to a predetermined staging area, the custody of another Field Team member, or the custody of a currier.
3. Sample labels shall be completed for each sample, using waterproof ink unless prohibited by weather conditions.

6.3.5 Transfer of Custody and Shipment

Samples are accompanied by a COC Record Form. When transferring the possession of samples, both the individual relinquishing and receiving the samples will sign, date, and note the time of transfer on the COC. The COC documents sample custody transfer from the sampler, often through another person, to the analyst in the laboratory.

Typically the COC is filled out in its entirety as follows:

1. Enter project specific information (i.e. project number and name, Contract Lab case No. or SAS No.). For each sample, record the applicable Sample Identification, date and time of sample collection, the sampler, preservation information, type of sample (composite/grab), number of containers transferred and the requested analytical parameters.
2. Sign, date, and enter the time under "Relinquished by" entry.
3. Ensure the person receiving the sample signs for the "Received by" entry, or enter the name of the carrier (e.g., UPS, Federal Express) under "Received by".
4. Record the bill-of-lading or Federal Express air bill number under "Remarks or Reason for Change of Custody", if appropriate.
5. Place the original (top, signed copy) of the Chain-of-Custody Recorded Form in the appropriate sample shipping package. Retain a copy with field records.

The custody record is completed using black waterproof ink. Any corrections are made by drawing a single line through the error and initialing and dating the change, then entering the correct information. Erasures are not permitted.

Common carriers (FedEx and UPS) will not accept responsibility for handling COC Forms, necessitating the package of the COC Record in the sample container (enclosed with the other documentation in a plastic zip-lock bag secured to the lid of the shipping cooler). As long as custody forms are secured inside the sample shipping container and the custody seals are intact, commercial carriers are not required to sign off on the custody form.

The laboratory representative who accepts the incoming sample shipment signs and dates the COC, completing the sample transfer process. It is then the laboratory's responsibility to maintain internal logbooks and custody records throughout sample preparation and analysis.

7.0 REFERENCES

Sanders, L. L., 1998. *Manual of Field Hydrogeology*, Prentice Hall, Inc., Upper Saddle River, New Jersey.

United States Army Corp of Engineers (USACE), 2001. *Requirements for the Preparation of Sampling and Analysis Plans*. EM 200-1-3. February.

United States Environmental Protection Agency (USEPA), 1992. *RCRA Ground Water Monitoring: Draft Technical Guidance.* November

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United States Environmental Protection Agency (USEPA), 2007. *Field Branches Quality System and Technical Procedures*. Region 4 Science and Ecosystem Support Division. SESDPROC-209-R1. November.

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8.0 DEFINITIONS

Carrier - A person or firm engaged in the transportation of passengers of property.

Chain-of-Custody Form - A Chain-of-Custody Form is a printed form that accompanies a sample or group of samples as custody of the sample(s) is transferred from one custodian to the subsequent custodian. Attachment 1 shows a typical Chain-of-Custody Form. Chain-of-Custody Form is a controlled document. One copy of the form must be retained in the project file.

Custodian - The person responsible for the custody of samples at a particular time, until custody is transferred to another person (and so documented), who then becomes custodian. A sample is under your custody if:

- You possess the sample.
- It is in your view, after being in your physical possession.
- It was in your physical possession and then you locked it up to prevent tampering.
- You have designated and identified a secure area to store the sample

Environmental Sample - A low concentration sample typically collected offsite and not requiring DOT hazardous waste labelling as a high hazard sample.

Marking - Applying the descriptive name, instructions, cautions, weight, or specification marks or combination thereof required to be placed outside containers of hazardous materials.

Packaging - The assembly of one or more containers and any other components necessary to assure compliance with the minimum packaging requirements of 49 CFR 172, including containers (other than freight containers or overpacks), portable tanks, cargo tanks, tank cars, and multi-unit tank car tanks.

Placard - Color-coded, pictorial sign depicting the hazard class symbol and name to be placed on all four sides of a vehicle transporting certain hazardous materials.

Sample - A sample is physical evidence collected from a facility or the environment, which is representative of conditions at the point and time that it was collected.

9.0 RECORD OF CHANGES

- 23 September 2009 – Updated SOP number.
- 20 March 2015 – Update Program Manager and Field QA Manager approvals, date, version number, and company logo

10.0 ATTACHMENTS

Attachment 1 – Example Chain of Custody Form

Attachment 2 – Example Custody Seal

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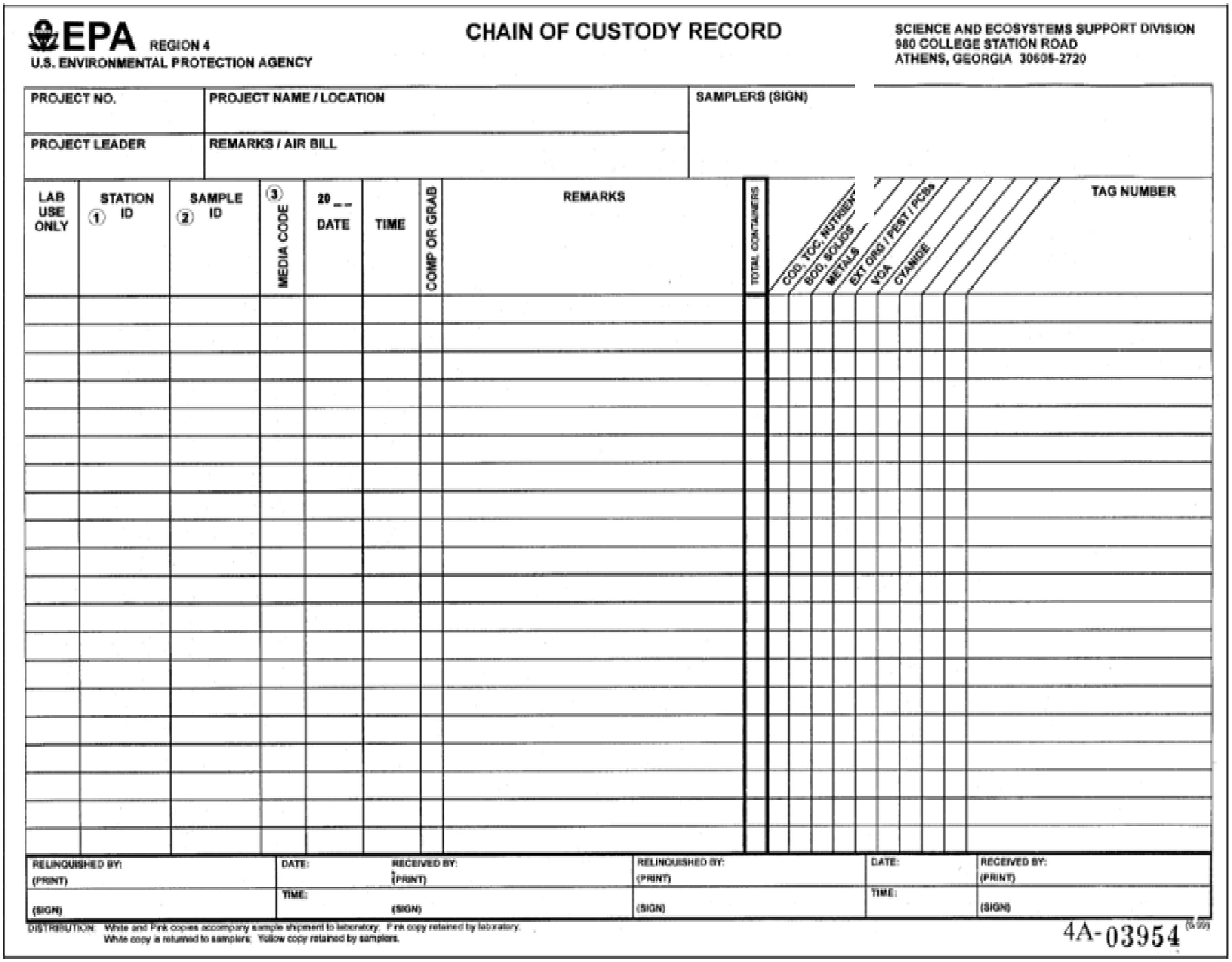
Attachment 1-Example Chain of Custody Form



EPA REGION 4
U.S. ENVIRONMENTAL PROTECTION AGENCY

CHAIN OF CUSTODY RECORD

SCIENCE AND ECOSYSTEMS SUPPORT DIVISION
980 COLLEGE STATION ROAD
ATHENS, GEORGIA 30605-2720

PROJECT NO.
PROJECT NAME / LOCATION
SAMPLERS (SIGN)
PROJECT LEADER
REMARKS / AIR BILL

LAB USE ONLY	STATION ① ID	SAMPLE ② ID	③ MEDIA CODE	20 _ _ DATE	TIME	COMP OR GRAB	REMARKS	TOTAL CONTAINERS	COD, TOC, NUTRIEN	BOD, SOLIDS	METALS	EXT ORG / PEST / PCBs	VOA	CYANIDE				TAG NUMBER

RELINQUISHED BY: (PRINT)	DATE:	RECEIVED BY: (PRINT)	RELINQUISHED BY: (PRINT)	DATE:	RECEIVED BY: (PRINT)
(SIGN)	TIME:	(SIGN)	(SIGN)	TIME:	(SIGN)

DISTRIBUTION: White and Pink copies accompany sample shipment to laboratory; Pink copy retained by laboratory.
White copy is returned to samplers; Yellow copy retained by samplers.

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Attachment 2 – Example Custody Seal

ATTACHMENT 2

CT Laboratories, LLC Analytical SOPs, Laboratory Quality Assurance Plan, and DoD Accreditation

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Scope of Accreditation
For
CT Laboratories, LLC

1230 Lange Ct
Baraboo, WI 53913
Christelle Newsome
608-356-2760

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (LABPR 403 DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM V5) based on the TNI Standard - Environmental Laboratory Sector, Volume 1 – Management and Technical Requirements for Laboratories Performing Environmental Analysis, Sept 2009 (EL-V1-2009); accreditation is granted to **CT Laboratories, LLC** to perform the following tests:

Accreditation granted through: **June 26, 2017**

Testing – Environmental

Non-Potable Water

Technology	Method	Analyte
Midi Dist / Colorimetric	EPA 9010 / EPA 9012	Total cyanide/Amen Cyanide
Distillation	EPA 9030 / EPA 9034	Sulfide
Titration	SM 4500-S2 F	Sulfide
GC	RSK-175	Carbon dioxide
GC	RSK-175	Ethane
GC	RSK-175	Ethylene
GC	RSK-175	Methane
Colorimetric	SM 3500-Fe B / UV-VIS	Iron
IC	SM 4500-CO2 D	Carbon dioxide (calc.)
IC	EPA 9056M	Acetic acid
IC	EPA 9056M	Butyric acid (Butanoic acid)
IC	EPA 9056M	Formic acid
IC	EPA 9056M	Lactic acid
IC	EPA 9056M	Propionic acid (Propanoic acid)
IC	EPA 9056M	Pyruvic acid
IC	EPA 9056M	Nitrocellulose
Automated Colorimetry	EPA 7.3.3.2	Reactive cyanide
Automated Colorimetry	EPA 7.3.4.2	Reactive sulfide
Probe	EPA 150.1	pH
Gravimetric	EPA 160.2	Residue-nonfilterable (TSS)

Non-Potable Water

Technology	Method	Analyte
Gravimetric	EPA 160.4	Residue-volatile
Nephelometry	EPA 180.1	Turbidity
ICP	EPA 200.7	Aluminum
ICP	EPA 200.7	Antimony
ICP	EPA 200.7	Arsenic
ICP	EPA 200.7	Barium
ICP	EPA 200.7	Beryllium
ICP	EPA 200.7	Boron
ICP	EPA 200.7	Cadmium
ICP	EPA 200.7	Calcium
ICP	EPA 200.7	Chromium
ICP	EPA 200.7	Cobalt
ICP	EPA 200.7	Copper
ICP	EPA 200.7	Iron
ICP	EPA 200.7	Lead
ICP	EPA 200.7	Lithium
ICP	EPA 200.7	Magnesium
ICP	EPA 200.7	Manganese
ICP	EPA 200.7	Nickel
ICP	EPA 200.7	Selenium
ICP	EPA 200.7	Silica as SiO2
ICP	EPA 200.7	Silver
ICP	EPA 200.7	Sodium
ICP	EPA 200.7	Strontium
ICP	EPA 200.7	Thallium
ICP	EPA 200.7	Tin
ICP	EPA 200.7	Titanium
ICP	EPA 200.7	Tungsten
ICP	EPA 200.7	Vanadium
ICP	EPA 200.7	Zinc
GFAA	EPA 200.9	Antimony
GFAA	EPA 200.9	Lead
GFAA	EPA 200.9	Selenium
GFAA	EPA 200.9	Thallium
CV	EPA 245.1	Mercury
GC	EPA 300.0	Bromide
GC	EPA 300.0	Chloride
GC	EPA 300.0	Fluoride
GC	EPA 300.0	Nitrate
GC	EPA 300.0	Nitrite
GC	EPA 300.0	Orthophosphate as P
IC	EPA 300.0	Sulfate
GC	EPA 300.0	Total nitrate-nitrite
Colorimetric	EPA 310.2	Alkalinity as CaCO3
Colorimetric	EPA 350.1	Ammonia as N

Non-Potable Water

Technology	Method	Analyte
Colorimetric	EPA 351.2	Kjeldahl nitrogen - total
IC	EPA 353.2	Nitrate as N
IC	EPA 353.2	Nitrate-nitrite
IC	EPA 353.2	Nitrite as N
Colorimetric	EPA 365.1	Orthophosphate as P
Colorimetric	EPA 365.1	Phosphorus, total
Colorimetric	EPA 365.4	Phosphorus, total
Titration	EPA 376.1	Sulfide
Colorimetric	EPA 410.4	Chemical oxygen demand
Oxidation Combustion	EPA 415.1	Total organic carbon
Pensky-Martens Closed-Cup	EPA 1010	Ignitability
Gravimetric	EPA 1664A	Oil & Grease
ICP	EPA 6010	Aluminum
ICP	EPA 6010	Antimony
ICP	EPA 6010	Arsenic
ICP	EPA 6010	Barium
ICP	EPA 6010	Beryllium
ICP	EPA 6010	Boron
ICP	EPA 6010	Cadmium
ICP	EPA 6010	Calcium
ICP	EPA 6010	Chromium
ICP	EPA 6010	Cobalt
ICP	EPA 6010	Copper
ICP	EPA 6010	Iron
ICP	EPA 6010	Lead
ICP	EPA 6010	Lithium
ICP	EPA 6010	Magnesium
ICP	EPA 6010	Molybdenum
ICP	EPA 6010	Nickel
ICP	EPA 6010	Potassium
ICP	EPA 6010	Selenium
ICP	EPA 6010	Silica as SiO2
ICP	EPA 6010	Silver
ICP	EPA 6010	Sodium
ICP	EPA 6010	Strontium
ICP	EPA 6010	Sulfur
ICP	EPA 6010	Thallium
ICP	EPA 6010	Tin
ICP	EPA 6010	Titanium
ICP	EPA 6010	Total hardness as CaCO3
ICP	SM 2340 B	Total hardness as CaCO3
ICP	EPA 6010	Tungsten
ICP	EPA 6010	Vanadium

Non-Potable Water

Technology	Method	Analyte
ICP	EPA 6010	Zinc
GFAA	EPA 7010	Antimony
GFAA	EPA 7010	Arsenic
GFAA	EPA 7010	Lead
GFAA	EPA 7010	Selenium
GFAA	EPA 7010	Silver
GFAA	EPA 7010	Thallium
Colorimetric	EPA 7196	Chromium VI
CV	EPA 7470	Mercury
GC	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
GC	EPA 8015	Diesel range organics (DRO)
GC	EPA 8015	Ethylene glycol
GC	EPA 8015	Gasoline range organics (GRO)
GC / GC/MS	EPA 8020	1,2,4-Trimethylbenzene
GC / GC/MS	EPA 8020	1,3,5-Trimethylbenzene
GC	EPA 8020	Benzene
GC	EPA 8020	Ethylbenzene
GC	EPA 8020	m+p-Xylenes
GC	EPA 8020	Methyl tert-butyl ether (MTBE)
GC / GC/MS	EPA 8020	Naphthalene
GC	EPA 8020	o-Xylene
GC	EPA 8020	Toluene
GC	EPA 8020	Xylene (total)
GC	EPA 8021	Benzene
GC	EPA 8021	Ethylbenzene
GC	EPA 8021	m-Xylene
GC	EPA 8021	Naphthalene
GC	EPA 8021	o-Xylene
GC	EPA 8021	p-Xylene
GC	EPA 8021	Toluene
GC	EPA 8021	Xylene (total)
GC	EPA 8081	4,4'-DDD
GC	EPA 8081	4,4'-DDE
GC	EPA 8081	4,4'-DDT
GC	EPA 8081	Aldrin
GC	EPA 8081	alpha-BHC (alpha-Hexachlorocyclohexane)
GC	EPA 8081	alpha-Chlordane
GC	EPA 8081	beta-BHC (beta-Hexachlorocyclohexane)
GC	EPA 8081	Chlordane (tech.)
GC	EPA 8081	delta-BHC
GC	EPA 8081	Dieldrin
GC	EPA 8081	Endosulfan I
GC	EPA 8081	Endosulfan II
GC	EPA 8081	Endosulfan sulfate
GC	EPA 8081	Endrin

Non-Potable Water

Technology	Method	Analyte
GC	EPA 8081	Endrin aldehyde
GC	EPA 8081	Endrin ketone
GC	EPA 8081	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)
GC	EPA 8081	gamma-Chlordane
GC	EPA 8081	Heptachlor
GC	EPA 8081	Heptachlor epoxide
GC	EPA 8081	Methoxychlor
GC	EPA 8081	Toxaphene (Chlorinated camphene)
GC	EPA 8082	Aroclor-1016 (PCB-1016)
GC	EPA 8082	Aroclor-1221 (PCB-1221)
GC	EPA 8082	Aroclor-1232 (PCB-1232)
GC	EPA 8082	Aroclor-1242 (PCB-1242)
GC	EPA 8082	Aroclor-1248 (PCB-1248)
GC	EPA 8082	Aroclor-1254 (PCB-1254)
GC	EPA 8082	Aroclor-1260 (PCB-1260)
GC/MS	EPA 8260	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260	1,1,1-Trichloroethane
GC/MS	EPA 8260	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260	1,1,2-Trichloroethane
GC/MS	EPA 8260	1,1-Dichloroethane
GC/MS	EPA 8260	1,1-Dichloroethylene
GC/MS	EPA 8260	1,1-Dichloropropene
GC/MS	EPA 8260	1,2,3-Trichlorobenzene
GC/MS	EPA 8260	1,2,3-Trichloropropane
GC/MS	EPA 8260	1,2,4-Trichlorobenzene
GC/MS	EPA 8260	1,2,4-Trimethylbenzene
GC/MS	EPA 8260	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260	1,2-Dichloro-1,1,2-trifluoroethane
GC/MS	EPA 8260	1,2-Dichlorobenzene
GC/MS	EPA 8260	1,2-Dichloroethane
GC/MS	EPA 8260	1,3,5-Trimethylbenzene
GC/MS	EPA 8260	1,3-Dichlorobenzene
GC/MS	EPA 8260	1,3-Dichloropropane
GC/MS	EPA 8260	1,4-Dichlorobenzene
GC/MS	EPA 8260	1,4-Dioxane (1,4-Diethyleneoxide)
GC/MS	EPA 8260	1-Chlorohexane
GC/MS	EPA 8260	2,2-Dichloropropane
GC/MS	EPA 8260	2,3-Dichloropropene
GC/MS	EPA 8260	2-Butanone (Methyl ethyl ketone, MEK)
GC/MS	EPA 8260	2-Chloroethyl vinyl ether
GC/MS	EPA 8260	2-Chlorotoluene
GC/MS	EPA 8260	2-Hexanone
GC/MS	EPA 8260	4-Chlorotoluene

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8260	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260	Acetone
GC/MS	EPA 8260	Acetonitrile
GC/MS	EPA 8260	Acrolein (Propenal)
GC/MS	EPA 8260	Acrylonitrile
GC/MS	EPA 8260	Benzene
GC/MS	EPA 8260	Bromobenzene
GC/MS	EPA 8260	Bromochloromethane
GC/MS	EPA 8260	Bromoform
GC/MS	EPA 8260	Carbon disulfide
GC/MS	EPA 8260	Carbon tetrachloride
GC/MS	EPA 8260	Chlorobenzene
GC/MS	EPA 8260	Chloroethane
GC/MS	EPA 8260	Chloroform
GC/MS	EPA 8260	cis-1,2-Dichloroethylene
GC/MS	EPA 8260	cis-1,3-Dichloropropene
GC/MS	EPA 8260	Cyclohexane
GC/MS	EPA 8260	Cyclohexanone
GC/MS	EPA 8260	Dibromochloromethane
GC/MS	EPA 8260	Dibromomethane
GC/MS	EPA 8260	Dichlorodifluoromethane
GC/MS	EPA 8260	Diethyl ether
GC/MS	EPA 8260	Di-isopropylether (DIPE)
GC/MS	EPA 8260	Ethyl acetate
GC/MS	EPA 8260	Ethylbenzene
GC/MS	EPA 8260	Hexane
GC/MS	EPA 8260	Iodomethane (Methyl iodide)
GC/MS	EPA 8260	Isopropylbenzene
GC/MS	EPA 8260	Methyl acetate
GC/MS	EPA 8260	Methyl bromide (Bromomethane)
GC/MS	EPA 8260	Methyl chloride (Chloromethane)
GC/MS	EPA 8260	Methylcyclohexane
GC/MS	EPA 8260	m-Xylene
GC/MS	EPA 8260	Naphthalene
GC/MS	EPA 8260	o-Xylene
GC/MS	EPA 8260	p-Isopropyltoluene
GC/MS	EPA 8260	Propylene oxide
GC/MS	EPA 8260	p-Xylene
GC/MS	EPA 8260	sec-Butylbenzene
GC/MS	EPA 8260	tert-Butyl alcohol
GC/MS	EPA 8260	tert-Butylbenzene
GC/MS	EPA 8260	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260	Toluene
GC/MS	EPA 8260	trans-1,2-Dichloroethylene
GC/MS	EPA 8260	trans-1,3-Dichloropropene

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8260	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260	Trichlorofluoromethane
GC/MS	EPA 8260	Vinyl acetate
GC/MS	EPA 8260	Vinyl chloride
GC/MS	EPA 8260	Xylene (total)
GC/MS	EPA 8260	n-Butylbenzene
GC/MS	EPA 8260	1,2-Dichloropropane
GC/MS	EPA 8260	Hexachlorobutadiene
GC/MS	EPA 8260	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260	Styrene
GC/MS	EPA 8260	Nitrobenzene
GC/MS	EPA 8270	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270	1,2,4-Trichlorobenzene
GC/MS	EPA 8270	1,2-Dichlorobenzene
GC/MS	EPA 8270	1,2-Dinitrobenzene
GC/MS	EPA 8270	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270	1,3-Dichlorobenzene
GC/MS	EPA 8270	1,4-Dichlorobenzene
GC/MS	EPA 8270	1,4-Dioxane (1,4-Diethyleneoxide)
GC/MS	EPA 8270	4-Nitrophenol
GC/MS	EPA 8270	4-Chloro-3-Methylphenol
GC/MS	EPA 8270	4-Bromophenyl Phenyl Ether
GC/MS	EPA 8270	Acenaphthene
GC/MS	EPA 8270	Acenaphthylene
GC/MS	EPA 8270	Acetophenone
GC/MS	EPA 8270	alpha-Terpineol
GC/MS	EPA 8270	Aniline
GC/MS	EPA 8270	Anthracene
GC/MS	EPA 8270	Atrazine
GC/MS	EPA 8270	Benzidine
GC/MS	EPA 8270	Benzo(a)anthracene
GC/MS	EPA 8270	Benzo(a)pyrene
GC/MS	EPA 8270	Benzo(b)fluoranthene
GC/MS	EPA 8270	Benzo(g,h,i)perylene
GC/MS	EPA 8270	Benzo(j)fluoranthene
GC/MS	EPA 8270	Benzo(k)fluoranthene
GC/MS	EPA 8270	Benzoic acid
GC/MS	EPA 8270	Benzyl alcohol
GC/MS	EPA 8270	Biphenyl
GC/MS	EPA 8270	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270	bis(2-Chloroethyl) ether
GC/MS	EPA 8270	bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))
GC/MS	EPA 8270	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270	Butyl benzyl phthalate

Non-Potable Water

Technology	Method	Analyte
GC/MS	EPA 8270	Caprolactam
GC/MS	EPA 8270	Carbazole
GC/MS	EPA 8270	Chrysene
GC/MS	EPA 8270	Cyanazine (Bladex)
GC/MS	EPA 8270	Dibenz(a,h)anthracene
GC/MS	EPA 8270	Dibenzofuran
GC/MS	EPA 8270	Diethyl phthalate
GC/MS	EPA 8270	Dimethyl phthalate
GC/MS	EPA 8270	Di-n-butyl phthalate
GC/MS	EPA 8270	Di-n-octyl phthalate
GC/MS	EPA 8270	Diphenylamine
GC/MS	EPA 8270	Hexachlorobenzene
GC/MS	EPA 8270	Hexachlorobutadiene
GC/MS	EPA 8270	Hexachlorocyclopentadiene
GC/MS	EPA 8270	Hexachloroethane
GC/MS	EPA 8270	Hexachlorophene
GC/MS	EPA 8270	Hexachloropropene
GC/MS	EPA 8270	Isophorone
GC/MS	EPA 8270	Naphthalene
GC/MS	EPA 8270	n-Nitrosodiethylamine
GC/MS	EPA 8270	n-Nitrosodimethylamine
GC/MS	EPA 8270	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270	n-Nitrosodiphenylamine
GC/MS	EPA 8270	n-Nitrosomethylethylamine
GC/MS	EPA 8270	n-Nitrosopyrrolidine
GC/MS	EPA 8270	Pentachlorophenol
GC/MS	EPA 8270	Phenanthrene
GC/MS	EPA 8270	Phenol
GC/MS	EPA 8270	Pyrene
GC/MS	EPA 8270	Pyridine
GC/MS	EPA 8270	Simazine
GC/MS	EPA 8270	2,3-DNT
GC/MS	EPA 8270	2,5-DNT
GC/MS	EPA 8270	3,4-DNT
GC/MS	EPA 8270	3,5-DNT
HPLC	EPA 8310	Acenaphthene
HPLC	EPA 8310	Acenaphthylene
HPLC	EPA 8310	Anthracene
HPLC	EPA 8310	Benzo(a)anthracene
HPLC	EPA 8310	Benzo(a)pyrene
HPLC	EPA 8310	Benzo(b)fluoranthene
HPLC	EPA 8310	Benzo(g,h,i)perylene
HPLC	EPA 8310	Benzo(k)fluoranthene
HPLC	EPA 8310	Chrysene
HPLC	EPA 8310	Naphthalene

Non-Potable Water

Technology	Method	Analyte
HPLC	EPA 8310	Phenanthrene
HPLC	EPA 8310	Pyrene
HPLC	EPA 8310	Dibenz(a,h)anthracene
HPLC	EPA 8310	Fluoranthene
HPLC	EPA 8310	Fluorene
HPLC	EPA 8310	Indeno(1,2,3-c.d)Pyrene
HPLC	EPA 8330	1,3,5-Trinitrobenzene (1,3,5-TNB)
HPLC	EPA 8330	1,3-Dinitrobenzene (1,3-DNB)
HPLC	EPA 8330	2,4,6-Trinitrotoluene (2,4,6-TNT)
HPLC	EPA 8330	2,4-Dinitrotoluene (2,4-DNT)
HPLC	EPA 8330	2,6-Dinitrotoluene (2,6-DNT)
HPLC	EPA 8330	2-Amino-4,6-dinitrotoluene (2-am-dnt)
HPLC	EPA 8330	2-Nitrotoluene
HPLC	EPA 8330	3-Nitrotoluene
HPLC	EPA 8330	4-Amino-2,6-dinitrotoluene (4-am-dnt)
HPLC	EPA 8330	4-Nitrotoluene
HPLC	EPA 8330	Nitrobenzene
HPLC	EPA 8330	Nitroglycerin
HPLC	EPA 8330	Nitroguanidine
HPLC	EPA 8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC	EPA 8330	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
HPLC	EPA 8330	Tetryl (methyl-2,4,6-trinitrophenylnitramine)
GC	EPA 8332	Nitroglycerin
PROBE	EPA 9040	pH
PROBE	EPA 9041	pH
Conductivity	EPA 9050	Conductivity
IC	EPA 9056	Bromide
IC	EPA 9056	Chloride
IC	EPA 9056	Fluoride
IC	EPA 9056	Nitrate
IC	EPA 9056	Nitrite
IC	EPA 9056	Orthophosphate as P
IC	EPA 9056	Total nitrate-nitrite
Oxidation Combustion	EPA 9060	Total organic carbon
Colorimetric	EPA 9066	Total phenolics
Gravimetric	SM 2540 C	Residue-filterable (TDS)
Gravimetric	SM 2540 B	Residue-total
Gravimetric	SM 2540 D	Residue-nonfilterable (TSS)
IC	SM 4110 B	Bromide
IC	SM 4110 B	Chloride
IC	SM 4110 B	Fluoride
IC	SM 4110 B	Nitrate
IC	SM 4110 B	Nitrite
IC	SM 4110 B	Orthophosphate as P

Non-Potable Water

Technology	Method	Analyte
IC	SM 4110 B	Total nitrate-nitrite
Probe	SM 4500-H+-B	pH
Colorimetric	SM 4500-NH3 G	Ammonia as N
DO	SM 5210 B	Biochemical oxygen demand
Spectrometry	SM 10200 H	Chlorophyll a
Spectrometry	SM 10200 H	Chlorophyll b
Spectrometry	SM 10200 H	Chlorophyll c
GC	EPA 8081	2,4'-DDD
GC	EPA 8081	2,4'-DDE
GC	EPA 8081	2,4'-DDT

Solid and Chemical Materials

Technology	Method	Analyte
Colorimetric	EPA 9056M	Acetic acid
IC	EPA 9056M	Butyric acid (Butanoic acid)
IC	EPA 9056M	Formic acid
IC	EPA 9056M	Lactic acid
IC	EPA 9056M	Propionic acid (Propanoic acid)
IC	EPA 9056M	Pyruvic acid
IC	EPA 9056M	Nitrocellulose
IC	EPA 7.3.3.2	Reactive cyanide
IC	EPA 7.3.4.2	Reactive sulfide
IC	EPA 353.2	Total nitrate-nitrite
Pensky-Martens Closed-Cup	EPA 1010	Ignitability
Leaching	EPA 1311	Toxicity Characteristic Leaching Procedure
Leaching	EPA 1312	Synthetic Precipitation Leaching Procedure
ICP	EPA 6010	Sulfur
GFAA	EPA 7010	Antimony
GFAA	EPA 7010	Arsenic
GFAA	EPA 7010	Lead
GFAA	EPA 7010	Selenium
GFAA	EPA 7010	Silver
GFAA	EPA 7010	Thallium
Colorimetric	EPA 7196	Chromium VI
CV	EPA 7471	Mercury
GC	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
GC	EPA 8011	1,2-Dibromoethane (EDB, Ethylene dibromide)
GC	EPA 8015	Formic acid
GC	EPA 8015	Ethylene glycol
GC	EPA 8015	Gasoline range organics (GRO)

Solid and Chemical Materials

Technology	Method	Analyte
GC	EPA 8020	1,3,5-Trimethylbenzene
GC	EPA 8020	Benzene
GC	EPA 8020	Ethylbenzene
GC	EPA 8020	m+p-Xylenes
GC	EPA 8020	Methyl tert-butyl ether (MTBE)
GC	EPA 8020	Naphthalene
GC	EPA 8020	o-Xylene
GC	EPA 8020	Toluene
GC	EPA 8020	Xylene (total)
GC	EPA 8021	Benzene
GC	EPA 8021	Ethylbenzene
GC	EPA 8021	Methyl tert-butyl ether (MTBE)
GC	EPA 8021	Toluene
GC	EPA 8021	Xylene (total)
GC	EPA 8021	o-Xylene
GC	EPA 8021	m+p-Xylenes
GC/MS	EPA 8260	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260	1,1,1-Trichloroethane
GC/MS	EPA 8260	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260	1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)
GC/MS	EPA 8260	1,1,2-Trichloroethane
GC/MS	EPA 8260	1,1-Dichloroethane
GC/MS	EPA 8260	1,1-Dichloroethylene
GC/MS	EPA 8260	1,1-Dichloropropene
GC/MS	EPA 8260	1,2,3-Trichlorobenzene
GC/MS	EPA 8260	1,2,3-Trichloropropane
GC/MS	EPA 8260	1,2,4-Trichlorobenzene
GC/MS	EPA 8260	1,2,4-Trimethylbenzene
GC/MS	EPA 8260	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260	1,2-Dichloro-1,1,2-trifluoroethane
GC/MS	EPA 8260	1,2-Dichlorobenzene
GC/MS	EPA 8260	1,2-Dichloroethane
GC/MS	EPA 8260	1,3,5-Trimethylbenzene
GC/MS	EPA 8260	1,3-Dichlorobenzene
GC/MS	EPA 8260	1,3-Dichloropropane
GC/MS	EPA 8260	1,4-Dichlorobenzene
GC/MS	EPA 8260	1,4-Dioxane (1,4-Diethyleneoxide)
GC/MS	EPA 8260	1-Chlorohexane
GC/MS	EPA 8260	2,2-Dichloropropane
GC/MS	EPA 8260	2,3-Dichloropropene
GC/MS	EPA 8260	2-Butanone (Methyl ethyl ketone, MEK)
GC/MS	EPA 8260	2-Chloroethyl vinyl ether
GC/MS	EPA 8260	2-Chlorotoluene
GC/MS	EPA 8260	2-Hexanone

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8260	4-Chlorotoluene
GC/MS	EPA 8260	4-Methyl-2-pentanone (MIBK)
GC/MS	EPA 8260	Acetone
GC/MS	EPA 8260	Acetonitrile
GC/MS	EPA 8260	Acrolein (Propenal)
GC/MS	EPA 8260	Acrylonitrile
GC/MS	EPA 8260	Benzene
GC/MS	EPA 8260	Bromobenzene
GC/MS	EPA 8260	Bromochloromethane
GC/MS	EPA 8260	Bromoform
GC/MS	EPA 8260	Carbon disulfide
GC/MS	EPA 8260	Carbon tetrachloride
GC/MS	EPA 8260	Chlorobenzene
GC/MS	EPA 8260	Chloroethane
GC/MS	EPA 8260	Chloroform
GC/MS	EPA 8260	cis-1,2-Dichloroethylene
GC/MS	EPA 8260	cis-1,3-Dichloropropene
GC/MS	EPA 8260	Cyclohexane
GC/MS	EPA 8260	Cyclohexanone
GC/MS	EPA 8260	Dibromochloromethane
GC/MS	EPA 8260	Dibromomethane
GC/MS	EPA 8260	Dichlorodifluoromethane
GC/MS	EPA 8260	Diethyl ether
GC/MS	EPA 8260	Di-isopropylether (DIPE)
GC/MS	EPA 8260	Ethyl acetate
GC/MS	EPA 8260	Ethylbenzene
GC/MS	EPA 8260	Hexane
GC/MS	EPA 8260	Iodomethane (Methyl iodide)
GC/MS	EPA 8260	Isopropylbenzene
GC/MS	EPA 8260	Methyl acetate
GC/MS	EPA 8260	Methyl bromide (Bromomethane)
GC/MS	EPA 8260	Methyl chloride (Chloromethane)
GC/MS	EPA 8260	Methylcyclohexane
GC/MS	EPA 8260	m-Xylene
GC/MS	EPA 8260	Naphthalene
GC/MS	EPA 8260	o-Xylene
GC/MS	EPA 8260	p-Isopropyltoluene
GC/MS	EPA 8260	Propylene oxide
GC/MS	EPA 8260	p-Xylene
GC/MS	EPA 8260	sec-Butylbenzene
GC/MS	EPA 8260	tert-Butyl alcohol
GC/MS	EPA 8260	tert-Butylbenzene
GC/MS	EPA 8260	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260	Toluene
GC/MS	EPA 8260	trans-1,2-Dichloroethylene

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8260	trans-1,3-Dichloropropene
GC/MS	EPA 8260	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260	Trichlorofluoromethane
GC/MS	EPA 8260	Vinyl acetate
GC/MS	EPA 8260	Vinyl chloride
GC/MS	EPA 8260	Xylene (total)
GC/MS	EPA 8260	n-Butylbenzene
GC/MS	EPA 8260	1,2-Dichloropropane
GC/MS	EPA 8260	Hexachlorobutadiene
GC/MS	EPA 8260	Methyl tert-butyl ether (MTBE)
GC/MS	EPA 8260	Styrene
GC/MS	EPA 8260	Nitrobenzene
GC/MS	EPA 8270	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270	1,2,4-Trichlorobenzene
GC/MS	EPA 8270	1,2-Dichlorobenzene
GC/MS	EPA 8270	1,2-Dinitrobenzene
GC/MS	EPA 8270	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270	1,3-Dichlorobenzene
GC/MS	EPA 8270	1,3-Dinitrobenzene (1,3-DNB)
GC/MS	EPA 8270	1,4-Dichlorobenzene
GC/MS	EPA 8270	1,4-Dinitrobenzene
GC/MS	EPA 8270	1,4-Dioxane (1,4-Diethyleneoxide)
GC/MS	EPA 8270	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8260	tert-Butyl alcohol
GC/MS	EPA 8260	tert-Butylbenzene
GC/MS	EPA 8260	Tetrachloroethylene (Perchloroethylene)
GC/MS	EPA 8260	Toluene
GC/MS	EPA 8260	trans-1,2-Dichloroethylene
GC/MS	EPA 8260	trans-1,3-Dichloropropene
GC/MS	EPA 8260	Trichloroethene (Trichloroethylene)
GC/MS	EPA 8260	Trichlorofluoromethane
GC/MS	EPA 8260	Vinyl acetate
GC/MS	EPA 8260	Vinyl chloride
GC/MS	EPA 8260	Xylene (total)
GC/MS	EPA 8270	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270	1,2,4-Trichlorobenzene
GC/MS	EPA 8270	1,2-Dichlorobenzene
GC/MS	EPA 8270	1,2-Dinitrobenzene
GC/MS	EPA 8270	1,2-Diphenylhydrazine (as Azobenzene)
GC/MS	EPA 8270	1,3-Dichlorobenzene
GC/MS	EPA 8270	1,4-Dichlorobenzene
GC/MS	EPA 8270	1,4-Dioxane (1,4-Diethyleneoxide)
GC/MS	EPA 8270	4-Nitrophenol
GC/MS	EPA 8270	4-Chloro-3-Methylphenol

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8270	4-Bromophenyl Phenyl Ether
GC/MS	EPA 8270	Acenaphthene
GC/MS	EPA 8270	Acenaphthylene
GC/MS	EPA 8270	Acetophenone
GC/MS	EPA 8270	alpha-Terpineol
GC/MS	EPA 8270	Aniline
GC/MS	EPA 8270	Anthracene
GC/MS	EPA 8270	Atrazine
GC/MS	EPA 8270	Benzidine
GC/MS	EPA 8270	Benzo(a)anthracene
GC/MS	EPA 8270	Benzo(a)pyrene
GC/MS	EPA 8270	Benzo(b)fluoranthene
GC/MS	EPA 8270	Benzo(g,h,i)perylene
GC/MS	EPA 8270	Benzo(j)fluoranthene
GC/MS	EPA 8270	Benzo(k)fluoranthene
GC/MS	EPA 8270	Benzoic acid
GC/MS	EPA 8270	Benzyl alcohol
GC/MS	EPA 8270	Biphenyl
GC/MS	EPA 8270	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270	bis(2-Chloroethyl) ether
GC/MS	EPA 8270	bis(2-Chloroisopropyl) ether (2,2'-Oxybis(1-chloropropane))
GC/MS	EPA 8270	bis(2-Ethylhexyl) phthalate (DEHP)
GC/MS	EPA 8270	Butyl benzyl phthalate
GC/MS	EPA 8270	Caprolactam
GC/MS	EPA 8270	Carbazole
GC/MS	EPA 8270	Chrysene
GC/MS	EPA 8270	Cyanazine (Bladex)
GC/MS	EPA 8270	Dibenz(a,h)anthracene
GC/MS	EPA 8270	Dibenzofuran
GC/MS	EPA 8270	Diethyl phthalate
GC/MS	EPA 8270	Dimethyl phthalate
GC/MS	EPA 8270	Di-n-butyl phthalate
GC/MS	EPA 8270	Di-n-octyl phthalate
GC/MS	EPA 8270	Diphenylamine
GC/MS	EPA 8270	Hexachlorobenzene
GC/MS	EPA 8270	Hexachlorobutadiene
GC/MS	EPA 8270	Hexachlorocyclopentadiene
GC/MS	EPA 8270	Hexachloroethane
GC/MS	EPA 8270	Hexachlorophene
GC/MS	EPA 8270	Hexachloropropene
GC/MS	EPA 8270	Isophorone
GC/MS	EPA 8270	Naphthalene
GC/MS	EPA 8270	n-Nitrosodiethylamine

Solid and Chemical Materials

Technology	Method	Analyte
GC/MS	EPA 8270	n-Nitrosodimethylamine
GC/MS	EPA 8270	n-Nitrosodi-n-propylamine
GC/MS	EPA 8270	n-Nitrosodiphenylamine
GC/MS	EPA 8270	n-Nitrosomethylethylamine
GC/MS	EPA 8270	n-Nitrosopyrrolidine
GC/MS	EPA 8270	Pentachlorophenol
GC/MS	EPA 8270	Phenanthrene
GC/MS	EPA 8270	Phenol
GC/MS	EPA 8270	Pyrene
GC/MS	EPA 8270	Pyridine
GC/MS	EPA 8270	Simazine
GC/MS	EPA 8270	2,3-DNT
GC/MS	EPA 8270	2,5-DNT
GC/MS	EPA 8270	3,4-DNT
GC/MS	EPA 8270	3,5-DNT
HPLC	EPA 8330	1,3,5-Trinitrobenzene (1,3,5-TNB)
HPLC	EPA 8330	1,3-Dinitrobenzene (1,3-DNB)
HPLC	EPA 8330	2,4,6-Trinitrotoluene (2,4,6-TNT)
HPLC	EPA 8330	2,4-Dinitrotoluene (2,4-DNT)
HPLC	EPA 8330	2,6-Dinitrotoluene (2,6-DNT)
HPLC	EPA 8330	2-Amino-4,6-dinitrotoluene (2-am-dnt)
HPLC	EPA 8330	2-Nitrotoluene
HPLC	EPA 8330	3,5-Dinitroaniline
HPLC	EPA 8330	3-Nitrotoluene
HPLC	EPA 8330	4-Amino-2,6-dinitrotoluene (4-am-dnt)
HPLC	EPA 8330	4-Nitrotoluene
HPLC	EPA 8330	Nitrobenzene
HPLC	EPA 8330	Nitroglycerin
HPLC/GC	EPA 8330	Nitroguanidine
HPLC	EPA 8330	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC	EPA 8330	Pentaerythritoltetranitrate (PETN)
HPLC	EPA 8330	RDX (hexahydro-1,3,5-trinitro-1,3,5-triazine)
HPLC	EPA 8330	Tetryl (methyl-2,4,6-trinitrophenylnitramine)
GC	EPA 8332	Nitroglycerin
Probe	EPA 9040	Corrosivity (pH)
IC	EPA 9056	Chloride
Physical	EPA 9095	Paint Filter Liquids Test
GC	EPA 8081	2,4'-DDD
GC	EPA 8081	2,4'-DDE
GC	EPA 8081	2,4'-DDT
ICP	EPA 6010	Aluminum
ICP	EPA 6010	Antimony
ICP	EPA 6010	Arsenic
ICP	EPA 6010	Barium

Solid and Chemical Materials

Technology	Method	Analyte
ICP	EPA 6010	Beryllium
ICP	EPA 6010	Boron
ICP	EPA 6010	Cadmium
ICP	EPA 6010	Calcium
ICP	EPA 6010	Chromium
ICP	EPA 6010	Cobalt
ICP	EPA 6010	Copper
ICP	EPA 6010	Iron
ICP	EPA 6010	Lead
ICP	EPA 6010	Lithium
ICP	EPA 6010	Magnesium
ICP	EPA 6010	Molybdenum
ICP	EPA 6010	Nickel
ICP	EPA 6010	Potassium
ICP	EPA 6010	Selenium
ICP	EPA 6010	Silica as SiO2
ICP	EPA 6010	Silver
ICP	EPA 6010	Sodium
ICP	EPA 6010	Strontium
ICP	EPA 6010	Sulfur
ICP	EPA 6010	Thallium
ICP	EPA 6010	Tin
ICP	EPA 6010	Titanium
ICP	EPA 6010	Tungsten
ICP	EPA 6010	Vanadium
ICP	EPA 6010	Zinc
GFAA	EPA 7010	Antimony
GFAA	EPA 7010	Arsenic
GFAA	EPA 7010	Lead
GFAA	EPA 7010	Selenium
GFAA	EPA 7010	Silver
GFAA	EPA 7010	Thallium
Colorimetric	EPA 7196	Chromium VI
CV	EPA 7470	Mercury

Biological Tissue

Technology	Method	Analyte
ICP	EPA 6010	Aluminum
ICP	EPA 6010	Antimony
ICP	EPA 6010	Arsenic
ICP	EPA 6010	Barium
ICP	EPA 6010	Beryllium

Biological Tissue

Technology	Method	Analyte
ICP	EPA 6010	Boron
ICP	EPA 6010	Cadmium
ICP	EPA 6010	Calcium
ICP	EPA 6010	Chromium
ICP	EPA 6010	Cobalt
ICP	EPA 6010	Copper
ICP	EPA 6010	Iron
ICP	EPA 6010	Lead
ICP	EPA 6010	Lithium
ICP	EPA 6010	Magnesium
ICP	EPA 6010	Molybdenum
ICP	EPA 6010	Nickel
ICP	EPA 6010	Potassium
ICP	EPA 6010	Selenium
ICP	EPA 6010	Silver
ICP	EPA 6010	Sodium
ICP	EPA 6010	Strontium
ICP	EPA 6010	Thallium
ICP	EPA 6010	Tin
ICP	EPA 6010	Titanium
ICP	EPA 6010	Tungsten
ICP	EPA 6010	Vanadium
ICP	EPA 6010	Zinc
CV	EPA 7471	Mercury
GC	EPA 8081	4,4'-DDD
GC	EPA 8081	4,4'-DDE
GC	EPA 8081	4,4'-DDT
GC	EPA 8081	2,4'-DDD
GC	EPA 8081	2,4'-DDE
GC	EPA 8081	2,4'-DDT
GC	EPA 8081	Alachlor
GC	EPA 8081	Aldrin
GC	EPA 8081	alpha-BHC (alpha-Hexachlorocyclohexane)
GC	EPA 8081	alpha-Chlordane
GC	EPA 8081	beta-BHC (beta-Hexachlorocyclohexane)
GC	EPA 8081	Chlordane (tech.)
GC	EPA 8081	delta-BHC
GC	EPA 8081	Endosulfan I
GC	EPA 8081	Endosulfan II
GC	EPA 8081	Endosulfan sulfate
GC	EPA 8081	Endrin
GC	EPA 8081	Endrin aldehyde
GC	EPA 8081	Endrin ketone
GC	EPA 8081	gamma-BHC (Lindane, gamma-Hexachlorocyclohexane)

Biological Tissue

Technology	Method	Analyte
GC	EPA 8081	gamma-Chlordane
GC	EPA 8081	Heptachlor
GC	EPA 8081	Heptachlor epoxide
GC	EPA 8081	Methoxychlor
GC	EPA 8081	Toxaphene (Chlorinated camphene)
GC	EPA 8082	Aroclor-1016 (PCB-1016)
GC	EPA 8082	Aroclor-1221 (PCB-1221)
GC	EPA 8082	Aroclor-1232 (PCB-1232)
GC	EPA 8082	Aroclor-1242 (PCB-1242)
GC	EPA 8082	Aroclor-1248 (PCB-1248)
GC	EPA 8082	Aroclor-1254 (PCB-1254)
GC	EPA 8082	Aroclor-1260 (PCB-1260)
GC/MS	EPA 8270	Alachlor
GC/MS	EPA 8270	2,3-DNT
GC/MS	EPA 8270	2,5-DNT
GC/MS	EPA 8270	3,4-DNT
GC/MS	EPA 8270	3,5-DNT

Preparation for Above Matrices	Method	Type
Florisil Cleanup	EPA 3620C	Extraction Clean Up
Gel-Permeantion CleanUp	EPA 3640A	Extraction Clean Up
SulfurcAcid/Permanganate Cleanup	EPA 3665A	Extraction Clean Up
Sulfur	EPA 3660B	Extraction Clean Up
TCLP	EPA 1311	Leaching
SPLP	EPA 1312	Leaching
Acid Digestion	EPA 3005A	Metals Prep
Acid Digestion	EPA 3010A EPA 3020A	Metals Prep
Hot Block	EPA 3050B	Metals Prep
Microwave Extraction	EPA 3546	Organic Extraction
Liquid/Liquid Extraction	EPA 3510C	Organic Extraction
Solid Phase Extraction	EPA 3535A	Organic Extraction
Soxhlet Extraction	EPA 3540C	Organic Extraction
Waste Dilution	EPA 3580A	Organic Extraction
Purge-and-Trap and Extraction	EPA 5035	Volatiles Prep
Purge-and-Trap and Extraction	EPA 5030B	Volatiles Prep

Notes:

1) This laboratory offers commercial testing service.

Approved by: ______________________

R. Douglas Leonard
Chief Technical Officer

Date: June 26, 2014

Initial: 6/26/14

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STANDARD OPERATING PROCEDURE

MT 009 Inductively Coupled Plasma (ICP) Emission – ICP-OES 6000

Review Date:
03/02/2016

Randy

03/02/2016

Technical Review by: Date

Colleen

03/31/2016

Approved by: Quality Assurance Date

1.0 Identification of Test Method

1.1 This procedure is used for the analysis of trace elements (metals) following EPA SW 846 Methods 6010B and/or 6010C (Inductively Coupled Plasma – Atomic Emission Spectrometry) and Method 200.7 (SDWA).

2.0 Applicable Matrix or Matrixes

2.1 This method is applicable to the determination of various metals in drinking water, surface water, groundwater, sludge, soils, and industrial wastes.

3.0 Detection Limits

3.1 Method detection limits (MDLs) are determined annually and results vary from element to element. For DOD-QSM, ACOE-LCG work MDL's and LOQ's are verified initially and quarterly thereafter. MDL checks are analyzed up to 3x the calculated MDL (to verify sensitivity). If MDL checks are not detected at the spiked level for any given element than increase the level of the spike until the element is detected (minimum 2 successful analyses). The level at which an element was successfully detected becomes the reported MDL. LOQ checks are used for accuracy verification and shall meet project or client specific control limits. Failing LOQ checks may also need elevation of the spike levels for a given element. The concentration that successfully meets control criteria is used as the reported LOQ. Procedures for conducting MDL studies can be found in CT Laboratories Initial Method Performance and Reporting SOP (CL-2, rev. 5).

4.0 Scope and Application

4.1 Metals in solution can be readily analyzed by atomic emission using an Inductively Coupled Plasma (ICP) spectrometer. All matrices, excluding filtered groundwater samples and drinking waters with a turbidity less than 1 NTU, will require a digestion prior to analysis.

5.0 Method Summary

5.1 If necessary, prior to analysis, samples are digested using an approved method. See SOPs 6205B, 6225B, 6230B, M200.2, and M-soluble for further information on sample digestion.

5.2 This method describes multi-element determinations using an iCAP 6000 Series ICP-OES which use an Echelle optical design and a Charge Injection Device (CID) solid-state detector to provide elemental analysis. Most Samples are liquids that are pumped through a nebulizer to produce a fine spray. The large droplets are removed by a spray chamber and the small droplets then pass through to the plasma. The solvent is evaporated. The residual sample is decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving measurement of the concentration of each element type in the original sample. Control of the spectrometer is provided by the PC based iTeva software (refer to the Thermo ICP-OES software manual). Samples are routinely analyzed using an internal standard solution of 50 mg/L Yttrium to eliminate certain matrix interference problems. Line switching is also used to extend the dynamic range of an element.

5.3 The data is exported to the LIMS system and reviewed by the analyst. Following analyst review, the data is given to a qualified reviewer for complete data review. After the data has been reviewed and it is determined that it is valid data, the reviewer sends the data to the "validated" mode in the LIMS system.

6.0 Definitions

6.1 Reagent Blank- A solution of de-ionized water, (containing in correct proportion, all reagents required by the method), used with the calibration standards to standardize the instrument, as a calibration blank, and for sample dilution.

6.2 Calibration Standards - A series of known standard solutions, which shall include the reagent blank, used for calibration of the instrument within the measurable linear range. Calibration standards shall contain, in correct proportion, all reagents required by the method. Acceptance of the calibration requires a correlation coefficient (r) of 0.995 or better. No samples shall be analyzed without acceptable calibration. For DOD-QSM data the low calibration standard shall be equal to or less than the MRL.

6.3 Calibration Verification Standard-Initial (ICV) & Continuing (CCV) - A midpoint calibration standard which is analyzed at the beginning of the run (ICV), at a frequency of 1 per 10 samples during a run (CCV), and at the end of a run to verify calibration throughout the run. The ICV must be from a second source different than that of the calibration standards.

Note for method 200.7 that limits for ICV are tighter than those for the CCV (see section 16).

6.4 Low-Level Calibration Check Standard – If a single point calibration is used (for DOD-QSM data) then a low level standard shall be analyzed. The acceptance criterion is +/- 20% of the expected value.

6.5 CB (Calibration Blanks- Initial and Continuing) - A reagent blank solution, which is analyzed immediately following the ICV (Initial Calibration Blank-ICB), at a frequency of 1 per 10 samples during a run (Continuing Calibration Blank-CCB), and at the end of a run to check for drifts in calibration, or possible analyte carry-over. Warning criteria include that the value be less than or equal to the three times the IDL for a given analyte for SW-846 work, less than the MDL for DOD-QSM data, or less than ½ the MRL for ACOE work. Control criteria consist of the CB value being less than or equal to 2 times the MDL for a given analyte for SW-846 and is determined by the QAPP for DOD/ACOE work. If this range is exceeded, a new calibration will be necessary.

6.6 LCS (Laboratory Control Sample) - A mid-range standard, prepared from a source different from that used for calibration standards, which is carried through the entire preparation and analytical method. The LCS is used to verify the accuracy of the preparation method. A minimum of one LCS is prepared per batch and is analyzed at the beginning of an analytical batch.

6.7 MB (Method Blank) - A Reagent Blank (see 3.1) which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation. A minimum of one MB is prepared per batch and is analyzed at the beginning of an analytical batch. Blank recovery must be less than 2x the MDL for SW846, less than the MDL for SWDA samples, less than ½ the RL for DOD-QSM, and is determined by the QAPP for ACOE samples.

6.8 MS-MSD (Matrix Spike-Matrix Spike Duplicate): - Two separate sample aliquots to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. An MS-MSD pair is prepared for every 20 samples of a given matrix per day for 6010B and once for every 10 samples of a given matrix per day for 200.7. For ACOE work only an MS is prepared and a duplicate sample is prepared rather than an MSD. Failure to meet criteria may be due to poor recovery during the preparation method or due to matrix interference within the digestate. To be considered acceptable, MSD must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample.

6.9 Duplicate sample- A separate sample aliquot which is carried through the entire preparation and analytical procedure. A duplicate is prepared for every 20 samples for ACOE/LCG work and per batch for DOD-QSM (in replacement of an MSD).

6.10 Method Reporting Limit (MRL) or Contract Required Detection Limit (CRDL) Standard: Detection level standard at a level near the reporting limit, or at a level specified by client contract. When required, it is to be analyzed following the ICB, and prior to the last CCV standard in the run.

6.11 Interelement Correction Factors (IEC) – These correction factors are determined by analyzing a concentration range of known interferents (Al, Ca, Co, Cr, Cu, Fe, Mg, Mn, Ni, V and Zn) and examining all other lines for a significant linear response. A line is considered to be significantly affected when the correlation coefficient for the interference is 0.99 or better and the correction factor multiplied by ten is greater than the MDL of the affected line. For interferents known to occur at high levels in environmental samples (Al, Ca, Cr, Cu, Fe, Mg and Zn) the interference will be considered to be significant when the correction factor multiplied by 100 is over the MDL of the affected line and the correlation coefficient in 0.99 or better. Interelement correction is used where applicable.

6.12 Linear Dynamic Range (LDR) – The upper limit of the linear dynamic range is established for each wavelength utilized by determining the signal responses from a minimum of three different concentration standards across the range. One of these will be near the upper limit of the range. The ranges used for the analysis of samples are judged by the analyst from the resulting data. The data and calculations are kept on file. The upper range limit is an observed signal no more than 10% below the level extrapolated from the lower standards. Determined analyte concentrations above the upper range limit are diluted and reanalyzed. New dynamic ranges are determined whenever there is a significant change in instrument response. For analytes that routinely approach the upper limit of the range, the range will be checked biannually. For analytes that are known interferents and exceed the dynamic range, the analyst will check that IEC's have been correctly applied. DOD-QSM requires that a LDR (or high level check standard) study be perform at least every six months.

Note: for ACOE/LCG work, analyte concentrations above the upper calibration limit are diluted and reanalyzed.

6.13 ICS-A (Interelement Correction Standard-A): A standard containing the elements Al, Ca, Fe, and Mg at 500mg/L. This standard is analyzed when using method 200.7 or performing ACOE work following the ICV at the beginning of the run to determine that interelement correction factors are correctly compensating for interference from these elements on other analyte lines. The ICSA may be required to be run before the last CCV of the run for ACOE work. Check the QAPP to determine if this is necessary.

Note: For ACOE work, the ICSA should be within the absolute value of two times the MDL for all analytes except Al, Ca, Fe and Mg unless a different requirement is specified within the contract.

6.14 ICS-AB (Interelement Correction Standard-AB): A standard containing the elements Al, Ca, Mg, and Fe at 500mg/L and all other elements at 500ug/L. This standard is analyzed following the ICV at the beginning of each run. It is analyzed to determine that the IEC are correctly preventing interference by these elements on the measurement of other analytes. The ICSAB may be required to be run before the last CCV of the run for ACOE work. Check the QAPP to determine if this is necessary

6.15 PDS (Post Digestion Spike): When a serial dilution or matrix spike falls outside of the acceptance limits a post digestion spike is used to determine if the sample digestion matrix is interfering with the analysis of the analyte. The sample is spiked at a level similar to that of the matrix spike.

Note: For ACOE/LCG work, a PDS will be conducted at a minimum rate of one per prep batch per unique matrix.

6.16 SD (Serial Dilution Analysis): A sample is diluted 5x with method blank solution and analyzed. The diluted result and the undiluted result should agree within a limit of precision defined by the program (SW846, CLP, 200.7) or client QAPP.

Note: For ACOE/LCG work, a SD will be conducted at a minimum rate of one per prep batch per unique matrix.

6.17 Batch- A batch consists of 20 samples of the same matrix analyzed on the same day or 20 samples of the same medium that have been prepared together.

6.18 IDL (Instrument Detection Limit); A series of blanks analyzed during initial setup or after significant changes, or as per DOD-QSM requirements. The limits established shall be < LOD for any given element.

6.19 Method detection limit (MDL): The minimum concentration of an analyte that can be identified measured and reported with 99% confidence that the analyte concentration is greater than zero.

6.20 LOQ Check: An internally prepared standard at a level near the Limit of Quantitation (LOQ) or at a level specified by a specific program or contract. An LOQ Check is required after MDL studies and quarterly thereafter for QSM work. Recovery limits are required for LOQ Checks and are usually program/contract specific.

6.21 Method Detection Limit (MDL) Check: An internally prepared standard prepared at approximately 1-2 time the calculated MDL for a given analyte (1-4 times for multi-component analyses). The MDL check sample is used as verification of the calculated MDL’s. Detection of the individual analytes in the MDL check is the only requirement. The MDL check is required after MDL studies and on an analytical run for LCG work when there are failures on the MRL spikes or quarterly for QSM.

7.0 Interferences

7.1 Background emission and stray light are corrected using background correction. See ICP 6000 Series operator’s manual for further instructions on background correction application.

7.2 Spectral overlaps are corrected for using interelement correction factors (IEC). When IEC are used, the interfering elements must be analyzed along with the elements of interest. The accuracy of IEC shall be verified daily by analyzing the ICSAB. All IEC factors shall be updated every six months or when an instrumentation change occurs; such as, changing a torch, nebulizer, injector, or plasma conditions.

7.3 Physical interferences such as viscosity are minimized by using an internal standard. Post digestion spike and serial dilutions help to determine if physical interferences are present.

7.4 Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Chemical interferences are not normally seen during ICP analysis and are highly matrix dependent.

7.5 Memory interferences occur when a sample of high analyte concentration does not thoroughly rinse prior to the analysis of the next sample. This causes elevated readings for that analyte in the subsequent sample. Memory effects can be minimized by rinsing at least 60 seconds between samples.

8.0 Safety

8.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

8.2 Insure that waste collection vessels contain enough room to accommodate all wastes that will be produced during the operation of the instrument.

9.0 Equipment and Supplies

9.1 ICP 6500, Cetac autosampler, computer, & network printer.
9.2 Argon (Airgas-liquid high purity or gaseous pre-purified grade or equivalent).
9.3 Class A volumetric flasks and pipettes (Chemglass or equivalent).
9.4 Disposable 15-mL polystyrene culture tubes.
9.5 100 ul pipette (Eppendorf or equivalent).
9.6 10-mL pipette (Eppendorf or equivalent).

10.0 Reagents and Materials

10.1 Reagents

10.1.1 Mixed and single element stock metals standards. See Section 9 and Appendix A, B, C and D for instructions on making the working standards.
10.1.2 Nitric acid, conc. (Fisher, Trace Metals grade or equivalent)
10.1.3 Hydrochloric acid, (Fisher, Trace Metals grade or equivalent)
10.1.4 Deionized water (Milli Q, > 10 mega ohm).

11.0 Sample Preservation and Storage

11.1 Samples must be preserved and analyzed within holding times stated in chart. Liquid samples are stored on shelves in the Laboratory warehouse and soil samples are stored in a walk-in refrigerator unit.

	Liquids	Solids
Preservative:	pH <2 with HNO_3	4^oC (+/-2)
Hold Time:	180 days	180 days

12.0 Quality Control

12.1 This SOP is designed to follow a variety of different projects and programs requirements. Table 3. is designed to illustrate the control steps and provisions required to adequately producing acceptable data.

12.2 Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.3 For the routine analysis of groundwater, wastewater, leachate, surface

water, soil, sludge, TCLP/SPLP extracts following method 6010B:
Required QC following instrument calibration is as follows:

12.3.1 ICV (initial calibration verification); The ICV is prepared from an alternate source standard whose concentrations are within the linear working range of the instrument. The results of the ICV should agree within 10% of the expected value for a given analyte. The relative standard deviation (RSD) between the replicate integrations should be <3%. If results are outside of this range, corrective action must be taken before samples can be analyzed.

12.3.2 ICB (initial calibration blank); analyze the calibration blank. The results of the initial calibration blank must be < 3x IDL for a given analyte. If the average of the replicates is not < 3x IDL, terminate the analysis, correct the problem and recalibrate or appropriately qualify the data. If the blank is less than 1/10th the concentration of the action level of interest and no sample is within ten percent of the action limit, analyses need not be rerun and re-calibration is not necessary before continuing with the analysis.

12.3.3 ICSAB (interference check solution); analyze a solution containing 500 mg/L Al, Ca, Mg, and Fe and all other analytes of interest at 0.50 mg/L. Recovery for analytes of interest is +/- 20% true value. If recovery is outside this range, corrective action must be taken before samples can be analyzed. Check placement of background correction points and IECs as a place to start troubleshooting .

12.3.4 LCS (laboratory control sample); analyze an alternate source reference sample. Control limits are +/- 20% of true value or in-house limits, whichever is more restrictive, or as specified in a client QAPP or the DOD-QSM manual.

12.3.5 MB (method blank); analyze a reagent blank. The method blank is a reagent blank that has been taken through the preparations steps alongside the samples being analyzed. Control limits are the MDL. If the average of the replicates is not < MDL terminate the analysis, correct the problem and recalibrate or appropriately qualify the data that falls within the MDL and twenty times the concentration of the analyte in the method blank.

12.3.6 CCV (continuing calibration verification); analyze a check standard after every ten samples and following the last sample in the run. This standard should be at a level approximately mid-scale. Control limits are +/- 10% of true value. If values fall outside this range, all samples back to the last acceptable ICV or CCV must be repeated.

12.3.7 CCB (continuing calibration blank); The results of the continuing calibration blank must be < 3x IDL for a given analyte. If the result falls outside this, reanalyze all samples back to the last acceptable CCB or qualify all sample <20 times the blank and greater than the MDL.

12.3.8 MS/MSD (matrix spike/matrix spike duplicate); for non-digested samples, prepare a bench spike in duplicate at a frequency of 5% or per analytical batch, whichever is more frequent. Control limits are +/- 25% of true value, and 20% RPD, or use calculated limits, whichever is tighter. See Sec. 18.0 for bench spike preparation. For digested samples, see "Predigestion Spike" chart in section 18.0. For digested samples, analyze the MS/MSD samples as they apply to each digestion set. Follow the above for control limits. For digested spikes with sample results greater than four times the digested spike level, prepare and analyze a PDS sample if the MS and/or MSD are outside the control limits. Prepare the PDS at a level approximately two times the sample level.

12.4 For SDWA analysis following method 200.7
Required QC following instrument calibration is as follows:

12.4.1 ICV: Referred to in 200.7 as LPC (laboratory performance check); analyze the mid-cal standard. Control limits are +/- 5% of true value. If values fall outside this range, recalibrate for the affected analytes.

12.4.2 ICB (initial calibration blank); analyze the calibration blank. The absolute value of the result should be below the MDL for the analyte(s) of interest. If the blank result falls outside this, evaluate the effect on the sample results and/or recalibrate for the affected analytes. Samples with results >10x the associated blank value need not be reanalyzed.

12.4.3 ICSA (interference check solution: interference only) analyze a solution containing 500 mg/L Al, Ca, Mg, and Fe. This sample must be analyzed at the beginning of the analytical run before the ICSAB. Recovery for interfering analytes is +/- 20% true value. All other analytes need to be ± 2x the MDL. If recovery/result is outside the acceptable range, corrective action must be taken before samples can be analyzed. Check placement of background correction points and IECs as a place to start trouble shooting.

12.4.4 ICSAB (interference check solution); analyze a solution containing 500 mg/L Al, Ca, Mg, Fe and all other analytes of interest at 0.50 mg/L. Recovery for analytes of interest is +/- 20% true value. If recovery is outside this range, corrective action must be taken before samples can be analyzed. Check placement of background correction points and IECs as a place to start trouble shooting.

12.4.5 LCS (laboratory control sample); analyze an alternate source reference sample per batch of 20 samples of the same matrix. Control limits are +/- 10% of true value, or manufacturer's limits, whichever is tighter.

12.4.6 MB (method blank); analyze a reagent blank per 20 samples of the same matrix. The method blank is a reagent blank that has been taken through the preparations steps alongside the samples being analyzed. If the average of the two replicates is not < MDL terminate the analysis, correct the problem and recalibrate or appropriately qualify the data that falls within the MDL and twenty times the concentration of the analyte in the method blank .

12.4.7 CCV: Referred to in 200.7 as LPC (laboratory performance check); analyze the mid-cal standard after every 10 samples. Control limits are +/- 10% of true value. If values fall outside this range, reanalyze all samples back to the last acceptable ICV or CCV.

12.4.8 CCB (continuing calibration blank); analyze the calibration blank immediately after each CCV. The value of the result should be below the MDL for the analyte(s)of interest. If the result falls outside this, evaluate the effect on the sample results. Sample results >10x the associated blank value need not be reanalyzed. Otherwise, reanalyze all samples back to the last acceptable CCB or qualify data that is >LOD and <10x the associated blank.

12.4.9 MS/MSD (matrix spike/matrix spike duplicate); for non-digested samples, prepare a bench spike in duplicate at a frequency of 5% or per analytical batch, whichever is more frequent. Control limits are +/- 25% of true value, and 20% RPD, or use calculated limits, whichever is tighter. See Sec. 18.0 for bench spike preparation. For digested samples, see “Predigestion Spike” chart in section 18.0. For digested samples, analyze the MS/MSD samples as they apply to each digestion set. Follow the above for control limits. For digested spikes with sample results greater than four times the digested spike level, prepare and analyze a PDS sample if the MS and/or MSD are outside the control limits. Prepare the PDS at a level approximately two times the sample level.

12.5 For the CLP-like level 4 analysis of groundwater, surface water, wastewater and soil

Note: A default of three replicate exposures per sample should be used for ACOE/LCG work unless specified differently in the QAPP.

Required QC following instrument calibration:

12.5.1 ICV (initial calibration verification): analyze the alternate source check standard immediately following calibration. Control limits +/- 10 % true value.

12.5.2 ICB (initial calibration blank): analyze the calibration blank. The absolute value of the result must be below the contract required detection limit (CRDL) or the limit stated within the QAPP for the project. The ICB results shall be less than the LOD/MDL for ACOE/ DOD-QSM data (or project specific). If a result falls outside this, reanalyze (recalibration may be necessary).

12.5.3 CRDL (contract required detection limit standard) or MRL (Method Required Limit): analyze a standard at a level two times the contract-required detection limits (CRDL) or at the level stated within the QAPP for the project. Follow limits stated within the QAPP as there are no EPA specified control limits for this standard. This sample must be analyzed at the beginning and the end of the run for ACOE samples.

12.5.4 ICSA (interference check solution: interference only) analyze a solution containing 500 mg/L Al, Ca, Mg, and Fe. This sample must be analyzed at the beginning of the analytical run prior to the ICSAB. Refer to QAPP for acceptability criteria. For ACOE work, the default criteria is the absolute value of two times the MDL for all analytes except Al, Ca, Mg and Fe which must have a recovery between 80-120%. Refer to QAPP to determine if the ICSA must also be analyzed at the end of the run.

12.5.5 ICSAB (interference check solution: interference plus analytes); analyze a solution containing 500 mg/L Al, Ca, Mg, and Fe, and all other analytes of interest at 0.50 mg/L. Recovery for analytes of interest is +/- 20% true value. If recovery is outside this range, corrective action must be taken before samples can be analyzed. Check placement of background correction points as a place to start troubleshooting. This sample must be analyzed at the beginning of the run. Refer to the QAPP to determine if the ICSAB must be analyzed at the end of the run.

12.5.6 Digested Sample set to include MB (S or W), LCS (S or W), MS, and DUP.

12.5.7 Serial Dilution: Analyze a x5 dilution of a sample from the digestion set. For sample results > 50x the MDL, the %RSD between the serial dilution result and the sample result must be ≤ 10.

12.5.8 Post digestion spike addition (bench spike): An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within +/- 25% of the known value or as specified by the client QAPP. The spike addition should produce a minimum level of 10 times the instrumental detection limit. If the spike recovery falls outside the limits, a matrix effect should be suspected.

12.5.9 CCV (continuing calibration verification): Analyze a mid-level standard after every ten samples. The CRDL/MRL, ICSA, ICSAB and batch QC all count as samples. Control limits are +/- 10% of true value. If any result falls outside this, all samples back to the last acceptable ICV/CCV must be reanalyzed.

12.5.10 MB (method blank); analyze a reagent blank per 20 samples of the same matrix. The method blank is a reagent blank that has been taken through the preparations steps alongside the samples being analyzed. If the average of the two replicates is not < MDL (< ½ the RL for ACOE/DOD-QSM, or < project specified limits), terminate the analysis, correct the problem and recalibrate or appropriately qualify the data that falls within the MDL and ten times the concentration of the analyte in the method blank.

12.5.11 CCB (continuing calibration blank); Analyze the calibration blank after each CCV (or every ten samples). Refer to the QAPP for CCB acceptance limits. The CCB results shall be less than the LOD for DOD-QSM data and <1/2 the RL for ACOE data (or project specific). If any result falls outside these limits, all samples with results less 10 times the CCB must be reanalyzed back to the last acceptable CCB or appropriately qualified.

12.5.12 MS/DUP (matrix spike/matrix duplicate); for non-digested samples, prepare a bench spike and a duplicate at a frequency of 5% or per analytical batch, whichever is more frequent. Control limits are specified in client QAPP. or see "Predigestion Spikes"(Table 1). For digested samples, analyze the MS/DUP samples as they apply to each digestion set and follow Table 3 control limits.

12.6 New or unusual matrices: It is recommended that whenever a new or unusual sample matrix is encountered, a serial dilution and post digestion (bench) spike be performed prior to reporting results. These tests will ensure that neither positive nor negative interferences are affecting sample results.

Note: For ACOE work, a serial dilution and a post digestion spike will be performed at a rate of one per matrix with each prep batch.

12.6.1 Serial Dilution: If the analyte concentration is sufficiently high (minimally a factor of ten above the instrumental detection limit after dilution), an analysis of a 1:5 dilution should agree within +/- 10% of the original determination. If not, a chemical or physical interference effect should be suspected.

12.6.2 Post digestion/bench spike addition (Table 2): An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within +/- 25% of the known value. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike recovery falls outside these limits, a matrix effect should be suspected..

13.0 Calibration and Standardization

13.1 The default calibration for TAL plus list (excluding Na and K) of metals for ACOE, LCG, DOD-QSM and routine work is a multi-point calibration method called 'DOD calibration' which uses 12 mixed standards and a calibration blank.

13.2 The default calibration for the metals Sodium and Potassium for ACOE/ LCG, DOD-QSM and routine work is a multi-point calibration method called 'Sodium and Potassium' which uses 8 mixed standards and a calibration blank.

13.3 The default calibration for the metal Boron for ACOE, LCG, DOD-QSM and routine work is a multi-point calibration method called 'Boron' which uses 5 standards and a calibration blank

13.4 The default calibration for the metal Lithium for ACOE, LCG, DOD-QSM and routine work is a multi-point calibration method called ‘Lithium’ which uses 7 standards and a calibration blank. Refer to section 11.0 for further instructions on how to perform the calibration.
Note: See Appendix A for preparations of calibration standards and blank for the DOD calibration method calibration. See Appendix B for the preparation of the calibration standards and blank for the Sodium and Potassium method calibration. See Appendix C for the preparation of the calibration standards and blank for the Boron calibrations. See Appendix D for the preparation of the calibration standards and blank for the Lithium calibration. See Tables 4 (a, b, & c), 5, 6, and 7 for individual element calibration concentrations/ranges.

13.5 Calibration Blank: Into a 1 L. volumetric flask, add 750 mL of Milli-Q water and 10mL of conc. HNO3 and 10mL HCl. Mix, dilute to volume with Milli-Q H2O. Transfer to a clean 1 L. Nalgene bottle. Prepare every 6 months or as needed.

13.6 Yttrium Internal std (Used for all methods) : Into a 2000mL volumetric flask, add 500mL of Milli-Q H2O, 10 ml 10,000 mg/L Yttrium standard dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean 2L Nalgene bottle. Prepare every 6 months or as needed. Concentration = 50 mg/L Yttrium solution.

13.7 Initial/Continuing Calibration Verification (ICV/CCV): Into a 1000mL volumetric flask, add 500mL of Milli-Q water, 10 mL of conc. HNO3 and 10mL HCl. Add the following:

10 mL SPEX Certiprep Spike Sample Standard 1 or Equivalent
0.5mL Mo 1000mg/L
0.05ml Ag 1000mg/L
2 ml Interferents-A-SPEX Certiprep or equivalent

Dilute to volume with Milli-Q water, mix and transfer to a clean 1 L Nalgene bottle. Make new every 6 months or as needed.

Concentration	Analyte
50ug/L	Cd, Be
100ug/L	Ag
200ug/L	Cr
250ug/L	Cu
500ug/L	Co, Mn, Mo, Ni, Pb, Sb, V, Zn
2000ug/L	Ba, As, Tl, Se
5,000ug/L	Fe
10,000ug/L	Ca, Mg
12,000ug/L	Al

13.8 Interference Check Solution (ICSA): Into a 500 mL volumetric flask, add 300 mL of Milli-Q H2O, 5 mL of conc. HNO3 and 5mL conc. HCl. Add the following stock solutions in the volumes listed:

50 mL Spex Certiprep Interferents A or equivalent

15 ml Fe 10,000mg/L or equivalent

Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean 500 mL Nalgene bottle. Prepare every 6 months or as needed.

Concentration	Analyte
500,000 ug/L	Al, Ca, Mg, Fe

13.9 Interference Check Solution (ICSAB): Into a 500 mL volumetric flask, add 300 mL of Milli-Q H2O, 5 mL of conc. HNO3 and 5 mL conc. HCl. Add the following stock solutions in the volumes listed:

50 mL Spex Certiprep Interferent A or equivalent
15 ml Spex Certiprep Fe 10,000 mg/L or equivalent
2.5 ml Spex Certiprep QC-21 or equivalent
0.25 ml Ultra Scientific Ag 1000 mg/L or equivalent
0.25 ml Ultra Scientific Ba 1000 mg/L or equivalent

Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean 500 mL Nalgene bottle. Prepare every 6 months or as needed.

Concentration	Analyte
500,000 ug/L	Al, Ca, Mg, Fe
500 ug/L	Ag, As, Ba, Be Cd, Co, Cr, Cu, Mn, Mo, Ni, Pb, Se, Sb, Tl, V, Zn

13.10 CRDL/MRL solution: Concentrations needed depend on the CRDL/MRL of a given contract.

14.0 Procedure

14.1 Instrument start-up procedure:

14.1.1 Open valve at argon tank, turn on chiller and instrument.. For best results, the instrument should be on and with an argon purge for at least 24 hours

14.1.2 Inspect pump tubing on instrument and on autosampler and change if necessary.

14.1.3 Fill DI rinse reservoir with DI water and preserve with HNO3 to1%.

14.1.4 Open up the ITeva software on the PC. Choose user, wait until instrument initializes.

14.1.5 Plasma startup and shutdown: Refer to the ICAP 6000 Series ICP-OES Spectrometer Operator manuals pages 11-1 thru 11-4. After plasma startup check the “Debug Wavelength Check” at the bottom of the ITeva control center. The absolute value of x and y #s should be less than 5, if not, stop and call Thermo service.

14.1.6 After a 30-minute warm-up period, check condition of nebulizer. Put pump tube into a 100mg/L solution Yttrium Std. With the lights off and after enough time has elapsed for the Yttrium standard to reach the plasma, a red cone should be noticeable in the center of the plasma. If the nebulizer is in good condition and the nebulizer gas flows are set properly, the red cone should project about 2mm beyond the coils. If not, check the settings, the pump tubes, and inspect the nebulizer under a microscope for any obstructions or breakage.

14.2 To create an autosampler sequence:

- 14.2.1 Refer to the ICAP 6000 Series ICP-OES Spectrometer Operator manuals pages 11-9 thru 11-11 to start an autosampler sequence.
- 14.2.2 Add all samples, LCS, Blanks, MS-MSD, etc. in order of program, agency or client request.
- 14.2.3 Print Autosampler Table: This will be used when preparing all samples and standards.
- 14.2.4 Using the printed autosampler table sheet, prepare all standards, QC samples, and samples in their designated positions in the autosampler. Prepare any bench spikes and place them in autosampler. Calibration standards, CCV, ICV, CCB, ICB and ICSAB all go into 50 ml vials in the designated S-# positions of the autosampler. All others are poured into 15 ml plastic vials into the designated areas within the 60 position racks.

14.3 Calibration and Analysis.

- 14.3.1 Once all calibration standards have been placed in the autosampler make sure the autosampler is initialized by clicking on the autosampler icon and the sequence is saved and then press the yellow arrow icon to start the calibration and prepare the remaining samples as the calibration is being carried out.

14.4 Instrument shutdown

- 14.4.1 If run will not be finished during work hours, program the instrument to shut down at the end of the analytical run. When setting up on the sequence page click the “End Action (after all sequences) Box to “Shutdown Plasma”. This will shut down the plasma.
- 14.4.2 For manual shutdown go to the flame icon in the bottom right corner and click, and then select the plasma off icon. After the plasma is shutdown loosen the pump tubes and shut off the chiller.

15.0 Calculations

15.1 Sample Calculations:

Liquid Concentration (ug/L) = A x C

Solid Concentrations (mg/kg) = $\frac{A \times B \times C}{D \times E}$,

Where:
A = instrument reading for sample (ug/L)
B = total volume of digestion (L)
C= dilution factor, if necessary (ex. For a 1 to 10 dilution, C = 10)
D = amount of sample used in digestion (g)

E = percent solids/100, if necessary (fraction equivalent)

15.2 Spike Recovery Calculations:

$$\text{LCS Recovery (\%)} = \frac{\text{(Result obtained) x 100}}{\text{(Spike amount)}}$$

$$\textit{MS/MSD Spike Recovery (\%)} = \frac{\text{(Spiked sample conc. – Sample conc.) x 100}}{\text{(Spike amount)}}$$

$$\textit{\%RPD} = \frac{\text{(MS – MSD) x 100}}{\text{(MS + MSD)/2}},$$

Where: MS = Matrix spike concentration obtained
MSD = Matrix spike duplicate concentration obtained

15.2 "<u>Total Hardness"</u> (by calculation) can also be determined by using the values for calcium and magnesium obtained by this procedure. The "Total Hardness" value is calculated in the LIMS system using the following equation:

Total Hardness (mg/L) or Hardness equivalent $CaCo^3$/L =

2.497[Ca mg/L} + 4.118[Mg mg/L]

16.0 Method Performance

16.1 Certified standard solutions, properly used instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

16.2 Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4-5 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (AR) and the relative standard deviation (RSD) of the recoveries for each analyte. In the absence of specific criteria found in the EPA methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated.

16.3 Proper instrument maintenance is another means to ensure adequate method performance.

16.3.1 Pump tubing and rollers: Ensure that the pump rollers turn freely. Inspect pump tubing daily and replace when it starts failing to retain its shape.

16.3.2 Drain line: Spray chamber drain line must flow unimpeded directly into waste jug. Make sure line is draining properly.

16.3.3 Spray Chamber: If the spray chamber becomes dirty, the sample waste may not drain properly. Remove and wash with hot soapy water, then rinse with DI H_2O.

16.3.4 Torch and O-rings: The O-rings surrounding the torch may need to be replaced if the plasma becomes unstable or internal standard emission counts fall off. See ICP6000 manual for technique. Torch needs to be cleaned occasionally with aqua regia followed by sonication. See sections 5 and 6 in the "ICAP 6000 Series ICP-OES Spectrometer Operating Manuals" for further assistance if needed.

17.0 Pollution Prevention

17.1 Pollution prevention encompasses any technique that reduces or eliminates the

quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation.

17.2 The quantity of chemicals purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

18.0 Data Assessment and Acceptance Criteria for QC Measures

18.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. The analyst will review the data to ensure QC is acceptable and that exceedances are addressed. Acceptable data is then captured into the LIMS system (See SOP ICP6000 Data Capture for instructions on data capture).

18.2 After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist (FMT9-01) for data review guidance.

18.3 Once analyst has reviewed and approved the data, it is given to a peer or supervisor for review.

18.4 After the second reviewer approves the data, the reviewer sends the data to "validated" status in LIMS.

18.5 A paper hard copy of the data is then filed or archived. The package includes the checklist, the sequence run log, and a copy of the bench sheet (if applicable), the LIMS run log, and verification of calibration data.

19.0 Corrective Measures for Out-of-Control Data

When data is out of control, a number of corrective actions may need implementing. If the nonconformities involve failing QC within the analytical sequence batch, then reanalysis of samples may eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data may correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument may need cleaning and decontamination. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions should be taken for different QC-type failures or nonconformities (Table 3.).

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

20.1 Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data may be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their effect on data.

20.1.1 The first thing addressed is prevention of producing unacceptable data. When limited sample volume is the issue, the analyst should determine if splitting the sample into lesser volumes or weights is an option. To avoid sample hold time issues, the analyst's first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises, reanalysis is an option. Analyst error is prevented by a second analyst confirmation and validation. If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.

20.1.2 When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more samples can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

20.1.2.1 If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

20.1.2.2 In all instances, if results are reported from data that is out of control or unacceptable, that data should be qualified accordingly. Once the client has been notified and he or she instructs us to report the data, flag the data indicating what type of nonconformity has occurred.

20.1.2.3 Out of control data is still retained by the laboratory and filed and archived along with acceptable data. The file folder should be labeled as such, indicating that the data is out of control.

20.1.2.4 A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

21.0 Waste Management

Samples are routinely held for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).

22.0 REFERENCES

22.1 Test Methods for Evaluating Solid Waste, EPA, SW-846, Method 6010B, rev. 2, December 1996.
22.2 Test Methods for Evaluating Solid Waste, EPA, SW-846, Method 6010B, rev. 3, February, 2000.
22.3 Test Methods for Evaluating Solid Waste, EPA, SW-846, Method 6010C, rev. 3, February, 2007.
22.4 Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, Method 200.7, rev. 4.4, 1991.
22.5 USEPA - Contract Laboratory Program, Statement of Work for Inorganic Analysis, ILM04.0.
22.6 ICAP 6000 Series ICP-OES Spectrometer Operator’s Manual, Thermo Electron (registration number 441506).
22.7 Thermo Electron Corporation Training Manual iCAP 6000 Series.
22.8 Standard Methods For the Examination of Water and Wastewater, 21st Edition, 2340B-Hardness by Calculation, 2005.
22.9 CT Laboratories Quality Manual, current revision.
22.10 Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.
22.11 National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.
22.12 ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.
22.13 Department of Defense, Quality Systems Manual for Environmental Laboratories,
Version 4.2, October 2010.

23.0 Appendices

Table1: Spike, LCS, & LFB Analysis- ICP
Pre-digestion Spikes

Element	Spike Amt. mL of	Spike Stock A, B, C	Stock Conc. mg/L	Final Vol. mL	Expected Conc. ug/L
Aluminum	1	**A**	200	50	4000
Antimony	1	**A**	50	50	1000
Arsenic	1	**A**	200	50	4000
Barium	1	**A**	200	50	4000
Beryllium	1	**A**	5	50	100
Cadmium	1	**A**	5	50	100
Calcium	0.5	**C**	20,000	50	200000
Chromium	1	**A**	20	50	400
Cobalt	1	**A**	50	50	1000
Copper	1	**A**	25	50	500
Iron	1	**A**	100	50	2000
Lead	1	**A**	50	50	1000
Manganese	1	**A**	50	50	1000
Magnesium	0.5	**C**	10,000	50	100000
Molybdenum*	0.1	**B**	1000	50	2000
Nickel	1	**A**	50	50	1000
Selenium	1	**A**	200	50	4000
Silver	1	**A**	5	50	100
Thallium	1	**A**	200	50	4000
Vanadium	1	**A**	50	50	1000
Zinc	1	**A**	50	50	1000

Spike Solutions ***

Supplier	Lot #/ std	Stock
SPEX Certiprep or equiv.	SPIKE 1-500	**A**
Molybdenum * or equiv.	1000 mg/L	**B**
Custom Std***	SPEX	**C**

- * Addition of Boron, Lithium, Silicon, Tin, Strontium, Titanium, and Tungsten at 0.1 ml each of a 1000 mg/L solution from SPEX Certiprep or equivalent.
- ** Addition of Potassium at 0.5 ml of a 10,000 mg/L solution from SPEX Certiprep or equivalent.
- *** Sodium is also included in this standard at 10,000 mg/L

Table 2: Bench Spike Analysis-ICP
Post Digestion/ Bench Spikes

Element	Spike Amt. ML of	Spike Soln.	Stock Conc. mg/L	Final Vol. mL	Expected Conc. ug/L
Aluminum	0.2	**A**	200	10	4000
Antimony	0.2	**A**	50	10	1000
Arsenic	0.2	**A**	200	10	4000
Barium	0.2	**A**	200	10	4000
Beryllium	0.2	**A**	5	10	100
Cadmium	0.2	**A**	5	10	100
Calcium	0.2	**B**	20,000	10	400000
Chromium	0.2	**A**	20	10	400
Cobalt	0.2	**A**	50	10	1000
Copper	0.2	**A**	25	10	500
Iron	0.2	**A**	100	10	2000
Lead	0.2	**A**	50	10	1000
Manganese	0.2	**A**	50	10	1000
Magnesium	0.2	**B**	10,000	10	200000
Molybdenum	0.02	**C***	1000	10	2000
Nickel	0.2	**A**	50	10	1000
Selenium	0.2	**A**	200	10	4000
Silver	0.2	**A**	5	10	10
Thallium	0.2	**A**	200	10	4000
Vanadium	0.2	**A**	50	10	1000
Zinc	0.2	**A**	50	10	1000

Standard Source ***	
Supplier	
SPEX	**A**
SPEX Custom Std	**B**
Molybdenum-1,000 mg/L Std*	**C***

- * Addition of Boron, Lithium, Silicon, Tin, Strontium, Titanium, and Tungsten at 0.02 ml each of a 1000 mg/L solution from SPEX Certiprep or equivalent.
- ** Addition of Potassium and Sodium at 0.1 ml of a 10,000 mg/L solution from SPEX Certiprep or equivalent.

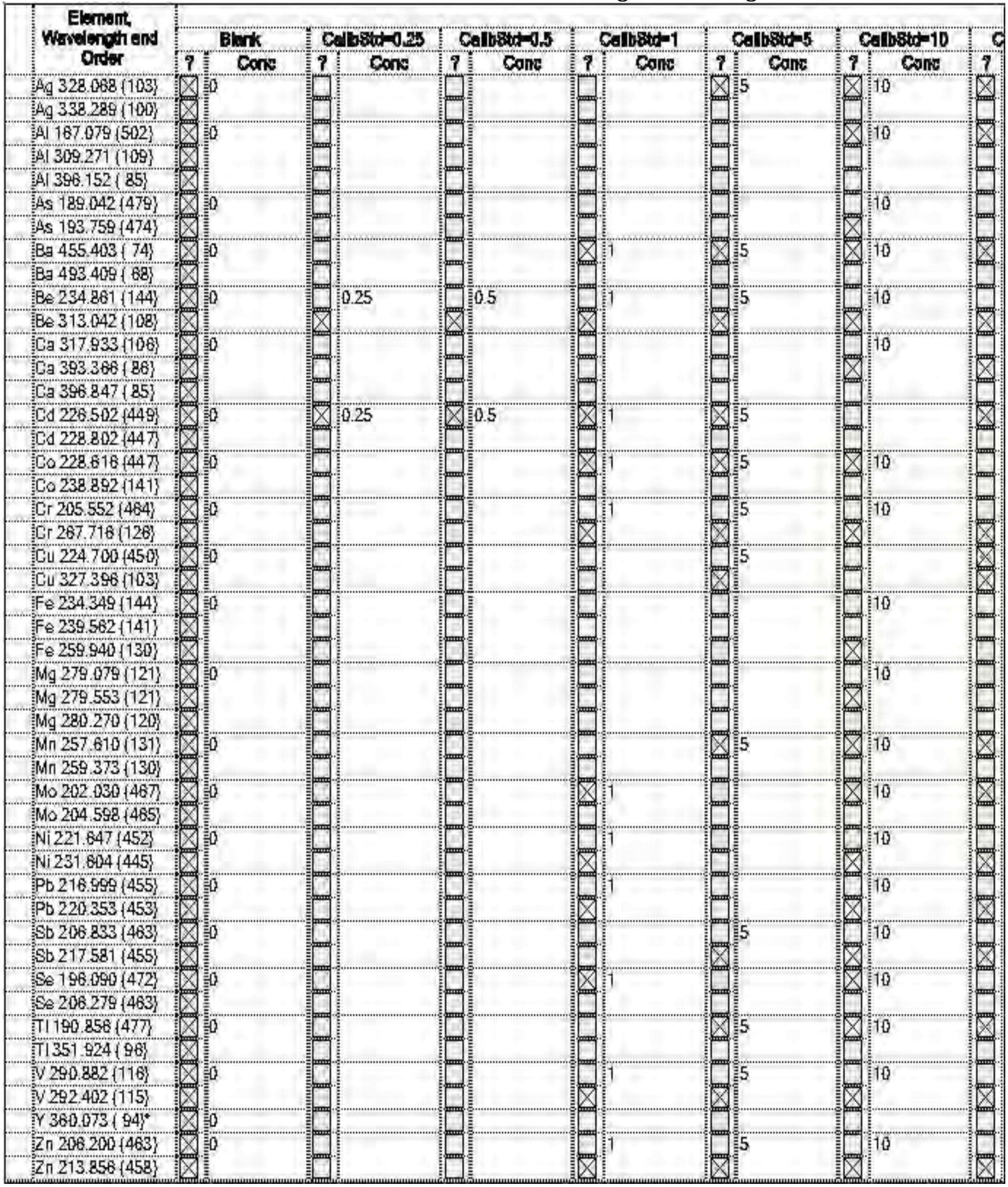
Table 3: Summary of Quality Control Requirements

QC Type	Frequency	Conc. Level	1. *Acceptance Criteria*	2. *Corrective Action*
Linear Dynamic Range(LDR) or high level check standard	At initial set up and checked every 6 months with a high standard at the upper limit of the range	High point of calibration curve	Within ± 10% of true value.	Data cannot be reported above the high calibration range without an a established/passing high level check standard
Initial Calibration (ICAL) for all analytes	Daily ICAL prior to sample analyst	Table. 4a.	If more than one calibration standard used, $r2 \geq 0.99$.	Minimum one high standard and a calibration blank. No samples shall be analyzed until ICAL has passed.
Initial Calibration Verification (ICV)	1 per calibration	Mid. Calib. Range	SDWA:95-105% SW846:90-110% Or Project/Program Specific	Terminate run. Correct the problem before proceeding
Initial Calibration Blank (ICB)	Immediately after the ICV	<MDL	SW846: < 3x IDL SDWA: < MDL DOD-QSM < MDL LCG: <1/2 MRL or Project / Program Specific	Terminate analysis and correct the problem before proceeding.
Method Blank (MB)	1 per batch of 20 samples	<MDL	SDWA: < MDL SW846: < 2x MDL DOD-QSM / LCG: <1/2 MRL or Project / Program Specific	Access data and reanalyze/re-prepare the MB and affected data or flag "B" analyte detected in Method Blank when insufficient sample remains
Laboratory Control Sample (LCS)	1 per batch of 20 samples	Mid. Calib. Range	In-house limits or, default 80-120% SDWA; 90-110% or Project / Program Specific	Terminate analysis: correct problem before proceeding.
Continuing Calibration Verification (CCV)	1 after every 10th sample	Mid. Calib. Range	SW846: 90-110% SDWA: 90-110% CLP:90-110%	Recalibrate and reanalyze all samples back to the last acceptable CCV or ICV

Continuing Calibration Blank (CCB)	Immediately following each CCV	<MDL	SW846: < 3x IDL SDWA: < MDL DOD-QSM < MDL LCG: < 2x MDL or Project / Program Specific	Reanalyze all samples back to the last acceptable CCB or flag "B" analytes detected
Interference Check Solution A (ICSA)	Immediately After LCS (& before final CCV if required by program / project specific QAPP)	500mg/L Al, CA,FE, Mg	80-120% for Interference Elements ABS of analytes not included must be < 2X MRL or Project / Program Specific	Terminate analysis, correct problem & reanalyze all samples back to last good ICSA/ICSAB
Interference Check Solution AB (ICSAB)	Immediately After ICSA (& before final CCV if required by program / project specific QAPP)	500mg/L Al, CA, FE,MG Other elements 500ug/L	80-120% for All Elements	Terminate analysis, correct problem & reanalyze all samples back to last good ICSA/ICSAB
MS	One per batch per matrix	See Table 1 spike chart	1. In-house limits: default 70-130 % Rec. 2. DOD-QSM: Use specified LCS limits. 3. LCG: 75-125 % Rec. when [matrix] is <4x[spike]	1. Reanalyze an alternative sample or perform a PDS, if MS and PDS fail qualify data as to matrix effect. 2. DOD-QSM: Used for matrix evaluation only. Determine source of difference (i.e. serial dilution/PDS). Reanalyze or qualify data as per client/ project requirement. 3. LCG: If MS fails and sample results are < 5x the MRL run a PDS. If sample results are > 5x the MRL perform a serial dilution.

MSD or Matrix Dup. (MD)	1. In-house & QSM-DOD: one MSD or MD per batch or matrix. 2. LCG: one MD per batch or matrix.	See Table 1 spike chart	1. In-house limits: default 70-130 % Rec. RPD = 20% 2. DOD-QSM: Use specified LCS limits for MSD. RPD = 20 % for MS/MSD or MD. 3. LCG: RPD = 20%	1. Perform PDS, if MSD and PDS fail qualify data as to matrix effect. 2. DOD-QSM: Used for matrix evaluation only. Determine source of difference (i.e. serial dilution/PDS). Reanalyze or qualify data as per client/ project requirement. 3. LCG: Qualify positive detects for precision failures between the sample and the MD.
Serial Dilution Analysis (SD)	1. In-house: Analyzed on the sample with a PDS failure. 2. DOD-QSM: Analyzed with each batch of samples. 3. LCG: After an MS failure with sample results > 5x the MRL.	5 fold dilution of chosen sample	RPD within 10% of value of diluted and undiluted sample, but only if sample conc. 1. In-house: > 10x LOQ 2. DOD-QSM: > 50x the MDL 3. LCG: > 5x the MRL	1. In-house: Qualify data only if sample result is > 10x the LOQ. 2. Analyze a PDS if sample result is > 50x the MDL. 3. Analyze a PDS if sample result is > 5x the MDL.
Post Digestion Spike (PDS)	1. In-house: Upon failure of MS or MSD. 2. DOD-QSM: When the SD test fails or all sample results < 50x the MDL and there is an MS or MSD failure. 3. LCG: When the MS fails and the sample result is <5x the MRL or when the SD test fails.	Between 10 and 100 times the MDL	1. In-house: 80-120% Rec. 2. DOD-QSM & LCG: 75-125% Rec.	1. In-house: Qualify for matrix interference or if requested analyze by MSA. 2. DOD-QSM: Run samples by MSA or ISA or qualify data using program/project specified criteria. 3. LCG: Run samples by MSA or qualify data using program/project specified criteria.
Method of Standard Additions (MSA) or Internal Standard Calibration (ISA)	1. In-house: When requested. 2. DOD-QSM & LCG: After the failure of a PDS.	Minimum of 3 standard levels and the unspiked sample	N/A	Document the use of an MSA or ISA

Table 4a. TAL list Concentrations/Ranges for ICP ug/L



Element, Wavelength and Order	Blank		CalibStd=0.25		CalibStd=0.5		CalibStd=1		CalibStd=5		CalibStd=10		C
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Ag 328.068 (103)	☒	0	☐		☐		☐		☒	5	☒	10	☒
Ag 338.289 (100)	☒		☐		☐		☐		☐		☐		☐
Al 167.079 (502)	☒	0	☐		☐		☐		☐		☒	10	☒
Al 309.271 (109)	☒		☐		☐		☐		☐		☐		☐
Al 396.152 (85)	☒		☐		☐		☐		☐		☐		☐
As 189.042 (479)	☒	0	☐		☐		☐		☐		☐	10	☐
As 193.759 (474)	☒		☐		☐		☐		☐		☒		☐
Ba 455.403 (74)	☒	0	☐		☐		☒	1	☒	5	☒	10	☒
Ba 493.409 (68)	☒		☐		☐		☐		☐		☐		☐
Be 234.861 (144)	☒	0	☐	0.25	☐	0.5	☐	1	☐	5	☐	10	☐
Be 313.042 (108)	☒		☒		☒		☒		☒		☒		☒
Ca 317.933 (106)	☒	0	☐		☐		☐		☐		☐	10	☐
Ca 393.366 (86)	☒		☐		☐		☐		☐		☒		☒
Ca 396.847 (85)	☒		☐		☐		☐		☐		☐		☐
Cd 226.502 (449)	☒	0	☒	0.25	☒	0.5	☒	1	☒	5	☐		☒
Cd 228.802 (447)	☒		☐		☐		☐		☐		☐		☐
Co 228.616 (447)	☒	0	☐		☐		☒	1	☒	5	☒	10	☐
Co 238.892 (141)	☒		☐		☐		☐		☐		☐		☐
Cr 205.552 (464)	☒	0	☐		☐		☐	1	☐	5	☐	10	☐
Cr 267.716 (126)	☒		☐		☐		☒		☒		☒		☒
Cu 224.700 (450)	☒	0	☐		☐		☐		☐	5	☐		☒
Cu 327.396 (103)	☒		☐		☐		☐		☒		☐		☒
Fe 234.349 (144)	☒	0	☐		☐		☐		☐		☐	10	☐
Fe 239.562 (141)	☒		☐		☐		☐		☐		☐		☐
Fe 259.940 (130)	☒		☐		☐		☐		☐		☒		☐
Mg 279.079 (121)	☒	0	☐		☐		☐		☐		☐	10	☐
Mg 279.553 (121)	☒		☐		☐		☐		☐		☒		☐
Mg 280.270 (120)	☒		☐		☐		☐		☐		☐		☐
Mn 257.610 (131)	☒	0	☐		☐		☐		☒	5	☒	10	☒
Mn 259.373 (130)	☒		☐		☐		☐		☐		☐		☐
Mo 202.030 (467)	☒	0	☐		☐		☒	1	☐		☒	10	☒
Mo 204.598 (465)	☒		☐		☐		☐		☐		☐		☐
Ni 221.647 (452)	☒	0	☐		☐		☐	1	☐		☐	10	☐
Ni 231.604 (445)	☒		☐		☐		☒		☐		☒		☒
Pb 216.999 (455)	☒	0	☐		☐		☐	1	☐		☐	10	☐
Pb 220.353 (453)	☒		☐		☐		☒		☐		☒		☒
Sb 206.833 (463)	☒	0	☐		☐		☐		☐	5	☐	10	☐
Sb 217.581 (455)	☒		☐		☐		☐		☒		☒		☒
Se 196.090 (472)	☒	0	☐		☐		☒	1	☐		☒	10	☐
Se 206.279 (463)	☒		☐		☐		☐		☐		☐		☐
Tl 190.856 (477)	☒	0	☐		☐		☐		☒	5	☒	10	☒
Tl 351.924 (96)	☒		☐		☐		☐		☐		☐		☐
V 290.882 (116)	☒	0	☐		☐		☐	1	☐	5	☐	10	☐
V 292.402 (115)	☒		☐		☐		☒		☒		☒		☒
Y 360.073 (94)*	☒	0	☐		☐		☐		☐		☐		☐
Zn 206.200 (463)	☒	0	☐		☐		☐	1	☐	5	☐	10	☐
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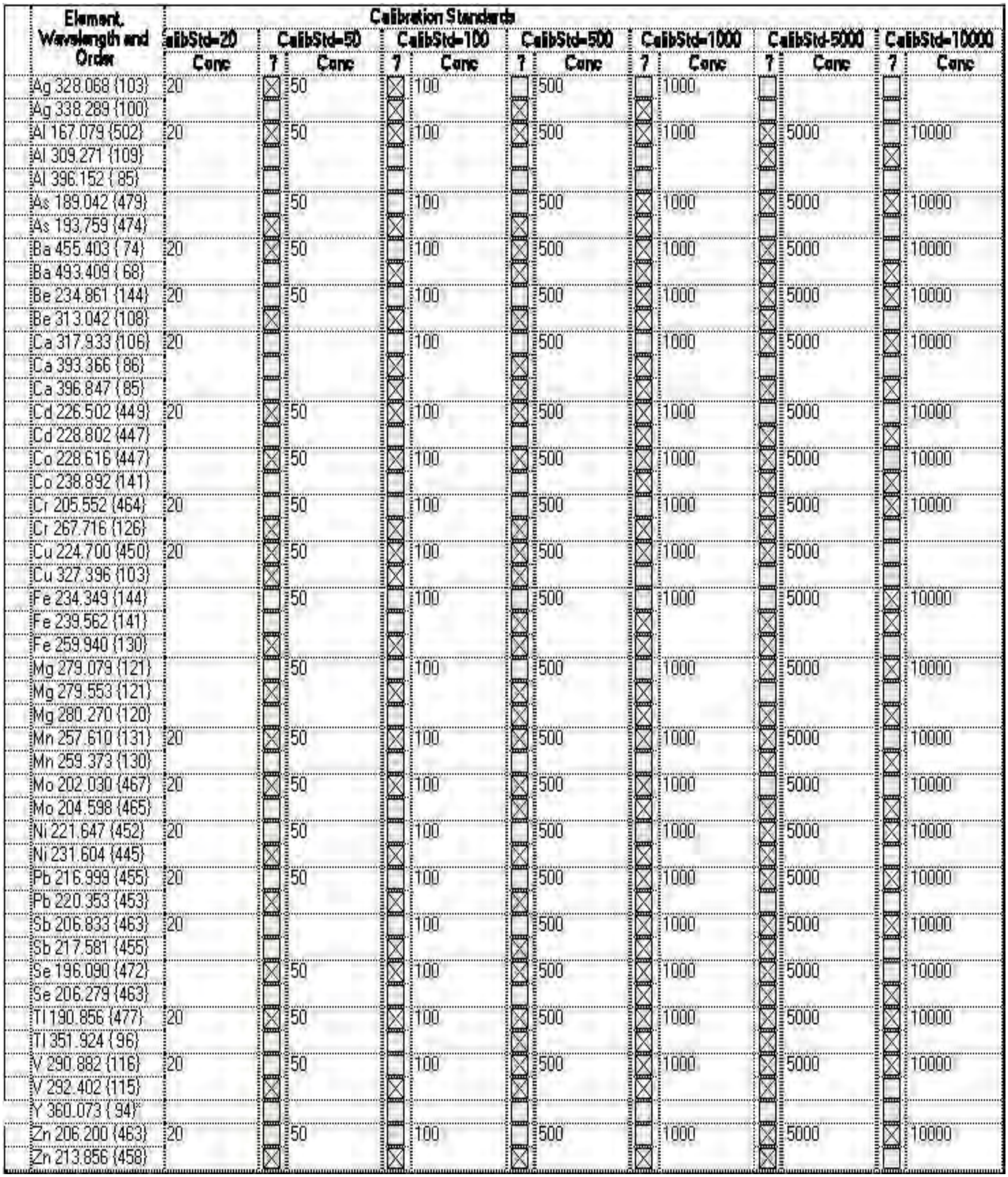

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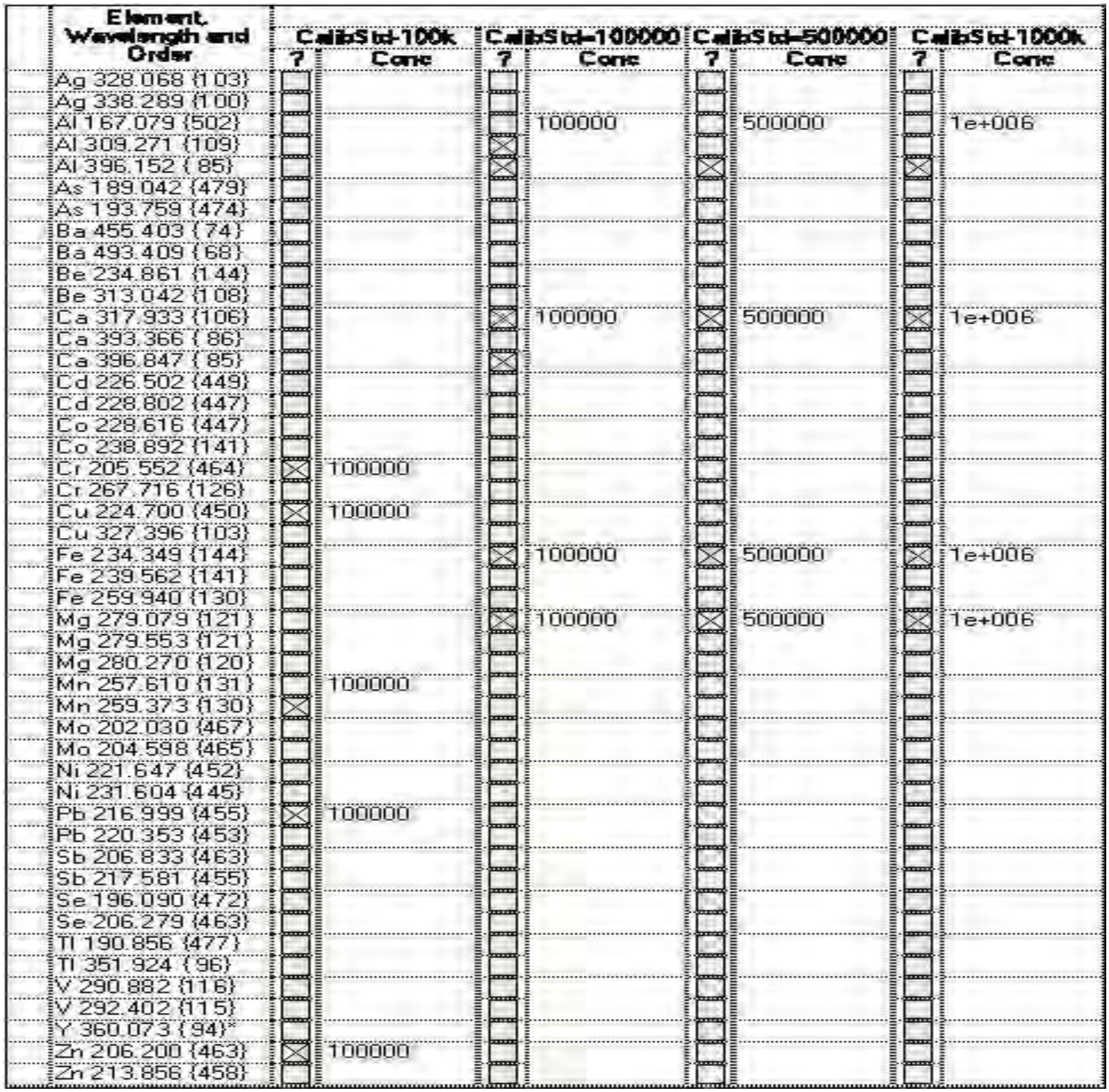
	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc
Ag 328.068 {103}	20	☒	50	☒	100	☐	500	☐	1000	☐		☐	
Ag 338.289 {100}		☐		☒		☒		☒		☐		☐	
Al 167.079 {502}	20	☒	50	☒	100	☒	500	☒	1000	☒	5000	☐	10000
Al 309.271 {109}		☐		☐		☐		☐		☒		☒	
Al 396.152 { 85}		☐		☐		☐		☐		☐		☐	
As 189.042 {479}		☐	50	☐	100	☐	500	☒	1000	☒	5000	☒	10000
As 193.759 {474}		☒		☒		☒		☒		☐		☐	
Ba 455.403 { 74}	20	☒	50	☐	100	☒	500	☒	1000	☒	5000	☐	10000
Ba 493.409 { 68}		☐		☒		☒		☒		☒		☒	
Be 234.861 {144}	20	☐	50	☐	100	☐	500	☒	1000	☒	5000	☒	10000
Be 313.042 {108}		☒		☒		☒		☒		☒		☐	
Ca 317.933 {106}	20	☐		☐	100	☐	500	☐	1000	☒	5000	☒	10000
Ca 393.366 { 86}		☐		☒		☒		☒		☐		☐	
Ca 396.847 { 85}		☐		☒		☒		☒		☒		☒	
Cd 226.502 {449}	20	☒	50	☒	100	☒	500	☒	1000	☐	5000	☐	10000
Cd 228.802 {447}		☐		☐		☐		☒		☒		☒	
Co 228.616 {447}		☒	50	☒	100	☒	500	☒	1000	☒	5000	☐	10000
Co 238.892 {141}		☐		☐		☐		☒		☒		☒	
Cr 205.552 {464}	20	☐	50	☐	100	☐	500	☐	1000	☒	5000	☒	10000
Cr 267.716 {126}		☒		☒		☒		☒		☒		☐	
Cu 224.700 {450}	20	☒	50	☒	100	☒	500	☒	1000	☒	5000	☐	
Cu 327.396 {103}		☒		☒		☒		☐		☐		☐	
Fe 234.349 {144}		☐	50	☐	100	☐	500	☐	1000	☐	5000	☒	10000
Fe 239.562 {141}		☐		☐		☒		☒		☒		☒	
Fe 259.940 {130}		☒		☒		☒		☒		☒		☐	
Mg 279.079 {121}		☐	50	☐	100	☐	500	☐	1000	☒	5000	☒	10000
Mg 279.553 {121}		☒		☒		☒		☒		☐		☐	
Mg 280.270 {120}		☐		☐		☒		☒		☒		☒	
Mn 257.610 {131}	20	☒	50	☒	100	☒	500	☒	1000	☒	5000	☐	10000
Mn 259.373 {130}		☐		☐		☐		☐		☒		☒	
Mo 202.030 {467}	20	☒	50	☒	100	☒	500	☒	1000	☐	5000	☐	10000
Mo 204.598 {465}		☐		☐		☒		☒		☒		☒	
Ni 221.647 {452}	20	☐	50	☐	100	☐	500	☐	1000	☒	5000	☒	10000
Ni 231.604 {445}		☒		☒		☒		☒		☒		☐	
Pb 216.999 {455}	20	☐	50	☐	100	☐	500	☒	1000	☒	5000	☒	10000
Pb 220.353 {453}		☒		☒		☒		☒		☐		☐	
Sb 206.833 {463}	20	☐		☐	100	☐	500	☒	1000	☒	5000	☒	10000
Sb 217.581 {455}		☐		☒		☒		☒		☒		☐	
Se 196.090 {472}		☒	50	☒	100	☒	500	☒	1000	☒	5000	☐	10000
Se 206.279 {463}		☐		☐		☐		☒		☒		☒	
Tl 190.856 {477}	20	☒	50	☒	100	☒	500	☒	1000	☒	5000	☒	10000
Tl 351.924 { 96}		☐		☐		☒		☒		☒		☒	
V 290.882 {116}	20	☐	50	☐	100	☒	500	☒	1000	☒	5000	☒	10000
V 292.402 {115}		☒		☒		☒		☒		☒		☐	
Y 360.073 { 94}		☐		☐		☐		☐		☐		☐	
Zn 206.200 {463}	20	☐	50	☐	100	☐	500	☐	1000	☒	5000	☒	10000
Zn 213.856 {458}		☒		☒		☒		☒		☒		☐	

Table 4b. TAL list Concentrations/Ranges for ICP ug/L cont.

Table 4c. TAL list Concentrations/Ranges for ICP ug/L cont.



Element, Wavelength and Order	CalibStd-100k		CalibStd=100000		CalibStd=500000		CalibStd-1000k	
	?	Conc	?	Conc	?	Conc	?	Conc
Ag 328.068 {103}	☐		☐		☐		☐	
Ag 338.289 {100}	☐		☐		☐		☐	
Al 167.079 {502}	☐		☐	100000	☐	500000	☐	1e+006
Al 309.271 {109}	☐		☒		☐		☐	
Al 396.152 { 85}	☐		☒		☒		☒	
As 189.042 {479}	☐		☐		☐		☐	
As 193.759 {474}	☐		☐		☐		☐	
Ba 455.403 { 74}	☐		☐		☐		☐	
Ba 493.409 { 68}	☐		☐		☐		☐	
Be 234.861 {144}	☐		☐		☐		☐	
Be 313.042 {108}	☐		☐		☐		☐	
Ca 317.933 {106}	☐		☒	100000	☒	500000	☒	1e+006
Ca 393.366 { 86}	☐		☐		☐		☐	
Ca 396.847 { 85}	☐		☒		☐		☐	
Cd 226.502 {449}	☐		☐		☐		☐	
Cd 228.802 {447}	☐		☐		☐		☐	
Co 228.616 {447}	☐		☐		☐		☐	
Co 238.892 {141}	☐		☐		☐		☐	
Cr 205.552 {464}	☒	100000	☐		☐		☐	
Cr 267.716 {126}	☐		☐		☐		☐	
Cu 224.700 {450}	☒	100000	☐		☐		☐	
Cu 327.396 {103}	☐		☐		☐		☐	
Fe 234.349 {144}	☐		☒	100000	☒	500000	☒	1e+006
Fe 239.562 {141}	☐		☐		☐		☐	
Fe 259.940 {130}	☐		☐		☐		☐	
Mg 279.079 {121}	☐		☒	100000	☒	500000	☒	1e+006
Mg 279.553 {121}	☐		☐		☐		☐	
Mg 280.270 {120}	☐		☐		☐		☐	
Mn 257.610 {131}	☐	100000	☐		☐		☐	
Mn 259.373 {130}	☒		☐		☐		☐	
Mo 202.030 {467}	☐		☐		☐		☐	
Mo 204.598 {465}	☐		☐		☐		☐	
Ni 221.647 {452}	☐		☐		☐		☐	
Ni 231.604 {445}	☐		☐		☐		☐	
Pb 216.999 {455}	☒	100000	☐		☐		☐	
Pb 220.353 {453}	☐		☐		☐		☐	
Sb 206.833 {463}	☐		☐		☐		☐	
Sb 217.581 {455}	☐		☐		☐		☐	
Se 196.090 {472}	☐		☐		☐		☐	
Se 206.279 {463}	☐		☐		☐		☐	
Tl 190.856 {477}	☐		☐		☐		☐	
Tl 351.924 { 96}	☐		☐		☐		☐	
V 290.882 {116}	☐		☐		☐		☐	
V 292.402 {115}	☐		☐		☐		☐	
Y 360.073 { 94}*	☐		☐		☐		☐	
Zn 206.200 {463}	☒	100000	☐		☐		☐	
Zn 213.856 {458}	☐		☐		☐		☐	

Note: These metals are calibrated using a blank and minimum of three standards. It is not allowable to remove any mid-levels to obtain an acceptable calibration; all points must be used. Multi-level calibrations must be sequential.

Appendix A.

Element specific standard prep for multipoint calibration of ICP.

A1 Calibration Standard 0.25 (Be, & Cd): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.25 ul of SPEX Quality Control Standard 7 and 0.25 ul of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 0.25 ug/L.

A2 Calibration Standard 0.50 (Be & Cd): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 50uL of SPEX Quality Control Standard 7 and 50uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 0.50 ug/L.

A3 Calibration Standard 1.0 (Ba, Be, Cd, Co, Cr, Mn, Mo, Ni, Pb, Se, Sb, Tl, V, & Zn): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1 ml of conc. HNO3 and 1mL conc. HCl. Add 1.0 ul of SPEX Quality Control Standard 7 and 1.0 ul of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1 ug/L

A4 Calibration Standard 5.0 (Ag, As, Ba, Be, Cd, Co, Cr, Cu, Mn, Sb, Tl, V, & Zn): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5 ul of SPEX Quality Control Standard 7 and 5 ul of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 5 ug/L

A5 Calibration Standard 10.0 (Ag, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Sb, Tl, V, & Zn): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 10 ul of SPEX Quality Control Standard 7 and 10 ul of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10 ug/L

A6 Calibration Standard 20 (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe Mn, Mo, Ni, Pb, Sb, Tl, & V): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 20uL of SPEX Quality Control Standard 7 and 20uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 20 ug/L

A7 Calibration Standard 50 (Ag, As, Ba, Be, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 50uL of SPEX Quality Control Standard 7 and 50uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 50 ug/L

A8 Calibration Standard 100 (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Sb, Tl, V, & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 100 ul of SPEX Quality Control Standard 7 and 100 ul of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 ug/L

A9 Calibration Standard 500 (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 500uL of SPEX Quality Control Standard 7 and 500uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L

A10 Calibration Standard 1000 (Ag, Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1000uL of SPEX Quality Control Standard 7 and 1000uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1000 ug/L

A11 Calibration Standard 5000 (Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5000uL of SPEX Quality Control Standard 7 and 5000uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 5000 ug/L

A12 Calibration Standard 10000 (Al, As, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, Mg, Mn, Mo, Ni, Pb, Se, Tl, V & Zn): Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 10000uL of SPEX Quality Control Standard 7 and 10000uL of SPEX Quality Control Standard 21. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10000 ug/L

A13 Calibration Standard 100000 (Al, Ca, Fe, & Mg): Into a 100mL volumetric flask, add 5mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL of HCl. Add 1mL of 10,000 mg/L Al, 1mL of 10,000 mg/L Ca, 1mL of 10,000 mg/L Fe and 1mL of 10,000 mg/L Mg . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100,000 ug/L Al, Ca, Fe, Mn, and Mg.

A14 Calibration Standard 100K (Cr, Cu, Co, Ni, Mn, & Pb): Into a 100mL volumetric flask, add 5mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL of HCl and add 10 mls of Cr 1000 mg/L, 10 mls Cu 1000 mg/L, 10 mls Pb 1000 mg/L, 10 mls Co 1000 mg/L,10 mls Ni 1000 mg/L and 10 ml of Mn 1000 mg/L and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration:100,000 ug/L Co, Cr, Cu, Ni, Mn, and Pb.

A15 Calibration Standard 500000 (Al, Ca, Fe, & Mg): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL of HCl. Add 5mL of 10,000 mg/L Al, 5mL of 10,000 mg/L Ca, 5mL of 10,000 mg/L Fe and 5mL of 10,000 mg/L Mg. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500,000 ug/L Al, Ca, Fe and Mg

A16 Calibration Standard 1000000 (Al, Ca, Fe, & Mg): Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL of HCl. Add 10mL of 10,000 mg/L Al, 10mL of 10,000 mg/L Ca, 10mL of 10,000 mg/L Fe and 10mL of 10,000 mg/L Mg. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1,000,000ug/L Al, Ca, Fe and Mg

A17 Calibration Blank: Into a 1 L. volumetric flask, add 750 mL of Milli-Q water and 10 mL of conc. HNO3 and 10mL HCl. Mix, dilute to volume with Milli-Q H2O. Transfer to a clean 1 L. Nalgene bottle. Prepare every 6 months or as needed.

A18 ICV/CCV: Into a 1000mL volumetric flask, add 10mL Milli-Q water, 1mL of conc. HNO3 and 10mL of conc. HCl. Add 10.0mL Spex Certiprep Spike Sample Standard 1 or equivalent and 2.0mL Interference A or equivalent, 0.5 mls of 1000 ug/L Mo and 0.05 mls 1000 ug/L Ag . Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 mg/L

A19 ICSAB: Into a 100mL volumetric flask, add 500mL Milli-Q water, 5mL of conc. HNO3 and 5mL of conc. HCl. Add 50 ml of SPEX Interferents A or equivalent, 15.0mLs of 10,000 mg/L Fe, 2.5mL Spex Certiprep QC 7 and 2.5 ml Spex Certiprep-QC 21 or equivalent. Dilute to volume with Milli-Q water and mix by inverting several times.

Table 5. Sodium and Potassium Concentrations/Ranges for ICP (mg/L)

Note: These metals are calibrated using a blank and minimum of three standards. It is not allowable to remove any mid-levels to obtain an acceptable calibration; all points must be used. Multi-level calibrations must be sequential.

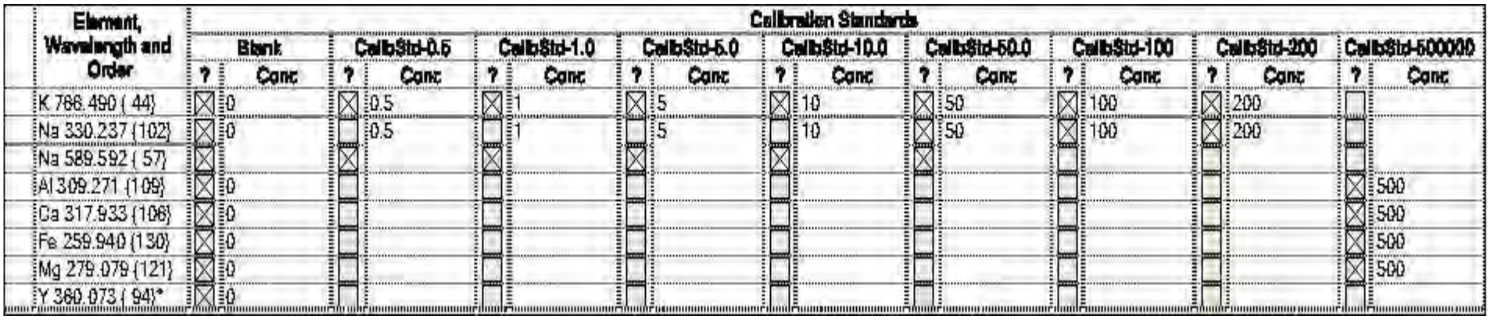
Appendix B

Standard Prep for Sodium and Potassium analysis.

B1 Calibration Standard 0.5: Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 2mL of conc. HNO3 and 2mL conc. HCl. Add 0.05mL Sodium 1000mg/L and 0.05mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 0.5 mg/L Na, K.

B2 Calibration Standard 1.0: Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.1mL Sodium 1000mg/L and 0.1mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1 mg/L Na, K.

B3. Calibration Standard 5.0: Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.5mL Sodium 1000mg/L and 0.5mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 5 mg/L Na, K.



Element, Wavelength and Order	Calibration Standards																	
	Blank		CalibStd-0.5		CalibStd-1.0		CalibStd-5.0		CalibStd-10.0		CalibStd-50.0		CalibStd-100		CalibStd-200		CalibStd-500000	
	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc
K 766.490 (44)	☒	0	☒	0.5	☒	1	☒	5	☒	10	☒	50	☒	100	☒	200	☐	
Na 330.237 (102)	☒	0	☐	0.5	☐	1	☐	5	☐	10	☒	50	☒	100	☒	200	☐	
Na 589.592 (57)	☒		☒		☒		☒		☒		☒		☐		☐		☐	
Al 309.271 (109)	☒	0	☐		☐		☐		☐		☐		☐		☐		☒	500
Ca 317.933 (106)	☒	0	☐		☐		☐		☐		☐		☐		☐		☒	500
Fe 259.940 (130)	☒	0	☐		☐		☐		☐		☐		☐		☐		☒	500
Mg 279.079 (121)	☒	0	☐		☐		☐		☐		☐		☐		☐		☒	500
Y 360.073 (94)*	☒	0	☐		☐		☐		☐		☐		☐		☐		☐	

B4. Calibration Standard 10: Into a 100mL volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1mL Sodium 1000mg/L and 5mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10 mg/L Na, K.

B5. Calibration Standard 50: Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5mL Sodium 1000mg/L and 5mL Potassium 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times.

Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 50 mg/L Na, K.

B6. Calibration Standard 100: Into a 100mL volumetric flask, add 10mL of Milli-Q H_2O, 1mL of conc. HNO_3 and 1mL conc. HCl. Add 10mL Sodium 1000mg/L and 10mL Potassium 1000mg/L. Dilute to volume with Milli-Q H_2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 mg/L Na, K.

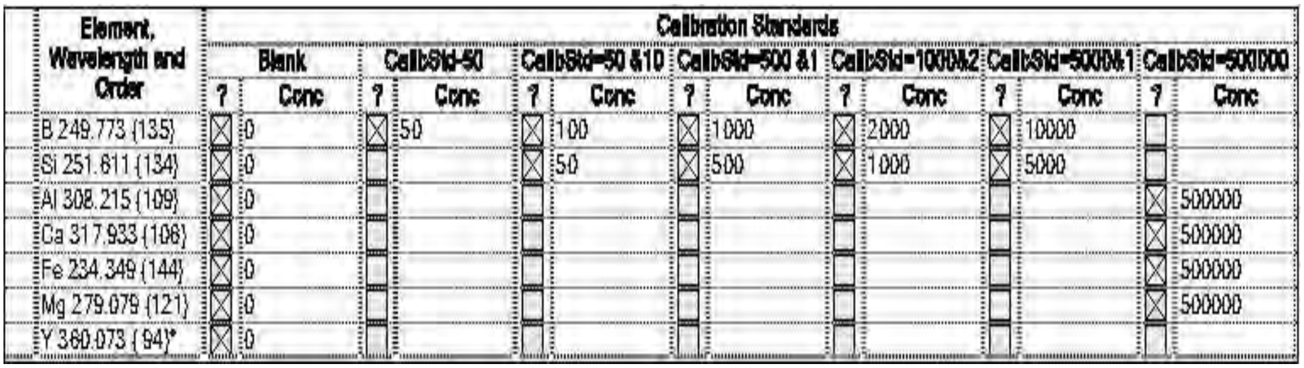
B7. Calibration Standard 200: Into a 100mL volumetric flask, add H_2O, 1mL of conc. HNO_3 and 1mL conc. HCl. Add 50mL Sodium 1000mg/L and 50mL Potassium 1000mg/L. Mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 mg/L Na, K.

B8. Calibration Standard 500 high std: Into a 100mL volumetric flask, add 10mL of Milli-Q H_2O, 1mL of conc. HNO_3 and 1mL conc. HCl. Add 5mL Aluminum 10,000mg/L, 5mL, Calcium 10,000mg/L, 5mL, Magnesium 10,000mg/L and 5mL Iron 1000mg/L. Dilute to volume with Milli-Q H_2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 mg/L Al, Ca, Mg, and Fe.

B9. ICV/CCV: Into a 100mL volumetric flask, add 50mL Milli-Q water, 1mL of conc. HNO_3 and 1mL of conc. HCl. Add 1.0mL 10,000mg/L Na and 1.0mL 10,000 K. Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 mg/L Na an K.

B8. ICSAB: Into a 100mL volumetric flask, add 10mL Milli-Q water, 1mL of conc. HNO_3 and 1mL of conc. HCl. Add 10 ml of SPEX Interferents A or equivalent, 3.0mLs of 10,000 mg/L Fe, 1.0mL 10,000mg/L Na and 1.0mL 10,000 K. Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100mg/L Na and K, 500,000ug/L Al, Ca, Fe and Mg.

Table 6. Boron and Silicon Concentrations/Ranges for ICP (ug/L)



Element, Wavelength and Order	Calibration Standards													
	Blank		CalibStd-50		CalibStd=50 &10		CalibStd=500 &1		CalibStd=1000&2		CalibStd=5000&1		CalibStd=500000	
	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc
B 249.773 {135}	☒	0	☒	50	☒	100	☒	1000	☒	2000	☒	10000	☐	
Si 251.611 {134}	☒	0	☐		☒	50	☒	500	☒	1000	☒	5000	☐	
Al 308.215 {109}	☒	0	☐		☐		☐		☐		☐		☒	500000
Ca 317.933 {106}	☒	0	☐		☐		☐		☐		☐		☒	500000
Fe 234.349 {144}	☒	0	☐		☐		☐		☐		☐		☒	500000
Mg 279.079 {121}	☒	0	☐		☐		☐		☐		☐		☒	500000
Y 360.073 {94}*	☒	0	☐		☐		☐		☐		☐		☐	

Note: Boron and Silicon are calibrated using a blank and a minimum three standards. It is not allowable to remove any mid-levels to obtain an acceptable calibration; all points must be used. Multi-level calibrations must be sequential.

3. Appendix C

Standard Prep for Boron and Silicon Analysis

C1. Calibration Standard 50 ug/L B: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.05 ml Spex Certiprep QC std. 7 or equivalent. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 50 ug/L B

C2. Calibration Standard 50 ug/L Si & 100 B ug/L: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 0.1 ml Spex Certiprep QC std. 7. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 50 ug/L Si & 100 ug/L B

C3. Calibration Standard 500 ug/L Si & 1000 B ug/L: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1.0 ml Spex Certiprep QC std. 7. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L Si &1000 ug/L B.

C4. Calibration Standard 1000 ug/L Si & 2000 B ug/L: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1.0 ml Spex Certiprep QC std. 7. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration:1000 ug/L Si &5000 ug/L B.

C5. Calibration Standard 5000 ug/L Si & 10000 B ug/L: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 1.0 ml Spex Certiprep QC std. 7. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 5000 ug/L Si &10000 ug/L B.

C6. Calibration Standard 500 high std: Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 1mL of conc. HNO3 and 1mL conc. HCl. Add 5mL Aluminum 10,000mg/L, 5mL, Calcium 10,000mg/L, 5mL, Magnesium 10,000mg/L and 5mL Iron 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 mg/L Al, Ca, Mg, and Fe.

C7. ICV/CCV: Into a 100mL **plastic** volumetric flask, add 50mL of Milli-Q water, 1 mL of conc. HNO3 and 1mL conc. HCl. Add 0.1mL of 1000mg/L Boron and 0.1mL of 1000mg/L Silicon (alternate sources from calibration source). Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1000 ug/L Boron and Silicon.

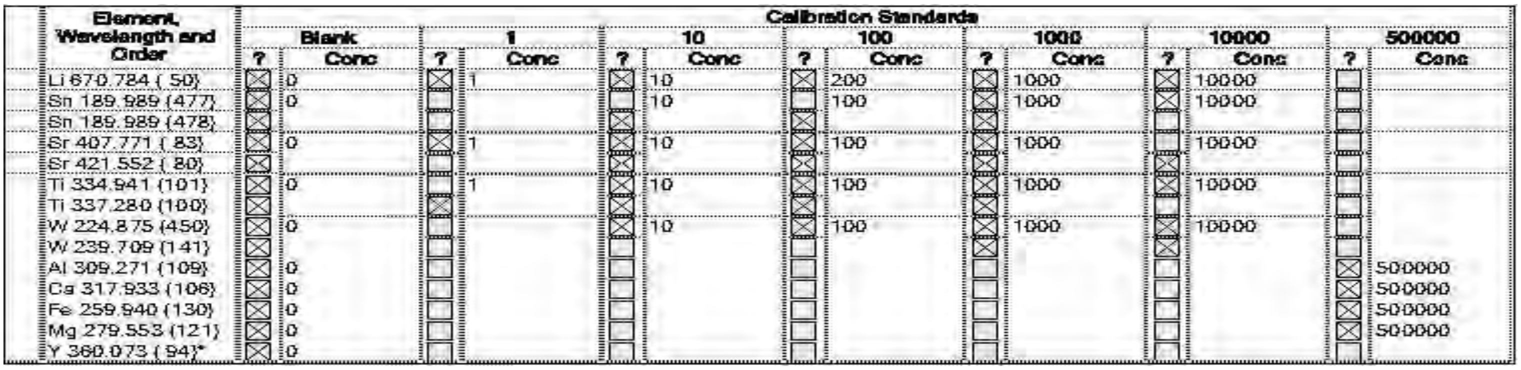
C8. ICSAB: Into a 100mL volumetric flask, add 10mL Milli-Q water, 1mL of conc. HNO3 and 1mL of conc. HCl. Add 10 ml of SPEX Interferents A or equivalent, 3.0mLs of 10,000 mg/L Fe, 0.05 ml 1000 mg/L B and 0.5 ml 1000 mg /L Si. Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L B and Si, 500,000ug/L Al, Ca, Fe and Mg.

Table 7. Lithium, Tin, Strontium, Titanium, and Tungsten Concentrations/Ranges for ICP ug/L

Appendix D

Standard prep for Lithium, Tin, Titanium, Strontium, and Tungsten

D1. Calibration Standard 1: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 0.01 ml Spex Certiprep QC std. 21 or equivalent and .001 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1 ug/L



Element, Wavelength and Order	Calibration Standards													
	Blank		1		10		100		1000		10000		500000	
	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc	?	Conc
Li 670.784 { 50}	☒	0	☒	1	☒	10	☒	200	☒	1000	☒	10000	☐	
Sn 189.989 {477}	☒	0	☐		☐	10	☐	100	☒	1000	☒	10000	☐	
Sn 189.989 {478}	☒		☐		☒		☒		☒		☐		☐	
Sr 407.771 { 83}	☒	0	☒	1	☒	10	☒	100	☒	1000	☐	10000	☐	
Sr 421.552 { 80}	☒		☐		☒		☒		☒		☒		☐	
Ti 334.941 {101}	☒	0	☐	1	☒	10	☒	100	☒	1000	☒	10000	☐	
Ti 337.280 {100}	☒		☒		☒		☒		☒		☐		☐	
W 224.875 {450}	☒	0	☐		☒	10	☒	100	☒	1000	☒	10000	☐	
W 239.709 {141}	☒		☐		☐		☐		☒		☒		☐	
Al 309.271 {109}	☒	0	☐		☐		☐		☐		☐		☒	500000
Ca 317.933 {106}	☒	0	☐		☐		☐		☐		☐		☒	500000
Fe 259.940 {130}	☒	0	☐		☐		☐		☐		☐		☒	500000
Mg 279.553 {121}	☒	0	☐		☐		☐		☐		☐		☒	500000
Y 360.073 { 94}*	☒	0	☐		☐		☐		☐		☐		☐	

D2. Calibration Standard 10: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 0.1 ml Spex Certiprep QC std. 21 or equivalent and .01 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10 ug/L Li, Sr, Sn, Ti, and W.

D3. Calibration Standard 100: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 1.0 ml Spex Certiprep QC std. 21 or equivalent and 0.1 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 100 ug/L Li, Sr, Sn, Ti, and W.

D4. Calibration Standard 1000: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 10.0 ml Spex Certiprep QC std. 21 or equivalent and 1.0 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 1000 ug/L Li, Sr, Sn, Ti, and W.

D5. Calibration Standard 10000: Into a 1000mLvolumetric flask, add 50mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 100.0 ml Spex Certiprep QC std. 21 or equivalent and 10.0 1000mg/L Sn and W . Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 10000 ug/L Li, Sr, Sn, Ti, and W.

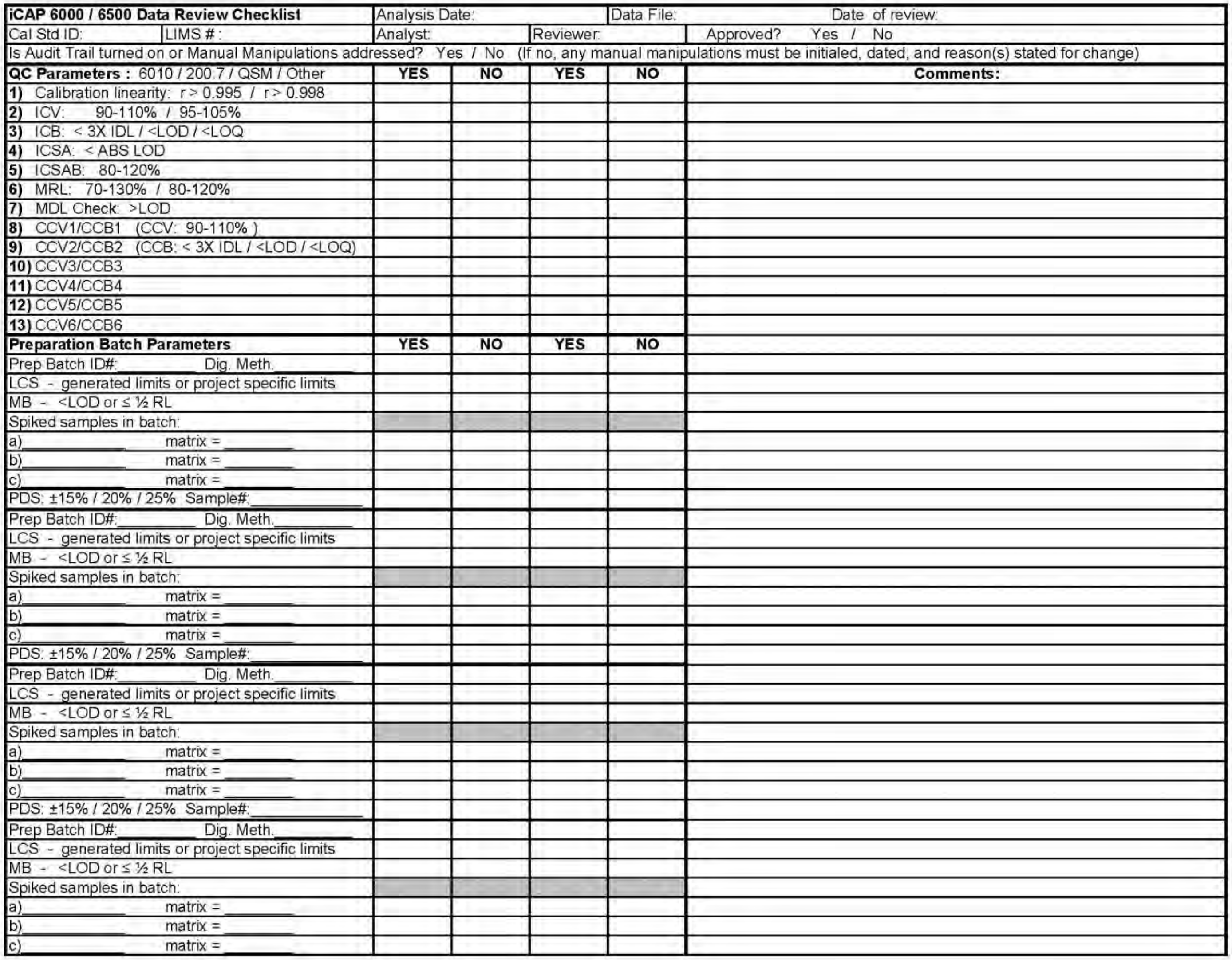
D6. Calibration Standard 500000 high std: Into a 100mL volumetric flask, add 10mL of Milli-Q H2O, 10mL of conc. HNO3 and 10mL conc. HCl. Add 5mL Aluminum 10,000mg/L, 5mL, Calcium 10,000mg/L, 5mL, Magnesium 10,000mg/L and 5mL Iron 1000mg/L. Dilute to volume with Milli-Q H2O and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 mg/L Al, Ca, Mg, and Fe.

D7. ICV/CCV: Into a 1000mL volumetric flask, add 50mL of Milli-Q water, 10 ml of conc. HNO3 and 10mL conc. HCl. Add 0.5mL of 1000mg/L Li, 0.5mL of 1000ug/L Sn, 0.5mL of 1000ug/L Sr, 0.5 ml of 1000mg/L Silicon, 0.5 ml 1000mg/L Tungsten and 0.5 ml of 1000mg/L Titanium. (all alternate sources from calibration source). Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L Li, Sr, Sn, Ti, and W.

D8. ICSAB: Into a 100mL volumetric flask, add 10mL Milli-Q water, 1mL of conc. HNO3 and 1mL of conc. HCl. Add 10 ml of SPEX Interferents A or equivalent, 3.0mLs of 10,000 mg/L Fe, 0.05 ml of 1000 mg/L Sr, 0.05 ml 1000 mg /L of Li, 0.05 ml 1000 mg /L of Sn, 0.05 ml 1000 mg /L of Ti, 0.05 ml 1000 mg /L of W. Dilute to volume with Milli-Q water and mix by inverting several times. Transfer to a clean Nalgene bottle. Prepare every 6 months or as needed. Concentration: 500 ug/L Li, Sr, Sn, Ti, and W and 500,000ug/L Al, Ca, Fe and Mg.

Note: For DOD-QSM data the lowest level (Calib. Level # 1) on a multi-point curve is prepared at concentrations equal to or less than the MRL for any given project and these levels are subject to change.

FMT9-01 iCAP 6000/6500 Data Review Checklist



iCAP 6000 / 6500 Data Review Checklist	Analysis Date:			Data File:	Date of review:
Cal Std ID: LIMS # :	Analyst:		Reviewer:		Approved? Yes / No
Is Audit Trail turned on or Manual Manipulations addressed? Yes / No (If no, any manual manipulations must be initialed, dated, and reason(s) stated for change)					
QC Parameters : 6010 / 200.7 / QSM / Other	**YES**	**NO**	**YES**	**NO**	**Comments:**
1) Calibration linearity: r > 0.995 / r > 0.998					
2) ICV: 90-110% / 95-105%					
3) ICB: < 3X IDL / <LOD / <LOQ					
4) ICSA: < ABS LOD					
5) ICSAB: 80-120%					
6) MRL: 70-130% / 80-120%					
7) MDL Check: >LOD					
8) CCV1/CCB1 (CCV: 90-110%)					
9) CCV2/CCB2 (CCB: < 3X IDL / <LOD / <LOQ)					
10) CCV3/CCB3					
11) CCV4/CCB4					
12) CCV5/CCB5					
13) CCV6/CCB6					
Preparation Batch Parameters	**YES**	**NO**	**YES**	**NO**	
Prep Batch ID#:__________ Dig. Meth.__________					
LCS - generated limits or project specific limits					
MB - <LOD or ≤ ½ RL					
Spiked samples in batch:					
a)__________ matrix = __________					
b)__________ matrix = __________					
c)__________ matrix = __________					
PDS: ±15% / 20% / 25% Sample#:__________					
Prep Batch ID#:__________ Dig. Meth.__________					
LCS - generated limits or project specific limits					
MB - <LOD or ≤ ½ RL					
Spiked samples in batch:					
a)__________ matrix = __________					
b)__________ matrix = __________					
c)__________ matrix = __________					
PDS: ±15% / 20% / 25% Sample#:__________					
Prep Batch ID#:__________ Dig. Meth.__________					
LCS - generated limits or project specific limits					
MB - <LOD or ≤ ½ RL					
Spiked samples in batch:					
a)__________ matrix = __________					
b)__________ matrix = __________					
c)__________ matrix = __________					
PDS: ±15% / 20% / 25% Sample#:__________					
Prep Batch ID#:__________ Dig. Meth.__________					
LCS - generated limits or project specific limits					
MB - <LOD or ≤ ½ RL					
Spiked samples in batch:					
a)__________ matrix = __________					
b)__________ matrix = __________					
c)__________ matrix = __________					

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Revision Number	**Description of Changes**	**Date**
02	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	03/12/2014
03	Added 4.2 QSM reference	3-9-2015
03	Added LDR and ICAL QC requirements to table 3	3-9-2015
04	Updated RSD requirement in section 12.3.1 from 5% to 3%.	03/02/2016

CT LABORATORIES
delivering more than data from your environmental analyses

STANDARD OPERATING PROCEDURE

MT 012 Mercury Cold Vapor Atomic Absorption (CV)

Review Date:
03/02/2016

Randy

03/02/2016

Technical Review by: Date

Colleen

03/31/2016

Approved by: Quality Assurance Date

Table of Contents

1. SCOPE OF APPLICABILITY

1.1. This method is appropriate for measuring mercury concentrations in groundwater, wastewater, drinking water, TCLP extracts, soils, sediments, and sludge-type materials.

2. SUMMARY OF METHOD

2.1. Prior to analysis, the samples must be prepared according to the procedures discussed in this Standard Operating Procedure (SOP).

2.2. This is a cold-vapor atomic absorption technique, based on the absorption of radiation at 254-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

3. DEFINITIONS

3.1. Reagent Blank - A solution of deionized water, (containing in correct proportion, all reagents required by the method), used with the calibration standards to standardize the instrument, as a calibration blank, and for sample dilution.

3.2. Calibration Standards - A series of known standard solutions used for calibration of the instrument within the measurable linear range. Calibration standards shall contain, in correct proportion, all reagents required by the method. A total of 5 calibration points are used for mercury calibration. Acceptance of the calibration requires a correlation coefficient of 0.995 or better. No samples shall be analyzed without an acceptable calibration.

3.3. Calibration Verification Standards, Initial (ICV) & Continuing (CCV) - A midpoint calibration standard which is analyzed at the beginning of the run (ICV), at a frequency of 1 per 10 samples during a run (CCV), and at the end of a run to verify calibration throughout the run. The ICV must be from a second source different than that of the calibration standards, while the CCV may be from the same source as the calibration standards. Note that limits for ICV are tighter than those for CCV (see section 13).

3.4. Calibration Blank Standards, Initial (ICB) & Continuing (CCB) - A reagent blank solution, which is analyzed immediately following the calibration standards (Initial Calibration Blank-ICB), at a frequency of 1 per 10 samples during a run (Continuing Calibration Blank-CCB), and at the end of a run to check for drifts in calibration or possible analyte carry-over. Control criteria consist of the highest of the following: the absolute value being less than or equal to the Method Detection Limit (MDL) for a given analyte for routine work, < Limit of Detection (LOD) for DOD-QSM, or <½ the MRL for ACOE work. If these ranges are exceeded, correct the problem and reanalyze affected data. A new calibration may be necessary to correct the problem.

3.5. Laboratory Control Sample (LCS) - A mid-range standard prepared from a source different from that used for calibration standards. The LCS is used to verify the accuracy of the digestion and is analyzed at the beginning of the analytical batch.

3.6. Method Blank (MB) - A Reagent Blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation. A minimum of one MB is prepared per batch, and is analyzed at the beginning of an analytical batch. Method blank value should be lower than the highest of the following: the absolute value being less than or equal to the MDL for a given analyte, five percent of the regulatory limit of five percent of the measure concentration in the sample. The MB results shall also be < ½ the LOQ or < 1/10th the amount in the samples or < 1/10th the regulatory limit, whichever is greater, for DOD-QSM/ACOE data.

3.7. Matrix Spike-Matrix Spike Duplicate (MS/MSD) - Two separate sample aliquots to which a known concentration of analyte has been added which is carried through the entire preparation and analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. An MS-MSD pair is prepared for every 20 samples per matrix of routine samples or for ACOE a DUP/MS pair is prepared for every 20 samples of a given matrix per day. Failure to meet criteria may be due to poor recovery during the preparation method or due to matrix interference within the digestate. To be considered acceptable, MSD must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample. MS/MSD %RPD and may be used as acceptance criteria for duplicate analysis.

3.8. Method Reporting Limit (MRL) or Contract Required Detection Limit (CRDL) Standard - Detection level standard at a level near but below the reporting limit, or at a level specified by client contract. When required, it is to be analyzed following the ICB, and prior to the last CCV standard in the run.

3.9. Duplicate (DUP) - A separate aliquot of sample which has been carried through the entire preparation and analytical procedure the same as the original sample. One duplicate per batch is prepared for ACOE/QSM work.

3.10. Serial Dilution Analysis (SD) - A sample is diluted 1:5 with method blank solution and analyzed. The diluted result and the undiluted result should agree within a limit of precision defined by the program (SW846, CLP, 200.7) or client QAPP. **For ACOE/QSM work, a SD will be conducted at a minimum rate of one per prep batch per unique matrix upon the failure of the MS.**

4. HEALTH AND SAFETY

4.1. Gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure shall utilize appropriate laboratory safety systems.

5. INTERFERENCES

5.1. Potassium permanganate is added to eliminate possible interference from sulfide. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from reagent water.

5.2. Copper has also been reported to interfere; however, copper concentrations as high as 10 mg/L had no effect on recovery of mercury from spiked samples.

5.3. Seawaters, brines, and industrial effluents high in chlorides require additional permanganate (as much as 25 mL) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 254 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine sulfate reagent (25 mL).

5.4. Certain volatile organic materials that absorb at this wavelength may also cause interference. A preliminary run without reagents should determine if this type of interference is present.

5.5. This method allows for detection of small quantities of mercury. All potential sources of mercury contamination should be avoided. This would include sources of mercury present in other lab areas.

6. EQUIPMENT AND SUPPLIES

6.1. Cetac M-6000A Mercury Analyzer with ASX-500 autosampler

6.2. Argon gas, HP grade

6.3. 50 mL disposable centrifuge tubes and caps. (Fisher p/n 05-539-9 or equivalent)

6.4. 25 mL Class A volumetric pipettes

6.5. 100 mL volumetric flasks

6.6. 25 mL glass Class A, TD, graduated cylinders

6.7. 1, 2, 3, 4, 5 mL Class A volumetric pipettes

6.8. Eppendorf pipette, 0.100 to 1.000 mL range

6.9. Environmental Express Hot Blocks set at 90-95 ^{0}C

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfuric acid, H_2SO_4, concentrated: Trace metal grade (Fisher p/n A300C-212)

7.1.2. Nitric acid, HNO_3, concentrated: Trace metal grade (Fisher p/n A509-212 or equivalent)

7.1.3. Hydrochloric acid, HCl, concentrated: Trace metal grade (Fisher p/n A508SK212 or equivalent)

7.1.4. Potassium permanganate solution, 5% w/v: Prepared by dissolving 50 g Potassium Permanganate (Fisher p/n P279-212 or equivalent) in 1000 mL of DI water. Prepare as needed. Expires 6 months from date of preparation. Store at room temperature in metals lab.

7.1.5. Potassium persulfate solution, 5% w/v: Prepared by dissolving 50 g Potassium Persulfate (Fisher p/n P282-500 or equivalent) in 1000 mL of DI water. Prepare as needed. Expires 6 months from date of preparation. Store at room temperature in metals lab.

7.1.6. Sodium chloride-hydroxylamine sulfate solution, 12% w/v: Prepared by dissolving 60 g Sodium Chloride (Fisher p/n) and 60 g of Hydroxylamine Sulfate in 500 mL of DI water. Prepare as needed. Expires 6 months from date of preparation. Store at room temperature in metals lab.

7.1.7. Stannous chloride (10% $SnCl_2$ w/v in 7% HCl v/v): to a 1000 mL volumetric flask dissolve 100 g Stannous Chloride (VWR part number MK817604) in 70 mL concentrated HCl. Stir until $SnCl_2$ is completely dissolved. Additional heat may be necessary to get complete dissolution. Once dissolved, dilute to line and cool. Prepare as needed. Expires 6 months from date of preparation. Store at room temperature in metals lab.

7.1.8. Aqua regia: In a fume hood, carefully add three volumes of concentrated HCl to one volume of concentrated HNO_3. Prepare fresh daily.

7.2. Stock Standards

7.2.1. Mercury stock standards, 1000 mg/L certified solutions, two sources. One is to be used for the calibration standards and the other for the LCS. (Ultra Scientific ICP-080 and JT Baker 6934-04 or equivalents). Store at room temperature in the metals lab. Expiration dates are given by the manufacturer.

7.3. Calibration Standards

7.3.1. Intermediate Stock #1 (10,000 µg/L) : To a 100 mL volumetric flask add 50 mL DI water and 0.2 mL concentrated HNO_3 and 0.2 mL HCl. Transfer 1.0 mL of

1000 mg/L Hg stock standard. Dilute to 100 mL with DI water and mix. Prepare fresh daily.

7.3.2. Intermediate Stock #2 (100 µg/L) :To a 100 mL volumetric flask add 50 mL DI water and 0.2 mL concentrated HNO_3 and 0.2 mL HCl. Transfer 1.0 mL of 10,000 µg/L intermediate stock #1. Dilute to 100 mL and mix. Prepare fresh daily.

7.3.3. Using 100 mL volumetric flasks, add 50 mL DI water and 0.2 mL concentrated HNO_3 and 0.2 mL concentrated HCl to each. Add the following volumes of 100 µg/L intermediate stock #2, dilute to volume with DI water, and mix well.

Standard Concentration (µg/L)	Volume (mL) of Intermediate Stock Std. #2 Added
0.5	0.5
1	1
2	2
4	4
5	5

Using a 25 mL volumetric pipette, transfer 25 mL of each to 50 mL centrifuge tubes. Add 25 mL reagent water to another centrifuge tube for the calibration blank.

7.3.4. <u>ICV/LCS and CCV: (ICV/LCS from second source, CCV from same source as standards)</u>

7.3.4.1. Intermediate Stock #1 (10,000 µg/L): To a 100 mL volumetric flask add 50 mL DI water and 0.2 mL concentrated HNO_3 and 0.2 mL HCl. Transfer 1.0 mL of 1000 mg/L Hg stock standard. Dilute to 100 mL and mix. Prepare fresh daily.

7.3.4.2. Intermediate Stock #2 (100 µg/L): To a 100 mL volumetric flask add 50 mL DI water and 0.2 mL concentrated HNO_3 and 0.2 mL HCl. Transfer 1.0 mL of 10,000 µg/L intermediate stock #1. Dilute to 100 mL and mix. Prepare fresh daily.

7.3.4.3. Check Standard/LCS: To a 100 mL volumetric flask add 50 mL DI and 0.2 mL concentrated

HNO_3 and 0.2 mL HCl. Add 3.0 mL of the 100 µg/L intermediate stock from the second source standard, dilute to the line and mix.

7.3.4.4. Using a 25 mL volumetric pipette, transfer 25 mL of each to 50 mL centrifuge tubes.

7.3.4.4.1. Note: Due to different digestion matrices for aqueous and solid samples, two sets of standards must be prepped to match the matrix for each digestion

8. SAMPLE HANDLING AND PRESERVATION

8.1. Aqueous

8.1.1. Preserved with HNO_3, pH < 2

8.1.2. 28 Day Hold Time

8.2. Solids

8.2.1. Preserved in Refrigerator (4°C)

8.2.2. 28 Day Hold Time

9. PROCEDURE

9.1. Turn on the Hot Block and allow it to heat to 95ºC while the samples are being prepared.

9.2. Sample Preparation-Aqueous:

9.2.1. Using a 25 mL graduated cylinder, transfer 25 mL of sample to a 50 mL polyethylene centrifuge tube. For drinking water analysis, a 25 mL Class A pipette must be used.

9.2.2. MS-MSD Prep: Add 0.50 mL of the 100 µg/L intermediate to a 25 mL final volume for a spike concentration of 2.0 µg/L(Table 1).

9.2.3. To each of the samples, MS-MSD, LCS, standards, and blanks add 1.25 mL concentrated H_2SO_4 and 0.625 mL concentrated HNO_3 under a hood.

9.2.4. To all samples, standards, and blanks add 3.75 mL Potassium permanganate ($KMnO_4$) solution.

9.2.5. Tightly cap the samples and mix by inverting several times.

9.2.6. The purple permanganate color should remain for at least 15 minutes. If it does not, add additional permanganate in 1 mL aliquots until the purple color remains for at least 15 minutes. Record any extra permanganate added on the mercury digestion bench sheet (Table 4). The same amount of extra permanganate will have to be added to all other samples and standards.

9.2.7. To all samples, standards, and blanks add 2.0 mL Potassium persulfate solution.

9.2.8. Place the samples and standards in the hot block. Heat at 90-95ºC for 2 hours. Record initial and final hot block temperatures on the mercury digestion bench sheet (Table 4).

9.2.9. Following digestion, remove the samples and place under a hood to cool. Alternately, the racks may be placed in a sink of cold water to hasten the cooling.

9.2.10. When the samples are cool, add 1.5 mL sodium chloride-hydroxylamine sulfate solution to all samples, standards, and blanks. Tightly cap and mix by inverting until samples are clear. Samples are now ready for analysis.

9.3. <u>Sample Preparation-Solids:</u>

9.3.1. Weigh triplicate 0.2 g (approximate) portions from separate areas of the sample container of the untreated sample into a 50 mL polyethylene centrifuge tube with a plastic spatula. Do not use metal spatulas. Record the weight on the mercury digestion bench sheet (Table 4). See the subsampling SOP FO-10 for further instructions on how to obtain a subsample for analysis.

9.3.2. Method Blank and LCS Prep: Weigh 0.50 g of sand blank into each of two 50 mL polyethylene centrifuge tubes. For the LCS, add 0.5 mL of the 100 µg/L second source intermediate stock solution #2.

9.3.3. MS-MSD Prep: Add 0.50 mL of the 100 µg/L intermediate to a 25 mL final volume for a spike concentration of 2.0 µg/L.

9.3.4. To all tubes, add 1.25 mL aqua regia reagent, and heat for 2 minutes in the hot block at 95ºC.

9.3.5. Cool, and then add 25 mL of DI water and 3.75 mL of Potassium Permanganate solution to each vial.

9.3.6. Tightly cap all vials and mix by inverting several times.

9.3.7. The purple permanganate color should remain for at least 15 minutes. If it does not, add additional permanganate in 1 mL aliquots until the purple color remains for at least 15 minutes. Record any extra permanganate added on the mercury digestion bench sheet (Table 4). The same amount of extra permanganate must be added to all other samples and standards.

9.3.8. Place the samples and standards in the hot block. Heat at 90-95ºC for 30 minutes. Record initial and final hot block temperatures on the mercury digestion bench sheet (Table 4).

9.3.9. Cool, and then add 1.5 mL of Sodium Chloride-hydroxylamine sulfate to each sample and mix by inverting. The samples should turn clear.

9.4. Instrument Set-up

9.4.1. Power up the M-6000A and autosampler and allow the instrument to warm up for one hour.

9.4.2. Turn on the lamp and gas supply and allow the lamp to warm up and the gas to flow for 15 minutes.

9.4.3. Place autosampler tubing into rinse water (1% HCl/1% HNO_3)

9.4.4. Verify that the sample capillary (inlet insert) is 0.5 mm above the gas/liquid separator center post.

9.4.5. Open vents on waste container

9.4.6. Inspect peristaltic pump tubing for wear and flat spots and replace if necessary.

9.4.7. Place the peristaltic pump tubing in their appropriate holes and holder clips. Do not lock shoe clamps at this time.

9.4.8. Initiate M-6000A program by clicking on the M6000 icon, then controls, and finally the autosampler page.

9.4.9. Start the autosampler rinse pump by clicking the pump on and the probe down.

9.4.10. Place reagent capillary in a beaker of DI water and start the peristaltic pump in a clockwise rotation.

9.4.11. Lock down the peristaltic shoe clamps.

9.4.12. Inspect liquid flows. The GLS drain should be flowing smoothly with no build up or pulsing of liquid. The waste line from the peristaltic pump to the waste container should be liquid/gas with no vibration. If this is not the case upon inspection, stop immediately and change the GLS drain line and/or waste line.

9.4.13. Wet the GLS center post. Pinch the drain line prior to the tee of the peristaltic pump drain tubing. Let two or three liquid bubbles go to the top of the GLS center post and release the drain line. If the liquid does not bubble, fill the GLS to the top of the center post and release the drain line.

9.4.14. Attach GLS exhaust tube to the GLS.

9.4.15. Place reagent capillary in the reagent bottle.

9.4.16. Open the appropriate worksheet and verify that the gas flow of the worksheet matches what is listed in the controls, if the flow is not the same make the necessary change and click set gas.

9.4.17. Zero the M-6000A using the autozero. Autozero is located under Instrument → Zero Instrument.

9.4.18. Peak profile the high standard and verify baseline and sample integration times. Do this by clicking on Analysis and then read then standard and then choose the highest standard. If there are any adjustments needed to the peak, refer to the M6000A software manual 5.6.12.

9.5. Analysis

9.5.1. Insert sample labels by clicking on labels and then entering the sample ID numbers.

9.5.2. Right click to enter the QC standards after all the samples are entered. Choose “QC standard” for the CCV and “QC blank for the CCB.

9.5.3. Click on Analysis and then Click on start.

9.5.4. Choose the appropriate box and then click OK.

9.5.5. After analysis, click on file and choose return to main index.

9.5.6. Choose reports.

9.5.7. Click on the data tab and choose the data that you want to report.

9.5.8. Click on the Reports tab.

9.5.9. Click on Write test to file and then enter the LIMS run number and make sure it is saved in the Cetac folder on the I drive.

9.6. Shutdown

9.6.1. Place the reagent capillary in a beaker of 10% nitric acid and cap the reagent bottle. Rinse the system for a minimum of ten minutes.

9.6.2. Place the reagent capillary in a beaker of DI water and rinse the system for one minute.

9.6.3. Raise sample probe by clicking on controls then autosampler and click probe up and pump off.

9.6.4. Remove reagent capillary from DI water.

9.6.5. Allow the drain and waste lines to run completely dry.

9.6.6. Turn off peristaltic pump.

9.6.7. Release peristaltic shoe clamps and release the pump tubing from their holder clips.

9.6.8. Close vents on waste container.

9.6.9. Remove GLS exhaust line from GLS.

9.6.10. Turn off gas and lamp.

9.6.11. Exit software and run off the autosampler and instrument.

10. CALCULATIONS AND DATA ANALYSIS AND REDUCTION

Liquid Concentration (µg/L) = A x C

Solid Concentrations (mg/kg) = $\frac{A \times B \times C}{D \times E}$

A = instrument reading for sample (µg/L)
B = total volume of digestion (L)
C= analyst dilution factor (ex. For a 1 to 10 dilution, C = 10)
D = amount of sample used in digestion (g)
E = percent solids/100, if necessary

Spike Recovery (%) = $\frac{\text{(Spiked sample concentration – Sample concentration) x 100}}{\text{(Spike amount)}}$

%RSD = $\frac{\text{(MS – MSD) x 100}}{\text{(MS + MSD)/2}}$

MS = Matrix spike concentration
MSD = Matrix spike duplicate concentration

11. CALIBRATION AND STANDARDIZATION

11.1. In the upper window menu bar select Instrument, then calibrate. Enter the calibration information, which consists of the standard number and standard concentration in µg/L, and them press continue. The calibration data will now be available for sample analysis. See section 9 for further calibration instructions.

12. QUALITY CONTROL

12.1. Personnel operating the CVAA shall have background knowledge of the scientific principles used during this application. All operators shall perform an initial demonstration of capability (IDC) prior to analyzing any samples. It is preferable for the operator to have at least two semesters of college chemistry.

12.2. This SOP is designed to follow a variety of different projects and programs requirements. Table 2 is designed to illustrate the control steps and provisions required to adequately producing acceptable data.

12.3. Contract Specific Sample Analysis: For certain samples, limits are specified by the Quality Assurance Project Plan (QAPP) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.4. Per QSM 5.0, LOD and LOQ checks must be analyzed for each element and matrix on a quarterly basis (or with each prep batch for infrequently performed analyses).

12.4.1. LOD checks should be analyzed at a concentration at least two times higher than the calculated MDL.

12.4.2. QSM 5.0 Appendix C LCS limits are used for recovery acceptability criteria for LOQ checks. In the absence of QSM-specific limits, in-house LCS limits are used.

13. DATA ASSESSMENT/ACCEPTANCE CRITERIA FOR QC MEASURES

13.1. When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. The analyst will review the data to ensure QC is acceptable and that exceedances are addressed. Acceptable data is then captured into the LIMS system.

13.2. After data has been captured by LIMS, it is reviewed by the analyst for accuracy and completeness by filling out the checklist (Table 3) for data review guidance.

13.3. Once the analyst has reviewed and approved the data, it is given to a peer or supervisor for review.

13.4. After the second reviewer approves the data, the reviewer sends the data to “validated” status in LIMS.

13.5. A paper hard copy of the data is then filed or archived. The package includes the checklist and data.

13.6. All calibrations are done within the linear range of the instrument. The acceptance criterion for linearity is a correlation coefficient of 0.995 or better. The highest standard used for each calibration is within the linear range for that element. Any sample measuring above the concentration of the highest standard is to be diluted and then reanalyzed.

13.7. Analysis data review checklist (Table 3) - the checklist will be completed by the analyst and the data reviewer and attached to the analytical data package.

13.8. Any results that fall outside the limits established are flagged and noted in the Metals Data Review form attached to the data package.

13.9. For each run a “QC Bench Sheet” is completed, summarizing accuracy, precision, spike preparation, and calibration standard references. Data from these sheets are used to create quarterly control limits.

13.10. <u>Non-CLP (Solid Waste and Wastewater-7470A &7471A):</u>

13.10.1. For every analytical run, calibrate with the blank and 0.50, 1.00, 2.00, 4.00, and 5.00 µg/L standards.

13.10.2. ICV: Following instrument calibration, analyze the 3.0 µg/L ICV/CCV standard. Control limits are ±10%. If the recovery exceeds this, terminate the run and correct the problem before proceeding.

13.10.3. CCV: A 3.0 µg/L standard is analyzed after every 10 samples and at the end of the run. Control limits are ± 20%.

13.10.4. ICB/CCB: After the ICV and any CCV, analyze a blank. The absolute value of the result for the blank must be below the highest of the following: the absolute value being less than or equal to the MDL for a given analyte, five percent of the regulatory limit or five percent of the measure concentration in the sample. If the result exceeds this, terminate the analysis and correct the problem before proceeding or appropriately qualify the data.

13.10.5. LCS: An alternate source standard from the same digestion set as the samples. It is prepared for every 20 samples per medium. Control limits are generated in-house control limits or as specified by client QAPP. If the recovery exceeds this, terminate the run, reprep, and reanalyze all samples.

13.10.6. MB: From the same digestion set as the samples. Blank recovery should be less than the highest of the following: the absolute value being less than or equal to the MDL for a given analyte, five percent of the regulatory limit or five percent of the measure concentration in the sample. If the result is exceeded, reanalyze. If still exceeded, isolate and correct problem, reprep and reanalyze the blank and samples associated with the blank, or appropriately qualify results.

13.10.7. MS/MSD: A MS/MSD is required every analytical run at a frequency of 5% per digestion batch for 7000 series or at a frequency of 10% per digestion batch for 200 series. To be considered acceptable, MSD must meet both the same % recovery criteria as an MS, and the same % RPD as a duplicate sample. For routine work, use in-house generated limits for the recovery and RPD limits

13.11. <u>CLP-Like Protocol (DOD-QSM/ACOE work):</u>

13.11.1. For every analytical run, calibrate with the blank and 0.50, 1.00, 2.00, 4.00, and 5.00 µg/L standards.

13.11.2. ICV: Following instrument calibration, analyze the 3.0 µg/L ICV/CCV standard. Control limits are ±10%. If the recovery exceeds this, terminate the run and correct the problem before proceeding.

13.11.3. ICB: Following the ICV, analyze a blank. The absolute value of the result for the ICB must be below ½ the MRL for ACOE data, and < LOD for DOD-QSM data. If the result exceeds this, terminate the analysis and correct the problem before proceeding.

13.11.4. LCS: Following the calibration verification standard and the calibration verification blank, and every 20 samples thereafter, analyze the 3.0 µg/L alternate source standard. Control limits are specified within the client QAPP or use default limits of 80-120%. If the recovery exceeds this, terminate the run, reprep, and reanalyze all samples.

13.11.5. MRL/CRDL standard: Analyze a standard at the MRL or the CRDL as specified by the client QAPP. Limits are listed within the client QAPP or use a default of 70-130%.

13.11.6. MB: From the digestion set. If the result exceeds ½ the LOQ, 1/10th the amount in the samples, or 1/10th the regulatory limit, whichever is greatest, re-digest all samples associated with the MB or appropriately qualify associated results.

13.11.7. CCV: Analyze the 3.0 µg/L calibration standard following every ten samples, and at the end of the analysis. Control limits are ± 20% the true value. If the recovery exceeds this, recalibrate and reanalyze all samples back to the last acceptable CCV.

13.11.8. CCB: Analyze a blank following every CCV. The absolute value of the result for the CCB must be below ½ the MRL ACOE data, and < LOD for DOD-QSM data. If the result exceeds this, reanalyze all samples back to the last acceptable CCB or appropriately qualify results.

13.11.9. MS: A matrix spike is required for every sample delivery group of 20 samples or less. The matrix spike shall be prepared at the time of digestion. Default control limits are ± 25% true value or as specified in the projects QAPP, or within limits established by the DOD-QSM. If recovery is outside of limits, refer to project QAPP or DOD-QSM for further instruction.

13.11.10. Duplicate: A duplicate is required for every sample delivery group of 20 samples or less and is prepared at the time of digestion. For results exceeding five times the MRL, the default control limit is 20% RPD, or as specified in the projects QAPP, or within limits established by the DOD-QSM. For results that are less than five times the MRL, the default control limit is ± MRL. If precision is outside of limits, refer to project QAPP or DOD-QSM for further instruction.

13.12. SDWA Protocol (245.1)

13.12.1. For every analytical run, calibrate with the blank and 0.50, 1.00, 2.00, 4.00, and 5.00 µg/L standards

13.12.2. ICV: Following instrument calibration, analyze the 3.0 µg/L ICV/CCV standard. Control limits are ± 5%. If the recovery exceeds this, terminate the run and correct the problem before proceeding.

13.12.3. ICB: Following the ICV, analyze a blank. The absolute value of the result for the ICB must be below the MDL for the method. If the result exceeds this, terminate the analysis and correct the problem before proceeding or appropriately qualify the data.

13.12.4. LCS: Following the calibration verification standards and every 20 samples thereafter, analyze the 3.0 µg/L alternate source standard. Control limits are generated control limits. If the recovery exceeds this, terminate the run, reprep, and reanalyze all samples associated with the LCS.

13.12.5. MB: From the same digestion set as the samples. Limits: the highest of the MDL, 5% of the measured concentration in the sample, or 5% of the regulatory limit for that analyte. If the results exceed this, terminate the run, reprep and reanalyze all samples associated with MB or appropriately qualify the data.

13.12.6. CCV: Analyze the 3.0 µg/L calibration standard following every ten samples, and at the end of the analysis. Control limits are ± 10% true value. If the recovery exceeds this, recalibrate and reanalyze all samples back to the last acceptable CCV.

13.12.7. CCB: Analyze a blank following every CCV. The absolute value of the result for the CCB must be below the highest of the MDL, 5% of the measured concentration in the sample, or 5% of the regulatory limit for that analyte. If the result exceeds this, reanalyze all samples back to the last acceptable CCB or appropriately qualify results.

13.12.8. MS/MSD: A MS/MSD is required every analytical run at a frequency of 10% per matrix type. For digested samples, appropriate samples are designated and spiked at the time of the digestion. Control limits are in-house limits.

14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1. When data is out of control, a number of corrective actions may need implementing. If the nonconformities involve failing QC within the analytical sequence batch, then reanalysis of samples may eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of affected data may correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument may need cleaning and decontamination. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart can be used as a guide as to which corrective actions should be taken for different QC-type failures or nonconformities (Figure 2).

15. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

15.1. Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data may be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/or minimize their effect on data.

15.1.1. The first thing addressed is prevention of producing unacceptable data. When limited sample volume is the issue, the analyst should determine if splitting the sample into lesser volumes or weights is an option. To avoid sample hold time issues, the analyst’s first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises, reanalysis is an option. Analyst error is prevented by a second analyst confirmation and validation. If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.

15.1.2. When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client service personnel of the problem. Client services will inform the client and/or responsible parties. In some instances, more samples can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

15.1.2.1. If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

15.1.2.2. In all instances, if results are reported from data that is out of control or unacceptable, that data should be qualified accordingly. Once the client has been notified and he/she instructs us to report the data, flag the data indicating what type of nonconformity has occurred.

15.1.2.3. Out of control data is still retained by the laboratory and filed and archived along with acceptable data. The file folder should be labeled as such, indicating that the data is out of control.

15.1.2.4. A non-conformance/corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

16. DATA RECORDS MANAGEMENT

16.1. Records are stored for a minimum of 5 years in accordance with the Quality Manual.

16.2. See SOP QA 003 for specifics on document control.

17. WASTE MANAGEMENT

17.1. Samples are routinely held for up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP NO. FO-8, Rev. 4).

18. REFERENCES

18.1. Test Methods for Evaluating Solid Waste, EPA, SW-846, Methods 7470A, 7471A.

18.2. Methods for the Determination of Metals in Environmental Samples. EPA/600/R94/111, Method 245.1

18.3. See CT Laboratories Quality System Manual, Section 8 for general references.

18.4. CT Laboratories Quality Manual, current revision.

18.5. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.

18.6. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.

18.7. National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.

18.8. ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

18.9 Atomic Absorption Methods, SW846, Method 7000A, Revision 1, July 1992

19. Appendices

Table 1. Mercury Spike Prep

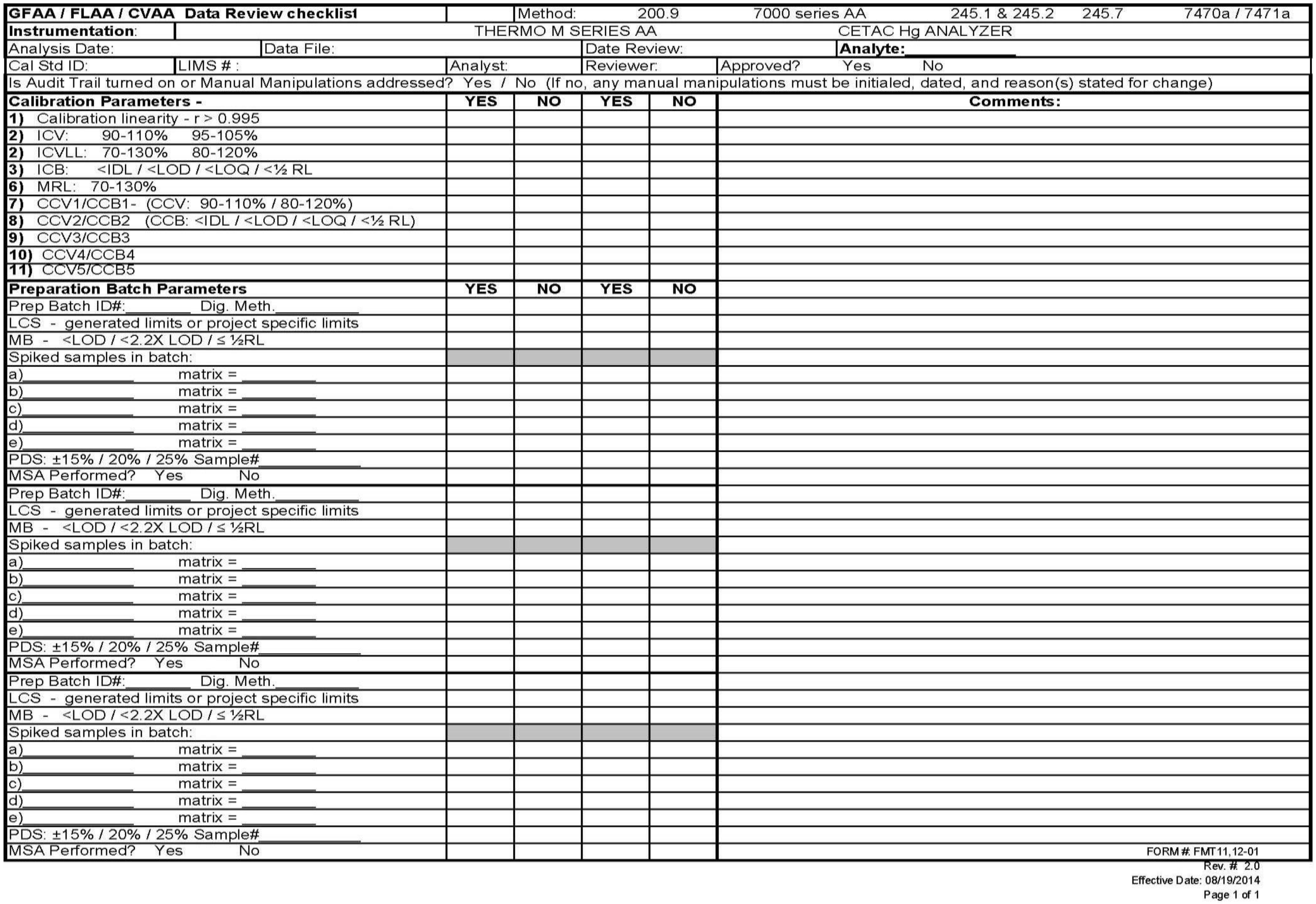
COLD VAPOR Element	**Spike Amt. mL**	**Spike Solution Supplier**	**Stock Conc. µg/L**	**Final Vol. mL**	**Expected Conc. µg/L**
Hg	0.50	Ultra 1000 mg/L	100.0	25.0	2.0

Table 2. Standard Quality Control Requirements and Corrective Action

QC Type	**Frequency**	**Conc. Level**	*Acceptance Criteria*	*Corrective Action*
ICal	Each time the instrument is set up. The ICal consists of five standards and a blank.	0 + 0.5 – 5.0 µg/L	Correlation coefficient of .995 or greater DOD-QSM: $r^2 \geq 0.99$.	Terminate analysis, correct problem and recalibrate.
ICV	Immediately after the ICal	3 µg/L	Second source standard, SDWA: 95-105% SW846:90-110% ACOE- see client QAPP DOD-QSM: 90-110%	Reanalyze once, if still unacceptable terminate analysis, correct problem and recalibrate
ICB	Immediately after the ICV	0	Routine work: < MDL, 5% of the Reg. Limit or 5% of the sample concentration. ACOE: ½ the MRL DOD-QSM: < LOD	Reanalyze once, if still unacceptable terminate analysis, correct problem and recalibrate.
LCS	1 per batch of ≤ 20 samples per matrix per day	mid cal. Range	SDWA; 85-115% SW846; 80-120% ACOE- see client QAPP DOD-QSM: See QSM Appendix C	Reanalyze once, if still unacceptable terminate analysis, correct problem and reanalyze all associated samples. High bias is acceptable for associated samples that have results less than the MDL for routine work or the MRL for ACOE work.

CCV	After every 10th sample and at the end of the analytical sequence	mid cal range	SDWA; 90-110% SW846; 80-120% ACOE- see client QAPP DOD-QSM: 90-110%	Reanalyze once, if still unacceptable recalibrate and reanalyze all samples back to the last acceptable CCV or ICV. High bias is acceptable for associated samples that have results less than the MDL for routine work or the MRL for ACOE work.
CCB	Immediately following each CCV	0	Routine work: < MDL, 5% of the Reg. Limit or 5% of the sample concentration. ACOE: ½ the MRL DOD-QSM: < LOD	Reanalyze once, if still unacceptable reanalyze all samples back to the last acceptable CCB or appropriately qualify results.
MS-MSD or MS-DUP (QSM)	5% of samples per matrix per day	See Figure 1.	≤ ± 20%, Applicable when spike level is >25% of original analyte level in the sample and RPD ≤ ± 20% 245.1 In-house limits if more stringent than Method Default of 70-130%, or as specified in DOD-QSM or ACOE QAPP	Perform PDS
Post Digestion Spike (PDS)	Upon failure of MS or per batch for ACOE work	Same level as MS	85-115% DOD-QSM: 80-120%	Qualify data as matrix interference or perform MSA
Method Blank	1 per batch of 20 samples	0	Routine work: < MDL, 5% of the Reg. Limit or 5% of the sample concentration. DOD-QSM/ACOE: < 1/2 LOQ or < 1/10th amount in samples or < 1/10th regulatory limit, whichever's highest	Investigate and isolate possible source and correct problem; then reanalyze all associated samples, if possible, or qualify data (B)

Table 3. CVAA Checklist FMT11,12-01 (Example)



GFAA / FLAA / CVAA Data Review checklist | Method: 200.9 | 7000 series AA | 245.1 & 245.2 | 245.7 | 7470a / 7471a

Instrumentation: THERMO M SERIES AA | CETAC Hg ANALYZER

Analysis Date: | Data File: | Date Review: | **Analyte:**____________

Cal Std ID: | LIMS # : | Analyst: | Reviewer: | Approved? Yes No

Is Audit Trail turned on or Manual Manipulations addressed? Yes / No (If no, any manual manipulations must be initialed, dated, and reason(s) stated for change)

Calibration Parameters -	**YES**	**NO**	**YES**	**NO**	**Comments:**
1) Calibration linearity - r > 0.995					
2) ICV: 90-110% 95-105%					
2) ICVLL: 70-130% 80-120%					
3) ICB: <IDL / <LOD / <LOQ / <½ RL					
6) MRL: 70-130%					
7) CCV1/CCB1- (CCV: 90-110% / 80-120%)					
8) CCV2/CCB2 (CCB: <IDL / <LOD / <LOQ / <½ RL)					
9) CCV3/CCB3					
10) CCV4/CCB4					
11) CCV5/CCB5					
Preparation Batch Parameters	**YES**	**NO**	**YES**	**NO**	
Prep Batch ID#:________ Dig. Meth.__________					
LCS - generated limits or project specific limits					
MB - <LOD / <2.2X LOD / ≤ ½RL					
Spiked samples in batch:					
a)____________ matrix = ________					
b)____________ matrix = ________					
c)____________ matrix = ________					
d)____________ matrix = ________					
e)____________ matrix = ________					
PDS: ±15% / 20% / 25% Sample#___________					
MSA Performed? Yes No					
Prep Batch ID#:________ Dig. Meth.__________					
LCS - generated limits or project specific limits					
MB - <LOD / <2.2X LOD / ≤ ½RL					
Spiked samples in batch:					
a)____________ matrix = ________					
b)____________ matrix = ________					
c)____________ matrix = ________					
d)____________ matrix = ________					
e)____________ matrix = ________					
PDS: ±15% / 20% / 25% Sample#___________					
MSA Performed? Yes No					
Prep Batch ID#:________ Dig. Meth.__________					
LCS - generated limits or project specific limits					
MB - <LOD / <2.2X LOD / ≤ ½RL					
Spiked samples in batch:					
a)____________ matrix = ________					
b)____________ matrix = ________					
c)____________ matrix = ________					
d)____________ matrix = ________					
e)____________ matrix = ________					
PDS: ±15% / 20% / 25% Sample#___________					
MSA Performed? Yes No					FORM #: FMT11,12-01

Rev. # 2.0
Effective Date: 08/19/2014
Page 1 of 1

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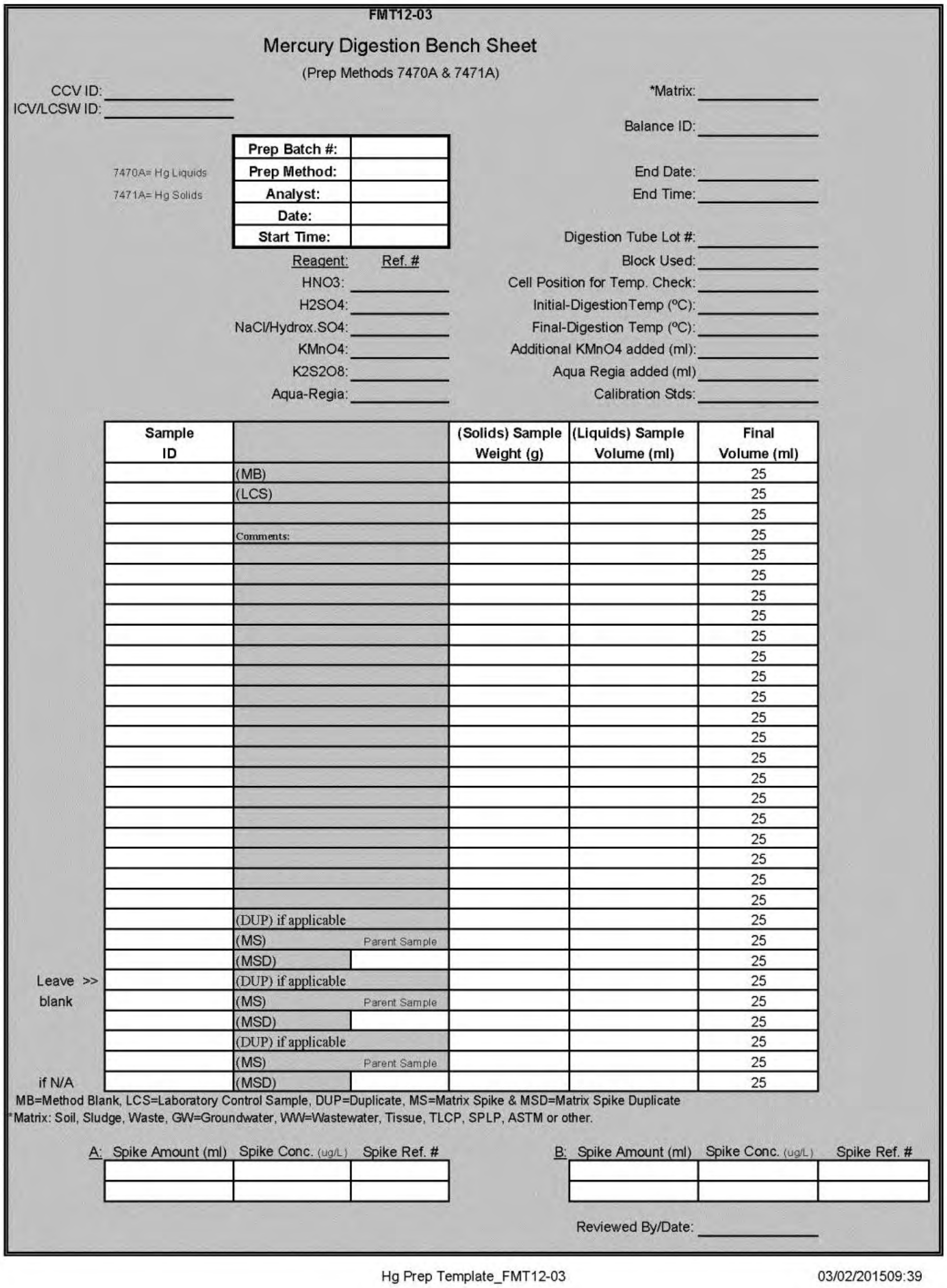
Table 4. FMT12-03(Example)
Mercury Digestion Bench Sheet



FMT12-03

Mercury Digestion Bench Sheet

(Prep Methods 7470A & 7471A)

CCV ID: ______________
ICV/LCSW ID: ______________

*Matrix: ______________

Balance ID: ______________

7470A= Hg Liquids
7471A= Hg Solids

Prep Batch #:	
Prep Method:	
Analyst:	
Date:	
Start Time:	

End Date: ______________
End Time: ______________

Digestion Tube Lot #: ______________
Block Used: ______________
Cell Position for Temp. Check: ______________
Initial-Digestion Temp (°C): ______________
Final-Digestion Temp (°C): ______________
Additional KMnO4 added (ml): ______________
Aqua Regia added (ml) ______________
Calibration Stds: ______________

Reagent:	Ref. #
HNO3:	
H2SO4:	
NaCl/Hydrox.SO4:	
KMnO4:	
K2S2O8:	
Aqua-Regia:	

Sample ID		(Solids) Sample Weight (g)	(Liquids) Sample Volume (ml)	Final Volume (ml)
	(MB)			25
	(LCS)			25
				25
	Comments:			25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
				25
	(DUP) if applicable			25
	(MS) Parent Sample			25
	(MSD)			25
	(DUP) if applicable			25
	(MS) Parent Sample			25
	(MSD)			25
	(DUP) if applicable			25
	(MS) Parent Sample			25
	(MSD)			25

Leave >>
blank
if N/A

MB=Method Blank, LCS=Laboratory Control Sample, DUP=Duplicate, MS=Matrix Spike & MSD=Matrix Spike Duplicate
*Matrix: Soil, Sludge, Waste, GW=Groundwater, WW=Wastewater, Tissue, TCLP, SPLP, ASTM or other.

A:

Spike Amount (ml)	Spike Conc. (ug/L)	Spike Ref. #

B:

Spike Amount (ml)	Spike Conc. (ug/L)	Spike Ref. #

Reviewed By/Date: ______________

Hg Prep Template_FMT12-03 03/02/201509:39

Revision Number	**Description of Changes** **EXAMPLE**	**Date**
08	Document changed to incorporated administrative requirements of ISO/IEC 17025:2005 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	03/14/2014
09	Update LCS and MS/MSD limits in Table 2 to meet method 245.1 criteria. And Included SW 846, Method 7000 reference to this SOP.	03/02/2016

CT LABORATORIES
delivering more than data from your environmental analyses

STANDARD OPERATING PROCEDURE
PR 002 TCLP and SPLP Extraction, Volatile Fraction (ZHE)

Review Date: 03/31/2016

03/31/2016

Technical Review by: Date

03/31/2016

Approved by: Quality Assurance Date

1.0 Identification of Test Method

The Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) are designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes. The following procedure will be used for performing the volatile organic ZHE procedure.

2.0 Applicable Matrix or Matrices

This procedure is used for the extraction of purgeable VOCs for a variety of liquid and solid matrices including soils, sludge, and waste samples.

3.0 Detection Limits

Method Detection Limits (MDLs) are compound, instrument, and matrix dependent. MDL analyses are performed annually for the instruments and matrices applicable to this procedure. The reporting limits (RLs) used are based on whether or not samples have been diluted prior to analyses. Default reporting limits are used for the standard TCLP/SPLP list used for the volatile organic analysis (see Scope and Application, figure 1).

4.0 Scope and Application

This procedure is applicable to a wide range of volatile organic compounds though typically only the standard TCLP/SPLP list is required (see figure 1). The reporting limits used reflect the Maximum Contaminant Level (MCL) allowed for TCLP samples. After performing the TCLP or SPLP extractions, the samples are analyzed by GCMS following the procedures outline in SOP 5280B (*Analysis of Volatile Organic Compounds by GC/MS*).

Figure 1.Standard TCLP/SPLP Compound List

Compound	Reporting limit (mg/L)
vinyl chloride	0.2
1,1-dichloroethene	0.7
1,2-dichloroethane	0.5
2-butanone	200
chloroform	6
carbon tetrachloride	0.5
benzene	0.5
trichloroethene	0.5
tetrachloroethene	0.7
chlorobenzene	100

5.0 Method Summary

5.1 TCLP

5.1.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

5.1.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.93 solution. A special extractor vessel (ZHE) is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

5.1.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

5.2 SPLP

5.2.1 For liquid samples (i.e., those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the SPLP extract.

5.2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of reagent DI water equal to 20 times the weight of the solid phase. A special extractor vessel (ZHE) is used when testing for volatile analytes. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

5.2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

6.0 Definitions

6.1 For definitions on all terms applicable to this method, see Appendix 10 of the Quality Assurance Manual (QAM).

6.2 For a list of common acronyms and abbreviations, see QAM Appendix 7.

7.0 Interferences

7.1 Carry over contamination is a problem when a highly contaminated sample is followed by a clean sample. Thorough cleaning and rinsing the ZHE device will eliminate the potential for carry over.

7.2 Other potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

8.0 Safety

8.1 Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

8.2 The toxicity and carcinogenicity of the chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, so care must be taken to prevent undue or extensive exposure.

8.3 For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psig or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced.

8.4 The ZHE should be checked periodically for leaks. If the device contains a built-in pressure gauge, pressurize the device to 50 psig, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psig, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device.

8.5 All personnel performing this analysis shall be instructed in the use of personal protective equipment prior to beginning analysis.

9.0 Equipment and Supplies

9.1 Rotator apparatus capable of turning at 30 +/- 2 rpm (Lars Land or equivalent).

9.2 ZHE extraction devices (Lars Land or equivalent).

9.3 Pressure filtration apparatus (Millipore or equivalent).

9.4 Glass fiber filters 0.6-0.8 micron 11.0 cm. (Environmental Express, Cat. # FG75110MM or equivalent).

9.5 Glass fiber filters 0.6-0.8 micron: 15.0 cm. (Environmental Express, Cat. # FG75150MM or equivalent).

9.6 Fluid pump (FMI Lab Pump, Model QSY or equivalent).

9.7 Gas pressure/vacuum pump (Gast, Model DOA-P104-AA or equivalent).

9.8 250 mL and 500 mL beakers (Pyrex or equivalent).

9.9 Transfer line, 1/8”ID x 1/4”OD (Nalgene, 280 Pur-ester tubing)

9.10 Tedlar Bag

9.11 1000 ml Graduated cylinders, Class A (Kimble or equivalent).

9.12 100mL glass gas tight syringe (Hamilton 7000 series or equivalent).

9.13 40 mL VOA vials (C&G or equivalent).

9.14 Analytical balance (Ohaus, Voyager Pro or equivalent).

9.15 Top-loading balance (Mettler-Toledo, Model BD-202 or equivalent).

9.16 TCLP/SPLP prep log (see Tables 2 & 3).

Note: The interior surface of the ZHE (9.2) and the pressure filtration apparatus (9.3) should be smooth and free of scratches. Clean using only a very soft bristled brush if necessary. In addition, the screen on which the filter is placed should be clear of debris. If any of the holes are clogged they can be cleaned by sonicating for 15 minutes.

10.0 Regents and Materials

10.1 Reagent grade DI water, organic free (Millipore, 18 mega ohm quality).

10.2 Glacial Acetic Acid (CH3CH2OOH), ACS Grade (Fisher, Cat.# A38S-212 or equivalent).

10.3 Sodium Hydroxide (NaOH), pellets

10.3.1 Fisher, Cat.# S318-3 or equivalent.

10.3.2 10 N NaOH solution. Into a 1 liter volumetric flask, add 500 mL of DI water. Dissolve 400g of NaOH pellets (caution: mixture will become very hot). When cool, dilute to volume with D.I. H2O and mix well.

10.4 Extraction Fluids

10.4.1 TCLP extraction fluid #1: (To prepare a 20 liter quantity): Fill a 20 L carboy with 19 L of DI water. Add 114 mL CH3CH2OOH and 128.6 mL 10N NaOH. Dilute to 20 L With D.I. H_2O and mix by stirring. When correctly prepared, the pH of this fluid will be 4.93 +/- 0.05.

Note: This extraction fluid should be monitored frequently for impurities. The pH should be checked prior to use to ensure that the fluid is made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

10.4.2 SPLP extraction fluid: Reagent DI water.

11.0 Sample Preservation and Storage

11.1 Preservatives shall not be added to samples before extraction.

11.2 Care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 0-6ºC. Samples shall be opened only immediately prior to extraction. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses.

11.3 Volatiles have 14 days from the date of collection to be extracted.

11.4 Volatiles have 14 days from the date of extraction to be analyzed.

12.0 Quality Control

12.1 This SOP is designed to follow a variety of different projects and programs requirements. Table 1 is designed to illustrate the control steps and provisions required to adequately producing acceptable data.

12.2 Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.3 Program Specific Limits: Samples analyzed under the guidance of certain programs; such as the Department of Defense Quality Systems Manual (DoD/QSM) or Louisville Chemistry Guidance (LCG), require their own specified limits. For these samples follow the limits specified in the manuals for that program.

13.0 Calibration and Standardization

See SOP 8260B (*Analysis of Volatile Organic Compounds by GC/MS*) for the appropriate analyses calibration.

14.0 Procedure

14.1 Determine sample % solids:

14.1.1 For solid samples which contain no free liquids, proceed to sec. 14.3.

14.1.2 For samples which are liquid, contain free liquids, or are multi-phasic, filtration or liquid/solid separation is required as follows:

14.1.2.1 Preweigh a GFF filter and record the weight.

14.1.2.2 Preweigh a receiving beaker and record the weight.

14.1.2.3 Preweigh a transfer beaker and record the weight.

14.1.2.4 Assemble the pressure filtration device with the GFF filter, and place the receiving beaker underneath.

14.1.2.5 Weigh out a subsample of the waste (100g. minimum) and record the weight. An additional minimum 25g will be needed for the extraction. For low volume samples, consult your supervisor.

14.1.2.6 Transfer the waste to the filtration device and secure the top.

14.1.2.7 Re-weigh the empty transfer beaker and record the weight.

14.1.2.8 Slowly apply air pressure to the filtration device in 10 psi increments up to 50 psi. or until air passes through the filter. Hold at each increment for 2 minutes before proceeding to the next higher increment.

Note: Some wastes, such as oily wastes and some paint wastes will contain material that appears to be a liquid. Even after applying pressure to 50 psi, this material may not filter. In this case, for the non-volatile extraction, the material in the filter holder is defined as the solid phase and is carried through the extraction procedure as a solid. However, since the volatile procedure requires utilizing the ZHE device to filter the sample following rotation, the ZHE filter may become plugged and filtration of the extract from the ZHE device may not be possible. Typically the % solids is predetermined in the preparation procedure for the non-volatile constituents.

14.1.2.9 Weigh the receiving beaker and record the weight.

14.1.2.10 The material in the filter holder is defined as the solid phase of the waste, and the material in the receiving beaker is defined as the liquid phase.

14.1.2.11 Determine and record the weight of the liquid phase.

14.1.1.12 Determine the weight of the solid phase by subtracting the weight of the liquid phase from the total weight of the waste.

14.1.1.13 Calculate the % solids as follows:

$$\% \text{ solids} = \frac{\text{weight of solid phase}}{\text{total weight of waste}} \times 100$$

14.2 Evaluation of % solids:

14.2.1 If the % solids are <0.5%, a fresh portion of the waste will be filtered through the ZHE device and collected into a Tedlar bag or 100 mL syringe. This filtrate will be defined as the TCLP extract. Proceed to sec.13.3, then 13.4.4

14.2.2 If the % solids are significantly >0.5%, proceed to section 14.3, then 14.4.1. or 14.4.2.

14.2.3 If the % solids are $\geq$0.5% or are very close, and it is noticed that the solid material is entrained in the filter, dry the filter at 80-120C until two successive weighings agree within +/- 1%. Determine the % dry solids. If the % dry solids are <0.5%, follow 14.2.1. If the % dry solids are >0.5%, see note below.

Note: There must be a significant level of % solids such that a minimum of 5-10g of solids can be generated for the extraction. This minimum amount of solids will yield 100-200 mL of extract.

14.3 ZHE device preparation:

14.3.1 Assemble the ZHE device as follows:

14.3.1.1 Place two O-rings on the piston.

14.3.1.2 Place an O-ring in the ZHE base.

14.3.1.3 Wet the O-rings of the piston and place the piston inside the ZHE body. Depress the piston into the ZHE body only far enough to allow room for the sample.

14.3.1.4 With the piston installed, place the ZHE body into the base.

14.3.1.5 Place an O-ring on the top of the ZHE body.

14.3.1.6 Place an 11.0 cm. GFF filter between the two filter screens and set aside.

14.3.1.7 The ZHE device is now ready to receive a sample.

14.4 Adding sample to the ZHE device:

14.4.1 For samples that are 100% total solids:

14.4.1.1 Weigh out 25.0g. of sample into a beaker or other suitable container and record the weight. If particle size reduction if required, chill the sample and reduction equipment to 4^0C to reduce the loss of volatile compounds. Proceed to chop, crush, or grind the sample to a minimum $1cm^2$ size. Minimize exposure to the atmosphere.

14.4.1.2 Transfer the waste material to the ZHE device and secure the top.

14.4.1.3 Attach the air line from the pressure pump to the lower valve. Open the upper and lower valves.

14.4.1.4 Slowly pressurize the ZHE device to 50 psi to force the piston to the top of the ZHE body, thereby removing any headspace. Remove the air line.

14.4.1.5 Determine the amount of extraction fluid to add follows: amount of fluid added = 20 x's the weight of the sample

14.4.1.6 Proceed to sec. 14.5

14.4.2 For samples >5% but <100% total solids:

14.4.2.1 Determine the amount of sample to add to the ZHE as follows:

$$\text{Weight of sample to use} = \frac{25}{\%\text{ total solids (sec. 14.1)}} \times 100$$

14.4.2.2 Proceed to sec. 14.4.3, using the amount of sample determined above.

14.4.3 For samples that are between 0.5% - 5% total solids:

14.4.3.1 Weigh a 500 mL beaker and record the weight.

14.4.3.2 Into the beaker, weigh out 500 g of sample and record the weight.

14.4.3.3 Transfer the waste to the ZHE device and secure the top.

14.4.3.4 Connect the air line to the lower valve. Open upper and lower valves.

14.4.3.5 Slowly pressurize the ZHE to expel any air. Close the top valve when liquid appears.

14.4.3.6 Pre-weigh a Tedlar bag and record the weight.

14.4.3.7 Slowly pressurize the ZHE to expel the liquid into the bag. Do not exceed 50 psi.

14.4.3.8 Close the valves. Remove the bag, weigh the bag and its contents, and record the weight.

14.4.3.9 Determine the amount of extraction fluid to add follows:

$$\text{weight of extraction fluid} = \frac{20 \times \text{weight of sample waste} \times \%\text{ solids}}{100}$$

11.4.3.10 Proceed to sec. 14.5

14.4.4 For samples that are <0.5% total solids:

14.4.4.1 The liquid portion of the waste, after filtration is defined as the TCLP extract. The ZHE device will be used to filter the sample.

14.4.4.2 Add an appropriate amount of sample to the ZHE device to complete the requested analyses.

14.4.4.3 Secure the top; connect the air line to the lower valve. Open upper and lower valves.

14.4.4.4 Slowly pressurize the ZHE to expel any air. Close the top valve when liquid appears.

14.4.4.5 Connect a 100 mL syringe or evacuated Tedlar bag to the top valve. Slowly pressurize the ZHE to force the liquid into the syringe or bag. Alternatively a transfer line can be connected to the outlet valve on the ZHE and the sample extract can be transferred directly into the VOA vial. If collecting the liquid with a syringe, carefully transfer, (after discarding the first 5 mL) with headspace, to 40 mL VOA vials. If using a bag, allow the liquid to flow into the bag until a sufficient quantity has been collected for analysis. Store the extract at 0-6^{0}C until analysis.

14.5 Adding extraction fluid to the ZHE device:

14.5.1 Transfer via a graduated cylinder the appropriate amount and type of extraction fluid to a 500 mL beaker.

14.5.2 Using the fluid pump, place the intake line into the beaker of extraction fluid. Turn on the pump, and allow the fluid to enter the pump. Stop the pump when the fluid appears at the end of the outlet line.

14.5.3 Connect the outlet line to the ZHE top valve. Turn on the pump, and allow the pump to charge the ZHE with the entire contents of the beaker. Stop the pump before any air reaches the ZHE. Close the top and bottom valves, and remove the inlet line. Manually rotate the ZHE device in an end over end fashion 2 or 3 times.

Note: While the ZHE is filling, check for fluid leaking out of the bottom valve. If this happens, stop the pump and use a different ZHE device with a fresh sample.

14.5.4 Attach the air line to the bottom valve. Open the top valve, and while holding a paper towel at the valve, slowly pressurize the ZHE to expel any remaining air. Close the valve at the first sign of fluid at the valve outlet.

14.5.5 With the air line still attached, re-pressurize the ZHE device to 10 psi. Check for leaks. The ZHE is now ready for the 18 +/- 2 hr. rotation. Proceed to sec. 14.6.

14.6 Extraction:

14.6.1 Secure the samples in the rotation apparatus.

14.6.2 Rotation time is 18 +/- 2 hours at 30 +/- 2 rpm. A room temperature of 23 +/- 2^0C shall be maintained during the extraction period. Begin rotating. Record the time, rotation rate, and room temperature in the Extraction Summary Logbook (Table 3).

14.6.3 Following the rotation period, record the end time of rotation and the room temperature. Proceed to sec 14.7 for filtering the extract.

14.7 Filtration following extraction:

14.7.1 Following the rotation period record the date, time, and temperature. Check the ZHE pressure gauge to ensure that the device did not leak. If pressure was not maintained, the sample must be re-extracted with a new portion of waste sample (Document in prep logbook).

14.7.2 If pressure has been maintained, the material in the ZHE device is separated into liquid and solid phases.

14.7.3 For samples with no initial liquid phase**:**

14.7.3.1 Attach the 100 mL gastight syringe to the ZHE outlet valve. Open the valve and carefully withdraw the extract into the syringe. Do not allow air bubbles into the syringe. Pressurizing the ZHE to 10 psi may help in withdrawing the extract. Alternatively, a transfer line can be connected to the outlet valve on the ZHE and the sample extract can be transferred directly into the VOA vial.

14.7.3.2 Discard the first 5 mL out of the syringe, and then transfer the remaining extract to 40 mL VOA vials (without headspace). Store at 0-6^0C until analysis.

14.7.4 For samples containing an initial liquid phase**:**

If an initial liquid phase was collected, determine if the ZHE liquid extract will mix with the initial liquid phase:

14.7.4.1 Using a transfer pipette and a small beaker, add a few drops of the liquid phase to a small quantity of DI water. Observe to see if the two liquids are miscible, or if they separate into layers.

14.7.4.2 If the two phases are miscible, combine the filtered extract with the initial liquid phase and analyze as one sample. Attach the Tedlar bag containing the initial liquid phase to the outlet valve of the ZHE device. Gradually apply pressure to the lower valve and slowly filter all of the extract into the Tedlar bag. Store at 0-6^{0}C until analysis.

14.7.4.3 If the two phases do not mix, the initial liquid phase and the filtered extract will need to be collected and analyzed separately. Collect all of the extract in a separate Tedlar bag.

14.8 Samples are now ready for analysis.

14.8.1 When the standard list and reporting limits are required the samples can be diluted at least 1:100 and still maintain the reporting limits. The dilution is recommended to reduce the amount of glacial acetic acid introduced into the GC/MS system in order to ensure a longer column lifetime. Samples can be analyzed undiluted but this greatly increases the peak for glacial acetic acid and will potentially shorten column lifetime and will increase the chances of background contamination.

14.8.2 Matrix spikes are prepared after extraction of the sample matrix and prior to analysis by Purge & Trap/GM-MS analysis.

14.8.2 Make sure all proper documentation was entered in the TCLP/SPLP Logbook (Table 3.

15.0 Calculations

15.1 Results are directly obtained from the analysis instrument in ug/L. If using the standard TCLP/SPLP list and reporting limits a dilution of 1:100 is typically perform. To obtain the final results calculate as follows:

$$\text{Results mg/L} = \frac{\text{Analytical result (ug/L)} \times \text{DF}}{1000}$$

15.2 Calculate the results for multiphasic as follows:

$$\text{Final analyte concentration} = \frac{(V1)\,(C1) + (V2)\,(C2)}{V1 + V2}$$

V1 = volume of the first phase liquid
C1 = concentration of the first phase in mg/L
V2 = volume of the second phase liquid
C2 = concentration of the second phase in mg/L

15.3 See Procedure (14.0) for all other applicable calculations.

16.0 Method Performance

Certified standard solutions, properly maintained extraction equipment and instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies.

17.0 Pollution Prevention

See QAM Appendix 9

18.0 Data Assessment & Acceptance Criteria for QC Measures

18.1 If the initial analysis of a sample or a dilution of the sample has a concentration of a particular target analyte that exceeds the calibration range, the sample must be reanalyzed at a dilution that will keep compounds within the calibration range of the instrument.

18.2 Refer to the analysis method (SOP 8260B: *Analysis of Volatile Organic Compounds by GC/MS*) for additional analysis criteria.

19.0 Corrective Measures for Out-of-Control Data

See QAM Appendix 9

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

See QAM Appendix 9

21.0 Waste Management

See QAM Appendix 9

22.0 Equipment / Instrument Maintenance, Computer Hardware & Software & Troubleshooting

See QAM Appendix 9

23.0 References

23.1 Test Methods for Evaluating Solid Waste, EPA-SW-846. September, 1994. Method 1311.

23.2 Test Methods for Evaluating Solid Waste, EPA-SW-846. September, 1994. Method 1312.

23.3 CT Laboratories Quality Manual, current revision.

23.4 Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.

23.5 National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.

23.6 ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

24.0 Tables

Table 1
Summary of Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
ZHE Pressure	Pressure applied and set @ 10 psi for all samples and blanks	Pressure must be maintained throughout the extraction process (pressure may increase due to affect from sample matrices)	Do not proceed with analysis until extraction meets criteria. Document problem and re-prepare sample for extraction.
Sample Rotation	30 rpm for 18 hours for all samples and blanks	Rotation rate: 30 ± 2 rpm. Rotation time: 18 ± 2 hours	Do not proceed with analysis until extraction meets criteria. Document problem and re-prepare sample for extraction.
Method Blank (MB)	1 / 20 samples per matrix or at contact/ program specific frequencies. The MB is used to document contamination resulting in the analytical process and shall be carried through the complete sample preparation and analytical procedure.	1. Concentration of analytes of concern shall be less than the highest of either : *Reporting Limit or MDL *Five percent of the measured concentration in the sample. 2. ACOE/QSM: ≤ ½ RL 3. Less than program/project specified limits.	Reanalyze to determine if instrument or laboratory background contamination was the cause. If the method blank is still non-compliant, re-prepare and reanalyze blank and samples.* For ACOE/QSM data if less than ½ MRL no action required.* *If reanalysis of blank still contains contamination above specified limits, affected data shall be qualified (B)

Table 2

FPR2-01
ZHE (Volatiles) Extraction Vessel Usage Log

ZHE (Volatiles)

Usage # >>>>
Blank Analyzed

Vessel #	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	Date / Batch #
1																					/
2																					/
3																					/
4																					/
5																					/
6																					/
7																					/
8																					/

****After 20 uses the vessel must be used for a blank check**

Table 3

FPR2-02
TCLP/SPLP/ASTM Extraction Summary

TCLP/SPLP/ASTM Extraction Summary

Batch:________ Date:________ *Analyst (set up):________* *Analyst (take down):________*

Sample #	Parameter	Test			Vessel / ZHE	Buffer Determination			Sample wt.	**Free Liquids Present (Y/N)	Vol. Ext. Fluid	Initial pH	ZHE Initial Press	Time In	Date / Time Out	Ext. Time	End pH	ZHE End Press	Date / Time Filtered	Volume Filtered
	(M / SV / V / WC)	TCLP	SPLP	ASTM	#	Initial pH	End pH	*Ext. Fluid	(g)		(mL)		(psi)			(hrs)		(psi)		(mL)
															/				/	
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**Extraction Fluid Used:____________* *Ext. Fluid ID #:____________* *Ext. Fluid pH:_________* *Ext. Start Temp (°C):_________*

Ext. End Temp(°C):___________ (min:________ / max:_________) *Filter Manufacturer:__________________* *Filter Lot #:______________*

Balance:____________ *pH Meter:_____________* 1N HCL:___________ *Tumbler Unit #(s):___________*

Acceptable rotation rate: 30 ±2 rpm *Verified Rotation rate: ________ rpm*

M=Metals, SV=Semi Volatiles, V=Volatiles, WC=Wet Chemistry

Table 4

FPR2-03
% Solids Calculation Worksheet

% Solids Calculation Worksheet

Tare weights (g) *Transfer weights (g)* *Separation weights (g)* *% Solids Calculations*

Sample #	A Filter wt.	B Filtrate Vessel wt.	C Transfer Vessel wt.	D Sample + Transfer Vessel	E Sample wt {D - C}	F Vessel wt. Post Transfer	G Residue wt. {F – C}	H Total Waste Amount {E – G}	I Filtrate Vessel + Filtrate	J Liquid Phase of Sample {I – B}	K Solid Phase of Sample {H – J}	L. % Solids (**) (K/H*100)	M Waste + Filter (Dry) wt. (g)	% Solids (Dry) ({M – A}/H)*100

(**) If Solids = <0.5%, treat filtrate as TCLP extract, if free liquid present use % Solids Calculation Worksheet

Revision Number	**Description of Changes**	**Date**
02	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	03/12/2014
03	Added section 14.8.2 as to when matrix spikes can be prepared for analysis. And updated the TCLP/SPLP/ASTM Extraction Summary form to include min/max temperatures (Table 3).	03/31/2016

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STANDARD OPERATING PROCEDURE

PR 003 TCLP/SPLP Extraction, Non- Volatile Fraction

Review Date: 02/18/2016

02/18/2016

Technical Review by: Date

03/31/2016

Approved by: Quality Assurance Date

1.0 SCOPE AND APPLICATION

1.1 The Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP) are designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 The following procedure will be used for performing the metals and semi-volatile extraction.

2.0 METHOD SUMMARY

2.1 TCLP

2.1.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

2.1.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.1.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

2.2 SPLP

2.2.1 For liquid samples (i.e., those containing less than 0.5 % dry solid material), the sample, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the SPLP extract.

2.2.2 For samples containing greater than 0.5 % solids, the liquid phase, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the region of the country where the sample site is located if the sample is a soil. If the sample is a waste or wastewater, the extraction fluid employed is a pH 4.2 solution. Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 DEFINITIONS

3.1 Batch - A batch consists of a maximum of 20 samples of similar matrix which are prepared and analyzed in the same manner. Each batch is given a unique prep batch number for tracking purposes.

3.2 PB or MB (Prep Blank/ Method Blank) - A Reagent Blank which is carried through the entire preparation and analytical method. The method blank is used to detect possible contamination that may occur prior to or during the sample preparation. A minimum of one MB is prepared per batch, and is analyzed at the beginning of an analytical batch.

3.3 MS (Matrix Spike): - A separate sample aliquot to which a known concentration of analyte has been added post extraction and is carried through the entire analytical procedure. The purpose of a matrix spike is to reveal any matrix effect from the sample on the recovery of the analyte by the method being used. One MS is prepared for each waste type in a given batch of samples. Failure to meet criteria may be due to poor recovery during the preparation method or due to matrix interference within the sample.

4.0 HEALTH AND SAFETY

Gloves and protective clothing should be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems.

5.0 CAUTIONS

There are no cautions

6.0 INTERFERENCES

Refer to the sample analytical methods for interferences.

7.0 PERSONNEL QUALIFICATIONS

7.1 All personnel performing this analysis should be instructed in the use of personal protective equipment prior to beginning analysis.

7.2 Personnel should know how to read a meniscus and how to use a balance correctly.

8.0 APPARATUS AND MATERIALS

8.1 Rotator apparatus capable of turning at 30 +/- 2 rpm.

8.2 2 liter glass extraction jars.

8.3 2 liter polyethylene extraction containers.

8.4 Millipore pressure filtration apparatus. Note: The interior surface of the pressure filtration apparatus should be free smooth and free of scratches. Clean using only a very soft bristled brush if necessary. Also, the screen on which the filter is placed should be clean of debris. If any of the holes are clogged they can be cleaned by sonicating for 15 minutes.

8.5 Glass fiber filters 0.7 micron: Environmental Express TCLP filters or equivalent.

8.6 Ceramic filtration funnel, 15 cm.

8.7 2 liter filtration flask.

8.8 pH meter.

8.9 Top-loading balance, 0.01 g capacity.

8.10 Hotplate.

8.11 Magnetic stirrer.

8.12 100 mL graduated cylinder.

8.13 5 mL oxford pipette.

8.14 Thermometer, 100^0C.

8.15 Reagents:

8.15.1 De-ionized water: Milli-Q type II

8.15.2 Hydrochloric acid 1N: Prepare in hood. Into a 1 liter volumetric flask, add 900 mL of D.I. H2O. Carefully add 83 mL ACS reagent grade conc. HCl. Dilute to volume with D.I. H2O and mix well.

8.15.3 Glacial acetic acid, conc. ACS reagent grade.

8.15.4 Sodium Hydroxide 10N: Into a 1 liter volumetric flask, add 500 mL of D.I. H2O. Dissolve 400g. ACS reagent grade NaOH pellets (caution: mixture will become very hot). When cool, dilute to volume with D.I. H2O and mix well.

8.15.5 SPLP extraction fluid acid mixture: To a 100 mL volumetric flask, add 50 mL D.I. H2O. Carefully add 6 g. Conc. H2SO4 and 4 g. Conc. HNO3. Mix by swirling and dilute to volume with D.I. H2O.

8.16 EXTRACTION FLIUDS

8.16.1 TCLP extraction fluid #1: (To prepare a 20 liter quantity): Fill a 20 L carboy with 19 L Of D.I. H_2O. Add 114 mL glacial acetic acid and 128.6 mL 10N NaOH. Dilute to 20 L With D.I. H_2O and mix by stirring. When correctly prepared, the pH of this fluid will be 4.93 +/- 0.05.

8.16.2 TCLP extraction fluid #2: (To prepare a 20 liter quantity): Fill a 20 L Carboy with 19 L of D.I. H_2O. Add 114 mL glacial acetic acid. Dilute to 20 L and mix by stirring. When correctly prepared, the pH of this fluid will be 2.88 +/- .05.

8.16.3 SPLP extraction fluid #1: To be used for sites that are east of the Mississippi River. Prepare a sufficient quantity of extraction fluid by adding the SPLP acid mixture (see step 8.15.5 of this SOP) to D.I. H_2O to obtain a pH of 4.20 +/- 0.05. Note: solutions are unbuffered and the exact pH may not be obtained.

8.16.4 SPLP extraction fluid #2: To be used for sites that are west of the Mississippi River. Prepare a sufficient quantity of extraction fluid by adding the SPLP acid mixture (see step 8.15.5 of this SOP) to D.I. H_2O to obtain a pH of 5.00 +/- 0.05. Note: solutions are unbuffered and the exact pH may not be obtained.

9.0 INSTRUMENT OR METHOD CALIBRATION

No calibration is necessary.

10.0 SAMPLE COLLECTION, HANDLING AND PRESERVATION

10.1 Preservatives shall not be added to samples before extraction. Extracts to be analyzed for metals shall be preserved following filtration with conc. HNO_3. Extracts to be analyzed for Semivolatile organic compounds (SVOCs) shall not be preserved following filtration. Extracts to be analyzed for Phenolics shall be preserved following filtration with conc. sulfuric acid.

10.2 Sample hold times are as follows (days):

	SVOCs	Mercury	Metals	Phenolics
From sample date to TCLP extraction:	14	28	180	28
From TCLP extraction to preparative extraction:	7	n/a	n/a	n/a
From preparative extraction to analysis:	40	28	180	28

See TCLP method 1311 sec. 6.0 and SPLP method 1312 sec. 6.0 for a detailed description of sample handling.

11.0 SAMPLE PREPARATION AND ANALYSIS

11.1 See appropriate SOP for sample analysis following extraction with this method.

11.2 DETERMINE SAMPLE % SOLIDS

11.2.1 For solid samples which contain no free liquids, proceed to sec. 11.3.2.

11.2.2 For samples which are liquid, contain free liquids, or are multi-phasic, filtration or liquid/solid separation is required as follows:

11.2.2.1 Preweigh a GFF filter and record the weight.

11.2.2.2 Preweigh a receiving beaker and record the weight.

11.2.2.3 Preweigh a transfer beaker and record the weight.

11.2.2.4 Assemble the pressure filtration device with the GFF filter, and place the receiving beaker beneath the outlet.

11.2.2.5 Weigh out a subsample of the waste (100g. minimum) and record the weight. An additional minimum 100g. will be needed for the extraction.

11.2.2.6 Transfer the waste to the filtration device and secure the top.

11.2.2.7 Re-weigh the empty transfer beaker and record the weight.

11.2.2.8 Slowly apply air pressure to the filtration device in 10 psi increments up to 50 psi. or until air passes through the filter. Hold at each increment for 2 minutes before proceeding to the next higher increment.

*Note: Some wastes, such as oily wastes and some paint wastes will contain material that appears to be a liquid. Even after applying pressure to 50 psi, this material may not filter. In this case, the material in the filter holder is defined as the solid phase and is carried through the extraction as a solid. Proceed to sec. 11.4.

11.2.2.9 Weigh the receiving beaker and record the weight.

11.2.2.10 The material in the filter holder is defined as the solid phase of the waste, and the material in the receiving beaker is defined as the liquid phase.

*Note: This subsample is not to be used for the extraction procedure.

11.2.2.11 Determine and record the weight of the liquid phase.

11.2.2.12 Determine the weight of the solid phase by subtracting the weight of the liquid phase from the total weight of the waste.

11.2.2.13 Calculate the % solids as follows:

$$\% \text{ solids} = \frac{\text{weight of solid phase}}{\text{total weight of waste}} \times 100$$

11.3 EVALUATION OF % SOLIDS

11.3.1 If the % solids is <0.5%, the filtrate is defined as the TCLP extract. Proceed to section 11.5 to prepare the extract for analysis.

11.3.2 If the % solids is significantly >0.5%, the solid portion must have a particle size which is smaller than 1cm. or have a surface area >3.1 cm^2 /g. (paper, cloth, etc.).If the above is not met, the material must be reduced to particles of the appropriate size by cutting, crushing, or grinding.

11.3.3 If the % solids is $\geq$0.5% or is very close, and it is noticed that the solid material is entrained in the filter, dry the filter at 80-120C until two successive weighings agree within +/- 1%. Determine the % dry solids. If the % dry solids is <0.5%, the filtrate is defined as the TCLP extract. Proceed to section 11.5 . If the % dry solids is >0.5%, see note below.

*Note: there must be a significant level of % solids such that a minimum of 25-50g of solids can be generated for the extraction. This minimum amount of solids will yield 500-1000 mL of extract.

11.4 DETERMINE THE APPROPRIATE EXTRACTION FLUID

11.4.1 TCLP

11.4.1.1 Transfer 5.0 g. of a representative subsample of the waste to a clean 250 mL beaker.

11.4.1.2 Add 96.5 mL D.I. H_2O and cover with a watchglass.

11.4.1.3 Stir vigorously for 5 min.

11.4.1.4 Measure and record the pH.

*Note: Accurate pH measurement is critical; calibrate the pH meter daily with fresh buffer solutions.

11.4.1.5 If the pH is <5.0, use extraction fluid #1.

11.4.1.6 If the pH is >5.0, add 3.5 mL of 1N HCl, cover with a watchglass, and heat on a hotplate to 50^0 C. Hold at 50^0 C for 10 min. Cool to room temperature, measure and record the pH. If the pH is < 5.0, use extraction fluid #1. If the pH is > 5.0, use extraction fluid #2.

11.4.2 SPLP

11.4.2.1 For samples collected from sites east of the Mississippi River, use extraction fluid #1.

11.4.2.2 For samples collected from sites west of the Mississippi River, use extraction fluid #2.

11.5 EXTRACTION PREPARATION

11.5.1 For samples that are 100% total solids

11.5.1.1 Perform particle size reduction if necessary.

11.5.1.2 Weigh **at least** 100 g of sample directly into the extraction vessel and record the weight. (For SVOC and phenolics analysis, a glass container must be used. For metals analysis, a glass or plastic container may be used.)

11.5.1.3 Record the number of the vessel being used and also check it off on the vessel usage sheet in front of the TCLP extraction log. A blank must be run on each container at a minimum of every 20th use.

11.5.1.4 Determine the amount of extraction fluid to add to the extraction container as follows:

Volume of extraction Fluid (mL) = 20 x mass of sample (g)

11.5.1.5 Measure and record the pH of the extraction fluid immediately prior to use.

11.5.1.6 Add the appropriate amount of extraction fluid.

11.5.1.7 Measure and record the initial pH.

11.5.1.8 Tightly cap the extraction container. The sample is now ready for extraction. Proceed to sec. 11.6.

11.5.2 For samples which are liquid, contain free liquids, or are multi-phasic, filtration or liquid/solid separation is required on a new portion of the waste:

11.5.2.1 Preweigh a GFF filter and record the weight. Filters should be prewashed with 1N HNO_3 followed by D.I. H_2O if metals are to be analyzed.

11.5.2.2 Preweigh a receiving beaker and record the weight.

11.5.2.3 Preweigh a transfer beaker and record the weight.

11.5.2.4 Assemble the pressure filtration device with the GFF filter, and place the receiving beaker underneath.

11.5.2.5 Weigh out at least 100 g of waste and record the weight. Ideally, enough sample should be filtered to allow for 100 g of solids to remain.

11.5.2.6 Transfer the waste to the filtration device and secure the top.

11.5.2.7 Re-weigh the empty transfer beaker and record the weight.

11.5.2.8 Slowly apply air pressure to the filtration device in 10 psi increments up to 50 psi. or until air passes through the filter. Hold at each increment for 2 minutes before proceeding to the next higher increment.

11.5.2.9 Weigh the receiving beaker and record the weight.

11.5.2.10 The material in the filter holder is defined as the solid phase of the waste, and the material in the receiving beaker is defined as the liquid phase.

11.5.2.11 Measure and record the weight and volume of the liquid phase.

11.5.2.12 Using a transfer pipette and a small beaker, add a few drops of the liquid phase to a small quantity of D.I. H_2O.

11.5.2.13 If the two phases are miscible, save the liquid phase for addition back to the filtered TCLP or SPLP extract. Store at 4^0 C in an appropriate container until the extraction is complete. If the two phases do not mix, the liquid phase will need to be analyzed separately and the results mathematically combined with the results from the extract.

Calculation:

$$\text{Final analyte concentration} = \frac{(V1)\,(C1) + (V2)\,(C2)}{V1 + V2}$$

V1 = volume of the first phase liquid
C1 = concentration of the first phase in mg/L
V2 = volume of the second phase liquid
C2 = concentration of the second phase in mg/L

11.5.2.14 Disassemble the filtration apparatus and carefully remove the filter and waste. Perform particle size reduction if necessary. Quantitatively transfer the filter and waste into the appropriate extraction container.

11.5.2.15 Determine the amount of extraction fluid to add to the extraction container as follows:

Volume of extraction Fluid (mL) = 20 x mass of sample (g)

11.5.2.16 Add the appropriate amount of extraction fluid.

11.5.2.17 Measure and record the initial pH.

11.5.2.18 Tightly cap the extraction container. The sample is now ready for extraction.

11.6 EXTRACTION

11.6.1 Secure the samples in the rotation apparatus which is located in the BOD incubator

11.6.2 Rotate for 18 +/- 2 hours. A room temperature of 23 +/- 2^0C must be maintained during the extraction period. This can be checked in the electronic temperature data logger.

11.6.3 Begin rotating. Record the time.

11.7 FILTRATION FOLLOWING EXTRACTION

11.7.1 Record the time and temperature at the end of the extraction period, as well as the minimum and maximum temperatures.
11.7.2 Following the rotation period, measure and record the pH of each bottle
11.7.3 Assemble the 2 L vacuum flask and ceramic filtration funnel.
11.7.4 Obtain the necessary sample containers and pH paper.
11.7.5 Place a 0.7 micron glass fiber filter (Environmental Express TCLP filters) in the filtration funnel. Record the lot # of filter that is used.
11.7.6 Filter the extract.
11.7.7 Transfer a suitable quantity of the filtrate to the appropriate sample containers:

Metals: 250 mL in polyethylene with HNO_3 preservative.
Semi-volatiles and phenolics: 1 L in amber glass jar.

11.7.8 For metals analysis, preserve the sample to pH <2 with conc. HNO_3.
11.7.9 For semi-volatile analysis, preservative is not added. Store at 4 °C.
11.7.10For phenolics analysis, preserve the sample to pH<2 with conc. H_2SO_4.

12.0 TROUBLESHOOTING AND MAINTENANCE

There is no troubleshooting or maintenance for this method.

13.0 DATA ACQUISITION, CALCULATION AND REDUCTION

See section 11.0 for any applicable calculations.

14.0 COMPUTER HARDWARE AND SOFTWARE

Computer with StarLIMS

15.0 DATA MANAGEMENT AND RECORD MANAGEMENT

15.1 Data is recorded in the TCLP/SPLP extraction log.
15.2 Prep data is entered into LIMS, and then the batch sheet is given to the metals prep analyst or the semi-volatile prep analyst.

16.0 QUALITY CONTROL/QUALITY ASSURANCE

16.1 A minimum of one blank, using the same extraction fluid as used for the samples, is required for every extraction batch. Also, a blank must be performed every 20th time an extractions is performed in a particular container to check for contamination.
16.2 A matrix spike is required for each waste type, with a minimum of one matrix spike per extraction batch. Matrix spikes are prepared at the time of digestion/analysis. Matrix spikes are to be added after filtration of the TCLP

extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

17.0 REFERENCES

17.1 *Test Methods for Evaluating Solid Waste*,. EPA-SW-846. September, 1994. Method 1311.
17.2 *Test Methods for Evaluating Solid Waste*,. EPA-SW-846. September, 1994. Method 1312.
17.3 CT Laboratories Quality Manual, current revision.
17.4 Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.
17.5 Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.
17.6 National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.
17.7 ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

18.0 TABLES AND CHARTS

FPR3-01
TCLP/SPLP/ASTM Extraction Plastic Vessel Usage Log

TCLP/SPLP/ASTM Extraction Vessel Usage Log

Plastic (Metals only)

Usage # >>>>
**Blank Analyzed

Vessel #	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	Date / Batch #
1P																					/
2P																					/
3P																					/
4P																					/
5P																					/
6P																					/
7P																					/
8P																					/
9P																					/
10P																					/
11P																					/
12P																					/
13P																					/
14P																					/
15P																					/
16P																					/
17P																					/
18P																					/
19P																					/
20P																					/
21P																					/
22P																					/
23P																					/
24P																					/
25P																					/
26P																					/
27P																					/
28P																					/
29P																					/
30P																					/

31P																				/

FPR3-02
Teflon Lined/Glass Extraction Vessel Usage Log

Teflon Lined (Metals / Wetchem / Semi Volatiles)

Usage # >>>>
Blank Analyzed

Vessel #	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	Date / Batch #
T1																					/
T2																					/

****After 20 uses the vessel must be used for a blank check** Reviewed By/Date:_______________

UNCONTROLLED WHEN PRINTED

FPR3-03
Glass Extraction Vessel Usage Log

Glass (Metals / Wetchem / Semi Volatiles)

Usage # >>>>
**Blank Analyzed

Vessel #	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	Date / Batch #
1G																					/
2G																					/
3G																					/
4G																					/
5G																					/
6G																					/
7G																					/
8G																					/
9G																					/
10G																					/
11G																					/
12G																					/
13G																					/
14G																					/
15G																					/
16G																					/
17G																					/
18G																					/

FPR3-04
TCLP/SPLP/ASTM Extraction Summary

TCLP/SPLP/ASTM Extraction Summary

Batch:________ Date:________ *Analyst (set up):________* *Analyst (take down):________*

Sample #	**Parameter**	**Test**			**Vessel / ZHE**	**Buffer Determination**			**Sample wt.**	****Free Liquids Present (Y/N)**	**Vol. Ext. Fluid**	**Initial pH**	**ZHE Initial Press**	**Time In**	**Date / Time Out**	**Ext. Time**	**End pH**	**ZHE End Press**	**Date / Time Filtered**	**Volume Filtered**
	(M / SV / V / WC)	TCLP	SPLP	ASTM	#	Initial pH	End pH	*Ext. Fluid	(g)		(mL)		(psi)			(hrs)		(psi)		(mL)
															/				/	
															/				/	
															/				/	
															/				/	
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															/				/	

**Extraction Fluid Used:___________* *Ext. Fluid ID #:___________* *Ext. Fluid pH:________* *Ext. Start Temp (°C):________*

Ext. End Temp(°C):__________ *(min:_______ / max:________)* *Filter Manufacturer:________________* *Filter Lot #:____________*

Balance:____________ *pH Meter:_____________* 1N HCL:___________ *Tumbler Unit #(s):__________*

Acceptable rotation rate: 30 ±2 rpm Verified Rotation rate: ________ rpm

M=Metals, SV=Semi Volatiles, V=Volatiles, WC=Wet Chemistry

FPR3-05
% Solid Calculation Sheet

% Solids Calculation Worksheet

	Tare weights (g)			*Transfer weights (g)*				*Separation weights (g)*				*% Solids Calculations*		
Sample #	**A Filter wt.**	**B Filtrate Vessel wt.**	**C Transfer Vessel wt.**	**D Sample + Transfer Vessel**	**E Sample wt. {D - C}**	**F Vessel wt. Post Transfer**	**G Residue wt. {F – C)**	**H Total Waste Amount {E – G}**	**I Filtrate Vessel + Filtrate**	**J Liquid Phase of Sample {I – B}**	**K Solid Phase of Sample {H – J}**	**L. % Solids (**) (K/H*100)**	**M Waste + Filter (Dry) wt. (g)**	**% Solids (Dry) ({M – A}/H)*100**

(**) If Solids = <0.5%, treat filtrate as TCLP extract, if free liquid present use % Solids Calculation Worksheet

Revision Number	**Description of Changes**	**Date**
04	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	03/26/2014
04	Document changed to incorporate preservation requirements for extracted samples.	02/27/2015
04	Reference to QSM 4.2 was added	03/03/2015
05	Added clarity to section 16.2 as to when TCLP matrix spikes are prepared.	02/18/2016

STANDARD OPERATING PROCEDURE
SV 004 Polychlorinated biphenyls (PCBs) as Aroclors by GC

Review Date: 03/31/2016

3/24/2015

Technical Review by: Date

Colleen Stuei

4/6/15

Approved by: Quality Assurance Date

Table of Contents

1.0 Identification of Test Method

This method is designed to follow procedures and QC requirements found in EPA SW-846 methods 3510, 3535, 3546, 3580 8000 and 8082 in order to determine quantities of semi-volatile organic compounds found in a variety of different sample matrices.

2.0 Applicable Matrix or Matrices

Semi-Volatile organic compounds are quantitated from a variety of matrices. This method is applicable to nearly all types of samples regardless of water content, including ground water, surface water, wastewater, waste oils, soils and sediments, as well as other matrices noted in SW-846 method 8082A.

3.0 Detection Limits

Method detection limits (MDLs) are determined annually and results vary from compound to compound. Water MDLs typically fall in the range of 0.020 to 0.20 ug/L. Soil MDLs are usually found to be between 2.0 to 16 ug/kg. Procedures for conducting MDL studies can be found in CT Laboratories Initial Method Performance and Reporting SOP Q23.

4.0 Scope and Application, including components to be analyzed

This method is used to quantify Semi-Volatile Polychlorinated Biphenyl's as aroclors in many types of solid waste matrices, soils, and groundwater. See Table 1.0 for typical target analyte list (TAL).

Table 1.0
Aroclor List

Aroclor Name
1016
1221
1232
1242
1248
1254
1260
1262
1268

5.0 Method Summary

5.1 This method describes procedures for isolating organic compounds through sample preparation from aqueous and soil matrices (reference methods SW846-3510, 3535, 3580 and 3546), concentration techniques that are suitable for preparing the extract, and the quantitative/ qualitative analysis for the determination of Polychlorinated Biphenyl's by method SW846-8082A.

5.2 A sample of a known volume or weight is extracted with solvent or diluted with solvent. Method applies for aqueous samples extracted by liquid-liquid separatory funnel (SW846-3510) or solid phase extraction (SW846-3535). Method applies for soil/sediment, and solid waste samples extracted by standard solvent extraction methods using microwave energy to produce elevated temperature and pressure conditions in a closed vessel containing extraction solvent (SW846-3546). This method includes the extraction for waste oil samples using SW846-3580.

5.3 The resultant extract is chemically dried and concentrated using a TurboVap system and/or Kuderna-Danish (K-D) apparatus in preparation for instrumental analysis.

5.4 Extracts for PCB analysis can be subjected to a variety of cleanup steps, depending on the nature of the matrix interferences and target analytes. Suggested cleanup methods include; sulfur cleanup (Method 3660) (attachment I), Florisil (Method 3620) (attachment II), and Gel-Permeation Chromatography (GPC) cleanup (Method 3640A) (GPC SOP Rev. 0). Alternative cleanup methods (refer to SW-846) are; alumina (Method 3610) and silica gel (Method 3630). After cleanup, the extract is analyzed by injecting a known aliquot into a gas chromatograph equipped with dual capillary columns and ECD detectors.

5.5 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

6.0 Definitions

6.1 Solid Phase Extraction (SPE): The separation ability of solid phase extraction is based on the preferential affinity of desired or undesired solutes in a liquid, mobile phase for a solid, stationary phase through which the sample is passed. Impurities in the sample are either washed away while the analyte of interest is retained on the stationary phase, or vice-versa. Analytes that are retained on the stationary phase can then be eluted from the solid phase extraction cartridge with the appropriate solvent.

6.2 For full definitions on all terms applicable to this method, see Section 25.6 in the Quality Assurance Manual (QAM).

6.3 For a list of common acronyms and abbreviations, see QAM front matter.

7.0 Interferences

7.1 Solvents, reagents, glassware, and other sample processing hardware can yield artifacts and /or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Refer to each method for specific guidance on quality control procedures.

7.2 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

7.3 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, can cause degradation of certain analytes.

7.4 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interference, further cleanup of the sample will be necessary.

7.5 Elemental sulfur (S_8) is readily extracted from soil samples. High concentrations of sulfur will cause chromatographic interferences in the determination of PCBs. Sulfur can be removed through cleanup procedures such as Method 3660.

8.0 Safety

8.1 Gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure must utilize appropriate laboratory safety systems.

8.2 The toxicity and carcinogenicity of the chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, so care must be taken to prevent undue or extensive exposure.

9.0 Equipment and Supplies

9.1 Gas Chromatograph- An analytical system complete with gas chromatograph (HP 6890) suitable for split-splitless injection and all required accessories including syringes, analytical columns, electron capture detectors, auto sampler, electronic pressure control, and EZ Chrom Elite (3.3.2 SP1) data acquiring system.

9.1.1 Carrier gas: Hydrogen at 37.0 psi at 12 mls/min
Mode: Ramped Flow
Injection Volume: 1 ul
Injector: 250 °C with Restek Septa 27142
Mode: Pulse Split
Pulse Pressure: 37.0 psi
Pulse Time: 0.20 min.
Split ratio: 1.0.
Total Flow: 77.7 ml/min

Initial Flow – 17.6 ml/min (hold 1.00 min)
Ramp – 2.50 ml/minute
Final – 19 ml/min (hold for 0.0 minutes)
Ramp – 3.2 ml/min
Final – 27 ml/min (hold for 2.0 minutes)

Detector (s): 300 °C
Mode: Constant Makeup
Make-up Gas: Nitrogen at 40 mL/min.
Range: 2 on A and 3 B channel

Oven: Initial – 185 °C (hold for 0.4 minute)
Ramp – 10.10 °C/minute
Final - 192 °C (hold for 0.0 minutes)
Ramp – 13 °C/minute
Final - 220 °C (hold for 0.0 minutes)

Ramp – 30°C/minute
Final - 300 °C (hold for 0.0 minutes)

Note: Instrument operating parameters are subject to change to improve overall chromatography (changes are noted in the Instrument Maintenance Log Book).

9.2 Analytical column pair:

9.2.1 30m x 0.32 mm ID bonded with 5% phenyl polysiloxane / 95% dimethylsiloxane, 0.25 um. (ZB-5, part # 7HM-G002-11 or equivalent).

9.2.2 30m x 0.32 mm ID bonded with 14% Cyanopropylphenyl/86% dimethylplysiloxane, 0.25 um (ZB-1701, part # 7HM-G006-11 or equivalent).

9.3 Water bath- heated and capable of accepting a Kuderna-Danish apparatus. (GlasCol 6 position heating mantel 100DRX30424 or equivalent)

9.4 CEM Microwave Accelerated Reaction System (MARS Xpress) extraction unit with Synergyprep software.

9.4.1 The CEM Mars extraction cycle:

Method 1
8-16 samples
Power: 100% at 800 watts
Ramp Time: 15 min
Pressure:0
Temp:110 C
Hold Time: 15 min

Method 2
17-48 samples
Power: 100% at 1600 watts
Ramp Time: 15 min
Pressure:0
Temp:110 C
Hold Time: 15 m

9.5 Organomation Nitrogen blow down concentrator (N-Evap).

9.6 TurboVap tubes: (Zymark 45817 or equivalent)

9.7 TurboVap; autoconcentrator

Temp: 45ºC
Pressure: 8-10psi

9.8 Analytical balance capable of accurately weighing to the nearest 0.01 gram (Fischer Scientific XD 2200 or equivalent).

9.9 Oven, muffle and drying.

9.10 Separatory funnel - 2000 mL Nalgene 4301, Teflon FEF lined with Teflon TFE stopcocks and Tefzel ETFE screw closures (MG Scientific #F847-2L or equivalent).

9.11 2-Platform shakers (Eberbach Model 6010 150 V shaker, or equivalent) fitted with trays to hold 6 Nalgene separatory funnels each.

9.12 Kuderna-Danish (K-D) apparatus:

- 9.12.1 Concentrator tube, 10.0 mL, graduated. (Fisher # K570051-1025).
- 9.12.2 Evaporation flask- 500 mL or 250 ml (Fisher # K570035-0250).
- 9.12.3 Synder column- Three-ball macro (Fisher # K503000-0121).
- 9.12.4 Teflon clamps to attach concentrator tube to evaporation.

9.13 Graduated cylinder (Class A TC) - 1000 mL. (Fisher 08-559G).

9.14 Beaker - 250 mL and 600 mL.

9.15 Vials - 2.0mL (National Scientific – C4000) 12mL (Kimble #60815-1965), and 60 mL screw cap vials with Teflon lined caps (C&G #LX64-A030-A01A) or equivalents.

9.16 Pasteur Pipets; 5 ¾" and 9" (VWR #14672-200 and -300).

9.17 Funnels – glass. (VWR #154-08 or equivalent).

9.18 Volumetric flask – (Class A TC) 10, 25, 50, and 100 mL.

9.19 Syringes 10 uL, 100 uL, 500 uL, and 1,000 uL. (Hamilton or equivalent).

9.20 Boiling chips, carborundum, approximately 10/40 mesh (methylene chloride rinsed) (Fisher #09-191-12) equivalent.

9.21 Filter- Glass Microfiber 12.5 cm (Ahlstrom, MG # F136-1250).

9.22 CEM-MARS Microwave extraction tubes with plugs and caps, 75mL (CEM #574127)

9.23 Spatulas- stainless steel. (VWR #57952-253 or equivalent)

9.24 pH indicator paper- pH 0-14. (Whatman #2613991) or equivalent. Stored in general lab storage area.

9.25 Aluminum foil.

9.26 Solid Phase Extraction Unit (SPEU). 3 station manifold assembly (47mm). (UCT: ECUCTVAC347) or equivalent.

10.0 Reagents and Materials

10.1 Deionized water (Milli-Q processed), analyte free or equivalent.

10.2 Sodium sulfate (granular, anhydrous 60/120 mesh, JT Baker # 3375-05) or equivalent. If sodium sulfate passes in house lot check, it can be used as is and stored in air tight glass jar. Otherwise condition sodium sulfate by heating to 400°C for 4 hours in a shallow glass tray loosely covered with foil and recheck for purity. Sodium sulfate will be stored in airtight glass jars and used within five years of opening or before the manufacturer's expiration date.

10.3 Silica sand- hydrocarbon free. Purify by heating to 400°C for 4 hours in a shallow glass tray, loosely covered with foil. Silica sand will be stored in airtight glass jars and used within five years of purifying.

10.4 Methylene chloride, pesticide grade, analyte free. Used within one year of opening or before the manufacturers expiration date. Or stored in large carboy tank provided by manufacturer and used within one year of opening or by the manufacturer's expiration date.

10.5 Acetone, pesticide grade, analyte free. Used within one year of opening or before the manufacturers expiration date.

10.6 Hexane, pesticide grade, analyte free. Used within one year of opening or before the manufacturers expiration date

10.7 Hydrogen (99.995% purity or greater).

10.8 Nitrogen (99.995% purity or greater)

10.9 Sulfuric Acid-1:1(v/v). ACS grade.Used within six months of mixing or before manufacturer's expiration date for any reagent used.

10.10 Sodium Hydroxide- 10N. ACS grade. Used within six months of mixing or before manufacturers expiration date for any reagent used.

10.11 Diatomaceous earth (Celite 545 – EMD #CX0574) or equivalent. Used within five years of opening or before the manufacturers expiration date.

11.0 Sample Collection, Preservation & Storage

11.1 Aqueous samples are collected in 1-L amber glass containers with Teflon lined lids. Aqueous samples are to be collected in duplicate. Solid samples are collected in 250-mL wide mouth glass containers with Teflon-lined lids. All samples are preserved by cooling to 4ºC. Soil samples must be extracted within 14 days and water samples must be extracted within 7 days from the date of collection.

11.2 Sample extracts are stored under refrigeration and analyzed within 40 days of extraction.

11.3 All soil samples are weighed on the top loading balance which is connected to a computer so that all weights can be automatically entered onto an Excel spread sheet. The spreadsheets are saved so the data can be transferred electronically to the LIMS system.

12.0 Quality Control

This SOP is designed to follow a variety of different projects and programs requirements. Table 3 is designed to illustrate the control steps and provisions required to adequately produce acceptable data.

13.0 Calibration and Standardization

13.1 Preparation of standards is documented in the Pest/PCB standards logbook. Each standard is labeled with a unique standard number to allow for tracking. Stock standards, once opened, expire in one year or sooner if routine QC indicates a problem, and are not to exceed the manufacturer's expiration date. Stock standards are saved in a capped vial in the original box in the freezer. Intermediate Stock Standards and Working Standards, which are subsequent dilutions made from the opened stock standard vial, expire in six months and are not to exceed the opened date of the stock standard or the manufacturer's expiration date.

13.2 Stock Standards -Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at -10ºC in a freezer reserved for standard solutions. Unopened standards shall have the manufactures suggested expiration date. Stock standards, once opened, expire within six months and are not to exceed the manufacturer's expiration date. The following list of stock standards are certified, commercially prepared standards, such as:

Aroclor 1016/1260: Restek Part # 32039 at 1000 ug/mL.
Aroclor 1016: Restek Part # 32006 at 1000 ug/ml.
Aroclor 1221: Restek Part # 32007 at 1000 ug/ml.
Aroclor 1232: Restek Part # 32008 at 1000 ug/ml.
Aroclor 1242: Restek Part # 32009 at 1000 ug/ml.
Aroclor 1248: Restek Part # 32010 at 1000 ug/ml.
Aroclor 1254: Restek Part # 32011 at 1000 ug/ml.
Aroclor 1260: Restek Part # 32012 at 1000 ug/ml.
Aroclor 1262: Restek Part # 32409 at 1000 ug/ml.
Aroclor 1268: Restek Part # 32410 at 1000 ug/ml.
Surrogate Mix: Restek Part # 32000 at 200 ug/ml.

13.3 Intermediate Working Stock Standards: The Aroclor intermediate standards are prepared at an optimum level from the preparation of the working stock standard. Each Aroclor with surrogate are prepared at an optimum level for the intermediate stock standard. The concentration of each Aroclor is 10 ug/ml with surrogate concentrations of 0.5 ug/ml in hexane. The following is a list of each Aroclor.

Aroclor 1016
Aroclor 1221
Aroclor 1232
Aroclor 1254
Aroclor 1260
Aroclor 1262

Aroclor 1242 Aroclor 1268

Table 2.0
Intermediate Working Stock Standard Concentration

Intermediate Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Aroclor(s)	1000	0.100	10.0	10.0
Surrogate	200	0.025	10.0	0.5

13.4 Calibration standards: PCB are to be determined as Aroclors, external calibration techniques will be used. An initial calibration is performed using a minimum of a five point calibration curve for Aroclors 1016/1260. These standard mixes will include many of the peaks represented in the other Aroclor mixtures. Standards for the other Aroclors are necessary for pattern recognition. These standards are also used to determine a single-point calibration factor (CF) for that specified Aroclor. The single-point calibration standard level is marked with an asterisk. In situations where only a few Aroclors are of interest for a specific project or program, the analyst will employ a minimum of a five point initial calibration of each of the Aroclors of interest. Alternative standard concentrations for the calibration curve can be prepared to meet client's or program's specified criteria.

Table 2.1
Calibration Points for the Aroclor(s) Linearity

Linearity Points	Spike Concentration (ug/ml) Aroclor(s)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml) Aroclor(s)	Final Concentration (ug/ml) Surrogate
1	10.0	0.030	10.0	0.03	0.0015
2	10.0	0.050	10.0	0.10	0.0025
3	10.0	0.200	10.0	0.20	0.010
4*	10.0	0.500	10.0	0.50	0.025
5	10.0	1.000	10.0	1.00	0.050
6	10.0	1.200	10.0	1.20	0.060

13.5 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacture from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of Aroclors 1016/1260 at a concentration of 0.5 ug/ml each time the initial calibration is performed. These are made by taking known aliquots of the ICV intermediate standard and diluting them to volume in hexane. If the analyst uses a project specified Aroclor in Section 7.4, then the analyst must verify that Aroclor with an ICV.

Table 2.2
ICV Working Standard

Working ICV Standard	Intermediate Standard Concentration	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)

	(ug/ml)			
Aroclor(s)	10.0	0.500	10.0	0.500
Surrogate	0.5	0.500	10.0	0.025

13.6 Continuing Calibration Verification Standard (CCV): A working standard solution for Aroclor 1016/1260 at a concentration of 0.5 ug/ml is used to check the validity of a calibration curve on a daily basis. If the analyst uses a project specified Aroclor in Section 7.4, then the analyst must use that Aroclor as the CCV. The variance of the Aroclor 1016/1260 mixture shall not be more than +/- 20% difference when compared to the mean calibration factor.

Table 2.3
CCV Working Standard

Working CCV Standard(s)	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Aroclor(s)	10.0	0.500	10.0	0.500
Surrogate	0.5	0.500	10.0	0.025

13.7 Surrogate standard: A commercially prepared certified solution of 2,4,5,6-Tetrachloro-m-xylene and Decachlorobiphenyl is diluted in acetone to produce a working surrogate solution of 0.50 ug/mL. 0.5 mL is added to each sample and QC. The surrogate concentration is normalized to 100% from the spiking solution in the initial calibration. This will provide percent recoveries that transfer directly to LIMS.

Table 2.4
Surrogate Spiking Solution

Surrogate Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Surrogate	200	0.250	100.0	0.500

13.8 Spiking standards (matrix and control samples): Prepare a spiking solution in acetone that contains Aroclor 1016/1260 at a concentration of 10.0 ug/mL for water and sediment /soil samples. 0.5 ml is added to quality control and matrix spike samples. If client request a specified Aroclor, a spiking solution will be altered to match the Aroclor of interest

Table 2.5
Aroclor Spiking Solution

Aroclor Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
Aroclor(s)	1000.0	0.500	50.0	10.00

13.9 The initial calibration for SW-846-8082 chromatographic method involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument across two dissimilar analytical columns. Each column must meet the initial calibration and continuing calibration criteria found in Table 3. In situations where only a few Aroclors are of interest for a specific project, the analyst will employ a multi-point initial calibration of each of the Aroclors of interest (e.g. five standards of Aroclor 1232 if this Aroclor is of concern and linear calibration models are employed) and not use the 1016/1260 mixture or the pattern recognition standards.

13.10 Quantitation is based upon indicator peaks in the Aroclor 1016/1260 mixture that are generally present in the Aroclors. The calibration factors (CF), and generated Aroclors 1016/1260 from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean calibration factor, the standard deviation (SD), and the relative standard deviation (RSD) for each Aroclor peak. When the Aroclors 1016/1260 mixture is used to demonstrate detector response, the calibration model (linear or non-linear models) chosen for this mixture must be applied to the other Aroclors for which only standards are analyzed. A minimum of five sets of calibration factors will be generated for the Aroclor 1016/1260 mixture, each set consisting of the calibration factors for each of the five (or more) peaks chosen for this mixture. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate the linearity.

Note: When selecting peaks for calibration, it is important to determine that common single-component pesticides such as DDT, DDD and DDE does not elute at the same retention time as the target peak.

13.11 The single point calibration standard will use a minimum of three to five peaks for each Aroclor. Choose peaks in the Aroclor standards that are at least 25% of the height of the largest Aroclor peak. For each Aroclor, the set of thee to five peaks are included and at least one peak that is unique to that Aroclor. The single standard for each of the other Aroclors will generate at least three calibration factors, one from each peak.

13.12 The identification of multi-peaked analytes such as PCBs as Aroclors can be performed by a combination of pattern recognition and retention time. Retention time windows are crucial to the identification of target compounds. Compare the retention time of each analyte in the calibration standard with the absolute retention time windows established in accordance with Method 8000 Section 11.6. Make three injections of target analyte standards over a course of 72-hour period. Each retention time must be to three decimal places. Calculate the mean and standard deviation of the target analytes. The width is 3x the standard deviation of the mean absolute retention time during the 72 hour period or 0.03 minutes, which ever is higher. If the standard deviation is 0.000, the laboratory will include more data points or use the default value of 0.01 minutes. Each analyte in each standard must fall within its respective retention time window. The retention time shall be set using the midpoint standard of the initial calibration curve or the value in the continuing calibration verification standard at the beginning of the analytical shift. If the analytes fall outside the established retention time window, the gas chromatographic system must either be adjusted so that a second analysis of the standard results in all analytes falling within their retention time windows, or a new initial calibration must be performed and new retention time windows established.

13.13 Prepare the Aroclor 1016/1260 calibration standards (in table 1.2) at a minimum of five different concentrations by adding known volumes of the intermediate stock standard to a volumetric flask and diluting to volume with hexane. Note: QSM requires that the LOQ or the lowest point in the curve, which ever is greater, be used for the reporting limit.

13.14 The lowest concentration calibration standard that is analyzed during an initial calibration curve, establishes the method's quantitation limit based on the final volume of the sample extract described in the preparative method or employed by the laboratory.

13.15 External standard calibration involves comparison of instrument response from the target compounds in the calibration standards. Sample peak areas are compared to peak areas of the standards. The ratio of the detector response to the amount of analyte in the calibration standard is defined as the calibration factor (CF). Aroclors are a multi-component standard. For the initial calibration curve, five peaks (or more) will be selected from each Aroclor 1016/1260 and calibrated using the average percent RSD of the peaks. If multi-point calibration is performed for individual Aroclors, use the calibration factors from those standards to evaluate the linearity.

13.16 Linear calibration using the average calibration or response factor. When calculating, both calibration factors and response factors are a measure of the slope of the calibration relationship and assume that the curve passed through the origin. If the relative standard deviation (RSD) is less than or equal to 20%, the use of the linear model is generally appropriate, and the calibration curve can be assumed to be linear and to pass through the origin

13.17 Linear calibration using a least squared regression. When the RSD of the calibration or response factors is greater than 20% over the calibration range, then linearity through the origin cannot be assumed. The approach is to employ a regression equation that does not pass through the origin. This approach can also be employed based on past experience of the instrument response.

13.18 Non-linear calibration. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable, and monotonic over the calibration range. The statistical considerations in using a non-linear calibration model require more data than the traditional linear approaches. A quadratic (second order) model requires the use of six initial calibration points.

13.19 Directly inject the prepared calibration standards into the gas chromatograph. An external calibration technique is employed. One of the external standards will be at a concentration near, but above the method detection limit. If the RSD $\leq$ 20%, the correlation of $r \geq 0.995$, or $r^2 \geq 0.990$ is obtained then the calibration is deemed acceptable.

13.20 Check the validity of the calibration by analyzing an ICV. The variance of any given compound shall not be more than +/- 20% difference. This standard must be run prior to sample analysis. If the percent drift or the percent difference criteria is not met, then the subsequent sample analysis for that analyte is not acceptable.

13.21 Continuous checking of the validity of the calibration by analyzing CCVs. The variance of any given compound shall not be more than +/- 20% difference. This standard must be ran at the beginning, every 20 samples or every 12-hour interval (which ever comes first), and at the end of the analysis run.

13.22 It is highly recommended to employ two standards (CCVs) at different concentrations to verify the calibration curve using non-linear calibration. One standard shall be near the quantitation limit or action limit. The choice of specific standards and concentrations is generally a method or project specific consideration.

13.23 When determining Polychlorinated Biphenyls by the external standard technique, calculate the calibration factor (CF) for each peak in each of the initial calibration standards using the equation below.

$$CF = \frac{\text{Peak Area (or Height) in the Standard}}{\text{Total Mass of the Standard Injected (in Nanograms)}}$$

13.24 The calibration factors from the initial calibration are used to evaluate the linearity of the initial calibration. This involves the calculation of the mean calibration factor, the standard deviation, and the relative standard deviation (RSD) for each multi-component peak.

$$\text{Mean } CF = \overline{CF} = \frac{\sum_{i=1}^{n} CF_i}{n}$$

$$SD = \sqrt{\left(\frac{\sum_{I=1}^{n} (CF_I - \overline{CF})^2}{\mathbf{n-1}}\right)}$$

$$RSD = \frac{SD}{\overline{CF}} \times 100$$

13.25 Linear Calibration: If the RSD of the calibration factor is greater than 20% over the calibration range, then linearity though the origin cannot be assumed. If this is the case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation in the form:

$$y = mx + b$$

y = instrument response (peak area or height)
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

13.26 The analyst will not force the line through the origin, but have the intercept calculated from the five data points. The use of linear regression will not be used as a rationale for reporting results below the calibration range.

13.26.1 A linear least squares regression attempts to construct a linear equation of the form: y= mx + b, by minimizing the differences between the observed results (y_i, the response calculated from the constructed equation). The regression equation is:

$$y_i' = ax_i + b$$

where:

a = Regression coefficient or the slope of the line
b = The y-intercept
y_i' = Predicted (or calculated) response for the ith calibration standard.
x_i = Mass of analyte in the ith calibration standard aliquot introduced into the instrument.

The sum of the squares of the differences is minimized to obtain a and b:

$$\sum_{i=1}^{n} (y_i - y_i')^2$$

where n is the total number of calibration points. The regression calculations attempt to minimize the sum of squares, hence the name “least squares regression.”

13.27 Weighting the sum of the squares of the differences can significantly improve the ability of the least squares regression to fit the linear model to the data. The general form of the sum of the squares of the differences containing the weighting factor is:

$$\sum_{i=1}^{n} w_i \ (y_i - y_i')^2$$

where:

w_i = Weighting factor for the calibration standard (w=1 for unweighted least squares regression.
y_i = Observed instrument response (area or height) for the ith calibration standard.
y_i' = Predicted (or calculated) response for the ith calibration standard.
n = Total number of calibration standards.

13.28 Least Squares Equation (LSQ) weighting method to be used for calculation of least squares regression fits, either 1/x or $1/x^2$, gives increased importance to smaller concentrations and areas. LSQ weight can be applied to linear, quadratic, and cubic fits only.

13.29 Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable, and monotonic over the calibration range. The model chosen shall have no more than four parameters, i.e., if the model is polynomial, it will be no more than third order as in the equation:

$$y = ax^3 + bx^2 + cx + d$$

13.30 The statistical considerations in developing a non-linear calibration model require more data that the more traditional linear approaches described above. Linear regression employ five calibration standards for the linear model, a quadratic model requires a minimum of six calibration standards.

13.31 The "goodness of fit" of the polynomial equation is evaluated by calculating the weighed coefficient of the determination (COD);

$$\mathrm{COD} = \frac{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2 - \left(\frac{n-1}{n-p} \right) \sum_{i=1}^{n} (y_{obs} - Y_i)^2}{\sum_{i=1}^{n} (y_{obs} - \bar{y})^2}$$

.

y_{obs} = Observed response (area) for each concentration from each calibration standard.

$\bar{y}$ = Mean observed response from the initial calibration
Y_I = Calculated response at each concentration from the initial calibrations.
n = Total number of calibration points (6 points for quadratic equation)
p = Number of adjustable parameters in the polynomial.

13.32 Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination will equal 1.0. In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99.

14.0 Procedure

14.1 Water Extraction (Method SW-846, 3510)

14.1.1 Pre-rinse all glassware to be used in the extraction with methylene chloride (Pesticide Grade).

14.1.2 Mark the meniscus on the bottle for later determination of sample volume. (Refer to section 11.1) From the glass sample collection bottle, quantitatively transfer sample into a 2 liter separatory funnel.

14.1.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare each by adding one liter of Milli-Q water to 2 liter separatory funnels.

14.1.4 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). In order of preference:

14.1.4.1 Select the sample where two full volume extra matrix was provided; use the extra volume supplied for a full volume MS and MSD.

14.1.4.2 Select a sample where one extra sample bottle was provided; quantitatively transfer half of the extra sample into a 2 liter separatory funnel and label MS. Transfer the other half of the sample into another 2 liter separatory funnel and label MSD.

14.1.4.3 Select a sample where no extra sample was provided and split it into three equal portions, one for the parent sample, one for the MS, and one for the MSD.

14.1.4.4For the last two situations concerning sacrificing a sample volume versus the inability to run a MS/MSD contact the project manager for proper procedure.

14.1.5 Add 0.5 mL of the surrogate standard mix to all samples by using a 0.5 ml syringe. In addition, add 0.5 mL of the proper spiking solution to the MS/MSD and LCS.

14.1.5.1 Surrogate and/or spike shall be added directly to the sample jar.

14.1.5.2 Reseal the sample jar and gently shake sample to mix.

14.1.5.3 If it is necessary to prepare split MS/MSD samples, the samples shall be quantitatively split using graduated cylinders and spiking shall occur directly into the graduated cylinder. Swirl gently to mix.

14.1.5.4 From the sample jar (or graduated cylinder), quantitatively transfer the sample into a 2-liter separatory funnel.

14.1.5.5 Check and adjust the extraction pH to between 5 and 9 with 10N sodium hydroxide and/or 1:1 sulfuric acid solutions.

14.1.5.6 Add 60 mL of methylene chloride to the sample jar (or graduated cylinder) and swirl to rinse sides of vessel. Transfer methylene chloride into the separatory funnel as well.

14.1.6 The sample is extracted by the automated shaker. Shake the samples vigorously for two minutes.

14.1.7 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. Decant the lower layer into a 250 ml beaker. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and will include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.

14.1.8 Repeat the extraction two more times using a fresh 60 mL portion of methylene chloride.

14.1.9 Determine the original sample volume by filling the sample bottle to the mark with water and transferring it to a "Class A TC" 1 liter graduated cylinder for measurement. Note all sample volumes on the extraction bench sheet (see Tables 5 and 6).

14.1.10 Record all lot numbers, prepping analyst, times and dates on prep bench sheet (see Tables 5 and 6).

14.1.11 Samples can potentially require cleanup prior to concentration/analysis. Refer to attachments I and II or the GPC SOP (Rev. 0) for sample cleanup procedure.

14.1.12 Refer to section 14.4 for sample concentration.

14.2 Soil Extraction (Method SW-846, 3546)

14.2.2 Microwave extraction

14.2.2.1 Preparing the extraction tubes for use: extraction tubes, caps and plugs are washed in the dishwasher, rinsed with methanol and baked in 110 C oven for 1 hour. After they have cooled, rinse the extraction cell (tubes, plugs and caps) with methylene chloride.

14.2.2.2 Decant and discard any water layer from sediment sample. Mix sample thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks.

14.2.2.3 Dry sediment/soil and dry waste samples amenable to grinding: Grind or otherwise reduce the particle size of the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make the sample more amenable to grinding. Dry samples as much as possible, as water will cause un-even heating of the tubes.

14.2.2.4 Gummy, fibrous, or oily materials not amenable to grinding, shall be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make the sample easier to mix. Wipe samples can be placed directly into the cell.

14.2.2.5 Weigh approximately 10.0 g of sample to the nearest 0.01 g in a 250-mL beaker and record the final weight on prep bench sheet (See FSV4-02). Add 2.5 g of diatomaceous earth to the sample. Mix well. The samples shall be a free flowing powder. If sample is not free flowing, add more diatomaceous earth and/or sodium sulfate until the sample has a dry texture. This powder is mixed so that it will allow the sample to pass through a 1 mm sieve.

14.2.2.6 Transfer the ground sample in a 75 mL extraction cell. There should be a minimum head space of 25%.

14.2.2.7 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding 10.0 g of sand and 2.5g of diatomaceous earth to a clean 250 ml beaker. Transfer sample to extraction cell.

14.2.2.8 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and transfer approximately 40 grams to a 250 ml beaker. Mix well. Weigh two individual 10.0 grams aliquots of sample. Add drying agent. Transfer each sample aliquot to separate extraction cells. If there is no sample available to perform a matrix spike/matrix spike duplicate, contact project management. Default QC is a laboratory control spike duplicate.

14.2.2.9 Add 0.5 mL of the surrogate standard mix to all samples by using a 0.5 mL syringe. In addition, add 0.5 mL PCB spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

14.2.2.10 Add 20 ml of (1:1) methylene chloride: acetone extraction solution to each tube. Insert tube plug and attach the cap to the extractor cell, making sure the cap is straight, screw on and torque with wrench**.** Shake each tube for 10 seconds to ensure the soil is mixed with the extraction solvent.

14.2.2.11 Place the extractor tube on the carousel in the appropriate slots for the number of tubes being used. Less than 16 use inside ring, greater than 16, use the outside ring then fill the inside ring. Schedule CEM Mars and begin the cycle. (Note: There must be a minimum of 8 samples, if less, use sand/solvent blanks to make up the shortage.)

14.2.2.12 Record all lot numbers, prepping analyst, times and dates on prep bench sheet (see FSV4-02).

14.2.2.13 Samples can potentially require cleanup prior to concentration/analysis. Refer to attachments I and II or GPC SOP (Rev. 0) for sample cleanup procedure.

14.2.2.14 Samples need to be shaken for 10 seconds to ensure sample residue is removed from tube wall prior to being poured out for concentration. Refer to section 14.4 for sample concentration.

14.3 Waste Dilution Extraction (SW846-3580)

14.3.1 (Refer to SOP FO-10 for subsampling guidance). Samples consisting of multiphase separations.

14.3.2 Pre-rinse "Class A TC" 10 ml volumetric with hexane.

14.3.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). If adequate sample is unavailable for a MS/MSD, contact project management for proper procedure.

14.3.4 Place the 10 ml volumetric on analytical balance (capable of accurately recording weight to the 0.001 g). Using a Pasteur pipet, transfer 1.0 g (to the nearest 0.001 g) to the volumetric. Record the weight.

14.3.5 Fill the volumetric half way with hexane.

14.3.6 Add 0.5 mL of the surrogate standard mix to all samples by using a 0.5 ml syringe. In addition, add 0.5 mL of the Aroclor 1016/1260 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

14.3.7 Bring samples up to volume with hexane. Transfer sample to a appropriately labeled 12 mL amber vial and cap. If samples are not analyzed immediately, store the sample extract in a refrigerator.

14.3.8 Add 2.0 grams of conditioned sodium sulfate to a 15ml amber vial with a Teflon cap. Transfer sample from the 10 ml volumetric flask to the 15ml vial.

14.3.9 Record all lot numbers, prepping analyst, times and dates on prep bench sheet (see FSV4-02).

14.3.10 Shake sample for two minutes.

14.3.11 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extracts through the glass wool and collect 5ml of the extract in a tube or vial.

14.3.12 No concentration step is need for this extraction. Sample can potentially require cleanup prior to analysis. Refer to attachments I and II or the GPC SOP (Rev. 0) for sample cleanup options.

14.4 Sample Concentration Methods 3510 and 3546

14.4.1 Place glass microfiber filter paper into a glass funnel. Fill the filter paper two-thirds with sodium sulfate. Rinse filter paper, sodium sulfate, funnel, and TurboVap tube with methylene chloride. (Kuderna-Danish (K-D) apparatus can be employed at this step)

14.4.2 Quantitatively pour the extract through the filter and funnel seated on a 200mL TurboVap concentrator tube (Kuderna-Danish (K-D) apparatus can be employed at this step). For microwave extraction, shake the tube for 30 seconds then pour both the extraction solution and the sample matrix from the microwave tube into the funnel and filter paper seated on the TurboVap apparatus, being careful to not allow the extract to splash out of the funnel as the sample matrix pours into it. Rinse the beaker, VOA vial or microwave tube three times with methylene chloride. Add these rinses through the filter and funnel into the concentrator tube.

14.4.3 Before placing the concentrator tube into the unit, make sure that the water is at the appropriate level and temperature. Place the concentrator tube into the TurboVap unit and then lower gently so the tubes rest firmly on the sensor tray. The proper nitrogen pressure condition is set between 6-8 and the temperature 45ºC. It is critical that the analyst watch the extract as it concentrates. THE EXTRACT CANNOT GO TO DRYNESS.

14.4.4 When the extract volume reaches approximately 4-6 mL, remove the concentrator tube from the bath and add 40 mLs of hexane. Use a pipet to aid in mixing of the solvents by drawing up the extract and expelling solvent, then gently stir the solvent to ensure that the methylene chloride and hexane are well mixed. Place the concentrator tube back into the unit. Concentrate the extract to approximately 5 mL. Remove the concentrator tube from the bath and allow the concentrator tube to cool completely.

14.4.5 With a disposable Pasteur pipet draw up the extract and rinse the sides of the concentrator tube. Repeat this rinsing several times. (Note: If the sample extract appears highly colored or has a strong petroleum type odor, a sulfuric acid clean-up will be required.) Transfer the extract from the concentrator tube using a Pasteur pipet to a 10mL TC volumetric flask. Rinse the concentrator tube with approximately 3mLs of hexane. Transfer the rinse to the volumetric flask. Bring sample up to a 10 mL volume with hexane. Transfer sample into a labeled 12mL amber vial. Highly contaminated samples may not adequately concentrate down to 10ml. Such samples shall be transferred to an appropriate volume volumetric flask and brought to volume with hexane. Record the final extract on the injection extraction bench sheet.

14.4.6 Record all lot numbers, prepping analyst, times and dates on prep bench sheet (see FSV4-02).

14.4.7 The sample extract is now ready for analysis. If samples are not analyzed immediately store the sample extract in a refrigerator (0-6 C).

14.4.8 Samples can potentially require cleanup prior to analysis. Refer to attachments I and II or the GPC SOP (Rev. 0) for sample cleanup options.

15.0 Data Analysis and Calculations

15.1 Before initial calibration or sample analysis, a priming standard will be injected at a suggested level 2x the highest linearity point. A clean hexane blank must be analyzed to determine baseline characteristics. If the instrument has been in use within 24 hours prior to analysis, this step can be omitted.

15.2 Verify calibration each twelve hour shift by injecting a Continuing Calibration Verification standard (CCV), containing Aroclors 1016/1260, prior to conducting any sample analysis. A CCV must be injected at the beginning of the sequence, and at intervals of no less than one per 20 samples injections or twelve hours (after every 10 samples is recommended to minimize the number of samples requiring re-injection when QC limits are exceeded), and at the end of each sequence. The variance of any given Aroclor 1016/1260 shall not be more than +/- 20% difference. If a different Aroclor is chosen for the continuing calibration verification, that CCV is subjected to the same QC criteria as the original CCV of Aroclors 1016/1260. The calibration verification process does not require analysis of the other Aroclor standards used for pattern recognition, but the analyst can include a standard for one or all of these Aroclors after the 1016/1260 CCV for pattern reference.

15.3 Before the initial sample batch analysis, inject the sample prep batch method blank to demonstrate that the instrument, as well as the extraction procedure, is free from contamination. All compounds must be below the method detection limit.

15.3.1 Samples can be directly injected after the successful analyses of the calibration curve, ICV, CCV and method blank. There can be up to 20 samples in an analytical batch. A matrix spike/matrix spike duplicate and laboratory control spike must be analyzed with every analytical batch. Recoveries will be within laboratory generated QC limits or client specified limits for all surrogate, matrix spike/matrix spike duplicate and laboratory control spike injections.

15.3.2 All positive detects in the associated sample must be confirmed by a second column. Positive detects are identified and quantitated using both detectors. The results between primary and secondary columns must be within +/- 40 % difference, (if greater than 40% the target analyte is qualified with a “P”). If there is no evidence of chromatographic problems, then it is appropriate to report the lower result (method 8000C). Specific projects or programs will require that the higher of the two columns results be reported.

15.3.2.1 QSM: Report from the primary column unless overlapping peaks are causing erroneously high results, then report the non-affected result. Qualify the data with a “Q” flag if target analyte is not confirmed by second column confirmation.

15.3.3 Each sample analysis must be bracketed with passing continuing calibration verification standards. If the CCV standard fails to meet QC criteria, all samples that were injected after the last standard which met QC criteria must be re-analyzed.

15.3.4 The data acquisition software generates a ug/ml concentrations which can then be applied to the final calculation to give sample concentrations. Samples with results exceeding calibration range are diluted accordingly in hexane and re-analyzed.

15.4 Using the slope (m) and the intercept (b) from this equation, the concentration of the sample (ug/ml) is calculated and appears on the report page following the chromatogram.

Water samples:

$$C_S = [(C_C)(V_E)(D)]/V_S$$

Soil samples:

$$C_S = [(C_c)(V_E)(D)]/[(W)(S)]$$

Wipe samples:

$$C = [(C_c)(V_E)(D)]$$

Where: C_S = Concentration of sample in ug/L for waters and mg/kg on a dry weight basis for soils

C_c = Concentration from the curve (ug/ml)

C = Concentration in total ug

V_E = Total volume of sample extract (after concentration) in ml

V_S= Volume of water sample in liters

D = Dilution factor if extract was diluted

W = Weight of wet soil sample in grams

S = Total percent solids, expressed as: percent total solids/100

16.0 Method Performance

See QAM Appendix 9.

17.0 Pollution Prevention

See QAM Appendix 9.

18.0 Data Assessment & Acceptance Criteria for QC Measures

18.1 If the initial analysis of a sample or a dilution of the sample has a concentration of a particular PCB that exceeds the calibration range, the sample must be re-analyzed at a dilution. If the initial sample exhibits interference such as sulfur, petroleum hydrocarbons, or other baseline interference, the sample must under go sample clean-up. Refer to Attachments I and II or the GPC SOP (Rev. 0) for sample clean-up procedures. The method blank and laboratory control sample must accompany the sample during the clean-up procedure.

18.1.1 Samples suspected of containing high levels of contamination or samples with known historical data may need to be diluted prior to analysis. Multiple dilutions may be needed to cover the entire working range of the current calibration.

18.2 The qualitative identification of PCBs as Aroclors determined by this method is based on retention time and pattern recognition. This method employs a dual ECD detector with two dissimilar columns. There is a potential for many non-target compounds present in samples to interfere with this analysis, therefore sample extracts can undergo cleanup procedures. The retention times are updated at the midpoint of each new calibration and they are continually updated with the daily continuing calibration verification standards (CCVs). A new method ID is provided for each new sequence with updated retention times or alteration to the operating initial calibration method. If an alteration occurs (other than daily retention time updates), project management and the QA officer will be notified. A description of the alteration will be addressed in the client notes or case narrative.

18.3 Reporting Quantitative Analysis:

18.3.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the standard retention times are compared and the sample retention times have been made, the sample data can be reported. Assessments of all spiked, calibration control samples and standards will also be scrutinized before reporting the data.

18.3.2 When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors, and percent solids adjustments are made. Once the final results have been verified, a checklist (FSV4-01.) is filled out and signed confirming that all the data has been thoroughly scrutinized. At this point, the data is turned over to another qualified analyst for final validation. The second analyst confirms the results, electronically marks them validated, and signs his or her portion of the checklist (the checklist is included in this SOP). Finally, the validated results are made available to the client services personnel in order for the data to be given to the client or appropriate agencies.

18.3.3 An electronic copy of the data is then filed and archived. The package includes; the sequence run log, checklist, a copy of the bench sheet, the LIMS run log, LIMS prep sheet, verification of calibration data, each sample’s chromatogram. All the data is initialed and dated by the analyst. Each sequence file header is labeled with the date of sequence.

19.0 Corrective Measures for Out-of-Control Data

See QAM Appendix 9.

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

See QAM Appendix 9.

21.0 Waste Management

See QAM Appendix 9.

22.0 Equipment / Instrument Maintenance, Computer Hardware & Software & Troubleshooting

See QAM Appendix 9.

23.0 References

23.1 USEPA, SW-846, 3rd Ed. Chapters 2&4, Method 8000C. March 2003.

23.2 USEPA, SW-846, “Polychlorinated Biphenyls(PCBs) by Gas Chromatography”, Method 8082A Revision 0. December 1996.

23.3 USEPA, SW-846, “Organic Extraction and Sample Preparation”, Method 3500B Revision 2. December 1996.

23.4 USEPA, SW-846, “Separatory Funnel Liquid-Liquid Extraction”, Method 3510C Revision 3. December 1996.

23.5 USEPA, SW-846 “Microwave Extraction”, Method 3546 Revision 0. February 2007.

23.6 USEPA, SW-846 “Gel-Permeation Cleanup” Method 3640A Revision 1. September 1994.

23.7 USEPA, SW-846 “Sulfur Cleanup”, Method 3660B Revision 2, December 1996.

23.8 USEPA, SW-846 “Florisil Cleanup”, Method 3620 Revision 3, February 2007.

23.9 USEPA, SW-846 “Solid Phase Extraction” Method 3535A Revision 1, February 2007.

23.10 USEPA, SW-846 “Waste Dilution” Method 3580A Revision 1, July 1992.

23.11 CT Laboratories Quality Manual, most recent version

23.12 Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Version 5.0, DoD QSM, March 2013 or most recent revision

23.13 National Environmental Laboratory Accreditation Conference (NELAC), 2003 *NELAC Standard Chapters 1 to 6*, EPA/600/R-04/003, June 5, 2003 or most recent version

23.14 ISO. 2005. *General requirements for the competence of testing and calibration laboratories*. ISO.IEC 17025:2005

ATTACHMENT I
SULFUR CLEANUP
METHOD 3660

1.0 Identification of Test Method

This method is designed to follow the procedures and QC requirements found in EPA SW-846 method 3660.

2.0 Applicable matrix or matrices

Semi-Volatile organic compounds are quantitated from a variety of matrices. This method is applicable to nearly all types of samples regardless of water content, including ground water, surface water, wastewater, soils and sediments, as well as other matrices noted in SW-846 method 8081B, 8082A.

3.0 Detection Limits

NA

4.0 Scope & Application, including components to be analyzed

This method is used to clean up Semi-Volatile Organochlorine Pesticides and Polychlorinated Biphenyls in many types of solid waste matrices, soils, and groundwater. See Tables 1.0 for typical target analyte list (TAL) in SOP 8081 and 8082.

5.0 Method Summary

This method covers the procedures used to eliminate sulfur interference from pesticide extracts. Elemental sulfur is indicated by the presence of white crystals in the sample extract, or, upon analysis, a broadband interference from the solvent front.

6.0 Definitions

NA

7.0 Interferences

7.1 Solvents, reagents, glassware, and other sample processing hardware can yield artifacts and /or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Refer to each method for specific guidance on quality control procedures.

7.2 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

7.3 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, can cause degradation of certain analytes.

8.0 Safety

8.1 Gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure must utilize appropriate laboratory safety systems.

8.2 The toxicity and carcinogenicity of the chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, so care must be taken to prevent undue or extensive exposure.

9.0 Equipment & Supplies

9.1 Mechanical shaker or mixer

9.2 Pasteur Pipettes; 5 ¾" and 9" (VWR #14672-200 and -300) or equivalent.

9.3 Disposable culture tubes – 25 mL with Teflon-lined screw caps.
(vials - Fischer 1496226F, Caps - Fisher 1495936A) or equivalent

9.4 Volumetric flask (Class A TC) 100 mL.

10.0 Reagents & Materials

10.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades shall be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2 De-ionized water (Milli-Q processed), analyte free or equivalent.

10.3 Acetone, pesticide grade, analyte free. Used within one year of opening or before the manufacturers expiration date.

10.4 Hexane, pesticide grade, analyte free. Used within one year of opening or before the manufacturers expiration date.

10.5 2-Propanol, pesticide grade. Used within one year of opening or before the manufacturers expiration date.

10.6 Tetrabutylammonium (TBA) sulfite reagent (Aldrich Part # 86847-1EA-F or equivalent). Used within five years of opening or before the manufacturers expiration date.

10.7 Tetrabutylammonium hydrogen sulfate (Aldrich Part # 155837-25G or equivalent). Used within five years of opening or before the manufacturers expiration date.

10.8 Sodium sulfite, (Aldrich Part# 239321-500G or equivalent). Used within five years of opening or before the manufacturers expiration date.

10.9 Nitric Acid, HNO_3, 1:1(v/v). Used within six months of mixing or before the manufacturers expiration date.

10.10 Copper powder, (UCT Part# ECCU01K or equivalent).

10.11 Tetrabutylammonium (TBA) – sulfite reagent (ACROS 42010-5000 or equivalent). Used within five years of opening or before the manufacturers expiration date.

10.12 Sodium sulfate (granular, anhydrous 60/120 mesh, JT Baker # 3375-05) or equivalent. If sodium sulfate passes in house lot check, it can be used as is and stored in air tight glass jar. Otherwise condition sodium sulfate by heating to 400^{o}C for 4 hours in a shallow glass tray loosely covered with foil and recheck for purity. Sodium sulfate will be stored in airtight glass jars and used within five years of opening or before the manufacturer's expiration date.

11.0 Sample Collection, Preservation & Storage

NA

12.0 Quality Control

This SOP is designed to follow a variety of different projects and programs requirements. Table 3 in SOP's 8081B and 8082A are designed to illustrate the control steps and provisions required to adequately producing acceptable data.

13.0 Calibration & Standardization

NA

14.0 Procedure

14.1 TBA Solution

14.1.1 Prepare reagent by dissolving 3.39g tetrabutylammoniuhydrogen sulfatete in 100ml organic-free water. To remove impurities, extract this solution three times with 20ml portions of hexane. Discard the hexane extracts. Add 25g sodium sulfite to the water solution. Store the resulting solution, which is saturated with sodium sulfite, in an amber bottle with a PTFE-lined screw cap. This solution can be stored at room temperature for at least one month.

14.1.2 Pipet 1.0 mL of pesticide extract into a 25 mL culture tube.

14.1.3 Add 1.0 mL TBA – sulfite reagent and 2 mL iso-propanol to the tube and cap.

14.1.4 Shake for at least 1 minute. If the sample is colorless or if the initial color is unchanged, and if crystals are observed, sufficient sodium sulfite is present. If the precipitated sodium sulfite disappears, add more crystalline sodium sulfite in approximately 100 mg portions until a solid residue remains after repeated shaking.

14.1.5 Add 5 mL distilled water and shake for at least 5 minutes. Allow the sample to stand for 5-10 minutes. Transfer the hexane layer to injection vials and/or storage vials.

14.1.6 Analyze the extracts by gas chromatography.

14.1.7 Verify that the TBA-sulfite is free from contamination by shaking out 1.0 mL of TBA – sulfite with 10 mL of hexane and analyze.

14.1.8 Process all quality control samples (e.g., spikes, blanks and duplicates) along with any samples.

14.1.9 Verify that recoveries of pesticides are greater that 80% by processing a standard (CCV) through the procedure.

14.2 Copper Powder

14.2.1 Remove oxides by treating the copper powder with dilute nitric acid, rinse with organic free reagent water to remove all traces of acid, rinse with acetone and dry under a stream of nitrogen.

14.2.2 Concentrate the sample to exactly 1.0ml or other known volume. Perform concentration using the techniques described in the appropriate 3500 series method.

14.2.3 If the sulfur concentration is such that crystallization occurs, centrifuge to settle the crystals, and carefully draw off the sample extract with a disposable pipet leaving the excess sulfur in the concentration tube. Transfer 1.0ml of the extract to the calibrated centrifuge tube.

14.2.4 Add approximately 2g of cleaned copper powder to the centrifuge tube. Vigorously mix the extract and the copper powder for at least 1 minute on the mechanical shaker. Allow phases to separate.

14.2.5 Separate the extract from the copper by drawing off the extract with a disposable pipet and transfer to a clean vial. The volume remaining still represents 1 ml of extract. This step is necessary to prevent further degradation of the pesticides.

15.0 Data Analysis & Calculations

NA

16.0 Method Performance

NA

17.0 Pollution Prevention

See QAM Appendix 9.

18.0 Data Assessment & Acceptance Criteria for QC Measures

NA

19.0 Corrective Measures for Out-of-Control Data

NA

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

NA

21.0 Waste Management

See QAM Appendix 9.

22.0 Equipment / Instrument Maintenance, Computer Hardware & Software & Troubleshooting

NA

23.0 References

USEPA, SW-846, 3rd Ed. Method 3660. December, 1996.

ATTACHMENT II
FLORISIL CARTRIDGE CLEANUP
METHOD 3620

1.0 Identification of the test method

This method is designed to follow the procedures and QC requirements found in EPA SW-846 method 3620.

2.0 Applicable matrix or matrices

This method is applicable to nearly all types of samples regardless of water content, including ground water, surface water, wastewater, soils and sediments, as well as other matrices noted in SW-846 method 8081B, 8082A.

3.0 Detection Limits

NA

4.0 Scope & Application, including components to be analyzed

This method is used to clean up Semi-Volatile Organochlorine Pesticides and Polychlorinated Biphenyls in many types of solid waste matrices, soils, and groundwater. See Tables 1.0 for typical target analyte list (TAL) in SOP 8081 and 8082.

5.0 Method Summary

5.1 This method describes procedures for Florisil cleanup of solvent extracts of environmental samples using solid-phase extraction cartridges. The cartridge cleanup protocol uses solid-phase extraction cartridges containing 40 µm particles of Florisil (60 D pores). Each cartridge is rinsed with solvent immediately before use. The sample extract is loaded onto the cartridge which is then eluted with suitable solvent(s). A vacuum manifold is required to obtain reproducible results. The eluate will be further concentrated prior to gas chromatographic analysis.

5.2 Florisil has been used for the cleanup of pesticide residues and other chlorinated hydrocarbons; the separation of nitrogen compounds from hydrocarbons; the separation of aromatic compounds from aliphatic-aromatic mixtures; and similar applications for use with fats, oils, and waxes. Additionally, Florisil is considered good for separations of steroids, esters, ketones, glycerides, alkaloids, and some carbohydrates. Florisil cleanup can be accomplished using glass chromatographic column packed with Florisil or using solid-phase extraction cartridges containing Florisil. This method includes procedures for cleanup of sample extracts containing the following analyte groups:

Chlorinated hydrocarbons
Organochlorine pesticides
Organophosphates
Organophosphorus pesticides
PCBs
Phthalate esters
Nitrosamines
Nitroaromatics
Haloethers
Aniline and aniline derivatives

6.0 Definitions

Florisil: a registered trade name of U. S. Silica Co., is a magnesium silicate with basic properties. It is used to separate analytes from interfering compounds prior to sample analysis by a chromatographic method.

7.0 Interferences

7.1 A reagent blank will be prepared and analyzed for the compounds of interest prior to the use of this method. The level of interferences must be below the method detection limit before this method is performed on actual samples. The procedures for reagent purification outlined here will be considered to be the minimum requirements for use of this method. More extensive procedures shall be necessary to achieve lower levels of interferences for some analytes. During the evaluation of the cartridge clean-up procedure, phthalate esters were detected in the Florisil cartridge method blanks at concentrations up to 400 ng per cartridge. Therefore, complete removal of the phthalates esters from Florisil cartridges will not be possible.

7.2 Solvents, reagents, glassware, and other sample processing hardware can yield artifacts and/or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Refer to each method for specific guidance on quality control procedures.

7.3 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

7.4 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, can cause degradation of certain analytes.

8.0 Safety

8.1 Gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure must utilize appropriate laboratory safety systems.

8.2 The toxicity and carcinogenicity of the chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, so care must be taken to prevent undue or extensive exposure.

9.0 Equipment & Supplies

9.1 Vacum manifold - VacElute Manifold SPS-24 (Analytichem International), Visiprep (Supelco, Inc.) or equivalent, consisting of glass vacuum basin, collection rack, funnel, collection vials, replaceable stainless steel delivery tips, built-in vacuum bleed valve and gauge. The system is connected to a vacuum pump or water aspirator through a vacuum trap made from a 500-mL sidearm flask fitted with a one-hole stopper and glass tubing. The manifold is required for use of the cartridge cleanup protocol.

9.2 Organomation Nitrogen blow down concentrator (N-Evap).

9.3 Pasteur Pipettes; 5 ¾” and 9” (VWR #14672-200 and -300) or equivalent.

9.4 Concentrator tube, 10.0 mL, graduated (Fisher # K570051-1025 or equivalent).

10.0 Reagents & Materials

10.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades shall be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

10.2 Acetone, pesticide grade, analyte free. Used within one year of opening or before the manufacturers expiration date.

10.3 Hexane, pesticide grade, analyte free. Used within one year of opening or before the manufacturers expiration date.

10.4 Methylene chloride, pesticide grade, analyte free. Used within one year of opening or before the manufacturers expiration date. Or stored in large carboy tank provided by manufacturer and used within one year of opening or before the manufacturer’s expiration date.

10.5 Florisil extraction cartridges, 1000MG/6ML (UCT EUFLSA1M6) or equivalent.

10.6 Florisil cartridge pesticide check solution - Prepare a solution containing the following analytes in hexane:

BHCs
Heptachlor
BHC
Endosulfan I
Dieldrin
Endrin
4,4'-DDD
4,4'-DDT
Methoxychlor
Tetrachloro-m-xylene
Decachlorobiphenyl

11.0 Sample Collection, Preservation & Storage

NA

12.0 Quality Control

12.1 This SOP is designed to follow a variety of different projects and programs requirements. Table 3 in SOP's 8081B and 8082A are designed to illustrate the control steps and provisions required to adequately producing acceptable data.

12.2 The analyst must demonstrate that the compounds of interest are being quantitatively recovered before applying this method to actual samples. A recovery check must be performed using standards of the target analytes at known concentrations. The efficiency of each lot of the solid-phase extraction cartridges must be verified. Only lots of cartridges from which the spiked analytes are quantitatively recovered are be used to process the samples. A check shall also be performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

12.3 Organochlorine pesticides - To check each new lot of Florisil cartridges before use, perform the following in duplicate. Combine 0.5 mL of the 2,4,5-trichlorophenol solution, 1.0 mL of the pesticide solution, and 0.5 mL of hexane in a vial. Condition the cartridge and then perform the cartridge cleanup Elute the cartridge with 9 mL of acetone/hexane (10/90, v/v) only. Reduce the volume to 1.0 mL and analyze. The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 110 %, if the recovery of trichlorophenol is less than 5 %, and if no peaks interfering with the target analytes are detected.

13.0 Calibration & Standardization

NA

14.0 Procedure

14.1 Whenever Florisil is used to fractionate groups of target compounds (rather than to simply remove potential interference) it is critical that the specific fractionation scheme be validated using spiked solutions or spiked sample extracts that contain most or all of the analytes of interest. This is particularly important when the Florisil cartridge techniques are employed, as the differences between the various cartridge formats and manufacturers can affect the fractionation patterns. In addition, it will be useful to archive any fractions not originally intended for analysis in the event that the fractionation scheme chosen does not yield the intended results. Once the determinative analysis has been performed and demonstrates that the fractionation has been successful, such archived fractions can be disposed of in an appropriate manner. However, if the fractionation did not perform as intended, the analytes of interest contained in the archived fractions will be able to be analyzed or combined with the other fraction(s) for reanalysis.

14.2 Following Florisil cleanup, extracts will require further concentration and/or solvent exchange.

14.3 Cartridge Evaluation

14.3.1 The efficiency of each lot of the solid-phase extraction cartridges must be verified. Only lots of cartridges from which the spiked analytes are quantitatively recovered are be used to process the samples. A check shall also be performed at least once on each individual lot of cartridges and at least once for every 300 cartridges of a particular lot, whichever frequency is greater.

14.3.2 To check each new lot of Florisil cartridges before use, perform the following in duplicate.

14.3.3 Combine 0.5 ml of 2,4,5-Trichlorophenol solution in Section 5.5 and 0.5 ml of pesticide spiking solution in Section 5.6 in a 2ml autosampler vial.

14.3.4 Arrange the cartridges on the manifold in the closed-valve position.

14.3.5 Turn on the vacuum pump and set the vacuum to 10 in (254 mm) of Hg. Do not exceed the manufacturer's recommendation for manifold vacuum. Flow rates can be controlled by opening and closing cartridge valves.

14.3.6 Condition the cartridges by adding 4 mL of hexane to each cartridge. Slowly open the cartridge valves to allow hexane to pass through the sorbent beds to the lower frits. Allow a few drops per cartridge to pass through the manifold to remove all air bubbles. Close the valves and allow the solvent to soak the entire sorbent bed for 5 minutes. Do not turn off the vacuum.

14.3.7 Slowly open cartridge valves to allow the hexane to pass through the cartridges.

14.3.8 Close the cartridge valves when there is still at least 1 mm of solvent above the sorbent bed.

14.3.9 Do not allow cartridges to become dry. If cartridges go dry, repeat the conditioning step.

14.3.10 Add the spiking solution in 6.2.3 to the Florisil cartridge.

14.3.11 Elute the cartridge with 9 ml of acetone/hexane (10/90, v/v). Collect the extraction in a 15 ml test tube.

14.3.12 Transfer extract to a concentrating thimble. Using the nitrogen blow down apparatus, concentration the extract down to 1ml and analyze by Method 8081A.

14.3.13 The lot of Florisil cartridges is acceptable if all pesticides are recovered at 80 to 110%, if the trichlorophenol is less than 5%, and if no peaks interfering with the target analytes are detected.

14.4 Handling sample extracts

14.4.1 Most sample extracts will have to be concentrated to a smaller volume prior to the use of Florisil cleanup. The extract volume is a function of the analytical sensitivity necessary to meet the project objectives. The extract volume will also affect the ability of the Florisil to separate target analytes from potential interferences. Applying large extract volumes to the cartridges will cause poor results. Consult the appropriate extraction and determinative methods for the details on final extract volumes, extract concentration techniques, and solvent exchange procedures.

14.4.2 Reduce the sample extract volume to 10.0 mL prior to cleanup. The extract solvent will be hexane for these analytes. In most cases, given the sensitivity of the determinative methods, only 1 mL of the 10.0 mL extract needs to be subjected to the Florisil cleanup procedure. The remaining 9 mL will be archived for later use, if needed.

14.4.3 Place Florisil cartridges on manifold. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Condition the cartridges by adding 4 ml of hexane. Slowly open the cartridge valve and collect the eluate into the collection vial. Allow a few drops of hexane to pass through the cartridge to remove any air bubbles. Close valve and allow solvent to soak the entire sorbent bed for five minutes. Do not turn off vacuum.

14.4.4 Slowly, open the valve to allow the hexane to pass through the cartridge. Close valve when about 1 mm of hexane is remaining above the sorbent bed.

14.4.5 Add 1.0 ml of extract to the Florisil cartridge. Allow the extract to pass through the cartridge at a rate about 2 ml per minute.

14.4.6 Before extract goes below the surface of the sorbent bed, add 9 ml of hexane/acetone (90/10, v/v) to the cartridge.

14.4.7 After extract has been collected, concentrate sample down to 1.0 ml. Refer to Section 9.5 for concentration techniques.

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14.4.8 The following procedures are used to separate the organochlorine pesticides from PCBs:

14.4.8.1 Add 3 mL of hexane to the cartridge. Turn on the vacuum pump and adjust the pump pressure to 10 inches (254 mm) of Hg. Allow the solvent to soak the sorbent bed for 1 minute or less. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 1 and it will contain the PCBs and a few of the organochlorine pesticides.

14.4.8.2 Close the cartridge valve, replace the collection vial, and add 5 mL of methylene chloride/hexane (26/74, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 2 and it will contain most of the pesticides.

14.4.8.3 Close the cartridge valve, replace collection vials, and add 5 mL of acetone/hexane (10/90, v/v) to the cartridge. Slowly open the cartridge valve and collect the eluate into the collection vial. This is Fraction 3 and it will contain the remaining pesticides.

14.4.8.4 As needed, perform a solvent exchange and adjust the final volume of the eluant to 1 ml.

15.0 Data Analysis & Calculations

NA

16.0 Method Performance

NA

17.0 Pollution Prevention

See QAM Appendix 9.

18.0 Data Assessment & Acceptance Criteria for QC Measures

NA

19.0 Corrective Measures for Out-of-Control Data

NA

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

NA

21.0 Waste Management

See QAM Appendix 9.

22.0 Equipment / Instrument Maintenance, Computer Hardware & Software & Troubleshooting

NA

23.0 References

USEPA, SW-846, 3rd Ed. Method 3620. December, 1996.

CT Laboratories Quality Manual, current revision.
Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.

National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.

ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

Table 3.0
Summary of Method Quality Objectives for Method 8082- PCBs by aroclors

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
Initial Calibration Aroclors 1016/1260 or client specified aroclor(s)	Initial Calibration prior to sample analysis.	1. RSD for each analyte $\leq$ 20% 2. Linear – least squares regression r $\geq$ 0.995. 3. Non-linear regression $r^2 \geq 0.99$. (6 points shall be used for second order)	For aroclor analysis, a mixture of aroclors 1016/1260 is normally used to establish detector calibration linearity, unless project specific arolclor(s) is required. Linearity must fit one of acceptance criteria. Correct the problem and repeat ICAL. Single level CFs for the remaining arolcors must be established with each initial calibration.
Initial Calibration Verification (ICV)	Immediately following the ICAL	Difference $\leq$20% From a second source (different lot or manufacturer)	If ICV falls outside QC criteria, reanalysis must take place. If ICV still fails, it will be necessary to correct to problem, or it will be appropriate to repeat the initial calibration curve or to qualify the analyte with “Z”. QSM: No samples will be analyzed until the problem has been corrected.
Continuing Calibration Verification (CCV)	Every twelve hours or every twenty samples (which one comes first) QSM: Every ten samples, Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	Difference $\leq$20%	If CCV falls outside QC criteria, reanalysis must take place. If CCV still fails, it will be necessary to correct to problem, or it will be appropriate to repeat the initial calibration curve or to qualify the analyte with “Z”. Exception to the above, if the acceptance limits are exceeded high and the analyte is not detected in the sample the verification standard has passed (analyte would have been detected if present). QSM: No samples will be analyzed until the problem has been corrected. Flagging is only appropriate in cases where the sample cannot be reanalyzed.
Retention Time Window (RTW)	Retention Times will be set using the midpoint standard in the ICAL or the RT in the CCV run at the beginning of each analytical shift.	Shift less than ± 3 times the absolute Standard Deviation from the 72 hour RTW study, with a minimum width of 0.03 minutes	

Table 3.0
Summary of Method Quality Objectives for Method 8082- PCBs by aroclors

Quality Control Element	Frequency of Implementation	Acceptance Criteria	Corrective Action
MRL Level Verification Check standard at Reporting Limit. (LCG only)	Beginning and End of 12 hr. sequence or program specified.	70-130% or project specific/client limits	Note failures in case narrative. If MDL check was run at the end and acceptable do not reject data.
Method Blank (MB)	1 per sample batch ≤ 20 samples of the same matrix	Analytes must not he higher than the highest of the following: 1/2 MRL, or 5% of the regulatory limit, or 5% of the associated sample concentration. QSM = ½ MRL	If sample is available and within holding times, sample associated with method blank needs to be reprepped. If no sample is available, qualify the data with a "B" to all associated positives when less than 5X blank concentration. QSM: Apply "B" to all results for the specific analytes in all samples in the associated preparatory batch.
Laboratory Control Sample (LCS)	1 per sample batch ≤ 20 samples of the same matrix	1.0 Client specified limits 2.0 QSM – use LCS criteria 3.0 In-house limits	If LCS fails percent recoveries, correct problem and re-analyze the LCS. If LCS recoveries are still outside QC control limits, and if there is sample remaining, the sample batch must be reprepped. If there is not sample available for reanalysis, qualify the failing analytes with a "Q".
Matrix Spike (MS)	1 per sample batch ≤ 20 samples of the same matrix	1.0 Client specified limits 2.0 QSM – use LCS criteria 3.0 In-house limits	No action is taken based on MS recovery alone, use of professional judgement. For recoveries outside QC criteria, qualify out lying analyte(s) in the parent sample with "M".
Matrix Spike Duplicate (MSD)		1.0 Client specified limits 2.0 QSM RPD < 30% 3.0 In-house limits	No action is taken based on MSD results alone, use of professional judgement. If RPD is outside QC criteria, then qualify the out lying analyte(s) in the parent sample with "Y".

Table 3.0
Summary of Method Quality Objectives for Method 8082- PCBs by aroclors

Quality Control Element	**Frequency of Implementation**	**Acceptance Criteria**	**Corrective Action**
Surrogates	Every sample and QC	1.0 Client specified limits 2.0 QSM – use LCS criteria 3.0 In-house limits	Rerun sample. If no apparent matrix interference noticed re-extract sample. If no sample is available, qualify the surrogate with "S". QSM: For QC and field samples; correct problem, reprep, and re-analyze all failed samples or failed surrogates in the associated batch, if sufficient sample material is available.
Target Analyte Confirmation	Whenever a positive is detected, check agreement between primary and secondary columns.	RPD $\leq$ 40% QSM: Discuss in case narrative about qualified data.	Report from primary column unless it can be scientifically excluded **If present and RPD >40%** Flag with "P" qualifier and discuss in case narrative if appropriate..

FSV4-01

Analytical Run # **8082 PCB Analysis Data Review Checklist (Example)**

Sequence Date	*Analyst / Data Interpreter*	*Independent Reviewer*	*Date of Review*	*Approved*
				Yes or No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the
Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	**Acceptance Criteria**	**Analyst Review**		**Independent Review**		**Comments:** (indicate reference to an attachment if necessary)
		Yes	**No**	**Yes**	**No**	
1. INITIAL CALIBRATION (ICAL)						
a. Was the PCB initial calibration (Aroclor 1016/1260) performed using a minimum of five varying standard concentration levels on two dissimilar columns?	Lowest standard at or near MRL					
b. Is the variation between calibration response factors for all concentration levels <20% RSD, is r^2 >.990, or r > 0.995 for the regression line?	RSD <20%, r^2 >.990, r>0.995					
c. Was each ICAL uniquely identified (i.e. Standard Number)?						
d. Were there Calibration Factors (CF) established for the remaining Aroclors?						
d. Was an initial calibration blank (ICB) analyzed?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there second source ICVs for all Aroclors analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the recoveries for the ICVs within program limits?	%Recovery					
c. Was the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs (Aroclor 1016/1260) analyzed at the beginning of the sequence, after every 12 hours or every 20 samples (which ever comes first) and at the end of the analytical run? QSM = every ten sample injections.						
b. Were the recoveries for the CCVs within program limits?	%Recovery					
c. Were confirmed Aroclor detects processed using the appropriate Aroclor method?						
d. Was each CCV uniquely identified (i.e. Standard Number)?						

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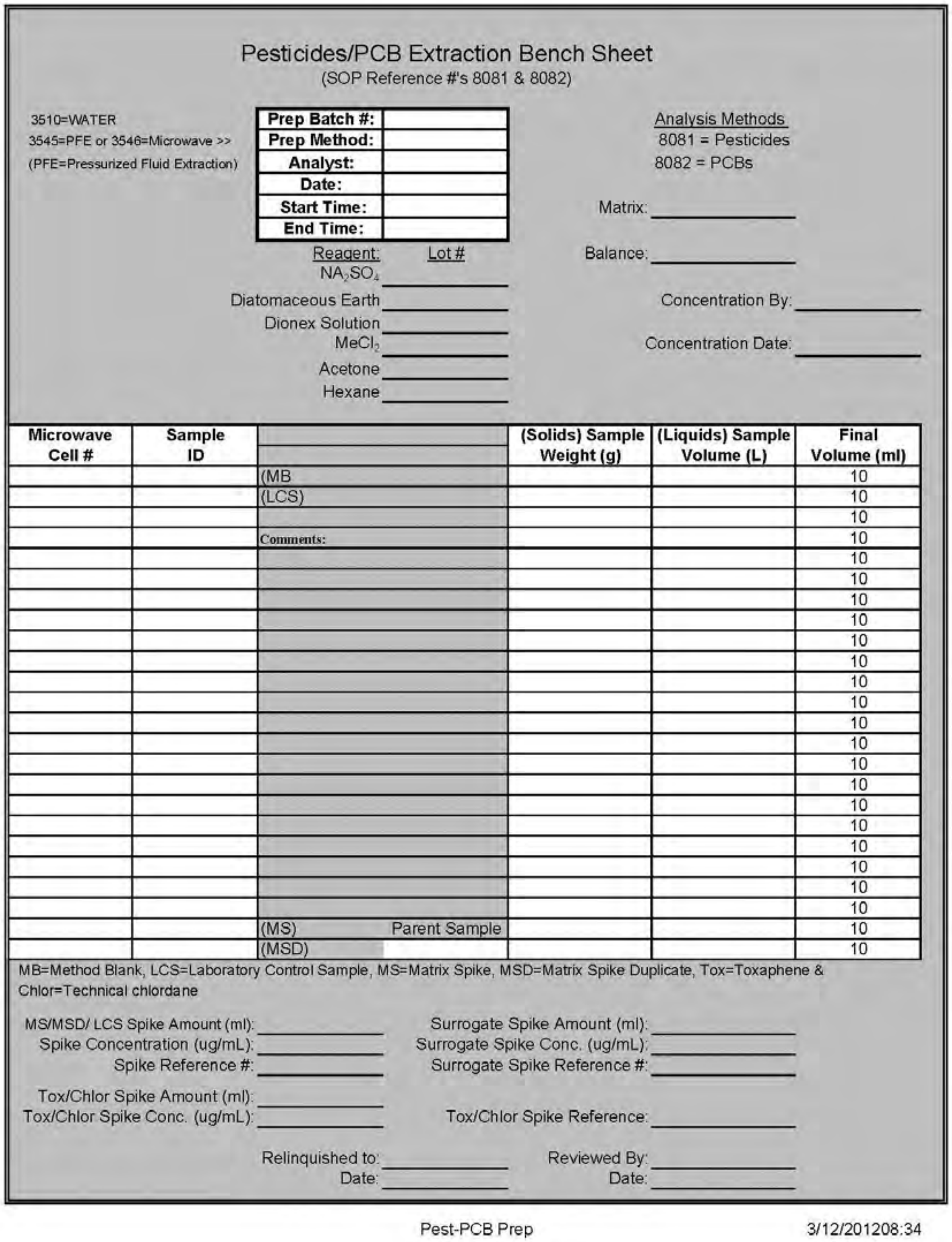
8082 PCB Analysis Data Review Checklist Continued

Requirement:	**Acceptance Criteria**	**Analyst Review**		**Independent Review**		**Comments:** (indicate reference to an attachment if necessary)
		Yes	**No**	**Yes**	**No**	
4. BLANKS						
a. Was the method blank (MB) analyzed prior to the analysis of samples?						
b. Was the MB result less than ½ the reporting limit (RL) or 5% of the sample amount?						
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch < 20 samples					
5. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits or client specified limits					
6. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits or client specified limits					
7. MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike sample analyzed at a frequency one per Prep Batch?	Batch < 20 samples					
b. Were MSD recoveries within the acceptance criteria?	In-house limits or client specified limits					
c. Is the relative percent difference (RPD) between a matrix spike (MS) and its' duplicate (MSD) within the acceptance criteria?	In-house limits or client specified limits					

8082 PCB Analysis Data Review Checklist Continued

Requirement:	**Acceptance Criteria**	**Analyst Review**		**Independent Review**		**Comments:** (indicate reference to an attachment if necessary)
		Yes	**No**	**Yes**	**No**	
8. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. acid treatment, florisil, and sulfur treatment)	Acid, Florisil, GPC, Sulfur Treatments					
f. Was there a hexane injection performed prior to sample analysis?						
g. Was there a priming standard injected prior to sample analysis?						
9. RECORDS AND REPORTING						
a. Is the Analytical Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, analytical data, and method transfer to PDF format?						
b. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier in LIMS and, if needed, a NCR completed?						
c. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
d. Is the ICAL method referenced on the Raw Data?						

FSV4-02 PCB Extraction Bench Sheet (Example)



Pesticides/PCB Extraction Bench Sheet

(SOP Reference #'s 8081 & 8082)

3510=WATER
3545=PFE or 3546=Microwave >>
(PFE=Pressurized Fluid Extraction)

Prep Batch #:	
Prep Method:	
Analyst:	
Date:	
Start Time:	
End Time:	

Analysis Methods
8081 = Pesticides
8082 = PCBs

Matrix: ______________

Balance: ______________

Reagent:	Lot #
NA$_2$SO$_4$	
Diatomaceous Earth	
Dionex Solution	
MeCl$_2$	
Acetone	
Hexane	

Concentration By: ______________

Concentration Date: ______________

Microwave Cell #	Sample ID		(Solids) Sample Weight (g)	(Liquids) Sample Volume (L)	Final Volume (ml)
		(MB			10
		(LCS)			10
					10
		Comments:			10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
					10
		(MS) Parent Sample			10
		(MSD)			10

MB=Method Blank, LCS=Laboratory Control Sample, MS=Matrix Spike, MSD=Matrix Spike Duplicate, Tox=Toxaphene & Chlor=Technical chlordane

MS/MSD/ LCS Spike Amount (ml): ______________
Spike Concentration (ug/mL): ______________
Spike Reference #: ______________

Surrogate Spike Amount (ml): ______________
Surrogate Spike Conc. (ug/mL): ______________
Surrogate Spike Reference #: ______________

Tox/Chlor Spike Amount (ml): ______________
Tox/Chlor Spike Conc. (ug/mL): ______________

Tox/Chlor Spike Reference: ______________

Relinquished to: ______________
Date: ______________

Reviewed By: ______________
Date: ______________

Pest-PCB Prep 3/12/201208:34

Revision Number	**Description of Changes**	**Date**

11	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	08/19/2013

CT LABORATORIES
delivering more than data from your environmental analyses

STANDARD OPERATING PROCEDURE
SV 006 Semi-volatile Organic Compounds by 8270D

Review Date: 03/31/2016

3/24/2015

Technical Review by: Date

Colleen Stei

4/6/15

Approved by: Quality Assurance Date

Table of Contents

1.0 Identification of the Test Method

This method is designed to follow procedures and QC requirements found in EPA SW-846 methods 3510C, 3545A, 3546, 3580A, 8000 and 8270D in order to determine quantities of semivolatile organic compounds in a variety of different sample matrices.

2.0 Applicable Matrix or Matrices

Semivolatile organic compounds are quantitated from a variety of matrices. This method is applicable to nearly all types of samples; including ground water, surface water, wastewater, soils, sediments, and tissue as well as other matrices noted in SW-846 method 8270D.

3.0 Detection Limits

Method detection limits (MDLs) are determined annually and results vary from compound to compound. Water MDLs are typically between 0.10 and 10 ug/L. Soil MDLs are typically between 0.01 and 0.50 mg/kg. Water MDLs for PAH compounds analyzed in SIM mode are typically between 0.005 ug/L and 0.015 ug/L. Soil MDLs for PAH compounds analyzed in SIM mode are typically between 1.0 and 5.0 ug/kg. Procedures for conducting MDL studies can be found in CT Laboratories Initial Method Performance and Reporting SOP.

4.0 Scope and Application, including components to be analyzed

4.1 Method SW-846 8270D is used to quantify solvent-extractable semivolatile organic compounds in water and soil. Most base-neutral and acidic organic compounds which are soluble in methylene chloride and capable of being eluted in a gas chromatograph without derivatization can be quantitated. See Table 1.0 for a typical target analyte list (TAL).

4.2 Examples of other compounds which have been analyzed by this method are listed in Table 1.1. SW846 method 8270D notes a number of other compounds amenable to this test.

4.3 The following compounds require special treatment when being determined by this method.

4.3.1 Benzidine is subject to oxidative losses during solvent concentration and exhibits poor chromatographic behavior.

4.3.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

4.3.3 Pentachlorophenol, 2,4-Dinitrophenol, 4-Nitrophenol, Benzoic Acid, N-Nitrosodimethylamine, 2-Naphthylamine, 4,6-Dinitro-2-Methylphenol, 4-Chloro-3-Methylphenol, 2-Nitroaniline, 3-Nitroaniline, 4-Chloroaniline, Pyridine, and Benzyl Alcohol are subject to erratic chromatographic behavior.

4.3.4 The analytes listed above are flagged when there are limitations caused by sample preparation and/or chromatographic problems.

4.3.5 N-nitrosodiphenylamine decomposes in the gas chromatograph and cannot be separated or differentiated from Diphenylamine. Both analytes are reported as a pair.

4.3.6 Azobenzene & 1,2-Diphenylhydrazine, 3&4-Methylphenol and 3&4-Chlorophenol are reported as a pair.

Table 1.0
8270 Compound List

Codes (Tables 1):
S = Surrogates
I = Internal Standards
CT= Target Compounds
CPT= System Performance Check Compounds

Table 1.0

PK#	***Compound Name***	***Retention Time***	***Relative RT***	***Primary Ion***	***Secondary Ion(s)***	***Code***
1	1,4-Dichlorobenzene d_4	3.881	1.00	152	150,115	I
2	N-Nitrosodimethylamine	2.011	0.52	42	74,44	CT
3	Pyridine	2.023	0.52	79	52	CT
4	2-Fluorophenol	2.736	0.70	112	64,57	S
5	Aniline	3.546	0.91	93	65,66	CT
6	Bis(2-chloroethyl) ether	3.614	0.93	93	63,95	CPT
7	Phenol d_5	3.517	0.91	99	42,71,100	S
8	Phenol	3.529	0.91	94	65,66	CPT
9	2-Chlorophenol	3.659	0.94	128	64,130	CPT
10	1,3-Dichlorobenzene	3.819	0.98	146	148,111	CT
11	1,4-Dichlorobenzene	3.901	1.01	146	148,111	CT
12	1,2-Dichlorobenzene	4.066	1.05	146	148,111	CT
13	Benzyl alcohol	4.054	1.04	108	79,77	CT
14	Bis(2-chloroisopropyl) ether	4.216	1.09	45	121,77	CPT
15	2-Methylphenol	4.194	1.08	107	108,77	CPT
16	N-Nitrosopyrrolidine	4.350	1.12	100	41,42	CT
17	Acetophenone	4.367	1.13	105	77,51	CPT
18	Hexachloroethane	4.466	1.15	117	201,199	CPT
19	N-Nitrosodi-n-propylamine	4.381	1.13	70	42,101	CPT
20	3 & 4-Methylphenol	4.398	1.13	107	108,77	CPT
21	Naphthalene d_8	5.540	1.00	136	68,108	I
22	Nitrobenzene d_5	4.546	0.82	82	128,54	S
23	Nitrobenzene	4.572	0.83	77	123,65	CPT
24	Isophorone	4.901	0.88	82	95,138	CPT
25	2-Nitrophenol	4.992	0.90	139	109,65	CPT
26	2,4-Dimethylphenol	5.106	0.92	122	107,121	CPT
27	Bis(2-chloroethoxy) methane	5.253	0.95	93	95,123	CPT
28	2,4-Dichlorophenol	5.370	0.97	162	164,98	CPT
29	1,2,4-Trichlorobenzene	5.475	0.99	180	182,145	CT

Table 1.0

PK#	***Compound Name***	***Retention Time***	***Relative RT***	***Primary Ion***	***Secondary Ion(s)***	***Code***
30	Benzoic Acid	5.336	0.96	122	105,77	CT
31	Naphthalene	5.569	1.01	128	129,127	CPT
32	4-Chloroaniline	5.680	1.03	127	129,65,92	CPT
33	2,6-Dichlorophenol	5.677	1.02	162	164,98	CT
34	Hexachloropropene	5.688	1.03	213	211,215	CPT
35	Hexachlorobutadiene	5.756	1.04	225	223,227	CPT
36	4-Chloro-3-methylphenol	6.262	1.13	107	144,142	CPT
37	2-Methylnaphthalene	6.353	1.15	142	141	CPT
38	1-Methylnaphthalene	6.441	1.16	141	142	CT
39	Acenaphthene d_{10}	7.188	1.00	164	162,160	I
40	Hexachlorocyclopentadiene	6.504	0.90	237	235,272	CPT
41	1,2,4,5-Tetrachlorobenzene	6.509	0.91	216	214,179	CPT
42	2,4,6-Trichlorophenol	6.623	0.92	196	198,200	CPT
43	2,4,5-Trichlorophenol	6.651	0.93	196	198,97	CPT
44	2-Fluorobiphenyl	6.694	0.93	172	171,170	S
45	2-Chloronaphthalene	6.774	0.94	162	127,164	CPT
46	2-Nitroaniline	6.873	0.96	65	92,138	CPT
47	Acenaphthylene	7.086	0.99	152	151,153	CPT
48	Dimethylphthate	7.027	0.98	163	194,164	CPT
49	2,6-Dinitrotoluene	7.063	0.98	165	63,89	CPT
50	Acenaphthene	7.214	1.00	154	153,152	CPT
51	3-Nitroaniline	7.180	1.00	138	108,92	CPT
52	2,4-Dinitrophenol	7.254	1.01	184	63,154	CPT
53	Dibenzofuran	7.331	1.02	168	139	CPT
54	2,4-Dinitrotoluene	7.339	1.02	165	63,89	CPT
55	4-Nitrophenol	7.313	1.02	109	139,65	CPT
56	2,3,5,6-Tetrachlorophenol	7.390	1.03	232	194,234,230	CT
57	2,3,4,6-Tetrachlorophenol	7.419	1.03	232	131,230,234	CPT
58	Fluorene	7.558	1.05	166	165,167	CPT
59	4-Chlorophenyl phenyl ether	7.561	1.05	204	206,141	CPT
60	Diethyl phthalate	7.501	1.04	149	177,150	CPT
61	4-Nitroaniline	7.592	1.06	138	65,108,92	CPT
62	2,4,6-Tribromophenol	7.714	1.07	330	332,141	S
63	Phenanthrene d_{10}	8.140	1.00	188	94,80	I
64	4,6-Dinitro-2-methylphenol	7.603	0.93	198	51,105	CPT
65	N-Nitrosodiphenylamine & Diphenylamine	7.643	0.94	169	168,167	CPT
66	Azobenzene & 1,2-Diphenylhydrazine	7.666	0.94	182	152,77	CT
67	4-Bromophenyl phenyl ether	7.868	0.97	248	250,141	CPT
68	Hexachlorobenzene	7.902	0.97	284	142,249	CPT
69	Pentachlorophenol	8.027	0.99	266	264,268	CPT
70	Phenanthrene	8.155	1.00	178	179,176	CPT
71	Anthracene	8.186	1.01	178	176,179	CPT
72	Carbazole	8.271	1.02	167	166,139	CPT

Table 1.0

PK#	***Compound Name***	***Retention Time***	***Relative RT***	***Primary Ion***	***Secondary Ion(s)***	***Code***
73	Di-n-butyl phthalate	8.430	1.04	149	150,104	CPT
74	Fluoranthene	8.748	1.07	202	101,203	CPT
75	Chrysene d_{12}	9.567	1.00	240	120,236	I
76	Benzidine	8.814	0.92	184	92,185	CT
77	Pyrene	8.868	0.93	202	200,203	CPT
78	Terphenyl d_{14}	8.944	0.93	244	122,212	S
79	Butyl benzyl phthalate	9.217	0.96	149	91,206	CPT
80	3,3-Dichlorobenzidine	9.541	1.00	252	254,126	CPT
81	Benzo (a) anthracene	9.558	1.00	228	229,226	CPT
82	Chrysene	9.581	1.00	228	226,229	CPT
83	Bis (2-ethylhexyl) phthalate	9.564	1.00	149	167,279	CPT
84	Di-n-octyl phthalate	9.973	1.04	149	167,43	CPT
85	Perylene d_{12}	10.456	1.00	264	260,265	I
86	Benzo (b) fluoranthene	10.223	0.98	252	253,125	CPT
87	Benzo (k) fluoranthene	10.240	0.98	252	253,125	CPT
88	Benzo (a) pyrene	10.422	1.00	252	253,125	CPT
89	Indeno (1,2,3-cd) pyrene	11.164	1.07	276	138,277	CPT
90	Dibenz (a,h) anthracene	11.175	1.07	278	139,279	CPT
91	Benzo (g,h,i) perylene	11.368	1.09	276	138,277	CPT

Table 1.1
Additional Analytes

Table 1.1

PK#	***Compound***	***Retention Time***	***Relative RT***	***Primary Ion***	***Secondary Ion***	***Code***
92	2-Ethoxyethanol	1.68	0.43	59	45,72	CT
93	N-Nitrosodiethylamine	3.468	0.76	102	42,57	CT
94	2-Chloro-5-Methylphenol	5.360	0.87	107	144,142	CT
95	2,5-Dichlorophenol	6.045	0.98	162	164,63,99	CT
96	2,3-Dichlorophenol	6.085	0.98	162	126,63,164,	CT
97	Quinoline	6.120	1.10	129	102,51	CT
98	3&4-Chlorophenol	6.176	1.11	128	130,65,100	CT
99	Alpha-terpineol	6.390	1.15	59	43,93	CT
100	N-Nitroso-di-n-butylamine	6.627	1.19	84	57,41	CT
101	2-Methyl-4-Chlorophenol	6.661	1.20	107	142,77	CT
102	3,4-Dichlorophenol	7.343	0.96	162	164,99,63	CT
103	2,5-Dinitrophenol	7.579	0.99	184	63,53,39	CT
104	Pentachlorobenzene	7.787	1.01	250	252,108	CT
105	Diallate	7.890	0.97	86	234,236	CT
106	2-Naphthylamine	7.895	1.03	143	115,116	CT
107	Benzaldehyde	4.287	0.90	106	105,77	CPT
108	Caprolactam	6.793	1.06	55	113,85	CPT
109	Biphenyl	7.430	0.95	154	153,152	CPT
110	Atrazine	8.518	0.98	200	215,58	CPT

Table 1.1

PK#	*Compound*	*Retention Time*	*Relative RT*	*Primary Ion*	*Secondary Ion*	*Code*
111	Hexachlorophene	10.27	0.98	196	209,211	CT
112	Benzo(a)anthracene-d12	10.329	1.00	240		I
113	o-Terphenyl-d14	8.550	0.83	244		S
114	1,4-Oxithiane	4.320	0.830	104	61	CT
115	1,4-Dithiane	5.632	1.08	120	92	CT
116	2-bromophenylmethylsulfide	7.548	1.183	202	108	S

Note: Retention time shifts can occur when instrument maintenance is performed.
Shifts in the retention times are reflected in the analytical method.

Table 1.2
Internal Standard For Each Target Compound

Table 1.2

1,4-Dichlorobenzene d_4	**Naphthalene d_8**	**Acenaphthene d_{10}**	**Phenanthrene d_{10}**	**Chrysene d_{12}**	**Perylene d_{12}**
N-Nitrosodimethylamine	Nitrobenzene d_5	Hexachlorocyclopentadiene	4,6-Dinitro-2-methylphenol	Benzidine	Benzo (b) fluoranthene
Pyridine	Nitrobenzene	1,2,4,5-Tetrachlorobenzene	N-Nitrosodiphenylamine & Diphenylamine	Pyrene	Benzo (k) fluoranthene
2-Fluorophenol	Isophorone	2,4,6-Trichlorophenol	Azobenzene & 1,2-Diphenylhydrazine	Terphenyl d_{14}	Benzo (a) pyrene
Aniline	2-Nitrophenol	2,4,5-Trichlorophenol	4-Bromophenyl phenyl ether	Butyl benzyl phthalate	Indeno (1,2,3-cd) pyrene
Bis(2-chloroethyl) ether	2,4-Dimethylphenol	2-Fluorobiphenyl	Hexachlorobenzene	3,3-Dichlorobenzidine	Dibenz (a,h) anthracene
Phenol d_5	Bis(2-chloroethoxy) methane	2-Chloronaphthalene	Pentachlorophenol	Benzo (a) anthracene	Benzo (g,h,i) perylene
Phenol	2,4-Dichlorophenol	2-Nitroaniline	Phenanthrene	Chrysene	Hexachlorophene
2-Chlorophenol	1,2,4-Trichlorobenzene	Acenaphthylene	Anthracene	Bis (2-ethylhexyl) phthalate	
1,3-Dichlorobenzene	Benzoic Acid	Dimethylphthate	Carbazole	Di-n-octyl phthalate	
1,4-Dichlorobenzene	Naphthalene	2,6-Dinitrotoluene	Di-n-butyl phthalate		
1,2-Dichlorobenzene	4-Chloroaniline	Acenaphthene	Fluoranthene		
Benzyl alcohol	2,6-Dichlorophenol	3-Nitroaniline	Atrazine		
Bis(2-chloroisopropyl) ether	Hexachloropropene	2,4-Dinitrophenol	Diallate		
2-Methylphenol	Hexachlorobutadiene	4-Nitrophenol			
N-Nitrosopyrrolidine	4-Chloro-3-methylphenol	2-Naphthylamine			
Acetophenone	2-Methylnaphthalene	Fluorene			
N-Nitrosodi-n-propylamine	1-Methylnaphthalene	4-Chlorophenyl phenyl ether			
3 & 4-Methylphenol	2-Chloro-5-methylphenol	Diethyl phthalate			
Hexachloroethane	2,5-Dichlorophenol	Dibenzofuran			
N-Nitrosodiethylamine	2,3-Dichlorophenol	2,4-Dinitrotoluene			
Benzaldehyde	3&4-Chlorophenol	4-Nitroaniline			
2-Ethoxyethanol	N-Nitroso-di-n-butylamine	2,4,6-Tribromophenol			

Table 1.2

1,4-Dichlorobenzene d_4	Naphthalene d_8	Acenaphthene d_{10}	Phenanthrene d_{10}	Chrysene d_{12}	Perylene d_{12}
	2-Methyl-4-chlorophenol	2,3,5,6-Tetrachlorophenol			
	Caprolactam	2,3,4,6-Tetrachlorophenol			
	Quinoline	3,4-Dichlorophenol			
	Alpha-terpineol	2,5-Dinitrophenol			
		Pentachlorobenzene			
		Biphenyl			

5.0 Method Summary

5.1 This method describes procedures for isolating organic compounds through sample preparation from aqueous, soil and tissue matrices (reference methods SW846-3510C, 3580A, 3545A and 3546), concentration techniques that are suitable for preparing the extract, and the quantitative/qualitative analysis for the determination of target analytes by method SW846-8270D.

5.2 A sample of a known volume or weight is extracted with solvent or diluted with solvent. This method applies to aqueous samples extracted by liquid-liquid separatory funnel (SW846-3510C) and to soil/sediment and solid waste samples extracted by standard solvent extraction methods utilizing pressurized extraction techniques as heated pressurized fluid extraction (SW846-3545A) and using microwave energy to produce elevated temperature and pressure conditions in a closed vessel containing extraction solvent (SW846-3546). This method includes the extraction for waste dilution samples (SW846-3580A).

5.3 The resultant extract is chemically dried and concentrated in a Kuderna-Danish (K-D) apparatus in preparation for instrumental analysis.

5.4 Extracts for 8270D analysis may be subjected to cleanup measures, depending on the nature of the matrix interferences and target analytes. The suggested method of cleanup is Gel-Permeation Chromatography (GPC) cleanup (SW846 3640A). Refer to GPC SOP Rev 0. After cleanup, the extract is analyzed by injecting a known aliquot into a gas chromatograph equipped with a mass spectrometer detector.

5.5 Identification of target analytes is accomplished by comparing their mass spectra with the spectra of certified commercially-prepared stock standards. Quantitation is accomplished by comparing the response of a major quantitation ion relative to an internal standard using a five point (minimum) calibration curve.

5.6 The PAH compounds may be analyzed using SIM (selected ion monitoring) signals for quantitation in order to achieve lower detection limits. This is referred to in this SOP as SIM+SCAN.

5.7 The procedures contained within this method are restricted to use by or under the supervision of trained analysts. Each analyst must demonstrate the ability to generate acceptable results.

6.0 Definitions

6.1 DFTPP: Decafluorotriphenylphosphine. This compound is used to verify that the GC/MS is properly tuned and ready for calibration and sample analysis. To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction to eliminate column bleed or instrument background noise is accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP. The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Benzidine and Pentachlorophenol must have tailing factors less than 2. Breakdown of DDT to DDD and DDE must be less than 20%.

6.2 System Performance Check Compounds (SPCC): SPCCs are system performance compounds that are a part of the Continuing Calibration Verification standard (CCV). The SPCCs must meet a minimum response factor of 0.050. The SPCC criteria also apply to the average response factor of the initial calibration curve.

6.3 Calibration Check Compounds (CCC): CCCs are calibration check compounds that are a part of the Continuing Calibration Verification standard (CCV). The CCC percent difference must be less than or equal to 20%.

6.4 For full definitions on all terms applicable to this method, see Section 25.6 in the Quality Assurance Manual (QAM).

6.5 For a list of common acronyms and abbreviations, see QAM front matter.

7.0 Interferences

7.1 Solvents, reagents, glassware, and other sample processing hardware can yield artifacts and /or interferences to sample analysis. All these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks. Refer to each method for specific guidance on quality control procedures.

7.2 Phthalate esters contaminate many types of products commonly found in the laboratory. Plastics, in particular, must be avoided because phthalates are commonly used as plasticizers and are easily extracted from plastic materials.

7.3 Soap residue (e.g. sodium dodecyl sulfate), which results in a basic pH on glassware surfaces, will cause degradation of certain analytes.

7.4 Interferences co-extracted from the samples will vary considerably from source to source. If analysis of an extracted sample is prevented due to interference, further cleanup of the sample extract or dilution of the sample extract will be necessary.

7.5 Mass spectrometer sensitivity, column degradation, and contamination can also contribute to background interferences. The presence of semivolatile hydrocarbons in the sample extracts may require an appropriate post analysis bake-out time to be incorporated in the method.

8.0 Safety

8.1 Safety glasses, gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure must utilize appropriate laboratory safety systems.

8.2 The toxicity and carcinogenicity of chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, and exposure to these chemicals shall be minimized.

9.0 Equipment and Supplies

9.1 GC-MS system - Hewlett Packard 6890 GC/7683 autosampler/5973 MSD - An analytical system complete with gas chromatograph suitable for split-splitless injection and all required accessories including syringes, analytical column, mass spectrometer detector, auto sampler, electronic pressure control, vacuum pumps, and HP Chemstation data acquisition system. The data acquisition system consists of an IBM compatible PC with an operating system of Windows XP Professional and Agilent Environmental Chemstation (MSD Chemstation Rev. D.03.00.611).

9.1.1 8270D GC Conditions
Carrier Gas: He at 1.6 mL/min, hold 5.0 min
Ramped to 1.2 ml/min (10 ml/min), hold 3.0 min
Ramped to 1.8 ml/min (10 ml/min), hold 4.96 min
Injector Temperature: 250° C
Mode: Pulsed Splitless
Inj. Volume: 0.5 uL
Pressure: 8.9 psi
Pulse Pressure: 30.0 psi
Pulse Time: 0.4 min
Purge Flow: 50.0 ml/min
Purge Time: 0.38 min
Total Flow: 53.5 ml/min
Gas Saver On: 20ml/min at 2 min
Oven: Initial – 20° (hold for 0.5 min)
Ramp – 45°/min
Final –70° (hold for 0 min)
Ramp – 14°/min
Final – 120° (hold for 0 min)
Ramp – 45°/min
Final – 220° (hold for 0 min)
Ramp – 40°/min
Final – 280° (hold for 0 min)
Ramp – 30° /min
Final – 325° (hold for 1.6 min)

9.1.2 8270D MS Conditions

MS Interface: 300°
MS Source: 280°
Mass range: 35-500 amu
Scan time: 0.317 sec/scan

9.1.3 Chemical Warfare Agents (1,4-Oxathiane and 1,4-Dithiane) G.C. Conditions

Carrier Gas: He at 1.2 mL/min, hold 5.0 min

Injector Temperature: 250° C
Mode: Pulsed Splitless
Inj. Volume: 1.0 uL
Pressure: 8.9 psi
Pulse Pressure: 30.0 psi
Pulse Time: 0.4 min
Purge Flow: 50.0 ml/min
Purge Time: 0.38 min
Total Flow: 53.5 ml/min
Gas Saver On: 20ml/min at 2 min
Oven: Initial – 20° (hold for 0.5 min)
Ramp – 25°/min
Final –300° (hold for 1.3 min)
Ramp – 45°/min
Final – 325° (hold for 1 min)

9.1.4 MS Conditions

MS Interface: 300°
MS Source: 280°
Mass range: 35-500 amu
Scan time: 0.317 sec/scan

9.1.4 SIM+SCAN GC Conditions

Carrier Gas: He at 1.6 mL/min
Ramped to 1.8 ml/min (10ml/min) at 14 min
Injector Temperature: 250° C
Mode: Pulsed Splitless
Inj. Volume: 0.5 uL
Pressure: 8.9 psi
Pulse Pressure: 30.0 psi
Pulse Time: 0.4 min
Purge Flow: 50.0 ml/min
Purge Time: 0.38 min
Total Flow: 53.5 ml/min

Gas Saver On: 20ml/min at 2 min
Oven: Initial – 20° (hold for 0.55 min)
Ramp – 45°/min
Final –70° (hold for 0 min)
Ramp – 14°/min
Final –120° (hold for 0 min)
Ramp – 35°/min
Final – 255° (hold for 0 min)
Ramp – 15°/min
Final – 290° (hold for 0 min)
Ramp – 4°/min
Final – 300° (hold for 0 min)
Ramp – 30°/min
Final – 325° (hold for 0.1 min)

9.1.5 SIM+SCAN MS Conditions
MS Interface: 300°
MS Source: 280°
See Table 2

Table 2
SIM+SCAN MS PARAMETERS

Table 1.3								
	START TIME	DWELL	LABEL	CYCLES/SEC	ION 1	ION 2	ION 3	ION 4
1	0	1	Auto_1	47.6	128			
2	5.26	50	NP	8.3	127	128		
3	5.66	1	Auto_2	47.6	142			
4	6.12	50	2MN,1MN	8.3	141	142		
5	6.38	1	Auto_3	47.6	152			
6	6.90	40	ACY,ACNE	7.1	1515	152	153	
7	7.27	1	Auto_4	47.6	166			
8	7.50	50	FLE	8.3	165	166		
9	7.90	1	Auto_5	47.6	178			
10	8.24	50	PHE,AN	8.3	176	178		
11	8.52	100	O-TER	8.3	244			
12	8.64	1	Auto_7	47.6	202			
13	9.05	50	FLA,PYR	8.2	101	202		
14	9.60	1	Auto_8	47.6	228			
15	10.20	40	BA,CHRY	7.1	226	228	240	
16	10.80	1	Auto_9	47.6	252			
17	11.40	40	BB,BK	7.0	125	252	264	
18	12.40	1	Auto_10	47.6	276			
19	13.00	40	IN,DB,BG	5.4	138	139	276	278

Retention time shifts can occur when instrument maintenance is performed. Shifts in the retention times are reflected in the analytical method.

Note: Instrument operating parameters are subject to change to improve chromatography. Changes are noted in the Instrument Maintenance Log

9.2 Analytical column: 30m x 0.25 mm ID, 0.25 um. (J&W DB-5.625 or equivalent).

9.3 CEM Microwave Accelerated Reaction System (MARS Xpress) extraction unit with Synergyprep software:

The CEM Mars extraction cycle:

Method 1
8-16 samples
Power: 100% at 800 watts
Ramp Time: 15 min
Pressure: 0
Temp: 110 C
Hold Time: 15 min

Method 2
17-48 samples
Power: 100% at 1600 watts
Ramp Time: 15 min
Pressure: 0
Temp: 110 C
Hold Time: 15 min

9.4 Organomation 12-position nitrogen concentrator. (N-Evap, Organomation #11250-RT)

9.5 Analytical balance capable of accurately weighing to the nearest 0.01 gram (Fischer Scientific XD 2200 or equivalent)

9.6 Oven, muffle and drying.

9.7 Separatory funnels - 2000 mL Nalgene 4301, Teflon FEF lined with Teflon TFE stopcocks and Tefzel ETFE screw closures (MG Scientific #F847-2L or equivalent).

9.8 Aluminum foil

9.10 2-Platform shakers (Eberbach Model 6010 150 V shaker, or equivalent) fitted with trays to hold 6 Nalgene separatory funnels each.

9.11 Water bath- heated and capable of accepting a Kuderna-Danish apparatus. (GlasCol 6 position heating mantle 100DRX30424 or equivalent)

9.12 Kuderna-Danish (K-D) apparatus:

9.12.1 Concentrator tube, 10.0 mL, graduated. (Fisher # K570051-1025).

9.12.2 Evaporation flask- 500 mL or 250 ml (Fisher # K570035-0250).

9.12.3 Snyder column- Three-ball macro (Fisher # K503000-0121).

9.12.4 Polyacetal 19/22 clamps (VWR #KT675300-019).

9.13 Graduated cylinder (Class A TC) - 1000 mL. (Fisher 08-559G).

9.14 Beakers - 250 mL (Pyrex, VWR #13912-207, or equivalent) and 600 mL (Pyrex, VWR #13912-240, or equivalent.

9.15 Vials - 2.0mL (National Scientific – C4000) 12mL (Kimble #60815-1965), and 60 mL screw cap vials with Teflon lined caps (C&G #LX64-A030-A01A) or equivalents.

9.16 Pasteur Pipettes; 5 ¾" and 9" (VWR #14672-200 and -300).

9.17 Funnels – glass. (VWR #154-08 or equivalent)

9.18 Volumetric flask – (Class A) 10 mL (VWR #29620-084, or equivalent), 25 mL (VWR #29620-109, or equivalent), 50 mL (VWR #29620-120, or equivalent), 100 mL (VWR #29620-142, or equivalent), and 200 mL (VWR #29620-164, or equivalent).

9.19 Syringes 10 uL (Hamilton #80300, or equivalent), 100 uL (Hamilton #80600, or equivalent), 500 uL (Hamilton #81243, or equivalent), 1 mL (Hamilton #81343, or equivalent), and 5 mL (Hamilton #81543, or equivalent).

9.20 Boiling chips, carborundum, approximately 10/40 mesh (methylene chloride rinsed) (Fisher # 09-191-12) equivalent. Stored in extractions lab.

9.21 Filter- Glass Microfiber 12.5 cm (Ahlstrom, MG # F136-1250).

9.22 CEM-MARS Microwave extraction tubes with plugs and caps, 75mL (CEM #574127)

9.23 Spatulas- stainless steel. (VWR #57952-253 or equivalent)

9.24 pH indicator paper- pH 0-14. (Whatman #2613991) or equivalent. Stored in general lab storage area.

10.0 Reagents and Materials

10.1 Deionized water (Milli-Q processed), analyte free or equivalent.

10.2 Sodium sulfate (granular, anhydrous 60/120 mesh, JT Baker # 3375-05) or equivalent. If sodium sulfate passes in house lot check, it can be used as is and stored in air tight glass jar. Otherwise condition sodium sulfate by heating to 400°C for 4 hours in a shallow glass tray loosely covered with foil and recheck for purity. Sodium sulfate is stored in airtight glass jars and used within five years of opening or before the manufacturer's expiration date.

10.3 Silica sand- hydrocarbon free. Purify by heating to 400°C for 4 hours in a shallow glass tray, loosely covered with foil. Silica sand is stored in airtight glass jars and used within five years of purifying.

10.4 Methylene chloride, pesticide grade, analyte free. Used within one year of opening or before the manufacturer's expiration date. Or stored in large carboy tank provided by manufacturer and used by the manufacturer's expiration date.

10.5 Acetone, pesticide grade, analyte free. Used within one year of opening or before the manufacturer's expiration date.

10.6 Methanol, pesticide grade. Used within one year of opening or before the manufacturer's expiration date.

10.7 Sulfuric Acid (Certified ACS)/Deionized Water-1:1(v/v). ACS grade. Used within six months of mixing or before manufacturer's expiration date for any reagent used. Log number recorded in Semivolatiles log book.

10.8 Sodium Hydroxide- 10 N (Certified ACS). Used within six months of mixing or before manufacturer's expiration date for any reagent used. Log number recorded in Semivolatiles log book.

10.9 Diatomaceous earth (Celite 545 – EMD #CX0574) or equivalent. Used within five years of opening or before the manufacturers expiration date.

10.10 Nitrogen (99.995% purity or greater).

10.11 Helium (99.995% purity or greater).

10.12 Standards and spikes

10.12.1 Preparation of standards is documented in the GC/MS standards logbook. Each standard is labeled by prep date to allow for tracking. Opened stock standards expire in one year or sooner if comparison with quality control check samples indicates a problem. Any subsequent dilutions made from the opened vial expire six months from the date of dilution (not to exceed one year from the initial opening of the vial). The cracking date of the stock standard vial will be recorded on the label along with the one year expiration date.

10.12.2 Stock Standards – Stock Standards are purchased from vendors who provide certified solutions. Standards are stored at –10ºC in a freezer reserved for standard solutions. Unopened standard shall have the manufacturer's suggested expiration date. Opened stock standards expire in one year or sooner if comparison with quality control check samples indicates a problem (Not to exceed the manufacturer's expiration date).The following list of stock standards (or equivalent) are commercially prepared standards which are certified by the manufacturer:

Benzidines:	SPEX Part # 605X at 2000 ug/ml
Balance Mix A:	SPEX Part # SV-X at 2000 ug/ml
Custom Mix:	SPEX Part # SVO-CTWI-6 at 2000 ug/ml
8270 Surrogate Mix BN:	Restek Part #31062 5000 ug/ml
8270 Surrogate Mix AE:	Restek Part #31063 10000 ug/ml
8270 Mega Mix:	Restek Part #31850 at 500/1000 ug/ml
8270 Benzidine Mix:	Restek Part #31834 at 2000 ug/ml
8270 Extra Analyte List:	SPEX Part # CT-SV-12 at 2000 ug/ml
TCLP B/N Mix:	Restek Part # 31028 at 2000 ug/ml
TCLP Acid Mix:	Restek Part # 31027 at 2000 ug/ml
8040 Phenol Mix #1:	Restek Part # 31088 at 2000 ug/ml
8040 Phenol Mix #2:	Restek Part # 31089 at 2000 ug/ml
PAH Mix:	Restek Part # 31622 at 2000 ug/ml
DFTPP:	Restek Part # 31615 at 1000 ug/ml
o-Terphenyl-d14:	Chem Service Part# FD1054-1,neat
Additions Mix	Restek Part # 31902 at 1000 ug/ml
Biphenyl	SPEX Part #S500 at 1000 ug/ml
1,4-Thioxane	Absolute standards part#71472 at 1000ug/ml
1,4-Dithiane	Absolute standards part# 71471 at 1000ug/ml
2-Bromophenylmethylsulfide	Absolute standards part# 72445 at 1000ug/ml

10.12.3 Intermediate Stock Standards: These standards are diluted stock standards so that the concentration levels are manageable for the preparation of working standards. The 8270D intermediate standard is prepared at an optimum level for the preparation of the working stock standard. Each 8270D target compound (or Surrogate) is at a concentration of 100.0 ug/ml in methylene chloride with the following exceptions: compounds that co-elute (listed in sec 4.3.6) are at a concentration of 200.0 ug/mL. For example, Azobenzene and 1,2-Diphenylhydrazine are each in the stock solution at 100.0 ug/mL. They are reported as a pair (Azobenzene&1,2-Diphenylhydrazine) with a concentration of 200.0 ug/mL. See Tables 3.0 and 3.1.

Table 3.0
Intermediate Stock Standard

Stock Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270 MegaMix	1000	1.000	10.0	100.0
Benzidines	2000	0.500	10.0	100.0
Customs Mix	2000	0.500	10.0	100.0
8270 Surr Mix BN	5000	0.200	10.0	100.0
8270 Surr Mix AE	10000	0.100	10.0	100.0

Table 3.1
Agents Intermediate Stock Standard

Stock Standard	Stock Standard Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
1,4-Thioxane	1000	100	5	20.0
1,4-Dithiane	1000	100	5	20.0
2-Bromophenylmethylsulfide	1000	100	5	20.0

10.12.4 Calibration standards: An initial calibration of the listed analytes in Table 1.0 is performed using a minimum of 5 points. The following concentrations correspond to the expected range of concentrations found in real samples and bracket the linear range of the detector. Standards are made by taking aliquots of the intermediate standard and diluting to volume in methylene chloride or by making dilutions directly from the stock standards (see Tables 3.2, 3.3 and 3.4). The following levels are repeated across all 8270D compounds. Note: due to low instrument response the following compounds are not calibrated from level 1: Benzoic acid, 2,4-Dinitrophenol, 4-Nitrophenol, 4,6-Dinitro-2-methylphenol and Pentachlorophenol.

Table 3.2
8270 Initial Calibration

Linearity Points	Spike Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
1	100.0	5	0.5	1.0
2	100.0	25	0.5	5.0
3	100.0	50	0.5	10
4	100.0	100	0.5	20
5	100.0	150	0.5	30
6	100.0	200	0.5	40
7	100.0	250	0.5	50

Table 3.3
8270 Agents Initial Calibration

Linearity Points	Spike Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
1	20	5	0.5	0.1
2	20	10	0.5	0.2
3	20	25	0.5	0.5
4	20	50	0.5	1.0
5	20	100	0.5	2.0
6	20	150	0.5	3.0
7	20	250	0.5	5.0

The PAH compounds may be analyzed using SIM (selected ion monitoring) signals for quantitation. In this case, two additional levels composed of PAH compounds only are acquired in the initial calibration at 0.02 ug/ml and 0.1 ug/mL. See Table 3.4.

Table 3.4
8270 SIM+SCAN Initial Calibration

Linearity Points	Spike Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
1*	20.0	0.010	10	0.020
2*	20.0	0.050	10	0.10
3	100.0	0.100	10	1.0
4	100.0	0.500	10	5.0
5	100.0	1.000	10	10
6	100.0	2.000	10	20
7	100.0	1.500	5	30
8	100.0	2.000	5	40
9	100.0	2.500	5	50

* SIM+SCAN linearity points 1 and 2 contain PAH compounds only. See Tables 2.0 and 2.1.

10.12.5 Initial Calibration Verification (ICV): The initial calibration verification standard shall verify the initial calibration curve (different manufacturer from the initial calibration standard, a second lot from the same manufacturer is acceptable if a second manufacturer cannot be found. The initial calibration verification standards involve the analysis of all target compounds at 20.0 ug/ml and 40.0 ug/mL (40.0 ug/mL and 80.0 ug/mL for Azobenzene & 1,2-Diphenylhydrazine and N-nitrosodiphenylamine & Diphenylamine) each time the initial calibration is performed. Standards are made by taking aliquots of the ICV intermediate standard or the purchased stock standards and diluting to volume in methylene chloride. The ICV stock standard is prepared in the same manner as the primary intermediate stock standard. See Tables 3.5, 3.6 and 3.7.

Table 3.5
8720 ICV Working Standards

Working ICV Standards	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
ICV 1	100.0	1.000	5.0	20.0
ICV 2	100.0	2.000	5.0	40.0

Table 3.6
8720 Agents ICV Working Standards

Working ICV Standards	Intermediate Standard Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
ICV 1	20	50	1	1.0
ICV 2	20	100	1	2.0

Table 3.7
8270 ICV SIM+SCAN Working Standards

Working ICV Standards	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
ICV 1*	20.0	0.250	10.0	0.50
ICV 2	100.0	0.500	5.0	10.0
ICV 3	100.0	1.500	5.0	30.0

* SIM+SCAN ICV 1 contains PAH compounds only. See Tables 3.0 and 3.1

10.12.6 Calibration Verification Standard (CCV): A working standard solution for 8270D at a concentration of 20.0 ug/ml and is used to check the validity of a calibration curve on a daily basis. Standard is made by taking an aliquot of the intermediate standard and diluting it to volume in methylene chloride. The CCV is prepared weekly and stored at -10ºC. The CCV for SIM+SCAN is the same as for normal 8270D. See Tables 3.8 and 3.9.

Table 3.8
8270 CCV Working Standard

Working CCV Standard	Intermediate Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270	100.0	1.00	5.0	20.0

Table 3.9
8720 Agents CCV Working Standards

Working ICV Standards	Intermediate Standard Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
CCV 1	20	50	1	1.0

10.12.7 Surrogate standard: Commercially prepared certified solutions of 2-Fluorophenol, Phenol d_5, and 2,4,6-Tribromophenol at 10000 ug/ml (AE Surrogates) and Nitrobenzene d_5, 2-Fluorobiphenyl, and p-Terphenyl d_{14} (BN

Surrogates) are diluted in acetone to produce a working surrogate solution of 40/20 ug/ml (AE/BN). 1.0 mL is added to each sample.
For Agents 2-Bromophenylmethylsulfide at 1000ug/ml is diluted with Acetone to produce 1.0ug/ml working surrogate solution. 1.0ml is added to each sample. The surrogate concentration is normalized to 100% from the spiking solution in the initial calibration. This will provide percent recoveries that transfer directly to LIMS. For SIM+SCAN analyses o-Terphenyl d_{14} is added at a final concentration of 1.0 ug/ml. See Tables 3.10, 3.11 and 3.12.

Table 3.10
8270 Surrogate Spiking Solution

Surrogate Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270 Surr. Mix BN	5000.0	0.400	100.0	20.0
8270 Surr. Mix AE	10000.0	0.400	100.0	40.0

Table 3.11
8270 Agents Surrogate Spiking Solution

Surrogate Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
2-Bromophenylmethylsulfide	1000	50	50	1.0

Table 3.12
8270 SIM+SCAN Surrogate Spiking Solution

Surrogate Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270 Surr. Mix BN	5000	0.400	100.0	20.0
8270 Surr. Mix AE	10000	0.400	100.0	40.0
o-Terphenyl-d14	1000	0.100	100.0	1.0

10.12.8 Internal standard solution: A Commercially prepared certified solution of 1,4-Dichlorobenzene d_4, Naphthalene d_8, Acenaphthene d_{10}, Phenanthrene d_{10}, Chrysene d_{12} and Perylene d_{12} at 2000 ug/mL in methylene chloride. 5 uL is added to each 500 uL aliquot of sample extract for a final concentration of 20 ug/mL. In addition, Benzo[a]anthracene d_{12} is added when the samples are analyzed for PAHs in selected ion monitoring (SIM) mode. See Table 3.13 for SIM+SCAN internal standard concentrations. See Table 3.14 for Agents internal standard concentrations.

Table 3.13
8270 SIM+SCAN Internal Standard Solution

Internal Standard Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
8270 IS Mix	2000	0.5	1.0	1000
Benz[a]anthracene-d12	1000	0.5	1.0	500

Table 3.14
8270 Agents Internal Standard Solution

Internal Standard Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
8270 IS Mix	2000	100	1.0	200

10.12.9 Spiking standards (matrix and control samples): Prepare a spiking solution in acetone or methanol that contains target compounds for water and sediment / soil samples. 1.0 ml is added to quality control and matrix spike samples. The concentration of these compounds are five times higher for waste samples. If other compounds of interest are to be monitored they can be added at an appropriate level and noted in the standard preparation log. If client requests, spiking solution can be altered to match the target analytes of interest. See Tables 3.15 and 3.16.

Table 3.15
8270 Analyte Spiking Solution

Spiking Solution	Stock Standard Conc. (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
MegaMix	1000	1.000	50.0	20.0
Benzidines	2000	0.500	50.0	20.0
Custom Mix	2000	0.500	50.0	20.0

Table 3.16
8270 Agents Analyte Spiking Solution

Spiking Solution	Stock Standard Conc. (ug/ml)	Standard Volume (ul)	Final Volume (ml)	Final Concentration (ug/ml)
1,4-Thioxane	1000	50	50.0	1.0
1,4-Dithiane	1000	50	50.0	1.0

10.12.10 DFTPP: Decafluorotriphenylphosphine solution in methylene chloride (See Table 3.17). This compound is used in tuning the GC/MS. To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged.

The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness.

Table 3.17
DFTPP Standard Solution

Spiking Solution	Stock Standard Concentration (ug/ml)	Standard Volume (ml)	Final Volume (ml)	Final Concentration (ug/ml)
DFTPP	1000	0.500	10.0	50.0

NOTE: All standards are stored at -10ºC. Opened stock standards expire in six months or sooner if comparison with quality control check samples indicates a problem. An intermediate stock standard or working standard shall not exceed expiration date criteria. All subsequent standards made from the intermediate stock standards expire on the same date as the stock standard. If more than one standard is added to a solution the expiration date will be the same as the stock standard with the earliest expiration date.

11.0 Sample Collection, Preservation, and Storage

11.1 Aqueous samples are collected in 1-L amber glass containers with Teflon lined lids. Aqueous samples are to be collected in duplicate. Solid samples are collected in 250-mL wide mouth glass containers with Teflon-lined lids. All samples are preserved by cooling to 4ºC. The soil samples must be extracted within 14 days and water samples must be extracted within 7 days from the date of collection.

11.1 Sample extracts are stored under refrigeration and analyzed within 40 days of extraction.

11.2 All soil samples are weighed on the top loading balance which is connected to a computer so that all weights can be automatically entered into an Excel spread sheet. The spreadsheets are saved so the data can be transferred electronically to the LIMS system.

12.0 Quality Control

12.1 This SOP is designed to follow a variety of different projects and program requirements. Table 5 is designed to illustrate the control steps and provisions required to adequately produce acceptable data.

12.2 Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.3 Program Specific Limits: Samples analyzed under the guidance of certain programs; such as the Department of Defense Quality Systems Manual (DoD/QSM) or Louisville Chemistry Guidance (LCG), require their own specified limits. For these samples follow the limits specified in the manuals for that program.

13.0 Calibration & Standardization

13.1 Calibration

13.1.1 The initial calibration for SW-846 chromatographic methods involves the analysis of standards containing the target compounds at a minimum of five different concentrations covering the working range of the instrument

13.1.2 For each compound and surrogate of interest, prepare calibration standards at a minimum of five different concentrations by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with methylene chloride.

13.1.3 The lowest concentration calibration standard that is analyzed during an initial calibration curve establishes the method's quantitation limit based on the final volume of the sample extract described in the preparative method or employed by the laboratory.

13.1.4 Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the response of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area or height of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and is also known as a relative response factor in other methods.

13.1.4.1 Internal standards are recommended in SW846-8270D. These internal standards are: 1,4-Dichlorobenzene $d_{4,}$ Naphthalene $d_{8,}$ Acenaphthene $d_{10,}$ Phenanthrene $d_{10,}$ Chrysene $d_{12,}$ and Perylene $d_{12.}$ In addition, Benzo[a]anthracene d_{12} is added when the samples are analyzed for PAHs in selected ion monitoring (SIM) mode. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

13.1.4.2 In preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. 5.0 uL of a solution containing the internal standards at a concentration of 2,000.0 ug/mL is added to each 500.0 uL of standard or sample extract. This results in an internal standard concentration of 20.0 ug/mL in the extract. For SIM+SCAN analysis the internal standard solution is at 1,000.0 ug/mL resulting in a concentration of 10.0 ug/mL in the extract, except for Benz[a]anthracene-d12 which is at half of this concentration (500.0 ug/mL in the IS solution, 5.0 ug/mL in the extract). The mass of each internal standard added to each sample extract immediately prior to injection into the instrument must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts is such that minimal dilution of the extract occurs (e.g., 5.0 uL of solution added to a 500.0 uL final extract results in only a negligible 0.1% change in the final extract volume which can be ignored in the calculations).

13.1.4.3 An ideal internal standard concentration would yield a response factor of 1 for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard shall produce an instrument response (area counts) that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This results in a minimum response factor of approximately 0.01 for the least responsive target compound.

13.1.5 For each of the initial calibration standards, calculate the RF values for each target compound relative to one of the internal standards as follows;

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

A_s = Peak area of the analyte or surrogate.
A_{is} = Peak area of the internal standard.
C_s = Concentration of the analyte or surrogate in ug/mL.
C_{is} = Concentration of the internal standard in ug/mL.

13.1.6 Linear calibration using the average response factor. Response factors are a measure of the slope of the calibration relationship and assume that the curve passes through the origin. Under ideal conditions, the factors will not vary with the concentration of the standard that is injected into the instrument. In practice, some variation is to be expected. However, when the variation, measured as the relative standard deviation (RSD), is less than or equal to 20%, the use of the linear model is generally appropriate, and calibration curve can be assumed to be linear and to pass through the origin. To evaluate the linearity of the initial calibration, calculate the RF, the standard deviation, and the relative standard deviation.

$$\text{Mean RF} = \overline{RF} = \frac{\sum_{i=1}^{n} RF_i}{n}$$

$$SD = \sqrt{\left(\frac{\sum_{I=1}^{n} (RF_I - \overline{RF})^2}{n-1} \right)}$$

$$RSD = \frac{SD}{\overline{RF}} \times 100$$

13.1.7 The average response factor (ARF) for all calibration levels is used when determining sample concentration and is calculated (along with the standard deviation) to evaluate the linearity of the curve (SW-846 Method 8000C sec. 11.5). When ARFs are not acceptable, results are sometimes calculated using linear (1st order) regression curves and/or quadratic (2nd order) curves. Internal standard quantitation is also used when generating linear and non-linear calibrations. All equations and acceptance criteria follow the examples in SW-846, Method 8000C (sec. 11.5).

13.1.8 Linear Calibration: If the RSD of the calibration factor is greater than 20% over the calibration range, then linearity though the origin cannot be assumed. If this is the case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation in the form:

$y = mx + b$

y = instrument response (peak area or height)
m = Slope of the line
x = Concentration of the calibration standard
b = The intercept

13.1.9 The use of origin (0,0) as a calibration point is not allowed. However, most data systems and many commercial software packages will allow the analyst to “force” the regression through zero. This is not the same as including the origin as a fictitious point in the calibration. It can be appropriate to force the regression through zero for some calibrations (SW-846 Method 8000C sec. 11.5.2.1). The use of linear regression cannot be used as a rationale for reporting results below the calibration range.

13.1.10 Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen shall have no more than four parameters, i.e., if the model is polynomial, it can be no more than third order as in the equation:

$$y = ax^2 + bx + c$$

13.1.1 The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches described above. Linear regression employs five calibration standards for the linear model, a quadratic model requires a minimum of six calibration standards. The coefficient of determination (COD) is calculated as follows:

$$\mathrm{COD} = \frac{\sum_{i=1}^{n} (y_{obs} - \overline{y})^2 - \left(\frac{n-1}{n-p} \right) \sum_{i=1}^{n} (y_{obs} - Y_i)^2}{\sum_{i=1}^{n} (y_{obs} - \overline{y})^2}$$

y_{obs} = Observed response (area) for each concentration of the calibration curve.

$\overline{y}$ = Mean observed response from the initial calibration.

Y_I = Calculated response at each concentration from the initial calibrations.

n = Total number of calibration points (6 points for quadratic equation).

p = Number of adjustable parameters in the polynomial.

13.1.2 Under ideal conditions, with a “perfect” fit of the model to the data, the coefficient of the determination will equal 1.0. In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99. Weighting in a calibration model can significantly improve the ability of the least squares regression to fit the data calibrations (SW-846 Method 8000C sec. 11.5.3).

13.2 Calibration Criteria

13.2.1 Before analysis of any samples or standards can begin, the GC/MS system must be hardware tuned so a 25 ng injection of Decafluorotriphenylphosphine (DFTPP) passes the tuning criteria listed in Table 4.0. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

Table 4.0
DFTPP Tuning Criteria

Mass	Ion Abundance Criteria
51	10-80% of Base Peak
68	<2% of mass 69
70	<2% of mass 69
127	10-80% of Base Peak
197	<2% of mass 198
198	Base peak, or >50% of Mass 442
199	5-9% of mass 198
275	10-60% of Base Peak
365	> 1.0% of mass 198
441	Present but < 24% of mass 442
442	Base peak or > 50% of mass 198
443	15-24% of mass 442

13.2.2 To acquire the mass spectrum of DFTPP, three scans (the peak apex scan and the scans immediately preceding and following the apex) are acquired and averaged. Background subtraction to eliminate column bleed or instrument background noise is accomplished using a single scan acquired no more than 20 scans prior to the elution of DFTPP.

13.2.3 The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and Pentachlorophenol shall be present at their normal responses and peak tailing shall be evaluated. Benzidine and Pentachlorophenol must have tailing factors less than 2.

13.2.4 Calibration Standards - Calibration standards are prepared at a minimum of five concentration levels and are prepared from the intermediate stock standards. One of the concentration levels shall be at a concentration near, but above, the detection limit and at or below the reporting limit. The remaining concentration levels shall correspond to the expected range of concentrations found in real samples and shall contain each analyte for detection by this method. If the measured relative standard deviation (RSD) is less than or equal to 20%, the use of the linear model is generally appropriate, and calibration curve can be assumed to be linear and to pass through the origin. Linear Calibration: If the RSD of the calibration factor is greater than 20% over the calibration range, then linearity though the origin cannot be assumed. In this case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response. The regression will produce the slope and intercept terms for a linear equation. In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range.

13.2.5 System Performance Check Compounds (SPCC) are the most common target analytes and are part of the initial calibration and the continuing calibration verification standard (CCV). A CCV must be made during each 12 hour shift. The SPCCs in the initial calibration and CCV must meet a minimum response factor. See Table 4.1.

13.2.6 Initial Calibration Verification (ICV): The initial calibration verification standard (different lot # or manufacturer from the initial calibration standard) shall verify the initial calibration curve. The initial calibration verification standard involves the analysis of all target analytes each time the initial calibration is performed. The SPCCs must meet minimum response factor given in Table 4.1. The percent drift of all target compounds must be less than or equal to 20%.

13.2.7 Calibration Verification Standard (CCV): A standard solution that is used to check the validity of a calibration curve on a daily basis. It also provides information on satisfactory maintenance and adjustment of the instrument during sample analysis. The SPCCs must meet minimum response factor given in Table 4.1. The percent drift of all target compounds must be less than or equal to 20%.

13.2.8 The relative retention time (RRT) of each compound in each calibration standard shall agree within 0.06 RRT units.

TABLE 4.1
System Performance Check Compounds and Minimum Response Factors

Table 4.1

#	Compound Name	Minimum Response Factor
1	Benzaldehyde	0.010
2	Bis(2-chloroethyl) ether	0.700
3	Phenol	0.800
4	2-Chlorophenol	0.800
5	Bis(2-chloroisopropyl) ether	0.010
6	2-Methylphenol	0.700
7	Acetophenone	0.010
8	Hexachloroethane	0.300
9	N-Nitrosodi-n-propylamine	0.500
10	3 & 4-Methylphenol	0.600
11	Nitrobenzene	0.200
12	Isophorone	0.400
13	2-Nitrophenol	0.100
14	2,4-Dimethylphenol	0.200
15	Bis(2-chloroethoxy) methane	0.300
16	2,4-Dichlorophenol	0.200
17	Naphthalene	0.700
18	4-Chloroaniline	0.010
19	Caprolactam	0.010
20	Hexachlorobutadiene	0.010
21	4-Chloro-3-methylphenol	0.200
22	2-Methylnaphthalene	0.400
23	Hexachlorocyclopentadiene	0.050
24	1,2,4,5-Tetrachlorobenzene	0.010
25	2,4,6-Trichlorophenol	0.200
26	2,4,5-Trichlorophenol	0.200
27	1,1’-Biphenyl	0.010
28	2-Chloronaphthalene	0.800
29	2-Nitroaniline	0.010
30	Acenaphthylene	0.900
31	Dimethylphthalate	0.010
32	2,6-Dinitrotoluene	0.200
33	Acenaphthene	0.900
34	3-Nitroaniline	0.010
35	2,4-Dinitrophenol	0.010
36	Dibenzofuran	0.800

Table 4.1		
37	2,4-Dinitrotoluene	0.200
38	4-Nitrophenol	0.010
39	2,3,4,6-Tetrachlorophenol	0.010
40	Fluorene	0.900
41	4-Chlorophenyl phenyl ether	0.400
42	Diethyl phthalate	0.010
43	4-Nitroaniline	0.010
44	4,6-Dinitro-2-methylphenol	0.010
45	N-Nitrosodiphenylamine & Diphenylamine	0.010
46	4-Bromophenyl phenyl ether	0.100
47	Hexachlorobenzene	0.100
48	Atrazine	0.010
49	Pentachlorophenol	0.050
50	Phenanthrene	0.700
51	Anthracene	0.700
52	Carbazole	0.010
53	Di-n-butyl phthalate	0.010
54	Fluoranthene	0.600
55	Pyrene	0.600
56	Butyl benzyl phthalate	0.600
57	3,3'-Dichlorobenzidine	0.010
58	Benzo(a)anthracene	0.800
59	Chrysene	0.700
60	Bis(2-ethylhexyl)phthalate	0.010
61	Di-n-octylphthalate	0.010
62	Benzo(b)fluoranthene	0.700
63	Benzo(k)fluoranthene	0.700
64	Benzo(a)pyrene	0.700
65	Indeno(1,2,3-cd)pyrene	0.500
66	Dibenz(a,h)anthracene	0.400
67	Benzo(g,h,i)perylene	0.500

14.0 Procedure

14.1 Water Extraction (Method SW-846,3510C)

14.1.1 Pre-rinse all glassware to be used in the extraction with methylene chloride (HPLC Grade).

14.1.2 Mark the meniscus on the bottle for later determination of sample volume.

14.1.3 One method blank (MB) and laboratory control spike (LCS) must be prepared with each batch of 20 samples or less. Prepare each by adding one liter of Milli-Q water to a clean amber glass sample jar.

14.1.4 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). In order of preference:

14.1.4.1 Select the sample where two full volume extra matrix was provided; use the extra volume supplied for a full volume MS and MSD.

14.1.4.2 Select a sample where one extra sample bottle was provided and split the sample into two equal portions, one for the MS and one for the MSD.

14.1.4.3 Select a sample where no extra sample was provided and split it into three equal portions, one for the parent sample, one for the MS, and one for the MSD.

14.1.4.4 For the last two situations concerning sacrificing a sample volume versus the inability to run a MS/MSD contact the project manager for proper procedure.

14.1.5 To all samples add 1.0 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 1.0 mL of the proper spiking solution to the MS/MSD and LCS.

14.1.5.1 Surrogate and/or spike shall be added directly to the sample jar.

14.1.5.2 Reseal the sample jar and gently shake sample to mix.

14.1.5.3 If it is necessary to prepare split MS/MSD samples, the samples shall be quantitatively split using graduated cylinders and spiking shall occur directly into the graduated cylinder. Swirl gently to mix.

14.1.5.4 From the sample jar (or graduated cylinder), quantitatively transfer the sample into a 2-liter separatory funnel.

14.1.5.5 Check and adjust the pH to <2 with 1:1 sulfuric acid.

14.1.5.6 Add 60 mL of methylene chloride to the sample jar (or graduated cylinder) and swirl to rinse sides of vessel. Transfer methylene chloride into the separatory funnel as well.

14.1.6 The sample is extracted by the automated shaker. Shake the samples vigorously for two minutes.

14.1.7 Allow the organic layer to separate from the water phase for a minimum of 10 minutes. Decant the lower layer into a 250 ml beaker. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and can include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Check sample pH to insure acidic conditions.

14.1.8 Repeat the extraction two more times using fresh 60 mL portions of methylene chloride.

14.1.9 After the third extraction of the acidified sample, adjust the pH >12 with 10N sodium hydroxide.

14.1.10 Extract three more times with 60 ml portions of methylene chloride as described above. The solvent from the basic pH extraction shall be collected into a separate 250 ml beaker from the acid extraction solvent.

14.1.11 Determine the sample volume by filling the sample bottle to the mark with water and transferring it to a "Class A TC" 1 liter graduated cylinder for measurement. Note all sample volumes on the extraction bench sheet (available on the network archive at Archive\Raw Date\Inprocess Data\Semi-Volatiles Bench Sheets\PREP TEMPLATES\8270 Prep).

14.1.12 Samples that are very dark or appear to have petroleum or other organic material may require cleanup prior to analysis. This will require further consultation with the analyst and/or project manager. Refer to GPC SOP Rev 0.

14.1.13 Record all lot numbers, prepping analyst, times and dates on prep bench sheet (see FSV6-02).

14.1.14 Refer to section 14.6 for sample concentration.

14.2 Soil and Tissue Extraction (Method SW-846, 3546) Microwave extraction

14.2.1 Preparing the extraction tubes for use: extraction tubes, caps and plugs are washed in the dishwasher, rinsed with Methanol and baked in 110 C oven for 1 hour. After they have cooled, rinse the extraction cell (tubes, plugs and caps) with Methylene chloride.

14.2.2 Decant and discard any water layer from sediment sample. Mix sample thoroughly, especially composite samples. Discard any foreign objects such as sticks, leaves, and rocks.

14.2.3 Dry sediment/soil and dry waste samples amenable to grinding: Grind or otherwise reduce the particle size of the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make the sample more amenable to grinding. Dry samples as much as possible, as water will cause un-even heating of the tubes.

14.2.4 Gummy, fibrous, or oily materials not amenable to grinding, shall be cut, shredded, or otherwise reduced in size to allow mixing and maximum exposure of the sample surfaces for the extraction. The addition of a drying agent (e.g. sodium sulfate or diatomaceous earth) can make the sample easier to mix. Wipe samples can be placed directly into the cell.

14.2.5 Weigh approximately 10.0 g of sample to the nearest 0.01 g in a 250-mL beaker and record the final weight on prep bench sheet (see FSV6-02). Add 2.5 g of diatomaceous earth to the sample. Mix well. The samples shall be a free flowing powder. If sample is not free flowing, add more diatomaceous earth and/or sodium sulfate so the sample is mixed in such a way that will allow the sample to pass through a 1 mm sieve.

14.2.6 Transfer the ground sample in a 75 mL extraction cell. There should be a minimum head space of 25%.

14.2.7 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. Prepare by adding 10.0 g of sand and 2.5g of diatomaceous earth to a clean 250 ml beaker. Transfer sample to extraction cell.

14.2.8 One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD). Select the sample and transfer approximately 40 grams to a 250 ml beaker. Mix well. Weigh three individual 10 grams aliquots of sample. Add drying agent. Transfer each sample aliquot to separate extraction cells. If there is no sample available to perform a matrix spike/matrix spike duplicate, contact project management. Default QC is a laboratory control spike duplicate.

14.2.9 To all samples, add 1.0 mL of the 8270 surrogate standard mix by using a 1.0 ml syringe. In addition, add 1.0 mL of the 8270 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

14.2.10 Add 20 ml of (1:1) methylene chloride: acetone extraction solution to each tube. Insert tube plug and attach the cap to the extractor cell, making sure the cap is straight, screw on and torque with wrench.

14.2.11 Shake each tube for 10 seconds to ensure the soil is mixed with the extraction solvent.

14.2.12 Place the extractor tube on the carousel in the appropriate slots for the number of tubes being used. Less than 16 use inside ring, greater than 16, use the outside ring then fill the inside ring. Schedule CEM Mars and begin the cycle. (NOTE: There must be a minimum of 8 samples, if less, use sand/solvent blanks to make up the shortage.)

14.2.13 Record all lot numbers, prepping analyst, times and dates on prep bench sheet (see FSV6-02)

14.2.14 Samples need to be shaken for 30 seconds to ensure sample residue is removed from tube wall prior to being poured out for concentration. Refer to section 14.6 for sample concentration.

14.2.15 Samples that are very dark or appear to have petroleum or other organic material may require cleanup prior to analysis. This will require further consultation with the analyst and/or project manager. Refer to GPC SOP Rev 0.

14.3 Waste Dilution Extraction (SW846-3580)

14.3.1 (Refer to SOP FO-10 for subsampling guidance) for samples consisting of multiphase separations.

14.3.2 Pre-rinse "Class A" 10 ml volumetric with Methylene chloride.

14.3.3 One method blank and laboratory control spike must be prepared with each batch of 20 samples or less. One sample from each batch of 20 samples or less must be selected for use in the preparation of a matrix spike (MS) and matrix spike duplicate (MSD).

14.3.4 Place the 10 ml volumetric on analytical balance (capable of accurately recording weight to the 0.001 g). Using a Pasteur pipet, transfer 1.0 g (to the nearest 0.1 g) to the volumetric. Record the weight on bench sheet (see FSV6-02).

14.3.5 Fill the volumetric half way with methylene chloride.

14.3.6 To all samples add 1.0 mL of the surrogate standard mix by using a 1.0 ml syringe. In addition, add 1.0 mL of the 8270 spiking solution to the matrix spike/matrix spike duplicate and laboratory control spike (laboratory control spike duplicate).

14.3.7 Bring samples up to volume with methylene chloride and cap for storage.

14.3.8 Add 2.0 grams of conditioned sodium sulfate to a 15ml amber vial with a Teflon cap. Transfer sample from the 10 ml volumetric flask to the 15ml vial.

14.3.9 Record all lot numbers, prepping analyst, times and dates on prep bench sheet (see FSV6-02)

14.3.10 Shake sample for two minutes.

14.3.11 Loosely pack disposable Pasteur pipets with 2-3 cm glass wool plugs. Filter the extracts through the glass wool and collect 5ml of the extract in a tube or vial.

14.3.12 No concentration step is need for this extraction. Samples that are very dark or appear to have petroleum or other organic material may require cleanup prior to analysis. This will require further consultation with the analyst and/or project manager. Refer to GPC SOP Rev 0.

14.4 Sample Concentration

14.4.1 Place glass microfiber filter paper into a glass funnel. Fill the filter paper two-thirds of the depth with Na_2SO_4. Rinse filter paper, Na_2SO_4, funnel, K-D apparatus, and concentrator tube with methylene chloride.

14.4.2 Quantitatively pour the extract through the filter and funnel seated on a 500 ml Kuderna-Danish (K-D) for water samples or a 250mL Kuderna-Danish (K-D) for soil samples, apparatus complete with concentrator tube. For Microwave extraction, shake tube for 30 seconds then pour both the extraction solution and sample matrix from the microwave tube into the funnel and filter paper seated on the K-D apparatus, being careful to not allow the extract to splash out of the funnel as the sample matrix pours into it. Rinse the beaker, VOA vial or Microwave tube three times with methylene chloride. Add these rinses through the filter and funnel into the K-D apparatus. Add a boiling chip to the K-D flask prior to placing it on the heated water bath. Wet a three ball Snyder column with approximately 2-mL of methylene chloride. Attach the Snyder column.

14.4.3 Place the K-D in the heated water bath so the concentrator tube is immersed in the water and the lower rounded surface of the K-D is bathed in steam. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood (set the knob of the temperature control to ~5 or 60ºC). It is critical that the analyst watch the extract as it distills. THE EXTRACT MUST NOT GO TO DRYNESS.

14.4.4 When the extract volume reaches approximately 5-7 mL, remove the K-D from the bath. Slightly tilt the apparatus and rotate to aid in solvent drainage from the Snyder column. Allow it to cool completely

14.4.5 Remove the Snyder column, rinse the ground glass joints with a small amount of methylene chloride and then remove the K-D flask. Turn on the heating unit for the Organomation. The water bath shall be about 35ºC. Place sample concentrator tube into the nitrogen blow down apparatus. Allow a gentle stream of nitrogen to interact with the extract. There shall be no splashing or excessive movement upon the surface of the extract. Allow the extract to evaporate down to 0.8 ml. Remove concentrator tube from water bath and by using a Pasteur pipet, bring sample extract up to 1.0 ml volume with methylene chloride.

14.4.6 Transfer the 1 mL of the extract to a labeled amber screw-cap injection vial. Record the final extract on the injection extraction bench sheet (see FSV6-02).

14.4.7 Record all lot numbers, prepping analyst, times and dates on prep bench sheet (see FSV6-02)

14.4.8 The sample extract is now ready for analysis. If samples are not analyzed immediately store the sample extract in a freezer.

14.4.9 Samples that are very dark or appear to have petroleum or other organic material may require cleanup prior to analysis. This will require further consultation with the analyst and/or project manager. Refer to attachment GPC SOP Rev 0.

15.0 Data analysis and Calculations

15.1 Sample Sequence

15.1.1 Allow the sample extract to warm to room temperature. Just prior to analysis, add 5.0 uL of the internal standard solution to 0.5 ml of the concentrated sample extract obtained from sample preparation. Alternatively, 2.0 uL of internal standard solution is added to 0.2 mL of sample extract in a vial insert.

15.1.2 Before initial calibration or sample analysis a priming standard (a standard solution at high concentration, such as the top point of the calibration curve) can be injected at a level up to twice the highest linearity point.

15.1.3 Before analysis of any samples or standards can begin, the GC/MS system must be hardware tuned so an injection (50 ng or less) of Decafluorotriphenylphosphine (DFTPP) passes the tuning criteria listed in Table 3. The DFTPP standard must also contain Pentachlorophenol, Benzidine, and DDT to assess GC column performance and injection port inertness. Degradation of DDT to DDE and DDD must not exceed 20%. Benzidine and Pentachlorophenol shall be present at their normal responses and peak tailing evaluated. Benzidine and Pentachlorophenol must each have tailing factors less than 2. These criteria must be demonstrated each 12-hour shift during which samples are analyzed.

15.1.4 Verify calibration each twelve hour shift by injecting a Continuing Calibration Verification standard (CCV), containing target analytes, prior to conducting any sample analysis. A CCV must be injected at the beginning of each twelve hour shift following the DFTPP tune. The SPCCs must meet a minimum response factor of 0.050. The percent drift of the CCCs must be less than or equal to 20%. If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large number of compounds that are analyzed by this method, it is expected that some compounds will fail to meet the criterion. In cases where compounds fail, they can still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations where the failed compound is present, the concentrations must be reported as estimated values.

15.1.5 The internal standard responses and retention times in the CCV standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last calibration check, the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the extracted ion chromatographic profile area for any of the internal standards changes by a more than a factor of two (-50% to +100%), when compared to the CCV level from the calibration, then the mass spectrometer must be inspected for malfunctions and corrections must be made. Reanalysis of CCVs and associated samples while the system was malfunctioning is necessary. The retention times and standard reference spectra in the method are updated from the CCV for each 12 hour sequence.

15.1.6 Samples can be directly injected after the successful analyses of the initial calibration curve, ICV, DFTPP, and CCV. There can be up to 20 samples in an analytical batch. A matrix spike/matrix spike duplicate and laboratory control spike must be analyzed with every analytical batch. Recoveries shall be compared to laboratory generated QC limits or client specified limits for all surrogate, matrix spike/matrix spike duplicate and laboratory control spike injections. Samples that are very dark or appear to have petroleum or other organic material may require cleanup prior to analysis. This will require further consultation with the analyst and/or project manager. Refer to attachment GPC SOP Rev 0.

15.2 Sample Calculations

15.2.1 Re-arranging the equation from sec. 10.1.5 to calculate the “as-analyzed” value yields:

$$C_s = \frac{A_s \text{ x } C_{is}}{A_{is} \text{ x } RF}$$

RF = Average Response Factor

A_s = Peak area of the analyte or surrogate.

A_{is} = Peak area of the internal standard.

C_s = Concentration of the analyte or surrogate in ug/mL.

C_{is} = Concentration of the internal standard in ug/mL.

15.2.2 Once the target components of the extract have been identified and quantitated, the "as-analyzed" value is converted to the "as-received" concentration as follows:

Water Matrix:

$$\frac{(ug/mL\ injected)\ x\,(mL\ extract\ \ final\ volume)x(dilutionfa\ ctor)}{(volume\ of\ \ sample\ extracted,\ in\ L)} = \mu g/L$$

Soil Matrix:

$$\frac{(ug/mL\ injected)\ x\,(mL\ extract\ \ final\ volume)x(dilutionfa\ ctor)}{(weight\ of\ \ sample\ extracted,\ in\ g)} = ug/g$$

16.0 Method Performance

See QAM Appendix 9.

17.0 Pollution Prevention

See QAM Appendix 9.

18.0 Data Assessment & Acceptance Criteria for QC Measures

18.1 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS, the sample extract must be diluted and reanalyzed. Additional internal standards must be added to the diluted extract to maintain the same concentration as in the calibration standards. Samples suspected of containing high levels of contamination or samples with known historical data may need to be diluted prior to analysis. Multiple dilutions may be needed to cover the entire working range of the current calibration.

18.2 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by the laboratory using the conditions of this method (SW846-8270D). The mass spectral library is updated with each new calibration. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

18.2.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

18.2.2 The relative retention times (RRT) of each compound in each calibration standard agree within 0.06 RRT units.

18.2.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum.

18.2.4 Structural isomers that produce very similar spectra are identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomeric peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs. Diastereomeric pairs that are separable by the GC are identified, quantitated and reported as the sum of both compounds by the GC.

18.2.5 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co- elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co eluting compound.

18.3 For samples containing components that are not a part of the normal target list, a library search may be required for the purpose of tentative identification. Tentatively identified compounds (TICs) are needed only when requested or required by a particular project or program. Data system library search routines shall not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Use the following as guidance for reporting TICs.

18.3.1 Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) shall be present in the sample spectrum.

18.3.2 The relative intensities of the major ions agree within ± 30%.

18.3.3 Molecular ions present in the reference spectrum shall be present in the sample spectrum.

18.3.4 Ions present in the sample spectrum but not in the reference spectrum shall be checked for possible background contamination. They shall also be reviewed for possible co elution with another compound.

18.3.5 Ions present in the reference spectrum but not in the sample spectrum shall be checked against the possibility of subtraction from the sample spectrum due to background contamination or co-eluting peaks. Some data reduction programs can create these discrepancies

18.4 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the extracted ion chromatographic profile. Quantitation is performed by the data system using the internal standard technique. The internal standard used shall be the one listed in Table 1.2.

Quantitation is performed using the RF averages from the initial calibration and not the continuing calibration check (CCV).

18.4.1 Where applicable, the concentration of any non-target analytes (TICs) identified in the sample shall be estimated. The same formulas that are used for target compounds are used with the following modifications: The areas A_x and A_{is} are from the total ion chromatograms, and the RF for the compound is assumed to be one.

18.4.2 The resulting TIC concentration is reported indicating: (1) that the value is an estimate, and (2) which internal standard was used to determine concentration. Use the nearest internal standard free of interferences.

18.5 Reporting Quantitative Analysis

18.5.1 When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the standard retention times and mass ions are compared to the sample retention times, the sample data can be reported. Assessments of all spiked and calibration control samples and standards shall also be finalized before reporting the data.

18.5.2 When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors and percent solids adjustments are made. Once the final results have been verified, a checklist (FSV6-01) is filled out and signed confirming that all the data has been thoroughly scrutinized. At this point the data is turned over to another qualified analyst for final validation. The second analyst confirms the results and electronically marks them validated and signs the checklist. Finally, the validated results are made available to the client services personnel in order for the data to be given to the client or appropriate agencies.

18.5.3 An electronic copy of the data is then filed and archived. The package includes: the sequence run log, checklist, bench sheet copy, the LIMS run log, LIMS PREP sheet, verification of calibration data and chromatograms/quant reports. The sequence run log and the analytical run are e-initialed and dated by the analyst. Each sequence file header is labeled with the date of sequence.

19.0 Corrective Measures for Out-of-Control Data

See QAM Appendix 9.

20.0 Contingencies for Handling Out-of-Control or Unacceptable Data

See QAM Appendix 9.

21.0 Waste Management

See QAM Appendix 9.

22.0 Equipment/Instrument Maintenance, Computer Hardware & Software & Troubleshooting

See QAM Appendix 9.

23.0 References

23.1 CT Laboratories Quality Manual, current revision.

23.2 Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.

23.3 National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.

23.4 ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

23.5 USEPA, SW-846, Method 8000C, Rev. 3, March 2003.

23.6 USEPA, SW-846, Method 8270D Rev. 4, January 1998.

23.7 USEPA, SW-846, Method 3510C Rev. 3, December 1996.

23.8 USEPA, SW-846, Method 3545A Rev. 1, February 2007.

23.9 USEPA, SW-846, Method 3546 Rev. 0, February 2007.

23.10 USEPA SW-846, Method 3580A, Rev. 1, July 1992

23.11 USEPA SW-846, Method 3640A Rev. 1, September 1994.

24.0 Tables, Diagrams, Flowcharts And Validation Data

Table 5
Semivolatile Organic Compounds - Method 8270D Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Tune Check (50ng or less DFTPP)	Every 12 hours.	Ensure correct mass assignment. DFTPP % Relative abundance criteria as specified in Table 4. Pentachlorophenol tailing ≤ 2, Benzidine tailing ≤ 2 DDT breakdown ≤ 20%.	Retune. Do not proceed with analysis until DFTPP spectrum meets criteria.
Initial Calibration	Each time the instrument is set up and when CCCs and SPCCs in the continuing calibration verification (CCV) do not meet criteria.	1. Average relative response factors (RRFs) for SPCCs (See Table 4.1) 2. % RSD for RRFs for all target compounds ≤15%. IF RF % RSD >15% use linear curve, r ≥.995, or quadratic curve, r2 ≥.990. 3. LGC, NELAC, QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.)	Correct system and recalibrate. Criteria must be met before sample analysis can begin. Any samples reported from data not meeting these criteria must be qualified (Z).
Initial Calibration Verification standards (ICV)	Immediately following the ICAL.	1. Second source (different lot or manufacturer than ICAL). 2. RRF for SPCCs ≥ minimum from Table 4.1 3. % Deviation. for RRFs for all target compounds ≤20%. ≤ 20% drift for linear curve and non-linear curves. 3 LCG,QSM, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	Correct system and recalibrate. Criteria must be met before sample analysis can begin. If %drift >20% then confirm the integrity of the second source standard by reanalysis, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. Sample results reported that have %drift failures must be qualified (Z). QSM allows no tolerances for % D. Problem compounds need to be addressed on a project to project basis.

Table 5 (Continued)
Semivolatile Organic Compounds - Method 8270D Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification standards (CCV)	Every 12 hours.	1. RRF for SPCCs ≥ minimum from Table 4.1 2. All target compounds- ≤20% Deviation for RRFs, ≤20 % Drift for linear curve and non linear curves- 3. LCG, QSM, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria are followed as stated in their documents.	Correct system and recalibrate. Criteria must be met before sample analysis can begin. If% drift >20% correct problem if determinable then reanalyze, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. Sample results reported that have %D failures must be qualified (Z). QSM allows no tolerance for % D. Problem compounds need to be addressed on a project to project basis
Internal Standards (ISTD)	Added to all blanks, standards, and samples.	1. Peak area within -50% to +100% of area in CCV level of ICAL. 2. Retention time (RT) within 30 sec of RT for associated CCV standard. 3. LCG, QSM, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria are followed as stated in their documents.	Inspect instrument for malfunctions; correct identified malfunctions, then reanalyze samples. If no instrument malfunction identified proceed as follows: * Re-extract and reanalyze sample, or reanalyze sample at a dilution. * If reanalysis is outside limits the data is qualified (S). Follow specified criteria as stated in Shell or other documentation.
Method Blank (MB)	One per prep batch/20 samples per matrix. The MB is used to document contamination resulting in the analytical process and is carried through the complete sample preparation and analytical procedure.	1. Concentration of analytes of concern are to be less than the highest of either: Method detection limit, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample. (10% for QSM) 2. ACOE/QSM: <1/2 MRL. 3. Follow criteria according to specific program/agency.	Reanalyze to determine if instrument or laboratory background contamination was the cause. If the method blank is still non-compliant, re-prepare and reanalyze blank and samples. For ACOE/QSM data, if <1/2 MRL no action required. If no sample remains for re-prepping, or if re-prepped data still contains contamination, flag data with (B) qualifier.

Table 5 (Continued)
Semivolatile Organic Compounds - Method 8270D Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Sample (LCS)	One per prep batch of 20 samples. Must undergo all sample preparation procedures. Spiking solution are to contain all target compounds with concentrations at or near the mid-point of the calibration range.	1. Client specified limits. 2. QSM – use LCS criteria. 3. In-house limits.	If LCS recoveries are within control limits then no action is required. If the LCS exceeds control limits, reanalyze the LCS. If LCS recoveries are still outside control limits, re-extract and reanalyze samples. If sample is not available for re-extraction then qualify data for the failing analytes with a (Q). Exception: If the LCS recoveries are high with no associated positives then no further action is taken.
Matrix Spike/Matrix Spike Duplicate	One set per prep batch of 20 samples. Must undergo all sample preparation procedures. Must be spiked with target compounds with concentrations at or near the mid-point of the calibration range.	1. Client specified limits. 1. QSM – use LCS criteria. 3 .In-house limits..	If LCS is acceptable, then report probable matrix interference. Qualify data if the recoveries are low (M). If recoveries are high and there are no detects in the un-spiked sample then that data does not require flagging. Qualify data for RPD failures (Y) when there is a detect for the failing compounds (non-detected compounds are not qualified). Exception: If a compound is already qualified for a LCS failure then no RPD qualifier is applied.

Table 5 (Continued)
Semivolatile Organic Compounds - Method 8270D Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Qualitative/Quantitative Issues	If detection level of any compound in a sample exceeds the detection level of that compound in the highest level standard, the sample must be diluted to approximately mid-level of the calibration range and reanalyzed.	The instrument level of all compounds must be within the calibration range for all samples. The sample analyzed immediately after a high level sample must display concentrations of the high level target compounds less than the RL or greater than 5X the RL	Dilute the sample to bring the level of the highest concentration of target compounds within the calibration range. If any data is reported with any results over range then those results are to be flagged (X). A sample displaying concentrations of target compounds between the RL and 5x the RL that was analyzed immediately after a high level sample must be re-analyzed. If the results do not agree within the RL, report only the second analysis.
Surrogate	1. Calibrated as target compounds. 2. Added to all blanks, samples, and QC samples, as a part of the internal standard-surrogate spiking mixture.	1. Client specified limits. 2. QSM – use LCS criteria. 3. In-house limits.	Rerun sample. If no apparent matrix interference is noticed, re-extract sample. If no sample is available, qualify the surrogate with "S". QSM – For QC and field samples, correct problem, re-prep and re-analyze all samples with failed surrogates in the associated batch, if sufficient sample material is available.
Retention Time Window (RTW)	Retention Times will be set using the midpoint of the calibration curve or the RTs in the CCV run at the beginning of the analytical sequence.	RTs of analytes must be within +/-.06 RRT units of the RRT of the CCV.	.

FSV6-01
8270D Analysis Data Review Checklist (Example)

8270D Analysis Data Review Checklist

Sequence Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes or No

Instructions: Complete one checklist per analytical run. Enter the appropriate response for each question. Each "No" response requires an explanation in the
Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. INITIAL CALIBRATION (ICAL)						
a. Was the initial calibration performed using a minimum of five standard concentration levels?	Lowest standard at or near MRL					
b. SPCC responses.	Avg. RRF ≥ Table 4.1					
c. Linearity.	RSD≤ 15%, or r ≥ 0.995, r2 ≥ 0.990 for regression.					
d. Were the standards used for the ICAL uniquely identified?						
e. Was there a DFTPP standard analyzed prior to the ICAL?						
2. INITIAL CALIBRATION VERIFICATION (ICV)						
a. Were there a second source ICVs for all target analytes analyzed after the initial calibration and prior to analysis of any samples?	Second source					
b. Were the SPCC within QC limits	RRF ≥ Table 4.1					
c. Were all compounds within QC limits	%D ≤ 20%					
d. Were the ICVs uniquely identified (i.e. Standard Number)?						
3. CONTINUING CALIBRATION VERIFICATION (CCV)						
a. Were CCVs for target analytes analyzed at the beginning of the sequence and after every 12 hours.						
b. Were SPCC compounds acceptable?	RRF ≥ Table 4.1					

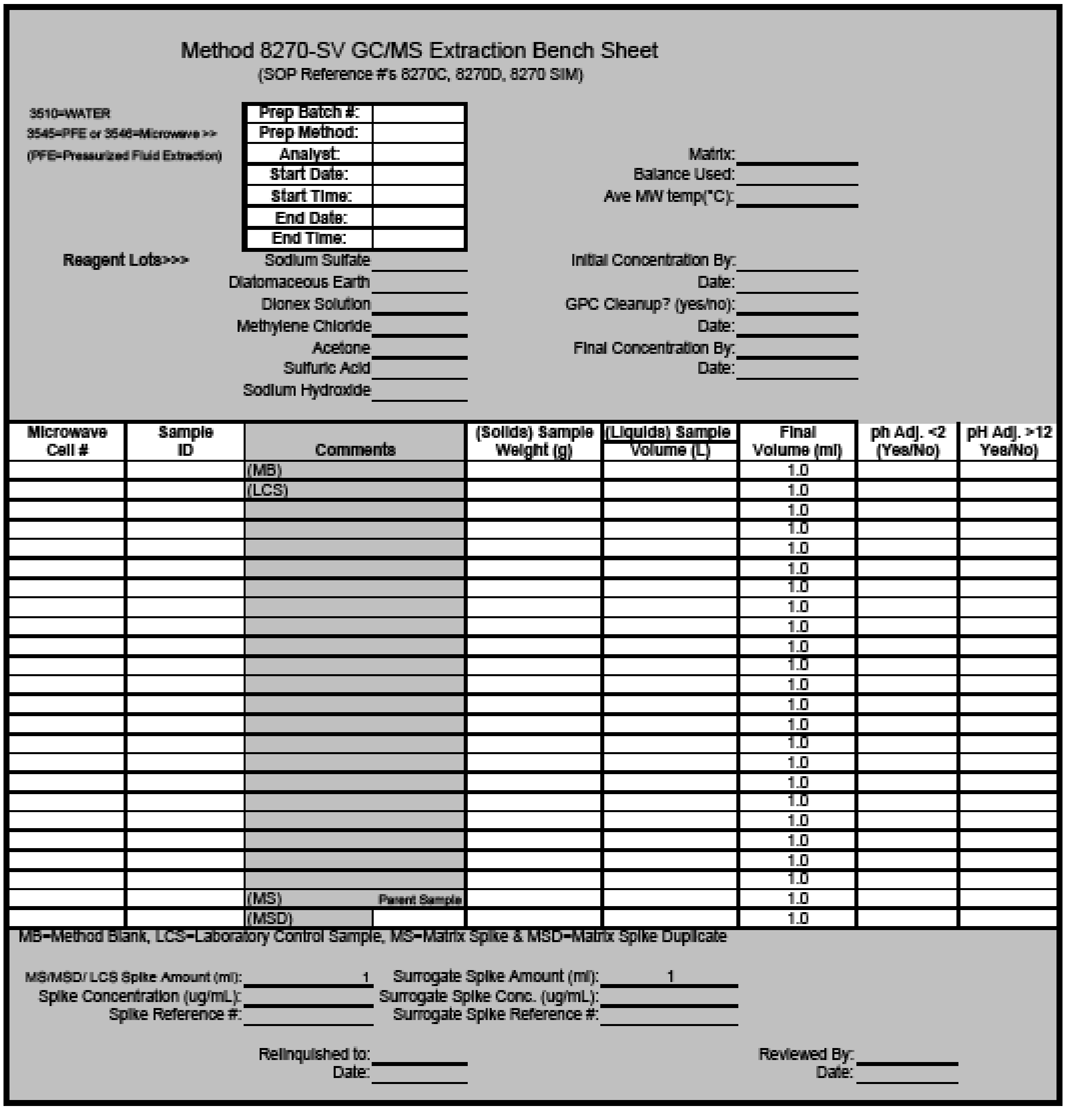
FSV6-01
8270D Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
c. Were the recoveries for the CCVs acceptable?	%D≤20%,					
d. Was each CCV uniquely identified (i.e. Standard Number)?						
4. DFTPP						
a. Was a DFTPP tune check ran at the beginning of every twelve hour shift?						
b. Were the relative abundance criteria met?						
c. Was the peak tailing acceptable for Pentachlorophenol and Benzidine?	Tailing factor ≤ 2					
d. Was the breakdown of DDT to DDE and DDD acceptable	≤ 20%					
5. BLANKS						
a. Was method blank (MB) analyzed prior to the analysis of samples?						
b. Were the MB results less than the detection limit (MDL)?	< MDL,(QSM<1/2RL)					
If no, were positive hits in the samples < 20x the amount in the blank flagged with a "B". (<10X for QSM).	< 20x (qualify data) > 20x (no action)					
c. Was a MB prepped and analyzed at a frequency of one per Prep Batch?	Batch ≤ 20 samples					
6. LABORATORY CONTROL SAMPLE (LCS)						
a. Was a LCS analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were the LCS recoveries in each LCS within the acceptance criteria?	In-house limits or client specified limits					
If no, and the recoveries were low, flag those analytes "Q". If the recoveries were high, only flag the detects (>RL) for those analytes "Q".						
7. MATRIX SPIKES						
a. Was a matrix spiked (MS) sample analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were MS recoveries in each MS within the acceptance criteria?	In-house limits or client specified limits					

FSV6-01
8270D Analysis Data Review Checklist (Continued)

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
8. LABORATORY CONTROL SPIKE / MATRIX SPIKE DUPLICATE						
a. Was a duplicate matrix spike or laboratory control spike sample analyzed at a frequency one per Prep Batch?	Batch ≤ 20 samples					
b. Were MSD or LCSD recoveries within the acceptance criteria?	In-house limits or client specified limits					
c. Is the relative percent difference (RPD) for each analyte between a matrix spike (MS) and matrix spike duplicate (MSD) within the acceptance criteria? (same criteria for LCS and LCSD)	In-house limits or client specified limits					
9. SAMPLES (INCLUDING BLANKS, STANDARDS, AND QC SAMPLES)						
a. Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?						
b. Are surrogate recoveries for all samples, blanks, standards, and QC samples within acceptance criteria?						
c. Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed?						
d. Were all samples extracted within holding times and analyzed within 40 days of extracting?	Analysis within 40 days of extraction					
e. Did the samples require additional cleanup steps? (i.e. GPC)	GPC, Treatments					
10. RECORDS AND REPORTING						
a. Are Run, Prep Batch and Extraction sheets, Summary sheets, Sequence file, initial and rerun raw and process data present in the data file?						
b. Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, a NCR completed?						
c. Do all values, dilution factors and qualifiers listed on the raw reports match the LIMS data?						
d. Is the ICAL method referenced on the Raw Data?						

FSV6-02
Semivolatiles 8270 Extraction Bench Sheet (Example)



Method 8270-SV GC/MS Extraction Bench Sheet
(SOP Reference #'s 8270C, 8270D, 8270 SIM)

3510=WATER
3545=PFE or 3546=Microwave >>
(PFE=Pressurized Fluid Extraction)

Prep Batch #:	
Prep Method:	
Analyst:	
Start Date:	
Start Time:	
End Date:	
End Time:	

Matrix: ____
Balance Used: ____
Ave MW temp(°C): ____

Reagent Lots>>>
Sodium Sulfate ____
Diatomaceous Earth ____
Dionex Solution ____
Methylene Chloride ____
Acetone ____
Sulfuric Acid ____
Sodium Hydroxide ____

Initial Concentration By: ____
Date: ____
GPC Cleanup? (yes/no): ____
Date: ____
Final Concentration By: ____
Date: ____

Microwave Cell #	Sample ID	Comments	(Solids) Sample Weight (g)	(Liquids) Sample Volume (L)	Final Volume (ml)	ph Adj. <2 (Yes/No)	pH Adj. >12 Yes/No)
		(MB)			1.0		
		(LCS)			1.0		
					1.0		
					1.0		
					1.0		
					1.0		
					1.0		
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					1.0		
					1.0		
					1.0		
					1.0		
					1.0		
					1.0		
					1.0		
		(MS) Parent Sample			1.0		
		(MSD)			1.0		

MB=Method Blank, LCS=Laboratory Control Sample, MS=Matrix Spike & MSD=Matrix Spike Duplicate

MS/MSD/ LCS Spike Amount (ml): 1
Spike Concentration (ug/mL): ____
Spike Reference #: ____

Surrogate Spike Amount (ml): 1
Surrogate Spike Conc. (ug/mL): ____
Surrogate Spike Reference #: ____

Relinquished to: ____
Date: ____

Reviewed By: ____
Date: ____

8270 Prep 4/10/20122:44 PM

Revision Number	**Description of Changes**	**Date**
00	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	04/22/2014

STANDARD OPERATING PROCEDURE

VO 004 Analysis of Volatile Organic Compounds by GC/MS (8260C)

Review Date: 03/31/2016

Randy

03/09/2015

Technical Review by: Date

Colleen

04/01/15

Approved by: Quality Assurance Date

Table of Contents

1. SCOPE OF APPLICABILITY

1.1. This method is used to quantify Volatile Organic Compounds (VOCs) with boiling points below 200° Celsius (°C) in water and soils. See Table 1 for typical target analyte list (TAL). This method is designed to follow procedures and QC requirements found in EPA SW-846 method's EPA 624, 5030B, 5035, 8000C and 8260C in order to determine quantities of volatile organic compounds found in a variety of different sample.

1.2. The scan mode is utilized by the instrument's software to identify and quantitate analysis results. When collecting data in the full scan mode, a target range of mass fragments is determined and put into the instrument's method. An example of a typical broad range of mass fragments to monitor would be *m/z* 35 to *m/z* 300. The determination of what range to use is largely dictated by what one anticipates being in the sample while being cognizant of the solvent and other possible interferences. A MS should not be set to look for mass fragments too low or else one may detect air (found as *m/z* 28 due to nitrogen), carbon dioxide (*m/z* 44) or other possible interferences. Additionally if one is to use a large scan range then sensitivity of the instrument is decreased due to performing fewer scans per second since each scan will have to detect a wide range of mass fragments. Full scan is useful in determining unknown compounds in a sample. It provides more information than Selected Ion Monitoring (SIM) when it comes to confirming or resolving compounds in a sample. During instrument method development it may be common to first analyze test solutions in full scan mode to determine the retention time and the mass fragment fingerprint before moving to a SIM instrument method.

1.3. Volatile organic compounds are quantitated from a variety of matrices. This method is applicable to nearly all types of samples regardless of water content, including ground water, surface water, wastewater, soils, sediments, and TCLP/SPLP extracts; as well as, other matrices noted in SW-846 method 8260C.

1.4. Examples of other compounds which have been analyzed by this method include: iodomethane, 2,3-dichloro-1-propene, 1-chlorohexane, acrolein, acrylonitrile, 1,1,2-trichloro-1,2,2-trifluoroethane, ethyl ether, hexane, ethyl acetate, 1-chlorohexane, 2-Chloroethylvinyl ether, methyl acetate, methyl methacrylate, cyclohexane, and cyclohexanone. Ethanol, 2-propanol, tert-butylalcohol, 1,4-dioxane may also be analyzed using this method but are poor responders. To achieve lower detection limits for these types of compounds, the SIM mode can be utilized. In selected ion monitoring certain ion fragments are entered into the instrument method and only those mass fragments are detected by the mass spectrometer. The advantages of SIM are that the detection limit is lower since the instrument is only looking at a small number of fragments (e.g. three fragments) during each scan. More scans can take place each second. Since only a few mass fragments of interest are being monitored, matrix interferences are typically lower. To additionally confirm the likelihood of a potentially positive result, it is relatively important to be sure that the ion ratios of the various mass fragments are comparable to a known reference standard.

1.5. SW-846 method 8260C notes a number of other compounds amenable to this test.

2. SUMMARY OF METHOD

2.1. A Purge & Trap system (including autosampler), a Gas Chromatograph (GC), and a Mass Spectrometer (MS) are utilized for the detection of VOCs. The autosampler introduces the sample to the purge and trap concentrator. The concentrator then removes the volatile constituents by purging the sample with an inert gas (helium or nitrogen). The constituents are then collected onto an adsorption trap. The trap is then rapidly heated and the volatilized compounds are introduced to the GC. The GC is temperature programmed to facilitate separation of the individual organic compounds. Finally the separated compounds enter the MS (which is interfaced with the GC) for quantitative and qualitative analyses.

2.2. Utilizing computer software, identification of target analytes is accomplished by comparing the mass spectra of the sample constituent with that of commercially purchased standards. Quantitation is achieved by comparing the response of a quantitation ion relative to an internal standard using a five point (minimum) calibration curve.

3. DEFINITIONS

3.1. For definitions on all terms applicable to this method, see Section 25.1 of the Quality Assurance Manual (QAM).

3.2. For a list of common acronyms and abbreviations, see QAM.

4. HEALTH AND SAFETY

4.1. Gloves and protective clothing shall be worn to protect against unnecessary exposure to hazardous chemicals and contaminants in samples. All activities performed while following this procedure must utilize appropriate laboratory safety systems.

4.2. The toxicity and carcinogenicity of the chemicals used in this method are not precisely defined. Each chemical and sample shall be treated as a potential health hazard, so care must be taken to prevent undue or extensive exposure.

5. INTERFERENCES

5.1. Volatile materials in the laboratory and impurities in the purging gas and sorbent trap can cause significant amounts of background contamination. Improper tubing such as certain plastics and rubber shall not be used. The analysis of IBs and MBs will indicate as to whether or not this type of contamination is present. Since subtraction on background contamination is not allowed, care must be taken to eliminate this type of contamination.

5.2. Carry over contamination is a problem when a highly contaminated sample is followed by a clean sample. Rinsing the autosampler and concentrator and adequate baking of the trap can greatly reduce contamination from carry over.

5.3. Some samples contain a lot of water soluble materials, suspended solids, compounds with high boiling points, or target analytes with very high concentrations which may contaminate some or all of the analytical system. Removing components of the system for cleaning or cleaning of the entire system may be required to eliminate the interferences.

5.4. Compounds with poor purging efficiencies may remain in the purge system, particularly with 25 ml purges. Ensuring adequate rinsing and increased line temperatures will help reduce this problem.

5.5. All chromatography gas/purge lines shall be stainless steel or copper to prevent permeation from possible background contaminants (i.e. Methylene chloride). Background levels of Methylene chloride are possible so care needs to be taken to reduce this possibility. Analyst clothing previously exposed to Methylene chloride must not be worn and isolating the instruments from possible air born contamination is essential in reducing Methylene chloride background contamination.

5.6. A trip blank normally accompanies sample in shipment and storage as a check on possible contamination from volatile organics by diffusion through the septum seal in sample vials/containers.

5.7. Mass spectrometer sensitivity, column degradation, and contamination can also contribute to background interferences. A proper maintenance procedure on instrumentation is essential to continually producing quality data. Maintenance manuals are provided with each piece of equipment and are essential for proper instrument care. The presence of semi-volatile hydrocarbons need also be taken into consideration, so appropriate post analysis bake out times need to be incorporated.

5.8. Cross-contamination can be a possibility when samples containing high concentrations of target analytes are stored in the same location as other samples. To prevent cross-contamination, samples suspected of containing high concentrations of volatiles organics should be isolated from other volatile organics samples. Storage Blanks are analyzed bi-weekly to determine whether cross-contamination has occurred.

6. EQUIPMENT AND SUPPLIES

6.1. 40 ml screw cap "VOA" vials-borosilicate glass with a Teflon faced silicone septum (C&G or equivalent)

6.2. 2 oz., 4 oz., or 60- ml Teflon lined screw top sample jars (C&G or equivalent).

6.3. 5 g or 25 g samplers for low level soils (Encore).

6.4. Top loading balance sensitive to 0.01 g (Mettler-Toledo, BD202).

6.5. pH paper to confirm water sample preservation(Color pHast, EM Reagents).

6.6. Stainless steel spatulas.

6.7. 10, 25, 50, 100, 500, and 1000 ul gas tight syringes for sample dilutions and standard preparation (Hamilton or equivalent).

6.8. 5.0, 10.0, 25.0, 50.0 ml syringes with luer-lok tips for methanol preserved soil sample preparation and sample dilutions (Hamilton/SGE or equivalents).

6.9. 10, 50 100, 200, 1000, and 2000 ml Volumetric flask for sample dilutions and standard preparation (Class A, Pyrex/Kimble or equivalents).

6.10. Auto pipetter – 2.5 to 25.0 ml – for dispensing methanol (Dispensette).

6.11. Sonicator used for methanol-preserved soil sample extraction (Fisher, FS-28 or equivalent).

6.12. Auto sampler used for sample introduction to the Purge and Trap (Archon, EST-Centurion or equivalents).

6.13. 3 ml standards vial (Mininert or equivalent).

6.14. Purge and Trap concentrator (EST-Encon Evolution or equivalent).

6.14.1. The glass purging tubes are of 5 ml or 25mL size. The all-glass purging device shall be designed to accept 5 or 25 ml samples with a water column at least 5 cm deep. The smaller (5 ml) purging device is recommended if the GC/MS system has adequate sensitivity to obtain the method detection limits required for a specific project or program.

6.14.2. The traps currently used are Supelco Type K or EST-EV1. As required by SW-846 methods, the trap must be at least 25 cm long and have an inside diameter of at least 0.105 inches. Starting from the inlet, the trap contains 1.0 cm of methyl silicone coated packing and the following amounts of adsorbents: 33% of 2,6-diphenylene oxide polymer, 33% of silica gel, and 33% of coconut charcoal.

6.15. Gas Chromatograph/Mass Spectrometer Data Systems (GC/MS).

6.15.1. Hewlett Packard Gas Chromatographs (5890 & 6890).

6.15.1.1. Columns, Supelco (SPB-624), Agilent (DB-624UI), or Zebron (ZB-624).

6.15.1.2. 30 Meter x 0.25 mm ID, 1.4 um film thickness or equivalents.

6.15.2. Hewlett Packard 5972 & 5973 Mass Spectrometers.

6.15.3. Hewlett Packard Chemstation Data Management System (version G1701AA v. A.03.02 for the 5972's) and MSD Chemstation (version D.01.02.16.15 for the 5973) with Enviroquant and Prolab data processing software.

7. REAGENTS AND STANDARDS

7.1. Purge and trap grade methanol: (Fisher, Purge & Trap grade or equivalent), stored in laboratory warehouse.

7.2. Reagent grade water, organic free (Milipore, 18 mega ohm quality).

7.3. Certified Calibration Standards:

7.3.1. (VOC Mix--2000 ug/ml, Ultra Scientific--#DWM-588; Addition mixes--1000/10,000 ug/ml, SPEX Certiprep--#'s VO-CTWI-4 & VO-CTWI-5 or equivalents), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.3.2. A 100 ug/mL[1] Continuing Calibration Verification (CCV/Calib.) working standard is prepared by adding 150 ul of the VOC mix and 300 ul of

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Addition mix VO-CTWI-4 and 300 ul of Additions Mix VO-CTWI-5 to 2250 ul of methanol into a 3 ml Mininert vial.

7.3.3. Calibration standards used for SIM mode calibrations can be prepared by further dilution of the working standards (7.3.2) or by purchasing individual compound standards (e.q. 1000 ug/mL 1,4-dioxane, SPEX Certiprep--#S175 or equivalent). For t-butyl alcohol and 1,2-dioxane the working CCV/Calib. standard concentration is 100 ug/ml.

7.4. Certified Calibration Check Standards:

7.4.1. (VOC Mix--2000 ug/ml, Accustandard--#M-502-10X; Addition mixes--1000/10,000 ug/ml, SPEX Certiprep--#'s VO-CTWI-4 & VO-CTWI-5 or equivalents), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.4.2. A 100 ug/mL[1] Initial Calibration Verification (ICV/Spiking) working standard is prepared by adding 150 ul of the VOC mix and 300 ul of Addition mix VO-CTWI-4 and 300 ul of Additions Mix VO-CTWI-5 to 2250 ul of methanol into a 3 ml Mininert vial. The ICV standard is prepared from standards of a different manufacturer or different lot than the standards used for calibration.

7.4.3. ICV standards used for SIM mode calibrations can be prepared by further dilution of the working standards (7.4.2) or by purchasing individual standards (e.q. 1000 ug/mL 1,4-dioxane, SPEX Certiprep--#S175 or equivalent). For t-butyl alcohol and 1,2-dioxane the working ICV/Spiking standard concentration is 100 ug/ml.

7.5. Certified Internal Standards (ISTD) and Surrogate Standards (SSTD):

7.5.1. ISTD/SSTD Mix (2500µg/mL): Ultra Scientific catalog # STM-540 or equivalent, stored in the Volatiles Standards Freeze at ≤ 10°C.

7.5.2. SSTD [2] 1,2-Dichlorobenzene-d_4 (2000µg/mL): Ultra Scientific catalog # STS-210 or equivalent, stored in the Volatiles Standards Freezer at ≤ 10°C.

7.5.3. A 20µg/mL ISTD/SSTD Working Standard is prepared by adding 200µL of ISTD/STD Mix and 250µL of SSTD 1,2-Dichlorobenzene-d_4 to 25mL of MeOH.

7.6. Certified Internal Standards (alternative to section 7.5 and used only with the Archon autosamplers):

7.6.1. (ISTD Mix--2500 ug/ml, Restek--#30241 or equivalent), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.6.2. An 80 ug/ml ISTD working standard is prepared by adding 320 ul of the ISTD mix to a 10 ml volumetric flask and brought to volume with methanol.

7.7. Certified Surrogate Standards (alternative to section 7.5 and used only with the Archon autosamplers):

7.8.

7.8.1. (SSTD Mix—2500 ug/ml, Restek--#30240; 1,2-DCA-d_4 --2000 ug/ml, Ultra Scientific--#STS210, or equivalents[2]), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.8.2. A 100 ug/ml SSTD working standard is prepared by adding 120 ul of the SSTD mix and 150 ul of 1, 2-DCA-d_4 to 2730 ul of methanol in a 3 ml mininert vial.

7.8.3. An 80 ug/ml ISTD/SSTD working standard is prepared by adding 320 ul of the ISTD Mix (sec. 10.5), 320 ul of the SSTD Mix (sec. 10.6), and 400 ul of 1,2-DCA-d_4 (sec. 10.6) to a 10 ml volumetric flask and brought to volume with methanol.

7.8.4. A 16 ug/ml ISTD/SSTD working standard is prepared by adding 2 ml of the 80 ug/ml ISTD/SSTD (sec. 10.6.2) to a 10 ml volumetric flask and brought to volume with methanol.

7.9. Certified Tuning Standard:

7.9.1. 4- bromofluorobenzene {BFB} (Ultra Scientific—2000 ug/ml, #STS-110N or equivalent), stored in VOC Standards Freezer in Volatiles laboratory at ≤ -10 °C.

7.9.2. A 50 ug/ml working standard is prepared by adding 75 ul of the certified standard to 2925 ul of methanol in a 3 ml mininert vial.

7.10. Sodium bisulfate (JT Baker--#3534-01 or equivalent), stored in cabinet in Volatiles laboratory.

7.11. All certified stock standards use the expiration date provided by the manufacturer/supplier.

7.11.1. The working standards (not including gases) expire one month after preparation. These standards include the BFB, ISTD, SSTD, and/or ISTD/SSTD.

7.11.2. The working standards (which include gases) expire one week after preparation. These standards include the ICV and CCV. When standards used for calibration are prepared from freshly open stock standard vials, the expiration of working standards used from that point on can be extended if the integrity of those standards can be confirmed and documented. For example, if a CCV/ICV standard continues to produce acceptable results after one week from preparation, it can be assumed still valid.

[1] Due to lower response or purging efficiencies, a number of compounds are purchased and prepared at concentrations greater than 100 ug/ml. Those compounds and concentrations are noted on the calibration curve.

[2] This surrogate compound is needed for Method 524.2 and is not used for this method/SOP.

8. Sample Handling and Preservation.

8.1. Water samples are stored at 0-6°C. The sample storage area must be free of organic solvent vapors and direct or intense light. Samples are stored in the Volatiles lab in a double door refrigerator (separate from analytical standards).

8.1.1. Analyze properly preserved samples (pH <2) samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected. If samples are not preserved then they must be noted (or qualified) as improperly preserved if not analyzed within 7 days.

8.1.2. Samples analyzed for Acrolein and Acrylonitrile are to be preserved at a pH of 4-5 and analyzed within 14 days (3 days if unpreserved).

8.1.3. If reactive compounds such as 2-Chloroethyl vinyl ether are target compounds than no preservatives are added and the sample needs to be analyzed as soon as possible.

8.1.4. Samples containing residual chlorine require alternative preservation (ascorbic acid or sodium thiosulfate) to reduce the chlorine. These sample shall be reduced to a pH of <2 (using HCL or $NaHSO_4$) to meet the 14 day is the hold time.

8.2. Soil samples are stored at 0-6° C. The sample storage area must be free of organic solvent vapors and direct or intense light. Samples are stored in a double door refrigerator located in the laboratory warehouse.

8.2.1. Samples received for low level analysis in “Encore” samplers must be preserved within 48 hours from time of collection. To preserve a sample, weigh it into a 40 ml VOA vial, record the weight, and then add 0.2 grams of sodium bisulfate per 1.0 gram of sample. Finally add 5.0 ml of DI H_2O and a stir bar. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

8.2.2. Samples received for low level analysis in “Terra Core” sampler vials are already preserved with bisulfate at 0.1 g per 1.0 gram of sample. Samples that are received for low level analysis in DI water are placed in a freezer at ≤-10° C. The pre-weighed vial weight (tare weight which includes the weight of vial + 5 ml of preservative/DI water & a stir bar) is subtracted from the total weight of the vial to determine sample weight. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

8.2.3. Samples received in filled 2 oz. or 4 oz. jars can be weighed and prepared for low level analysis as described in section 11.2.1. or they can be weighed into a VOA vial and preserved at a 1:1 ratio with methanol for medium/high- level analysis. Analyze all samples within 14 days of collection. Samples not analyzed within this period must be discarded and recollected.

8.2.4. Samples collected and preserved with methanol in the field in pre-weighed 60-ml jars are weighed as is. The pre-weighed jar weight, as well as the methanol weight (19.8 grams for 25 ml of methanol) is subtracted from the total weight of the jar to determine sample weight. For Wisconsin LUST samples if the weight to volume ratio is more than 1:1 then methanol is added using the auto-pipetter to correct the ratio to 1:1. Unless instructed by the client to do otherwise, the maximum acceptable weight for volume correction is 35 grams. If samples are being analyzed for the Wisconsin LUST program then the hold time is 21 days from collection. Otherwise hold time is 14 days.

8.2.5. Samples collected and preserved in the field using “Terra Core” sample vials are weighed as is. The pre-weighed jar weight (tare weight which includes weight of the vial + MeOH) is subtracted from the total weight of the vial to determine sample weight. Unless instructed to do so samples are not adjusted for volume to weight differences. Analyze sample within 14 days from collection.

8.2.6. All soil samples are weighed on the top loading balance which is connected to a computer so that all weights can be automatically entered onto an Excel spread sheet. The Excel spreadsheet is set up to record the weights as well as calculate the methanol to weight differences. The spreadsheets are saved so the data can be transferred electronically to the LIMS system. See forms FVO4-(2-7) for examples of the sample weight spreadsheets.

8.2.7. Each prepared methanol soil sample is then placed on a shaker table for 2 minutes and then sonicated for 20 minutes prior to preparation for analysis.

8.3. Most samples received are accompanied with a Trip Blank (TB). In most cases the TBs are prepared by the lab and are sent along with the vials used for sample collection. The intent of the TB is to accompany the sample vials through all collection, preservation, shipping, and storage procedures. The infusion of outside contamination in the TB is not common, but can be an indicator of incorrect preparation/sampling procedures or inadequate sample storage.

9. PROCEDURE

9.1. Prior to sample analysis a GC/MS tune and calibration check must be analyzed. Verify the MS tune and initial calibration at the beginning of each 12-hour work shift during which analyses are performed.

9.1.1. Introduce into the GC (by direct injection) 25 to 50 ng of BFB and acquire a mass spectrum that includes data for m/z 35-260. If the spectrum does not meet all criteria, the MS must be retuned and adjusted to meet all criteria before proceeding with the continuing calibration check.

9.1.2. The calibration curve integrity for each analyte must be confirmed with the use of a CCV standard once every 12 hours of analysis time. The CCV standard is prepared at concentrations near the midpoint of the calibration curves (10/100 ug/L for water-5 ml purge, 4.0/40 ug/L for water-25 ml purge, 0.010/0.10 mg/kg for low level soils, and 0.50/5.0 mg/kg for MeOH preserved soils). The QSM recommends the CCV's to be varied throughout an Analytical run. Typically the concentrations used are 10, 20, and 30 ppb for Low Level soil and water-5 ml purged; 0.5, 1.0 1.5 mg/kg for MeOH preserved soil; and 1.0, 2.0, and 4.0 ppb for water-25 ml purged (preparation procedures are the same as listed 14.1.3, using the appropriate amount of the 100/1000 ug/ml CCV Std).

9.1.3. QSM 5.0 states a CCV must be analyzed at the beginning of the sequence, every 12 hours, and at the end of each analytical batch. The criteria for the ending CCV is all targets analytes within 50%. If a compound in the ending CCV fails, then 2 additional CCV's may be analyzed within a 60 minute timeframe. If both CCV's have acceptable recoveries, then the data can be reported without qualification.

9.1.4. The CCV is placed on the autosampler in the same manner as the samples (sec. 14.2.3). Preparation of CCV's is as follows:

9.1.4.1. **Water (5 ml purge)** -- Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer to a VOA vial for analysis.

9.1.4.2. **Water (25 ml purge)** -- Spike 50 ml of DI water (volumetric flask) with 2.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer into a VOA vial for analysis.

9.1.4.3. **Low-level Soils** -- Spike 50 ml of DI water(volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer 5.0 ml into a VOA vial (containing and a stir bar) for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA vial (containing and a stir bar) for analysis.

9.1.4.4. **Med/high-level soils --** Spike 49 ml of DI water (volumetric flask) with 1.0 ml of MeOH and 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer into a VOA vial for analysis.

9.1.5. Each of the most common target compounds in the CCV should meet the minimum RFs as noted in Table 4. This is the same check that is applied during the initial calibration (sec. 13.4). If the minimum RFs are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins.

9.1.6. All target compounds of interest must be evaluated using a 20% variability criterion. Use percent deviation when performing the ARF model calibration. Use percent drift when calibrating using a regression fit model. If the percent difference or percent drift for a compound is less than or equal to 20%, then the initial calibration for that compound is assumed to be valid. Due to the large number of compounds that are analyzed by this method, it is expected that some compounds will fail to meet the criterion. In cases where compounds fail, they can still be reported as non-detects if it can be demonstrated that there was adequate sensitivity to detect the compound at the applicable quantitation limit. For situations where the failed compound is present, the concentrations must be reported as estimated values.

9.1.7. The internal standard responses and retention times in the CCV standard must be evaluated immediately after or during data acquisition. If the retention time for any internal standard changes by more than 30 seconds from the last check calibration (12 hours), the chromatographic system must be inspected for malfunctions and corrections must be made, as required. If the EICP area for any of the internal standards changes by a more than a factor of two (-50% to +100%), when compared to the average from the calibration, then the mass spectrometer must be inspected for malfunctions and corrections must be made. Reanalysis of CCV's and associated samples while the system was malfunctioning is necessary.

9.1.8. Samples can be directly injected after the successful analysis of the initial calibration curve, ICV, BFB, and CCV. There can be up to 20 samples in an analytical batch. A MS/MSD and LCS must be analyzed with every analytical batch. Recoveries shall be compared to laboratory generated QC limits or client specified limits for all surrogate, MS/MSD and LCS injections.

9.2. Sample Introduction and Purging.

9.2.1. BFB tuning criteria and daily GC/MS calibration criteria must be met before analyzing samples. Currently 18-25 (depending on the temperature program used) purged samples including QC can be analyzed within 12 hours of the BFB injection. The Archon or the Centurion autosampler can be programmed to accommodate the number of samples needed per analytical shift.

9.2.2. After the continuing calibration is verified, the system must be proven to be free of contamination by analyzing a MB. The MB shall not contain detects above the detection limits for any given compound. Some programs allow detects up to but not exceeding one half the MRL. If the MB contains detects above the detection limits or RL's, then corrective actions must be performed to ensure the system is free from contamination; all affected samples shall also reanalyzed. The MBs are also placed on the autosampler in the same manner as the samples. For QSM, 5.0 common contaminants must not be detected above the LOQ.

9.2.3. Analysis of samples begins by allowing the sample to come to ambient temperature prior to analysis. The VOA vials containing the water samples are placed on the autosampler where a 5.0 to 25.0 ml aliquot is withdrawn from the vial and added into the appropriate purge vessel. The same procedure is followed for methanol preserved soils (1.0 ml of soil extract/49.0 ml DI H2O is prepared and added into a 40 ml VOA vial prior to adding the samples to the autosampler). Low level soils are prepared by adding the VOA vial containing a magnetic stir bar and | 5 g sample/5.0 ml DI H2O to the autosampler (2-5 grams of sample is required for low-level analysis). The autosampler then adds an additional 5.0-mL of H2O containing the ISTD/SSTD mixture. The sample is heated to 40°C and purged in the VOA vial while being stirred, and the volatiles are collected onto the trap.

9.2.3.1. The ISTD/SSTD is added automatically by the Archon or the Centurion autosampler as the sample is transferred from the 40 ml sample vial to the sparge tube; the exception is for low level soils as noted above.

9.2.3.2. The sample is purged for 11 minutes at 32°C for waters and MeOH-preserved soils and 40°C for low-level soils using helium or nitrogen with a flow of 35-40 ml/min.

9.2.3.3. During the 11-minute purge time, the purge able volatile organics are adsorbed onto the Supelco Carbosieve K trap.

9.2.3.4. During desorption the trapped materials are rapidly heated while back-flushing the trap with helium or nitrogen at 35-40 ml/min. for 1 minute at 260°C and introduced in the GC/MS. After the valve to the GC is closed the trap is then baked and back flushed with helium for | 8 minutes at 265°C.

9.2.3.5. The GC is temperature programmed at 32°C for 2.5 minutes, then ramped to 165°C at 10°C/min, and finally ramped to 220°C at 15°C/min. The column flow is set at 1 ml/min. constant flow using helium as the carrier gas.
The transfer line to the MS is maintained at 250°C and the ion source is maintained at | 260°C while under constant vacuum. The GC injector is set at 200°C.

Note: Samples suspected of containing high levels of contamination or samples with known historical data may need to be diluted prior to analysis. Multiple

dilutions may be needed to cover the entire working range of the current calibration

9.2.4. For each sample batch a MS, MSD, and LCS is prepared and analyzed. The concentrations for water spikes are 10.0/100 ug/L for 5 ml purge and 4.0/40.0 ug/L for 25 ml purge. The spiked concentrations for soil samples are | 0.010/0.10 mg/kg for low level and | 0.50/5.0 mg/kg for MeOH preserved depending on sample weights and percent solids. One exception is for the analysis of samples with low sample volume. These samples may be analyzed with a LCS and a LCSD upon client request. All spikes are transferred into 40 ml VOA vials and added to the autosampler. The spike concentrations may vary depending on program/project specific criteria, but the preparation volumes are constant and only the spiking amount changes. 14.2.4.1 lists examples of spike preparation based on the concentrations above.

9.2.4.1. The preparation of the matrix spikes is performed as follows:

9.2.4.1.1. **Water (5 ml purge)**--Spike 40 ml of sample with 4.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer to a VOA vial for analysis. As an alternative the sample VOA vial may be spiked with 4.2 ul of the 100/1000 ug/ml CCV standard. When adequate sample amounts are not provided, one 40 ml aliquot of sample is spiked and split into two separate VOA vials containing 15 ml glass inserts.

9.2.4.1.2. **Water (25 ml purge)**-- Spike 50 ml of sample with 2.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer into a VOA vial for analysis. Alternatively, A MS/MSD can be prepared by spike ~1.6 ul of the 100/1000 ug/ml CCV standard directly into the sample vial. The sample is then inverted three times and then placed on the autosampler for analysis.

9.2.4.1.3. **Low-level Soils**--Spike 50 ml of DI water(volumetric flask) with 5.0 ul of the 100/100 ug/ml CCV standard, invert three times and transfer 5.0 ml into a VOA vial containing | 5 g of sample and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA vial containing | 5 g of sample and a stir bar for analysis.

9.2.4.1.4. **Med/high-level soils--**Spike | 10 g of sample contained in a VOA vial with 50 ul of the 100/1000 ug/ml CCV standard. Add 9.95 ml of methanol to the spiked sample and sonicate for 20 minutes. Add 1.0 ml of methanol extract to 49.0 ml DI water in a 50 ml syringe and then transfer into a VOA vial for analysis. For samples that are MeOH preserved in the field, take 1.0 mL of sample into 49 mL of DI water and add 5.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer to a VOA vial for analysis.

9.2.4.2. The preparation of a LCS is performed as follows:

9.2.4.2.1. **Water (5 ml purge)**--Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer to a VOA vial for analysis. The LCS and the CCV may be run as a single analysis.

9.2.4.2.2. **Water (25 ml purge)**-- Spike 50 ml of DI water (volumetric flask) with 2.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer into a VOA vial for analysis. The LCS and the CCV may be run as a single analysis.

9.2.4.2.3. **Low-level Soils**--Spike 50 ml of DI water(volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV standard, invert three times and transfer 5.0 ml into a VOA vial containing 5 g of control and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA vial containing 5 g of control sand and a stir bar for analysis.

9.2.4.2.4. **Med/high-level soils--**Spike 10 g of control sand contained in a VOA vial with 50 ul of the 100/1000 ug/ml CCV standard. Add 9.95 ml of methanol to the spiked sand and sonicate for 20 minutes. Add 1.0 ml of methanol extract to 49.0 ml DI water in a

50 ml syringe and then transfer into a VOA vial for analysis.

9.2.5. The data is collected by the Chemstation software using the RFs (or linear/second order regressions when necessary), and results are calculated using the internal standard method of quantitation. Response factors for each detected compound are compared with that obtained in calibration, and based on those comparisons, results are generated. Software manuals define the procedures for creating and understanding a specific method (Understanding Your Chemstation, Hewlett Packard, G2070-90100, October, 1994, Environmental Forms Software, Hewlett Packard, G1032-90021, November, 1992, and Productivity Enhancement Software for HP Chemstation, Prolab Resources Inc., XMS01A-002, Rev. G, 2001).

10. CALCULATIONS, DATA ANALYSIS AND REDUCTION.

10.1. The Chemstation software (using response factors) calculates the initial concentration (or raw result) of target compounds as follows:

10.1.1. **Liquids**

$$\text{Initial Concentration (ug/L)} = \frac{A_x \times I_{is}}{A_{is} \times RF}$$

Where:
A_X = Area of characteristic ion for compound being measured in the sample.
I_{IS} = Amount of internal standard injected (ug/L). Typical concentrations used are 20.0 ug/L for 5.0 ml purge, and 4.0 ug/L for 25.0 ml purge
A_{IS}= Area of characteristic ion for the internal standard.
RF = Response factor for compound being measured.

10.1.2. **Solids**

$$\text{Initial Concentration (ug/L)} = \frac{A_x \times I_{is}}{A_{is} \times RF}$$

Where:
A_X = Area of characteristic ion for compound being measured in the sample.
I_{IS} = Amount of internal standard injected (ug/L). Typical concentrations used are 20.0 ug/L for 5.0 ml purge, and 4.0 ug/L for 25.0 ml purge
A_{IS}= Area of characteristic ion for the internal standard.

RF = Response factor for compound being measured.

10.2. The Chemstation software (using linear regression) calculates the initial concentration of target compound as follows:

Response Ratio = slope * amount ratio + intercept
Where: Response Ration = response of target compound/response of associated ISTD.
Amount Ratio = target compound concentration/associated ISTD concentration.

Example: $Tr / ISr = m * Tc / ISc + b$

Where: Tr = response of target compound
ISr = Internal Standard Response
M = slope of the curve (for the target compound)
ISc = Internal Standard Concentration
Tc = Target compound concentration
B = y-intercept of the curve (for the target compound)

<u>Solve for "Tc"</u>

10.3. The initial concentration results are then transferred to the laboratory's LIMS system where the final concentrations are calculated.

10.3.1. The final concentration for water samples is calculated as follows:

Final Concentration (ug/L) = Initial concentration x Dilution Factor

10.3.2. The final concentrations for low-level and med/high-level soils are calculated by the following equation:

$$\text{Final concentration (mg/kg)} = \frac{\text{Initial concentration} \times \text{Sample Volume} \times \text{Dilution factor}}{\text{Sample weight} \times \%\ \text{solids}}$$

Where:

<u>Sample volume</u> = 5.0 mL for low –level soils, or volume of MeOH used for med/high-level soils preservation.
<u>Sample weight</u> = grams of sample in VOA vial for low level soils, or total grams of sample preserved for med/high-level soils.
<u>% Solids</u> = fraction equivalent (e.g. 97.1% = 0.971)

10.4. The spike percent recoveries (%R) and relative percent differences (RPD) are calculated in LIMS as follows:

10.4.1. Liquids – Concentration of spike added:

$$\text{ug/L} = \frac{\text{mL of spike added x concentration of spiking standard}}{\text{mL of sample (or DI } H_2O\text{) spiked}} \times 100$$

10.4.2. Solids – Concentration of spike added:

$$\text{mg/kg} = \frac{\text{mL of spike added x concentration of spiking standard}}{\text{grams of sample (or control sand) spiked}}$$

10.4.3. Final Calculations:

$$\text{MS/MSD \%R} = \frac{\text{Concentration of spike obtained - concentration of sample obtained}}{\text{Concentration of spike added}} \times 100$$

$$\text{MS/MSD RPD} = \frac{[\text{Concentration MS – Concentration MSD}]}{(\{\text{Concentration MS + concentration MSD}\}/2)} \times 100$$

$$\text{*LCS \% R} = \frac{\text{Concentration of Spike obtained}}{\text{Concentration of Spike added}} \times 100$$

Notes:

--Concentrations (conc.) of samples, MS/MSD, and LCS spikes are obtained directly from calibration curve.
--Soil spike concentrations and recoveries are calculated on a dry weight basis.
-- [ ] Signifies absolute values
--* Equation can also be used to calculate surrogate recoveries

11. Calibration and Standardization

11.1. To facilitate appropriate separation and provide adequate sensitivity, the entire operating system must be correctly set up and maintained before calibration and analyses can occur. Proper settings and programming of the GC/MS volatile system greatly increase the likelihood that calibrations will be acceptable. Generating and reproducing results will also be affected favorably in a well-maintained system.

11.1.1. The following tables provide instrument settings for the daily use of the Archon/Encon or Centurion/ Encon Purge and Trap Systems. Any modifications are noted in the specific instrument's maintenance log:

PARAMETER SETTINGS FOR ENCON EVOLUTION	
Trap Ready Temp.	≈35° C
Mort Ready Temp.	≈39° C
Purge Flow	40 ml/minute
Purge Time	11.00 minutes
Dry Purge Time	2.00 minutes
Desorb Preheat	255° C
Desorb Temp.	1.00 minutes at 260 ° C
Trap Bake Temp.	265° C
MoRT Bake Temp.	235 ° C
Bake Flow Rate	45 ml/minute
Gas	Helium or Nitrogen
Sample Purge Temp.	32 ° C
Sample Bake Temp.	75-90 ° C
Valve and Line Temp.	150 ° C

11.1.2. An example of the GC temperature program for the SPB/DB/ZB-624 columns used for the analysis of samples is as follows:

Start temp °C	End temp °C	Rate °C/minute	Time minutes
32	32	0.0	2.5
32	165	10.0	0.00
165	220	15.0	1.00

11.1.3. The injector is a split/split less injector operated in split mode ranging from 1:10 to 1:60. The injector temperature is 200 °C.

11.1.4. The MS detector parameters are subject to change to achieve optimum chromatography. See instrument maintenance logbook for recent changes regarding source maintenance, as well as filament and multiplier replacements. Current tune values and EM voltage settings are documented and can be found in the appropriate instrument's tuning logbook.

11.1.5. 4-Bromofluorobenzene (BFB) Standard:

11.1.5.1. A standard solution containing 50 ug/ml is used for the daily tune check. The BFB is directly injected onto the column in 25 to 50 ng injections (0.5 to 1.0 ul).

11.1.5.2. The GC/MS system tune must be verified at the beginning of any calibration or a sequence run and verified every 12 hours thereafter. The tuning compound is BFB which is injected directly onto the GC column The software is set up to check the tune by using the mean of three scans across the apex. Background subtraction is performed using a single scan no more than 20 scans prior to the elution of BFB. Manual scans can be checked by taking an average of scans across the BFB peak. The tuning acceptance criteria are listed below (m/z range 35-260):

Mass (m/z)	Abundance criteria
50	15 to 40% of mass 95.
75	30 to 60% of mass 95.
95	Base Peak, 100% Relative Abundance
96	5 to 9% of mass 95.
173	<2% of mass 174.
174	>50% of mass 95.
175	5 to 9% of mass 174.
176	>95% but <101% of mass 174.
177	5 to 9% of mass 176.

11.1.6. The preparation of working standards is routinely performed each week unless integrity is shown to be intact. All standards are assigned a unique identification number and preparations are documented in a Standards Logbook.

11.1.6.1. Calibration Standards - Calibration standards are prepared at a minimum of five concentration levels (in most instances, eight levels are currently used) and are prepared from the working standard dilutions of stock standards. One of the concentration levels shall be at a concentration near, but above, the detection limit and at or below the reporting limit. The remaining concentration levels shall correspond to the expected range of concentrations found in real samples and shall contain each analyte for detection by this method. For low-level soil calibrations, sodium bisulfate is added at a 0.2 g/1.0 g sample to the water to match sample matrix /acidity if the samples were collected and preserved with sodium bisulfate. Med/high-level soil calibrations have MeOH added at a 0.1 ml/5.0 ml H2O to match sample matrix/preservation. All final concentrations are brought to volume with DI water. The following tables outline the preparation calibration curves for water and soil samples (all calibration standards are transferred into 40 ml VOA vials for placement on the auto sampler):

1) Waters Curve (5.0 ml Purge)

Concentration (ug/L)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Final Volume (ml) (Volumetric flask)
0.5	1.0	200
2.0	2.0	100
5.0	5.0	100
10.0	10.0	100
20.0	20.0	100
30.0	30.0	100
40.0	40.0	100
80.0	80.0	100

2) Waters Curve (25.0 ml Purge)

Concentration (ug/L)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Final Volume (ml) (Volumetric flask)
0.1	0.5	500
0.5	1.0	200
1.0	1.0	100
2.0	2.0	100
4.0	4.0	100
6.0	6.0	100
8.0	8.0	100
16.0	16.0	100

3) Low Level Soils Curve

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Grams of sodium bisulfate added (if needed)	Final Volume (ml) (Volumetric flask)
0.001	1.0	1.0	100
0.002	2.0	1.0	100
0.005	5.0	1.0	100
0.010	10.0	1.0	100
0.020	20.0	1.0	100
0.030	30.0	1.0	100
0.040	40.0	1.0	100
0.080	80.0	1.0	100

4) Medium/High Soils Curve

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	ul of MeOH added	Final Volume (ml) (Volumetric flask)
0.050	1.0	999	100
0.100	2.0	998	100
0.250	5.0	995	100
0.500	10.0	990	100
1.000	20.0	980	100
1.500	30.0	970	100
2.000	40.0	960	100
4.000	80.0	920	100

5) Waters Curve (5.0 ml Purge-SIM)

Concentration (ug/L)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Final Volume (ml) (Volumetric flask)
1.0	1.0	100
2.0	2.0	100
5.0	5.0	100
10.0	10.0	100
20.0	20.0	100
40.0	40.0	100
80.0	80.0	100

6) Medium/High Soils Curve (SIM)

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	ul of MeOH added	Final Volume (ml) (Volumetric flask)
0.050	1.0	999	100
0.100	2.0	998	100
0.250	5.0	995	100
0.500	10.0	990	100
1.000	20.0	980	100
2.000	40.0	960	100
4.000	80.0	920	100

7) Low Level Soils Curve (SIM)

Concentration (mg/kg)	Amount added of the 100 ug/ml CCV/Calib. Std. (in ul)	Grams of sodium bisulfate added (if needed)	Final Volume (ml) (Volumetric flask)
0.001	1.0	1.0	100
0.002	2.0	1.0	100
0.005	5.0	1.0	100
0.010	10.0	1.0	100
0.020	20.0	1.0	100
0.040	40.0	1.0	100
0.080	80.0	1.0	100

11.1.6.2. Internal Standards - The internal standards used are Chlorobenzene-d_5, 1,4-Difluorobenzene, 1,2-Dichloroethane-d_4 and Fluorobenzene (sec. 7.6.1). Other compounds may be used as internal standards as long as they have retention times similar to the compounds being detected by GC/MS.

11.1.6.3. Surrogate Standards - The surrogate standards used are Toluene-d_8, 1,2-Dichloroethane-d_4, 4-Bromofluorobenzene, and Dibromofluoromethane. Other compounds may be used as surrogates, depending upon the analysis requirements. The 100 ug/ml working surrogate standard (sec. 7.7.2) may be used for calibration added at the same concentrations as the target compounds (see above).

11.1.6.4. ISTD/SSTD Combined Standard - A combination of internal standard and surrogate standard at 80ug/ml (sec. 7.7.3), or 20 ug/ml for Centurion auto-sampler (sec. 7.5.3) , is automatically added by the autosampler to all calibration levels, samples, blanks, CCV's and spikes used for any given sequence (actual volume for the archon autosamplers is determined by an ISTD/SSTD study and is documented in the maintenance logbooks for each instrument). Limits are generated internally or project/program limits are used.

11.1.6.5. Calibration curves are prepared fresh from newly made working standards to ensure accurate concentrations are maintained.

11.1.6.6. Secondary dilution standards (when necessary) - secondary dilution standards containing the compounds of interest (usually at 10.0/100 ug/ml) for SIM, low level, and MDL analyses may be prepared in methanol and stored with minimal headspace and shall be checked frequently for degradation. They are to be stored for one week only.

11.1.6.7. Preparation of standards is documented in the Volatile standards logbook. Each standard solution is documented with the standard name, concentration, preparation date, expiration date and a unique number given to that standard for future traceability.

11.2. The curve is generated using the relative response factor (RRF or RF). The data system tabulates the area response of the characteristic ions against the concentration of each compound and each internal standard. Calculate RFs for each compound relative to one of the internal standards. The internal standard selected for the calculation for the RF for a compound is the internal standard that has a retention time closest to the compound being measured.

11.2.1. The RF is calculated by the data system as follows:

$$RF = \frac{A_S \times C_{IS}}{A_{IS} \times C_S}$$

Where:

A_S = Area of the characteristic ion for the compound being measured in the calibration standard.
A_{IS} = Area of the characteristic ion for the specific internal standard.
C_{IS} = Concentration of the specific internal standard.
C_S = Concentration of the compound being measured in the calibration standard.

11.2.2. The average response factor (ARF) for all calibration levels is used when determining sample concentration and is calculated (along with the standard deviation) to evaluate the linearity of the curve (SW-846 Method 8000C. Sec. 11.5.1).

11.3. When ARFs are not acceptable, results are sometimes calculated using linear (1st order) regression curves and/or quadratic (2nd order) curves. Internal standard quantitation is also used when generating linear and non-linear calibrations. All equations and acceptance criteria follow the examples in SW-846, Method 8000C (sec. 11.5.2 and sec. 11.5.3).

11.4. If the RSD of the RFs is less than 20%, then the RF is assumed to be constant over the calibration range, and the average response factor may be used for quantitation. If the RSD of any analyte or surrogate mean RF exceeds 20% than linear regression or second order curves may be used for quantitation.

11.4.1. Linear Calibration: If the RSD of the calibration factor is greater than 20% over the calibration range, then the linearity through the origin cannot be assumed. If this is the case, the analyst can employ a regression equation that does not pass through the origin. This approach can also be employed based on the past experience of the instrument response.

11.4.2. The use of origin (0,0) as a calibration point is not allowed. However, most data systems and many commercial software packages will allow the analyst to “force” the regression through zero. This is not the same as including the origin as a fictitious point in the calibration. It can be appropriate to force the regression through zero for some calibrations (SW-846 Method 8000C sec. 11.5.2.1). The use of linear regression cannot be used as a rationale for reporting results below the calibration range.

11.4.3. The method of linear regression analysis has a potential for a bias to the lower portion of a calibration curve. If linear regression is used, then the lowest point in the calibration curve is calculated using the new curve. The recalculated concentration of the low calibration point should be within +/- 30% of the standard's true concentration.

11.4.4. Non-Linear Calibration: In situations where the analyst knows that the instrument response does not follow a linear model over a sufficiently wide working range, or when the other approaches described here have not met the acceptance criteria, a non-linear calibration model can be employed. When using a calibration model for quantitation, the curve must be continuous, continuously differentiable and monotonic over the calibration range. The model chosen shall have no more than four parameters, i.e., if the model is polynomial, it can be no more than third order.

11.4.4.1. The statistical considerations in developing a non-linear calibration model require more data than the more traditional linear approaches described above. Linear regression employs five calibration standards for the linear model; a quadratic model requires a minimum of six calibration standards.

11.4.4.2. Under ideal conditions, with a "perfect" fit of the model to the data, the coefficient of the determination (COD) will equal 1.0. In order to be an acceptable non-linear calibration, the COD must be greater than or equal to 0.99 Weighting in a calibration model can significantly improve the ability of the least squares regression to fit the data calibrations (SW-846 Method 8000C sec. 11.5.3).

11.5. Each of the most common target compounds ARFs in the ICV and the CCV should meet the minimum RF as noted in Table 4.

11.5.1. A number of compounds (primarily the ketones) do not respond well at normal concentrations, especially for low level analyses, resulting in RF's below the minimum requirement. These compounds are purchased at concentrations 10x the normal concentration to ensure adequate responses for working calibrations. Other poor responding compounds are commonly requested to be analyzed by this procedure and are purchased at concentrations that best ensure adequate responses to achieve successful calibrations.

11.5.2. If the minimum response factors are not met, the system should be evaluated, and corrective action should be taken before sample analysis begins. Examples of possible occurrences are as follows:

	Comment
Chloromethane	This compound is the most likely compound to be lost if the purge flow is too fast.
Bromoform	This compound is one of the compounds most likely to purge poorly if the purge flow is too slow. Cold spots and/or active sites in the transfer lines may adversely affect response. Response of the quantitation ion (m/z 173) is directly affected by the tuning of BFB at ions m/z 174/176. Increasing the m/z 174/176 ratio relative to m/z 95 may improve bromoform response.
1,1,2,2-Tetrachloroethane	The response of this compound is degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.
1,1-Dichloroethane	The response of this compound is also degraded by contaminated transfer lines in purge and trap systems and/or active sites in trapping materials.

11.6. All calibrations are confirmed by the analysis of a “second source” ICV standard before daily checks and analyses are performed. The RSD limit for all target compounds is ±20%, unless specified differently by any other applicable program or project’s criteria (QSM: ±25% RSD for all analytes). If these criteria are not met and a reanalysis of the ICV confirms the nonconformities, then corrective actions must be taken and the instrument recalibrated. Any outliers suggest a problem and poor performers shall be addressed. The concentrations of the ICV are near the midpoint of the curve (10/100 ug/L for water-5 ml purge, 4.0/40 ug/L for water-25 ml purge, 0.010/0.10 mg/kg for low level soils, and 0.50/5.0 mg/kg for MeOH preserved soils). The preparation of ICV’s is as follows:

Water (5 ml purge)--Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml ICV/Spiking standard, invert three times and transfer to a VOA vial for analysis.

Water (25 ml purge) -- Spike 50 ml of DI water (volumetric flask) with 2.0 ul of the 100/1000 ug/ml ICV/Spiking standard, invert three times and transfer into a VOA vial for analysis.

Low-level Soils--Spike 50 ml of DI water(volumetric flask) with 5.0 ul of the 100/1000 ug/ml ICV/Spiking standard, invert three times and transfer 5.0 ml into a VOA vial (containing and a stir bar) for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA (containing a stir bar) for analysis.

Med/high-level soils-- Spike 49 ml of DI water (volumetric flask) with 1.0 ml of MeOH and 5.0 ul of the 100/1000 ug/ml ICV/Spiking standard, invert three times and transfer into a VOA vial for analysis.

11.7. An Initial Calibration Blank (ICB) is analyzed to confirm that the instrument is free from contamination. Any detects in the ICB shall be less than the method detection limit and/or less than ½ the program/project limits. Any detects above MDL or program limits must be addressed before sample analyses begin. To prepare an ICB fill a 40 ml VOA vial preserved with 5% HCL with DI water.

11.8. Demonstration and documentation of an acceptable initial calibration is required before any samples are analyzed. Refer to EPA SW-846, Method 8000B, Section 7, for a detailed discussion of calibration procedures.

12. QUALITY CONTROL

12.1. Method Performance

12.1.1. Certified standard solutions, properly maintained instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by the appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

12.1.2. Initial demonstration of capability (IDC) is another technique used to ensure acceptable method performance.

12.1.2.1. An analyst must demonstrate initial precision and accuracy through the analysis of 4-5 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculates the average recovery (x) in μg/L and the relative standard deviation (RSD) of the recoveries for each analyte. In the absence of specific criteria found in the SW-846 methods or project specific limits, the default criteria of 70-130% recovery and 20 % RSD are used until internal limits are generated (Method 8000C, sec. 9.4.9)

12.1.3. Examples of the preparation of IDCs are as follows:

Water (5 ml purge)--Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer to a VOA vial for analysis.

Water (25 ml purge) -- Spike 50 ml of DI water (volumetric flask) with 2.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer into a VOA vial for analysis.

Low-level Soils--Spike 50 ml of DI water (volumetric flask) with 5.0 ul of the 100/1000 ug/ml CCV/Calib. standard, invert three times and transfer 5.0 ml into a VOA vial containing 5 g of control and a stir bar for analysis. As an alternative, prepare a 10.0/100 ug/ml working standard, then add 5.0 ul of this to 5.0 ml of DI water and transfer into a VOA vial containing 5 g of control sand and a stir bar for analysis.

Med/high-level soils--Spike 10 g of control sand contained in a VOA vial with 50.0 ul of the 100/1000 ug/ml CCV/Calib. standard. Add 9.95 ml of methanol to the spiked sand and sonicate for 20 minutes. Add 1.0 ml of methanol extract to 49.0 ml DI water in a 50 ml syringe and then transfer into a VOA vial for analysis.

12.1.4. Many projects require the analysis of MRL standards and MDL check samples as another means of checking method performance. The MRLs are analyzed at the beginning and end of each 12 hour shift and are typically prepared at concentrations equal to the lowest standard on the calibration curve. Recovery limits are program specific but are usually set at 70-130%. The MDL check sample is usually spiked at approximately 2x the method detection limit. The MDL check sample is analyzed quarterly (as a minimum) to confirm instrument sensitivity (e.g. to verify that the method detection limits are still achievable). The MDL check samples are taken through all preparation and extraction steps used for actual samples (e.g. spiking/preserving control sand for soil samples). In most instances, a method detection limit check sample is analyzed at the end of each sequence requiring an MRL standard. The recovery criteria for MDL check samples are the ability to detect all compounds. If any given compound is not detected, the MDL check is spiked at a higher level and analyzed again. Detection limits for those compounds not detected on the initial MDL check analysis need to be raised to match the MDL check analysis at which they were detected.

12.1.5. Creating and monitoring control charts is also important for maintaining and improving method performance. Currently all SSTD, MS, MSD, and LCS recoveries are monitored with the use of the LIMS system. The data collected is used to recognize trends in recovery performance, as well as for generating new in-house QC limits. Default accuracy limits of 70-130 % recovery and a precision limit 20 % RSD are used until enough data points are generated to provide usable internal limits. Other programs such as the WI UST program uses default accuracy and precision limits for surrogates and spikes of 80-120/20 %. Client and/or project specific limits are also used frequently in sample analyses. The Quality Control Requirements chart (Table 2.) also lists recovery limits specific to the method/project/program.

12.1.6. Performance Testing (PT's) must be done on all compounds on the list. If a compound is not available from a PT provider, the LCS studies must be performed and documented (at least 4 reps) twice a year to demonstrate proficiency.

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12.2. This SOP is designed to follow a variety of different projects and programs requirements. Table 2. is designed to illustrate the control steps and provisions required to adequately producing acceptable data.

12.3. Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.4. Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

13. DATA ASSESSMENT/ACCEPTANCE CRITERIA FOR QC MEASURES

13.1. If the initial analysis of a sample or a dilution of the sample has a concentration of a particular analyte that exceeds the calibration range, the sample must be reanalyzed at a dilution. Secondary ion quantitation is allowed only when there are sample interferences with the primary ion. When a sample is analyzed that has saturated ions from a compound, this analysis must be followed by a blank water analysis. If the blank analysis is not free of interferences, the system must be decontaminated. Sample analyses can not resume until a blank can be analyzed that is free of interferences.

13.2. After the analysis of water samples, the pH shall be taken to verify proper field preservation. pH strips are used to verify the pH which is then documented in the bench sheet logbook.

13.3. Qualitative Analysis:

13.3.1. The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum (ion scans) after background correction with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated (by the laboratory) using the conditions of this method. The mass spectral library is updated with each new calibration and is continually updated with the mass spectra from CCV's.

13.3.2. The characteristic ions from the reference mass spectrum are defined to be the three ions of greatest relative intensity or any ions over 30% relative intensity if fewer than three such ions occur in the reference spectrum. Table 3 lists compounds along with the Primary Ion (Quantitation ion) used for calculating results, and the Secondary Ions (Qualitative ions) used for qualitatively matching sample spectrums with reference spectrums for positive identifications. Compounds shall be identified as present when the criteria below are met.

13.3.2.1. The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other. Selection of a peak by a data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

13.3.2.2. The relative retention time (RRT) of the sample component is within +/- 0.06 RRT units of the RRT of the standard component.

13.3.2.3. The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

13.3.2.4. Structural isomers that produce very similar mass spectra shall be identified as individual isomers if they have sufficiently different GC retention times. Sufficient GC resolution is achieved if the height of the valley between two isomer peaks is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13.3.2.5. Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component (i.e., a broadened peak with shoulder(s) or a valley between two or more maxima), appropriate selection of analyte spectra and background spectra are important. Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co elute (i.e., only one chromatographic peak is apparent), the identification criteria can be met, but each analyte spectrum will contain extraneous ions contributed by the co eluting compound.

13.3.3. For samples containing compounds that are not a part of the normal target list, a library search may be required for the purpose of tentative identification. Tentative identified compounds (TICs) are needed only when requested or required by a particular project or program. Data system library search routines shall not use normalization routines that would misrepresent the library of unknown spectra when compared to each other. Use the following a guidance for reporting TICs.

13.3.3.1. Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) shall be present in the sample spectrum.

13.3.3.2. The relative intensities of the major ions agree within ± 20%.

13.3.3.3. Molecular ions present in the reference spectrum shall be present in the sample spectrum.

13.3.3.4. Ions present in the sample spectrum but not in the reference spectrum shall be checked for possible background contamination. They shall also be reviewed for possible co elution with another compound.

13.3.3.5. Ions present in the reference spectrum but not in the sample spectrum shall be check against the possibility of subtraction from the sample spectrum due to background contamination or co-eluting peaks. Some data reduction programs can create these discrepancies.

13.4. Ions present in the reference spectrum but not in the sample spectrum shall be check against the possibility of subtraction from the sample spectrum due to background contamination or co-eluting peaks. Some data reduction programs can create these discrepancies.

13.5. When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. Once the reference spectrums are compared and the sample spectrums and identifications have been made, the sample data can be reported. Assessments of all spiked and calibration control samples and standards shall also be finalized before reporting the data.

13.5.1. When the analyst has finished processing the analytical batch, the results are electronically transferred to the LIMS system where weight to volume corrections, dilution factors and percent solids adjustments are made. Once the final results have been verified, a checklist (FVO4-01) is filled out and signed confirming that all the data has been thorough scrutinized. At this point the data is turned over to another qualified analyst for final validation. The second analyst confirms the results and electronically marks them validated and signs his or her portion of the checklist. Finally, the validated results are made available to the client services personnel in order for the data to be given to the client or appropriate agencies.

13.5.2. A PDF copy of the data is then electronically filed and archived. The package includes the checklist, the sequence run log, a copy of the bench sheets, the LIMS run log, verification of tuning and system performance data, and verification of calibration data. For each sample, the chromatogram, quantitation and library spectra (ion scans) for all detected target compounds are also included. Each data file header shall contain the sample ID # and the date and time acquired.

14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1. See QAM Appendix 9.

15. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

15.1. See QAM Appendix 9.

16. DATA RECORDS MANAGEMENT

16.1. Records are stored for a minimum of 5 years in accordance with the Quality Manual.

16.2. See SOP QA 003 for specifics on document control.

17. WASTE MANAGEMENT

17.1. See QAM Appendix 9.

18. REFERENCES

18.1. Determinative Chromatographic Separations, USEPA SW-846 Methods 8000C, Rev. 3, March, 2003

18.2. Volatiles Organic Compounds by Gas Chromatography/Mass Spectrometry (GC/MS), USEPA SW-846 Method 8260C, Rev. 3, August, 2006.

18.3. Purge and Trap for Aqueous Samples, USEPA SW-846 Methods 5030B, Rev. 2, December, 1996.

18.4. Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, USEPA SW-846 Methods 5035 (Inc. App. A), Rev. 0, December, 1996.

18.5. Wisconsin DNR, Lust Guidance, July, 1993.

18.6. USEPA, Method 603, Acrolein and Acrylonitrile, July, 1982.

18.7. CT Laboratories Quality Manual, current revision.

18.8. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 4.2, October 2010.

18.9. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.

18.10. National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.

18.11. ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.

18.12. Appendix A to part 136, Methods for Organic Chemical Analysis of Municipal and Industrial Wastewaters, EPA method 624-Purgeable, 1984.

19. Attachments.

Table 1

Analyte List

Analyte	Analyte
Acetone	2,2-Dichloropropane
Benzene	1,1-Dichloropropene
Bromobenzene	cis-1,3-Dichlropropene
Bromochloromethane	trans-1,3-Dichloropropene
Bromodichloromethane	Diisopropyl ether
Bromoform	Ethylbenzene
Bromomethane	Hexachlorobutadiene
2-Butanone	2-Hexanone
n-Butylbenzene	Isopropylbenzene
sec-Butylbenzene	p-Isopropyltoluene
tert-Butylbenzene	Methylene chloride
Carbon disulfide	4-Methyl-2-pentanone
Carbon tetrachloride	Methyl tert butyl ether
Chlorobenzene	Naphthalene
Chloroethane	n-Propylbenzene
2-Chloroethylvinyl ether	Styrene
Chloroform	1,1,1,2-Tetrachloroethane
Chloromethane	1,1,2,2-Tetrachloroethane
2-Chlorotoluene	Tetrachloroethene
4-Chlorotoluene	Tetrahydrofuran
Dibromochloromethane	Toluene
1,2-Dibromo-3-chloropropane	1,2,3-Trichlorobenzene
1,2-Dibromoethane	1,2,4-Trichlorobenzene
Dibromomethane	1,1,1-Trichloroethane
1,2-Dichlorobenzene	1,1,2-Trichloroethane
1,3-Dichlorobenzene	Trichloroethene
1,4-Dichlorobenzene	Trichlorofluoromethane
Dichlorodifluoromethane	1,2,3-Trichloropropane
1,1-Dichloroethane	1,2,4-Trimethylbenzene
1,2-Dichloroethane	1,3,5-Trimethylbenzene
1,1-Dichloroethene	Vinyl chloride
cis-1,2-Dichloroethene	Vinyl acetate
trans-1,2-Dichloroethene	o-Xylene
1,2-Dichloropropane	m/p-Xylene
1,3-Dichloropropane	112Trichloro122trifluoroethane

Table 2
Volatile Organic Compounds by GC/MS
Summary of Quality Control Requirements

Quality Control Item	Frequency	Acceptance Criteria	Corrective Action
Tune Check (BFB)	Beginning of Every 12 hours shift.	Ensure correct mass assignment. BFB % Relative abundance criteria as specified in method 8260 or use program/project specific criteria.	Retune. Do not proceed with analysis until tune meets criteria.
Initial Calibration (ICAL)	Each time the instrument is set up and when compounds in the continuing calibration verification (CCV) do not meet criteria. Established initially at minimum five concentration levels (six concentration levels if a second order {quadratic} curve is used) - low standard at or below project required reporting limit (PRRL), near but above method detection limits (MDL). Heated purge for low-level soils.	1. Average relative response factors (RRFs) for compounds on Table 4. 2. % RSD for RRFs for all target compounds ≤20%. IF RF % RSD >20% use linear curve, r =.995, r2 = .990. 4. LCG, NELAC, DoD-QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents	Correct system and recalibrate. Criteria must be met before sample analysis may begin. Any samples reported from data not meeting these criteria must be qualified (Z).
Initial Calibration Verification standards (ICV)	After each initial calibration. Shall be at or near the mid-point of calibration range for all target compounds, and is prepared from second source standards. Typically use 10/100 ppb for H2O and Low Level Soils, 0.5/5.0 mg/kg for MeOH preserved soils. Two ICV's are required for 2^{Nd} order quadratic curves (one below and one above the inflection point). Heated purges for low-level soils.	1. RRF for compounds on Table 4. 2. %RSD <20% Deviation for RRFs, <20 % Drift for linear and nonlinear curves 3. LCG, NELAC, DoD-QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents (ex: ± 25% D. for QSM projects).	Correct system and recalibrate. Criteria must be met before sample analysis may begin. IF % relative standard deviation (RSD) >20%, then system must be inspected and problem corrected before sample analysis. If >20% RSD then confirm the integrity of the second source standard by reanalysis, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. ACOE allows no tolerances for % D. Problem compounds need to be addressed on a project to project basis.

Continuing Calibration Verification standards(CCV)	Beginning of Every 12 hour shift, after the BFB injection. Shall be at or near the mid-point of calibration range for all target compounds, and is prepared from standards used for calibration (Typically use 10/100 ppb for H2O and Low Level soils, 0.5/5.0 mg/kg for MeOH preserved soils). Varied CCV levels are required for QSM when multiple CCV's are necessary on a run (Typically use 10, 20, and 30 ppb for H2O and Low Level soils, 0.5, 1.0, and 1.5 mg/kg for MeOH preserved soils). Heated purges for low-level soils.	1. Average relative response factors (RRFs) for compounds on Table 4. 2. %RSD <20% Deviation for RRFs, <20 % Drift for linear curve and nonlinear curves 3. NELAC, DoD-QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	Correct system and recalibrate. Criteria must be met before sample analysis may begin. IF % RSD >20%, then system must be inspected and problem corrected before sample analyses. If >20% RSD correct problem if determinable then reanalyze, and/or determine if it's a sporadic problem involving compounds that are typically poor performers. In any case sample results reported that have %D failures must be qualified (Z). ACOE allows no tolerance for % D. Problem compounds need to be addressed on a project to project basis QSM 5.0 - Immediately analyze two additional consecutive CCVs. If both pass (for those compounds that initially failed), samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.
Internal Standards (ISTD)	Added to all blanks, standards, and samples.	1. Peak area within -50% to +100% of area in associated CCV standard. 2. Retention time (RT) within 10 sec of RT for associated CCV standard. 3. NELAC, DoD-QSM, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	Inspect instrument for malfunctions; correct identified malfunctions, then reanalyze samples. If no instrument malfunction identified proceed as follows: * Reanalyze sample. * If reanalysis is outside limits the data shall be qualified (S). Follow specified criteria as stated in Shell or other documentation.

Method Blank (MB)	1 / 20 samples per matrix or at program/project specific frequencies. The MB is used to document contamination resulting in the analytical process and shall be carried through the complete sample preparation and analytical procedure.	1. Concentration of analytes of concern shall be less than the highest of either : *Method Detection Limit *Five percent of the regulatory limit for that analyte or, *Five percent of the measured concentration in the sample. 2. DoD-QSM: ≤ ½ RL 3. Follow criteria according to specific program or project. 4. QSM5.0 – Know Lab contaminants = No detection above the LOQ	Reanalyze to determine if instrument or laboratory background contamination was the cause. If the method blank is still non-compliant, re-prepare and reanalyze blank and samples.* For ACOE/QSM data if less than ½ RL no action required.* *If reanalysis of blank still contains contamination above specified limits, affected data shall be flagged (B).
Laboratory Control Sample (LCS)	1 / 20 samples or at contact/ program specific frequencies. Must undergo all sample preparation procedures. Prepared from a second source and contain target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries (and RPDs, if applicable) within in-house generated limits. Default 70- 130% (20% RPD). 2. Use DoD-QSM, program/project specific, or client contract limits when applicable.	If LCS recoveries are within control limits or within SMF frequency and limits then no action is required. If the LCS exceeds control limits, as well as SMF criteria the reanalyze the LCS to confirm proper preparation procedure. If still exceeding limits then reanalyze associated samples with a new LCS.. If sample data is reported with LCS failures then that data must be qualified (Q). Exception: If the LCS recoveries are high with no associated positives then no further action is taken.

Matrix Spike/Matrix Spike Duplicate	One per set of 20 samples per matrix. Must undergo all sample preparation procedures. Must be spiked with target compounds with concentrations at or near the mid-point of the calibration range.	1. % Recoveries and RPDs within in-house generated limits. Default 70-130 / 20%RPD. 2. Use DoD-QSM, program/project specific, or client contract limits when applicable. 3. QSM 5.0 – RPD within 20% 4. QSM 4.2 – RPD within 30%	If LCS is acceptable, then report probable matrix interference. Qualify non-detects if the recoveries are low (M), and detects if the recoveries are low and the sample amount + the true spiked amount shall be within calibration range. Qualify detects if recoveries are high and the detects + the true spiked amount are within calibration range. If recoveries are high and there are no detects in the parent sample then that data does not require flagging. If spiked amount + sample amount for any given compound exceeds calibration range than the spike is considered invalid for that compound. Qualify data for RPD failures (Y) when there is a detect for the failing compounds (non-detected compounds are not qualified). Exception: If a compound is already qualified for a LCS failure then no RPD qualifier is applied.
Qualitative/Quantitative Issues	1. If detection level of any compound in a sample exceeds the detection level of that compound in the highest level standard, the sample must be diluted to approximately mid-level of the calibration range and reanalyzed. 2. If the concentration of the target analyte (that exceeded the calibration range) is present in the sample following the high level sample and is greater than the RL but ≤5x RL, then that sample must be reanalyzed to determine if carryover occurred.	1. The instrument level of all compounds must be within the calibration range for all samples. 2. The sample analyzed immediately after a high-level sample must display concentrations of the high level target compounds less than the RL or greater than 5x RL.	Dilute the sample to bring the level of the highest concentration of target compounds within the calibration range. If any data is reported with any results over range then those results shall be flagged (X). A sample displaying concentrations of target compounds between the RL and 5x the RL that was analyzed immediately after a high-level sample must be reanalyzed. If the results do not agree within the RL, report only the second analysis.

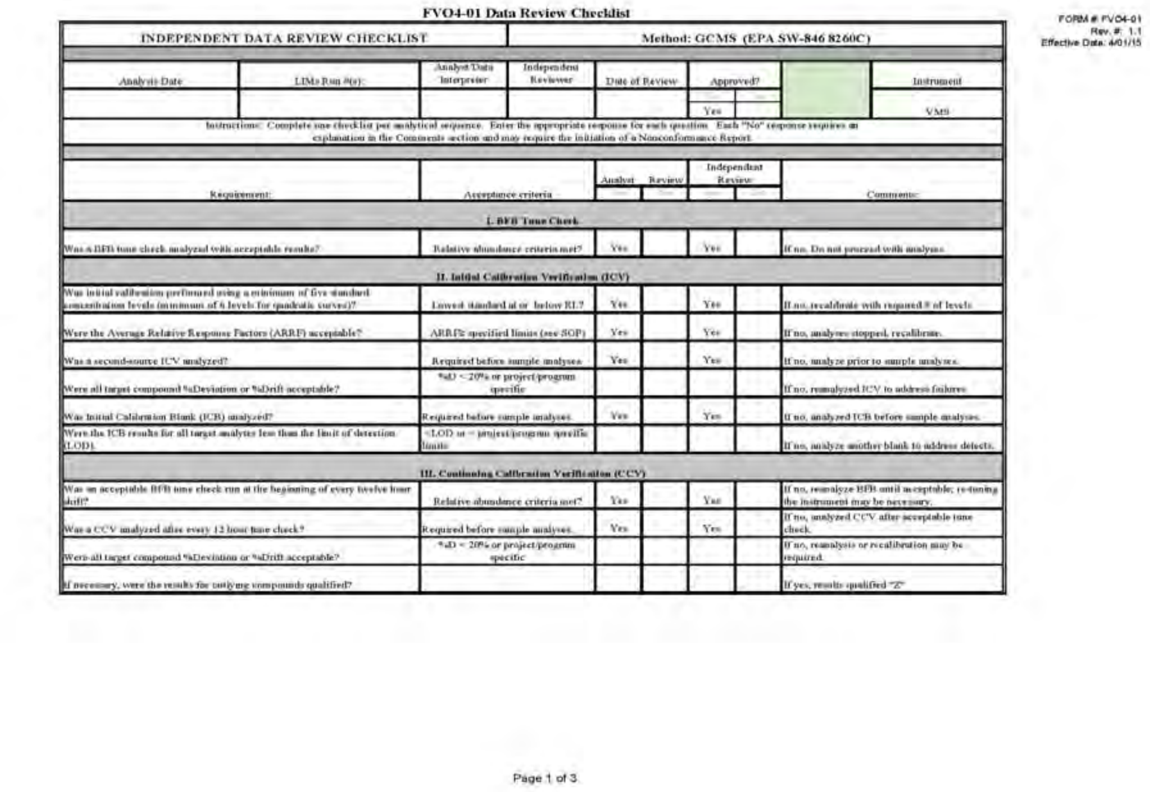
Surrogate	1. Typically use single point calibrations. 2. Added to all blanks, samples, and QC samples, as a part of the internal standard-surrogate spiking mixture.	1. All % Recoveries within in-house generated limits. Default 70-130%. 2. Use DoD-QSM, program/project specific, or client contract limits when applicable.	If recoveries are not within limits: Check to be sure that there are no errors in calculations, surrogate solutions, or internal standards. Also, check instrument performance. If no problem is found, re-prepare and reanalyze the sample. If the reanalysis is within limits, report only the reanalysis. If the reanalysis is still out of limits the sample shall be qualified (S). Due to matrix affect, no reanalysis is required if the MS and/or MSD are outside limits.
Sample Duplicate (Dup) – when required	1. Program/contract specific. 2. When limited sample is available a sample duplicate may be used in lieu of a MSD.	1. RPD < or = 10% (between sample and sample duplicate) for QSM projects. 2. RPD's within in-house limits. Default ± 20%. 3. DoD-QSM, NELAC, or other programs/agencies may require different criteria than stated here. Program and/or project specific criteria shall be followed as stated in their documents.	If RPDs are not within limits: Check to be sure that there are no errors in calculations. Also, check instrument performance and correct if necessary. If corrected or no problem is found, re-prepare and reanalyze the sample. If the reanalysis is within limits, report only the reanalysis. If the reanalysis is still out of limits the sample shall be qualified (Y).
Method Reporting Limit (MRL) Spike – when required	1. Program/contract specific. 2. Typically bracketing samples for every 12 hour analysis window.	1. % Recoveries within in-house generated limits. Default 70-130 %Rec. 2. Program or project/contract specific limits shall be followed as stated in their documents..	If there is a failure investigate problem. If system is in control run an MDL check sample to verify detection limits.
Continuing Calibration Verification Final(CCVF) (QSM 5.0 only)	For QSM 5.0: at the end of the analytical sequence for a batch of 20 or fewer samples. Shall be at or near the mid-point of calibration range for all target compounds, and is prepared from standards used for calibration (Typically use 10/100 ppb for H2O and Low Level soils, 0.5/5.0 mg/kg for MeOH preserved soils).	1. %RSD <50% Deviation for RRFs, <50 % Drift for linear curve and nonlinear curves	QSM 5.0 - Immediately analyze two additional consecutive CCVFs. If both pass (for those compounds that initially failed), samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCVF. (it is allowable, if needed, the two reanalysis of the ending CCVF can extend beyond the 12 hour analysis window.

Table 3
Characteristic ions

Analyte	Primary Ion	Secondary Ion	Analyte	Primary Ion	Secondary Ion
Acetone	43	58	2,2-Dichloropropane	77	97,79
Benzene	78	51,77	1,1-Dichloropropene	110	77,75
Bromobenzene	156	77,158	cis-1,3-Dichlropropene	75	110
Bromochloromethane	128	49,130	trans-1,3-Dichloropropene	75	77,110
Bromodichloromethane	83	85,129	Diisopropyl ether	45	87,43
Bromoform	173	175,171	Ethylbenzene	91	106
Bromomethane	94	96	Hexachlorobutadiene	225	223,227
2-Butanone	43	72,57	2-Hexanone	43	58,57
n-Butylbenzene	91	92,134	Isopropylbenzene	105	120
sec-Butylbenzene	105	134	p-Isopropyltoluene	119	134,91
tert-Butylbenzene	119	91,134	Methylene chloride	84	86,49
Carbon disulfide	76	78	4-Methyl-2-pentanone	43	58,57
Carbon tetrachloride	119	121	Methyl tert butyl ether	73	57,43
Chlorobenzene	112	77,114	Naphthalene	128	51,129
Chloroethane	64	66	n-Propylbenzene	91	120
2-Chloroethylvinyl ether	63	65,106	Styrene	104	78
Chloroform	83	85	1,1,1,2-Tetrachloroethane	131	133,119
Chloromethane	50	52	1,1,2,2-Tetrachloroethane	83	85
2-Chlorotoluene	91	126	Tetrachloroethene	166	168,129
4-Chlorotoluene	91	126	Tetrahydrofuran	42	72,71
Dibromochloromethane	129	127,131	Toluene	92	91
1,2-Dibromo-3-chloropropane	157	155	1,2,3-Trichlorobenzene	180	182,145
1,2-Dibromoethane	107	109	1,2,4-Trichlorobenzene	180	182,145
Dibromomethane	93	95,174	1,1,1-Trichloroethane	97	99,61
1,2-Dichlorobenzene	146	111,148	1,1,2-Trichloroethane	83	97,85,99
1,3-Dichlorobenzene	146	111,148	Trichloroethene	95	130,132
1,4-Dichlorobenzene	146	111,148	Trichlorofluoromethane	101	103,105
Dichlorodifluoromethane	85	87	1,2,3-Trichloropropane	75	110
1,1-Dichloroethane	63	65,83	1,2,4-Trimethylbenzene	105	120
1,2-Dichloroethane	62	98,64	1,3,5-Trimethylbenzene	105	120
1,1-Dichloroethene	96	61,63	Vinyl chloride	62	64
cis-1,2-Dichloroethene	96	61,98	Vinyl acetate	43	86
trans-1,2-Dichloroethene	96	61,98	o-Xylene	106	91
1,2-Dichloropropane	63	76,112	m/p-Xylene	106	91
1,3-Dichloropropane	76	78			
			SSTD		
ISTD			Dibromofluoromethane	113	111,192
Fluorobenzene	96	77	1,2-Dichloroethane-d_4	102	104
Chlorobenzene-d_5	117		Toluene-d_8	98	100
1,4-Dichlorobenzene-d_4	152		4-Bromofluorobenzene	95	174,176

***Refer to Method 8260C for characteristic ions not listed here**

FV04-01 (Example)
8260C Checklist



FVO4-01 Data Review Checklist

FORM # FVO4-01
Rev. #: 1.1
Effective Date: 4/01/15

INDEPENDENT DATA REVIEW CHECKLIST				Method: GCMS (EPA SW-846 8260C)			
Analysis Date	LIMs Run #(s):	Analyst Data Interpreter	Independent Reviewer	Date of Review	Approved?	🟩	Instrument
					Yes		VMS

Instructions: Complete one checklist per analytical sequence. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance criteria	Analyst	Review	Independent Review		Comments:
I. BFB Tune Check						
Was a BFB tune check analyzed with acceptable results?	Relative abundance criteria met?	Yes		Yes		If no, Do not proceed with analyses.
II. Initial Calibration Verification (ICV)						
Was initial calibration performed using a minimum of five standard concentration levels (minimum of 6 levels for quadratic curves)?	Lowest standard at or below RL?	Yes		Yes		If no, recalibrate with required # of levels
Were the Average Relative Response Factors (ARRF) acceptable?	ARRF≥ specified limits (see SOP)	Yes		Yes		If no, analyses stopped, recalibrate.
Was a second-source ICV analyzed?	Required before sample analyses	Yes		Yes		If no, analyze prior to sample analyses.
Were all target compound %Deviation or %Drift acceptable?	%D < 20% or project/program specific					If no, reanalyzed ICV to address failures
Was Initial Calibration Blank (ICB) analyzed?	Required before sample analyses.	Yes		Yes		If no, analyzed ICB before sample analyses.
Were the ICB results for all target analytes less than the limit of detection (LOD).	<LOD or = project/program specific limits					If no, analyze another blank to address detects.
III. Continuing Calibration Verification (CCV)						
Was an acceptable BFB tune check run at the beginning of every twelve hour shift?	Relative abundance criteria met?	Yes		Yes		If no, reanalyze BFB until acceptable; re-tuning the instrument may be necessary.
Was a CCV analyzed after every 12 hour tune check?	Required before sample analyses.	Yes		Yes		If no, analyzed CCV after acceptable tune check.
Were all target compound %Deviation or %Drift acceptable?	%D < 20% or project/program specific					If no, reanalysis or recalibration may be required.
If necessary, were the results for outlying compounds qualified?						If yes, results qualified "Z"

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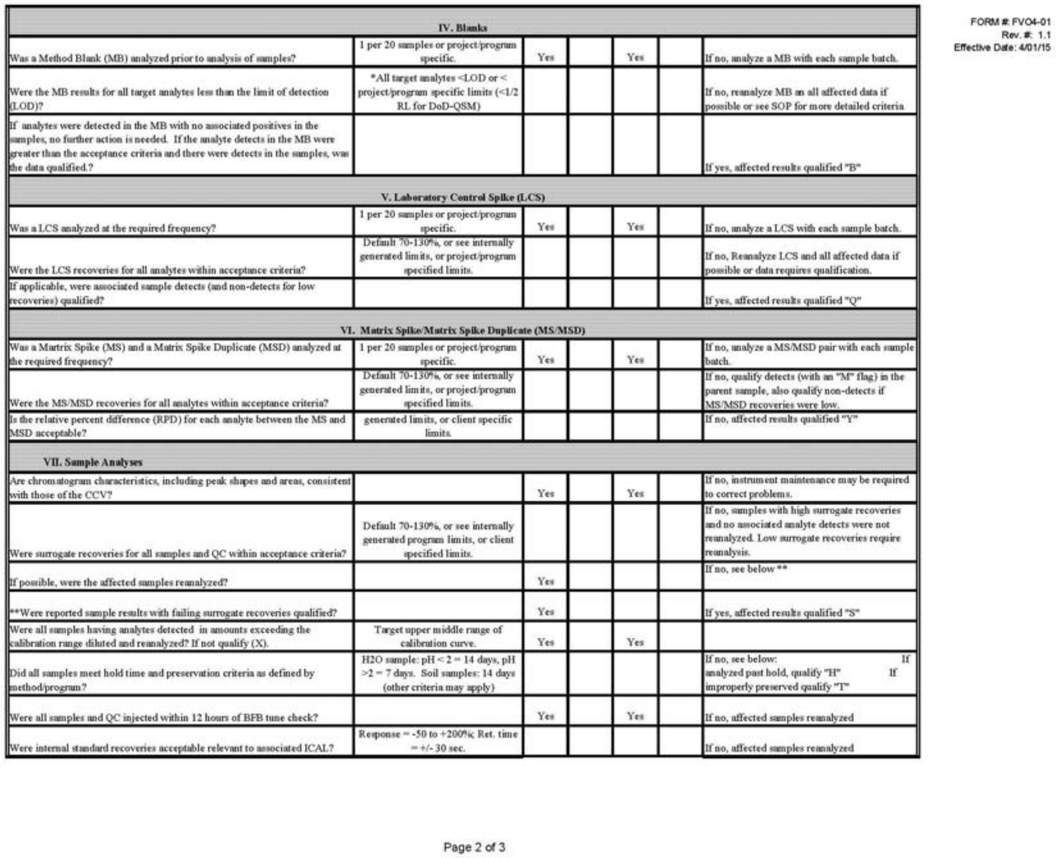

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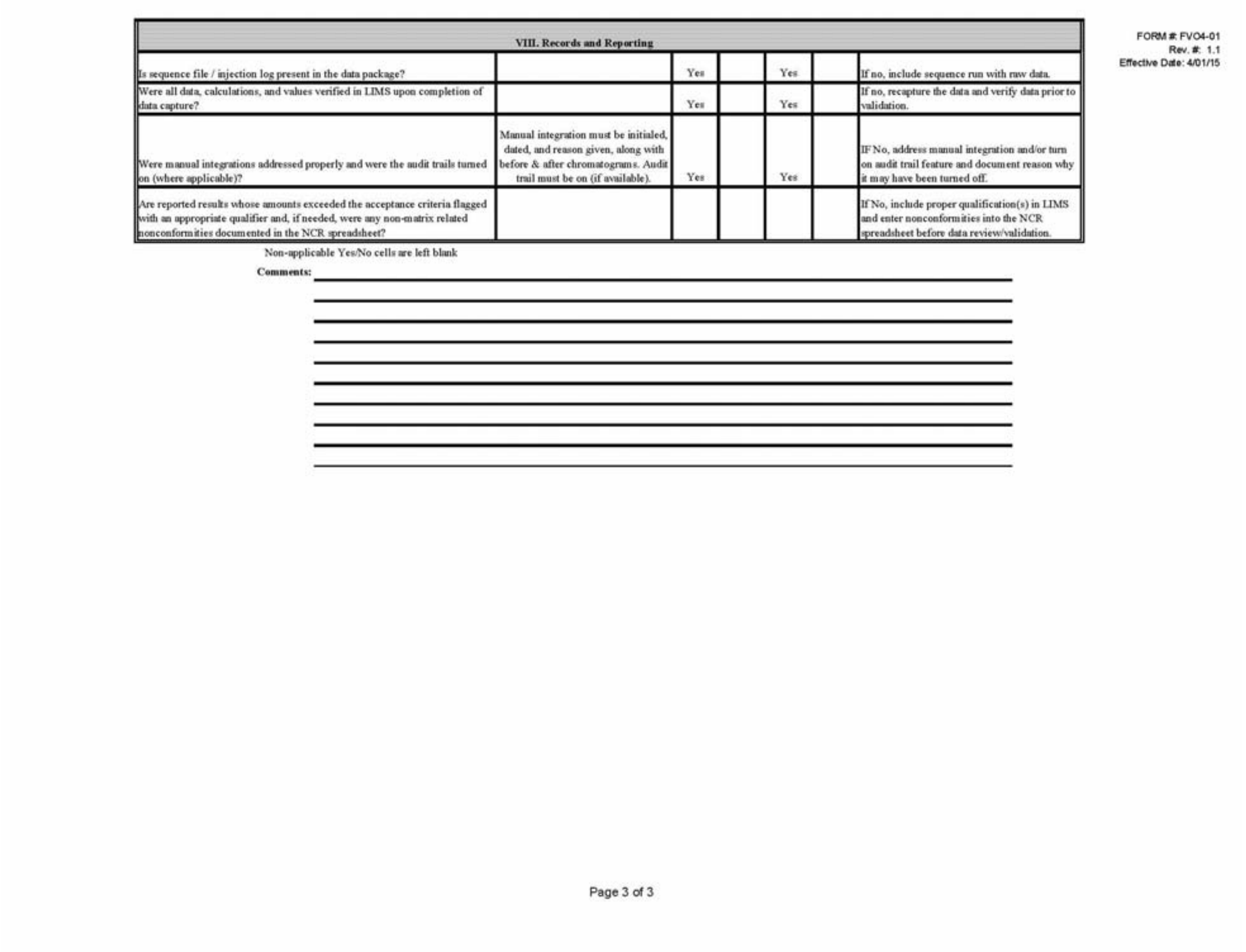
IV. Blanks						
Was a Method Blank (MB) analyzed prior to analysis of samples?	1 per 20 samples or project/program specific.	Yes		Yes		If no, analyze a MB with each sample batch.
Were the MB results for all target analytes less than the limit of detection (LOD)?	*All target analytes <LOD or < project/program specific limits (<1/2 RL for DoD-QSM)					If no, reanalyze MB an all affected data if possible or see SOP for more detailed criteria
If analytes were detected in the MB with no associated positives in the samples, no further action is needed. If the analyte detects in the MB were greater than the acceptance criteria and there were detects in the samples, was the data qualified.?						If yes, affected results qualified "B"
V. Laboratory Control Spike (LCS)						
Was a LCS analyzed at the required frequency?	1 per 20 samples or project/program specific.	Yes		Yes		If no, analyze a LCS with each sample batch.
Were the LCS recoveries for all analytes within acceptance criteria?	Default 70-130%, or see internally generated limits, or project/program specified limits.					If no, Reanalyze LCS and all affected data if possible or data requires qualification.
If applicable, were associated sample detects (and non-detects for low recoveries) qualified?						If yes, affected results qualified "Q"
VI. Matrix Spike/Matrix Spike Duplicate (MS/MSD)						
Was a Martrix Spike (MS) and a Matrix Spike Duplicate (MSD) analyzed at the required frequency?	1 per 20 samples or project/program specific.	Yes		Yes		If no, analyze a MS/MSD pair with each sample batch.
Were the MS/MSD recoveries for all analytes within acceptance criteria?	Default 70-130%, or see internally generated limits, or project/program specified limits.					If no, qualify detects (with an "M" flag) in the parent sample, also qualify non-detects if MS/MSD recoveries were low.
Is the relative percent difference (RPD) for each analyte between the MS and MSD acceptable?	generated limits, or client specific limits.					If no, affected results qualified "Y"
VII. Sample Analyses						
Are chromatogram characteristics, including peak shapes and areas, consistent with those of the CCV?		Yes		Yes		If no, instrument maintenance may be required to correct problems.
Were surrogate recoveries for all samples and QC within acceptance criteria?	Default 70-130%, or see internally generated program limits, or client specified limits.					If no, samples with high surrogate recoveries and no associated analyte detects were not reanalyzed. Low surrogate recoveries require reanalysis.
If possible, were the affected samples reanalyzed?		Yes				If no, see below **
**Were reported sample results with failing surrogate recoveries qualified?		Yes				If yes, affected results qualified "S"
Were all samples having analytes detected in amounts exceeding the calibration range diluted and reanalyzed? If not qualify (X).	Target upper middle range of calibration curve.	Yes		Yes		
Did all samples meet hold time and preservation criteria as defined by method/program?	H2O sample: pH < 2 = 14 days, pH >2 = 7 days. Soil samples: 14 days (other criteria may apply)					If no, see below: If analyzed past hold, qualify "H" If improperly preserved qualify "T"
Were all samples and QC injected within 12 hours of BFB tune check?		Yes		Yes		If no, affected samples reanalyzed
Were internal standard recoveries acceptable relevant to associated ICAL?	Response = -50 to +200%; Ret. time = +/- 30 sec.					If no, affected samples reanalyzed

Page 2 of 3



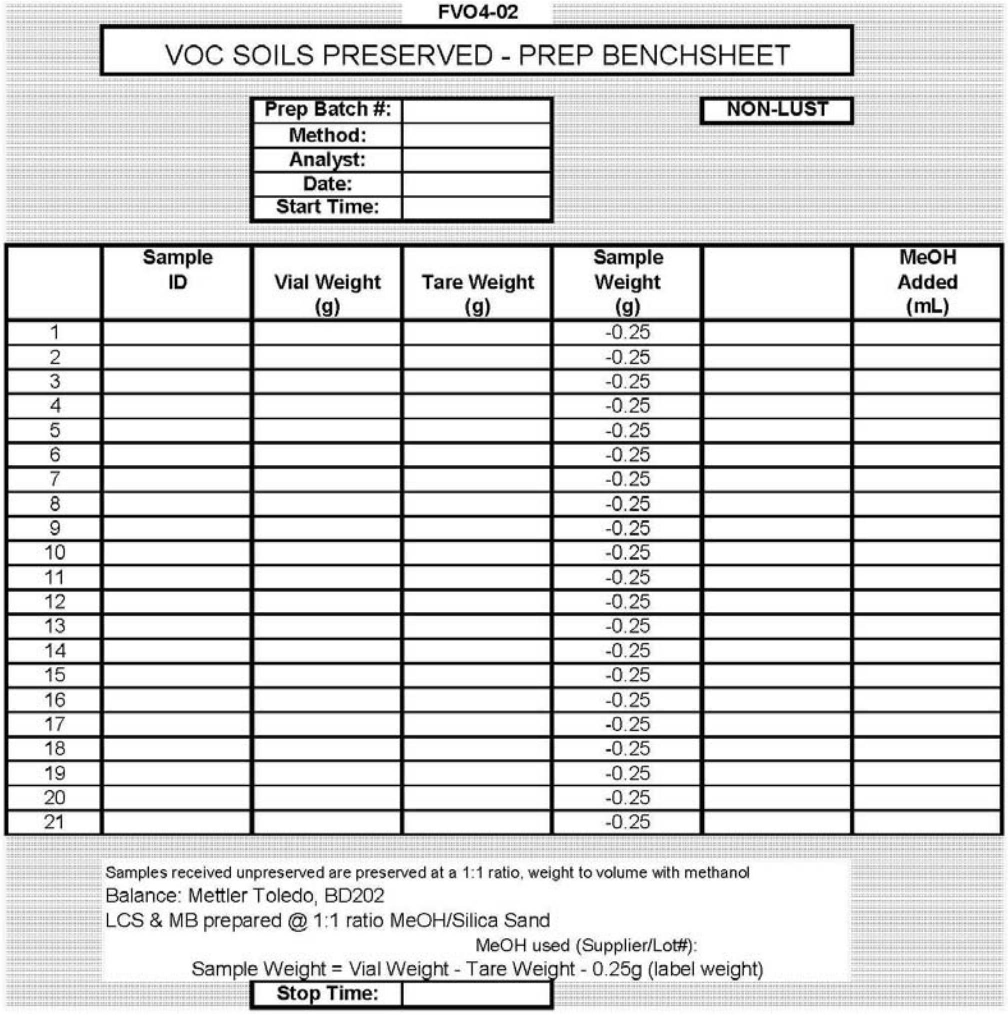
FORM # FVO4-01
Rev. #: 1.1
Effective Date: 4/01/15

VIII. Records and Reporting						
Is sequence file / injection log present in the data package?		Yes		Yes		If no, include sequence run with raw data.
Were all data, calculations, and values verified in LIMS upon completion of data capture?		Yes		Yes		If no, recapture the data and verify data prior to validation.
Were manual integrations addressed properly and were the audit trails turned on (where applicable)?	Manual integration must be initialed, dated, and reason given, along with before & after chromatograms. Audit trail must be on (if available).	Yes		Yes		IF No, address manual integration and/or turn on audit trail feature and document reason why it may have been turned off.
Are reported results whose amounts exceeded the acceptance criteria flagged with an appropriate qualifier and, if needed, were any non-matrix related nonconformities documented in the NCR spreadsheet?						If No, include proper qualification(s) in LIMS and enter nonconformities into the NCR spreadsheet before data review/validation.

Non-applicable Yes/No cells are left blank

Comments: ______________________________

FVO4-02 (Example)
VOC Soils Preserved Prep Bench Sheet (Non-Lust)



FVO4-02

VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:	
Method:	
Analyst:	
Date:	
Start Time:	

NON-LUST

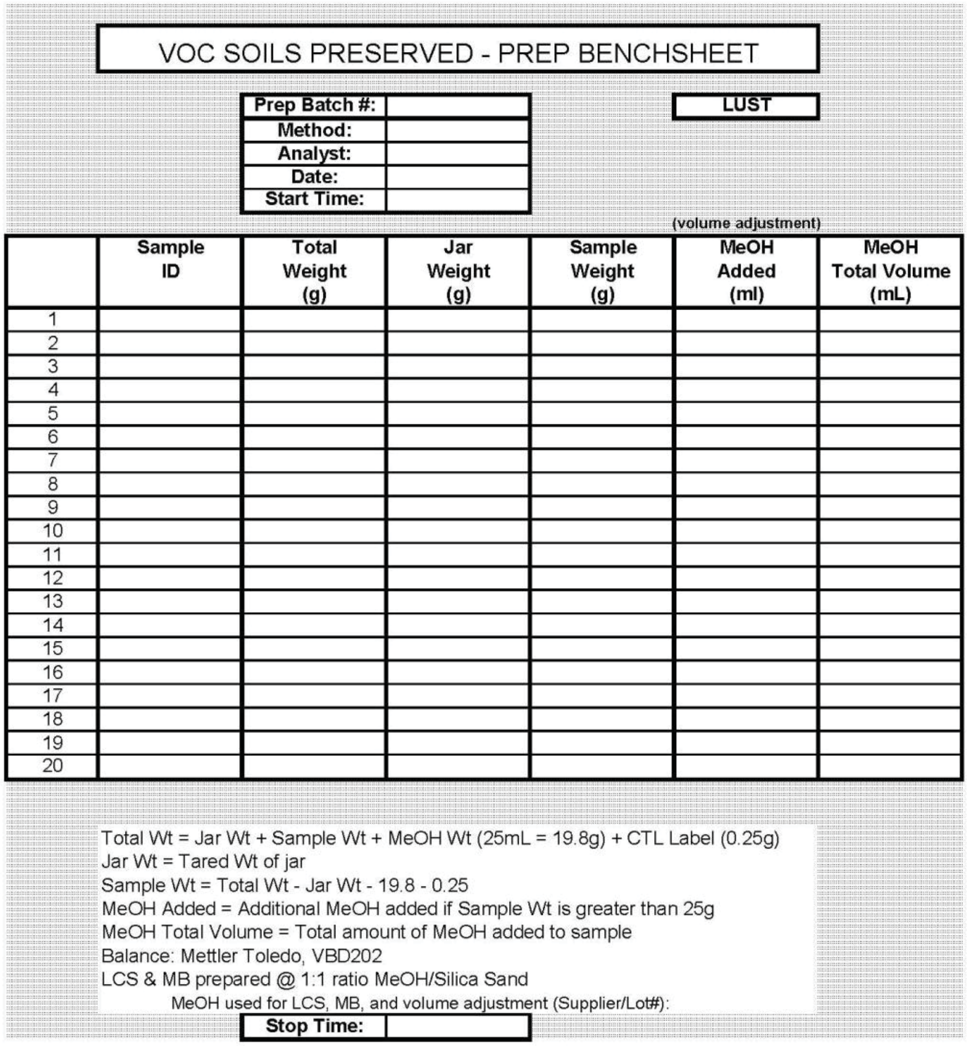
	Sample ID	Vial Weight (g)	Tare Weight (g)	Sample Weight (g)		MeOH Added (mL)
1				-0.25		
2				-0.25		
3				-0.25		
4				-0.25		
5				-0.25		
6				-0.25		
7				-0.25		
8				-0.25		
9				-0.25		
10				-0.25		
11				-0.25		
12				-0.25		
13				-0.25		
14				-0.25		
15				-0.25		
16				-0.25		
17				-0.25		
18				-0.25		
19				-0.25		
20				-0.25		
21				-0.25		

Samples received unpreserved are preserved at a 1:1 ratio, weight to volume with methanol
Balance: Mettler Toledo, BD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used (Supplier/Lot#):
Sample Weight = Vial Weight - Tare Weight - 0.25g (label weight)

Stop Time:

VocSoilPresNonLust_FVO3-02 05/07/201415:07

FVO4-03 (Example)
VOC Soils Preserved Prep Bench Sheet (Lust)



VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:
Method:
Analyst:
Date:
Start Time:

LUST

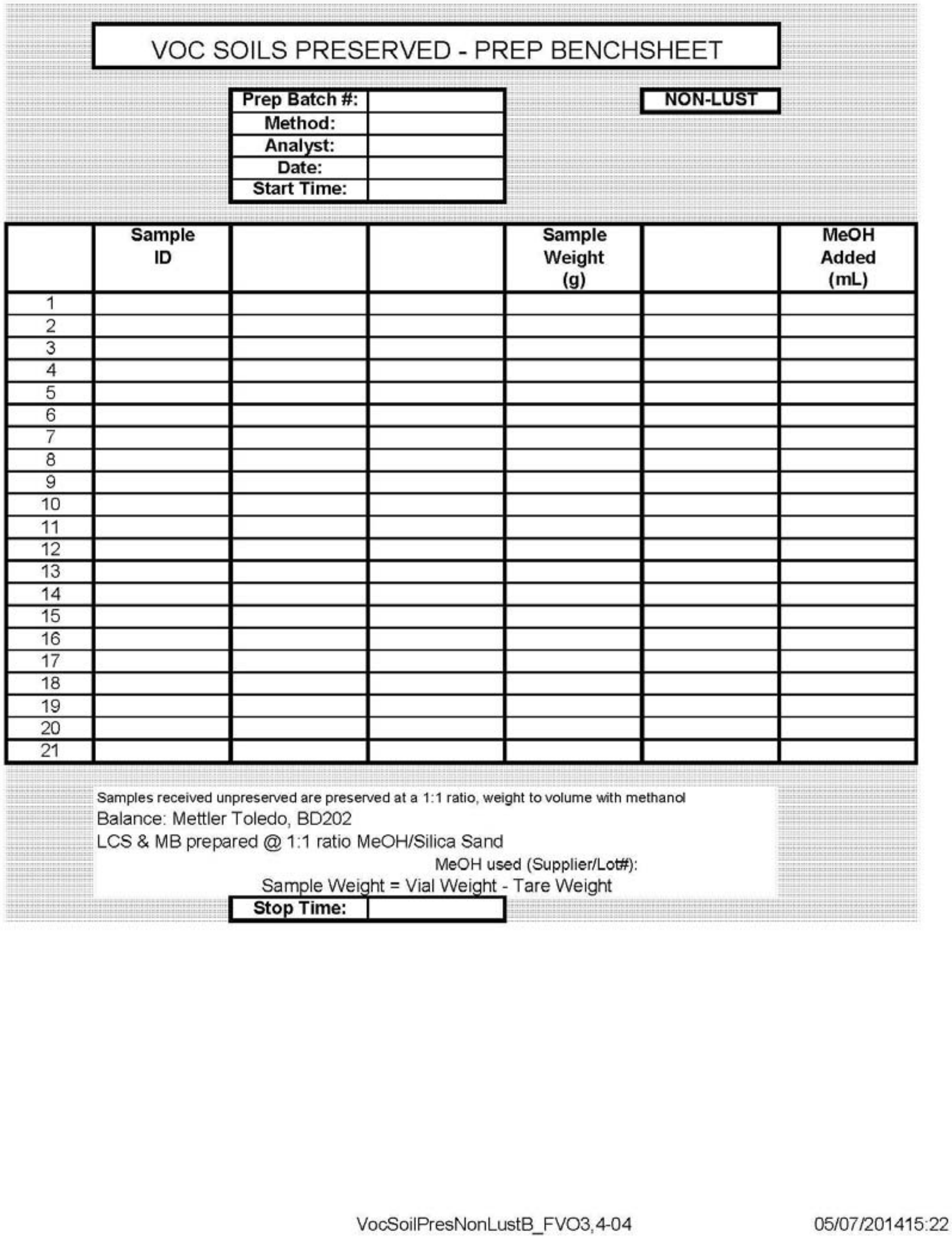
	Sample ID	**Total Weight (g)**	**Jar Weight (g)**	**Sample Weight (g)**	**(volume adjustment)** **MeOH Added (ml)**	**MeOH Total Volume (mL)**
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

Total Wt = Jar Wt + Sample Wt + MeOH Wt (25mL = 19.8g) + CTL Label (0.25g)
Jar Wt = Tared Wt of jar
Sample Wt = Total Wt - Jar Wt - 19.8 - 0.25
MeOH Added = Additional MeOH added if Sample Wt is greater than 25g
MeOH Total Volume = Total amount of MeOH added to sample
Balance: Mettler Toledo, VBD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used for LCS, MB, and volume adjustment (Supplier/Lot#):

Stop Time:

VocSoilPresLust_FVO3-03 05/07/201415:13

FVO4-04 (Example)
VOC Soils Preserved Prep Bench Sheet (Non-Lust) B



VOC SOILS PRESERVED - PREP BENCHSHEET

NON-LUST

Prep Batch #:
Method:
Analyst:
Date:
Start Time:

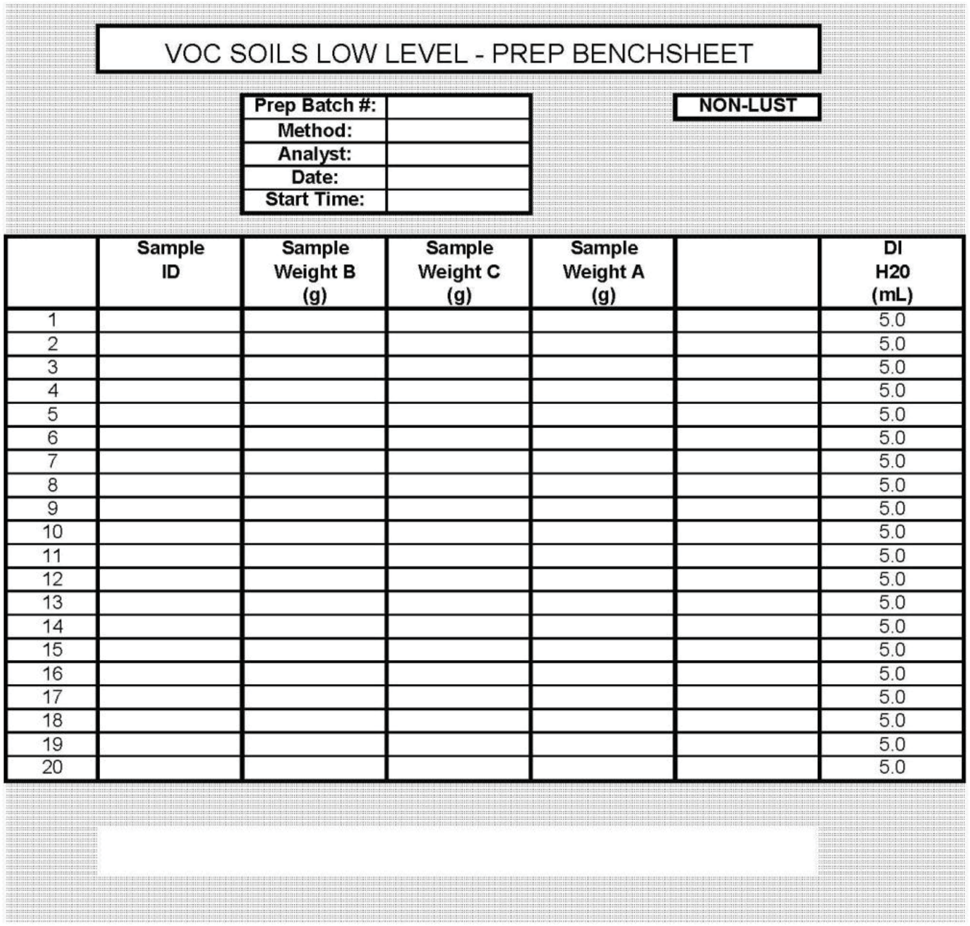
	Sample ID			Sample Weight (g)		MeOH Added (mL)
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						
21						

Samples received unpreserved are preserved at a 1:1 ratio, weight to volume with methanol
Balance: Mettler Toledo, BD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used (Supplier/Lot#):
Sample Weight = Vial Weight - Tare Weight

Stop Time:

VocSoilPresNonLustB_FVO3,4-04 05/07/201415:22

FVO4-05 (Example)
VOC Soils Low Level Prep Bench Sheet (Non-Lust)



VOC SOILS LOW LEVEL - PREP BENCHSHEET

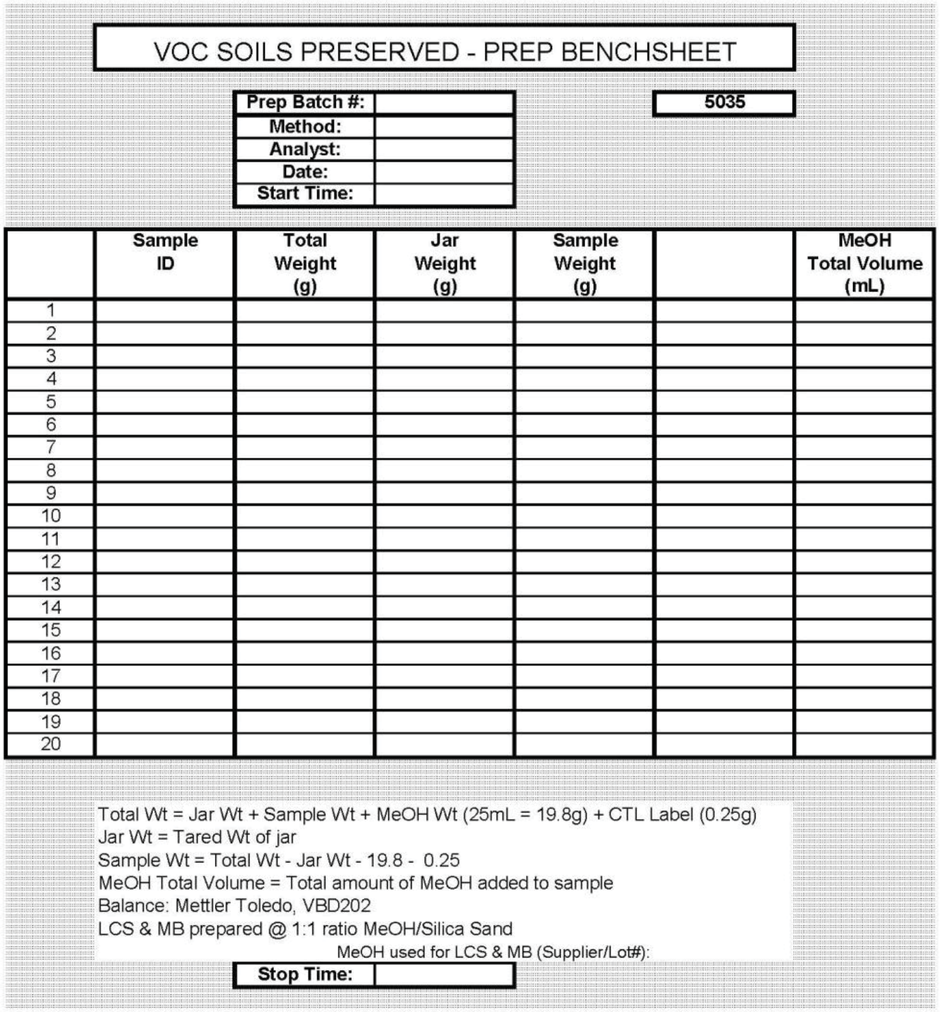
Prep Batch #:
Method:
Analyst:
Date:
Start Time:

NON-LUST

	Sample ID	**Sample Weight B (g)**	**Sample Weight C (g)**	**Sample Weight A (g)**		**DI H20 (mL)**
1						5.0
2						5.0
3						5.0
4						5.0
5						5.0
6						5.0
7						5.0
8						5.0
9						5.0
10						5.0
11						5.0
12						5.0
13						5.0
14						5.0
15						5.0
16						5.0
17						5.0
18						5.0
19						5.0
20						5.0

Voc LLSoil Temp_FVO3-05 05/07/201415:20

FVO4-06 (Example)
VOC Soils Preserved Prep Bench Sheet (5035)



VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:
Method:
Analyst:
Date:
Start Time:

5035

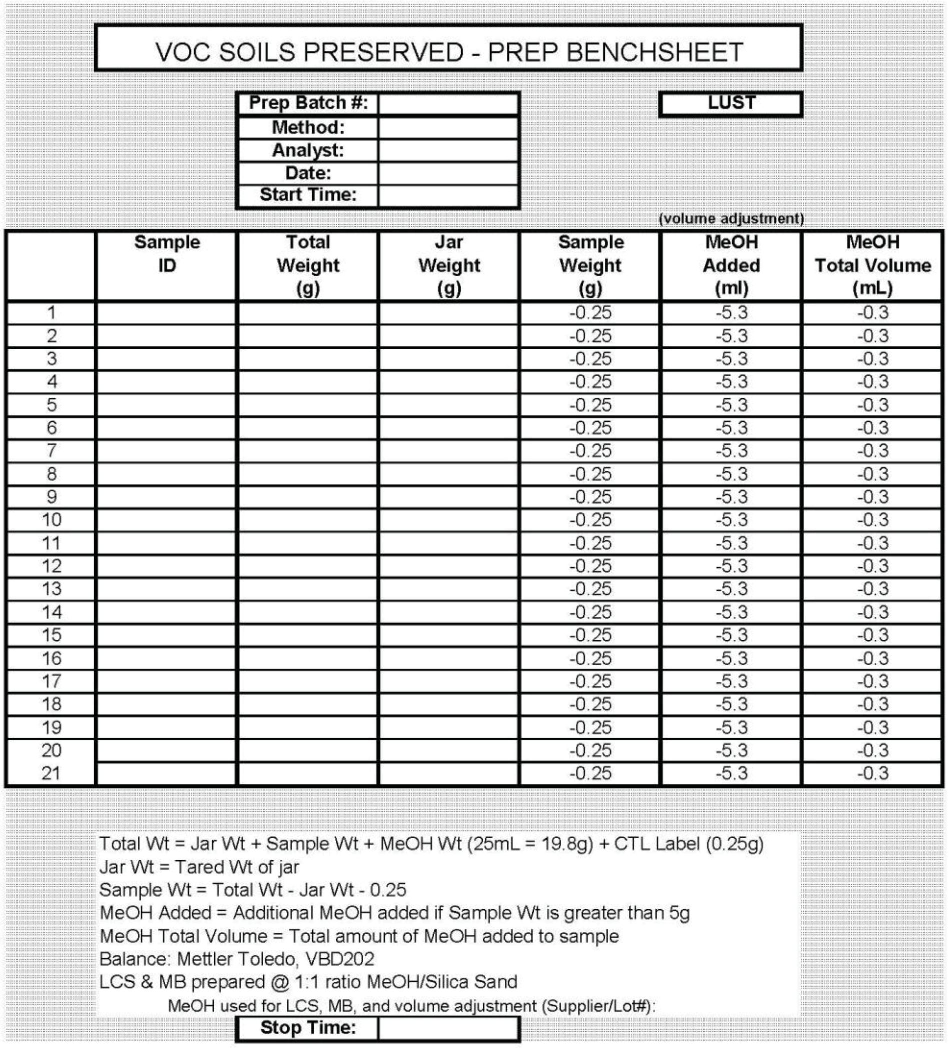
	Sample ID	**Total Weight (g)**	**Jar Weight (g)**	**Sample Weight (g)**		**MeOH Total Volume (mL)**
1						
2						
3						
4						
5						
6						
7						
8						
9						
10						
11						
12						
13						
14						
15						
16						
17						
18						
19						
20						

Total Wt = Jar Wt + Sample Wt + MeOH Wt (25mL = 19.8g) + CTL Label (0.25g)
Jar Wt = Tared Wt of jar
Sample Wt = Total Wt - Jar Wt - 19.8 - 0.25
MeOH Total Volume = Total amount of MeOH added to sample
Balance: Mettler Toledo, VBD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used for LCS & MB (Supplier/Lot#):

Stop Time:

VocSoilPres25mL_FVO3-06 05/07/201415:25

FVO4-07 (Example)
VOC Soils Preserved Prep Bench Sheet (Lust) 5 mL



VOC SOILS PRESERVED - PREP BENCHSHEET

Prep Batch #:	
Method:	
Analyst:	
Date:	
Start Time:	

LUST

(volume adjustment)

	Sample ID	**Total Weight (g)**	**Jar Weight (g)**	**Sample Weight (g)**	**MeOH Added (ml)**	**MeOH Total Volume (mL)**
1				-0.25	-5.3	-0.3
2				-0.25	-5.3	-0.3
3				-0.25	-5.3	-0.3
4				-0.25	-5.3	-0.3
5				-0.25	-5.3	-0.3
6				-0.25	-5.3	-0.3
7				-0.25	-5.3	-0.3
8				-0.25	-5.3	-0.3
9				-0.25	-5.3	-0.3
10				-0.25	-5.3	-0.3
11				-0.25	-5.3	-0.3
12				-0.25	-5.3	-0.3
13				-0.25	-5.3	-0.3
14				-0.25	-5.3	-0.3
15				-0.25	-5.3	-0.3
16				-0.25	-5.3	-0.3
17				-0.25	-5.3	-0.3
18				-0.25	-5.3	-0.3
19				-0.25	-5.3	-0.3
20				-0.25	-5.3	-0.3
21				-0.25	-5.3	-0.3

Total Wt = Jar Wt + Sample Wt + MeOH Wt (25mL = 19.8g) + CTL Label (0.25g)
Jar Wt = Tared Wt of jar
Sample Wt = Total Wt - Jar Wt - 0.25
MeOH Added = Additional MeOH added if Sample Wt is greater than 5g
MeOH Total Volume = Total amount of MeOH added to sample
Balance: Mettler Toledo, VBD202
LCS & MB prepared @ 1:1 ratio MeOH/Silica Sand
MeOH used for LCS, MB, and volume adjustment (Supplier/Lot#):

Stop Time:	

VocSoilPresLust5mL_FVO3,4-07 05/07/201415:29

Table 4
Recommended Minimum relative response factor criteria for Initial and Continuing Calibration Verification

Volatile Compounds	Minimum Response Factor (RF)
Dichlorodifluoromethane	0.100
Chloromethane	0.100
Vinyl chloride	0.100
Bromomethane	0.100
Chloroethane	0.100
Trichlorofluoromethane	0.100
1,1-Dichloroethene	0.100
1,1,2-Trichloro-1,2,2-trifluoroethane	0.100
Acetone[1]	0.100
Carbon disulfide	0.100
Methyl Acetate	0.100
Methylene chloride	0.100
trans-1,2-Dichloroethene	0.100
cis-1,2-Dichloroethene	0.100
Methyl tert-Butyl Ether	0.100
1,1-Dichloroethane	0.200
2-Butanone[1]	0.100
Chloroform	0.200
1,1,1-Trichloroethane	0.100
Cyclohexane	0.100
Carbon tetrachloride	0.100
Benzene	0.500
1,2-Dichloroethane	0.100
Trichloroethene	0.200
Methylcyclohexane	0.100
1,2-Dichloropropane	0.100
Bromodichloromethane	0.200
cis-1,3-Dichlorpropene	0.200
trans-1,3-Dichlorpropene	0.100
4-Methyl-2-pentanone[1]	0.100
Toluene	0.400
1,1,2-Trichloroethane	0.100
Tetrachloroethene	0.200
2-Hexanone[1]	0.100
Dibromochloromethane	0.100
1,2-Dibromoethane	0.100

Volatile Compounds	Minimum Response Factor(RF)
Chlorobenzene	0.500
Ethylbenzene	0.100
Meta-/para-Xylene	0.100
Ortho-Xylene	0.300
Styrene	0.300
Bromoform	0.100
Isopropylbenzene	0.100
1,1,2,2-Tetrachloroethane	0.300
1,3-Dichlorobenzene	0.600
1,4-Dichlorobenzene	0.500
1,2-Dichlorobenzene	0.400
1,2-Dibromo-3-chloropropane	0.050
1,2,4-Trichlorbenzene	0.200

[1] Due to low response at standard levels, these compounds are run at a concentration ten times the normal.

20.0 Description of Changes

Revision Number	Description of Changes	Date
00	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	03/12/2014
01	Changed SOP to new format, updated for QSM 5.0	01/28/2015
02	Added SIM mode analyses to Scope & Application	03/09/2015

STANDARD OPERATING PROCEDURE
WC 034 Flash-Point by Pensky-Martens Closed Cup Tester

Review Date: 04/01/16

Lauren Silvers

04/01/2016

Technical Review by: Date

Colleen Stei

04/01/2016

Approved by: Quality Assurance Date

Table of Contents

1. SCOPE OF APPLICABILITY

1.1. This procedure is used to determine the flash point of solids and liquids based on the method SW 846-1010A.

1.2. Method detection limits (MDLs) are not applicable to this test.

2. SUMMARY OF METHOD

2.1. The sample is heated at a slow, constant rate, in a closed vessel. A small flame is directed into the cup at regular intervals, while simultaneously interrupting stirring. The flash point is the lowest temperature at which the test flame ignites the vapor above the sample.

3. DEFINITIONS

3.1. For full definitions of many of the terms applicable to this method, see Appendix 10 of the Quality Assurance Manual (QAM).

3.2. For a list of common acronyms and abbreviations, see QAM Appendix 7.

4. HEALTH AND SAFETY

4.1. Gloves and protective clothing should be worn to protect against unnecessary exposure to possibly hazardous chemicals and contaminates in samples. All activities performed while following this procedure should utilize appropriate laboratory safety systems (see CTL Health and Safety Manual).

4.2. p-Xylene is flammable and should be handled with care around an open flame.

4.3. All samples are potentially flammable and should be treated with care around an open flame.

4.4. Samples that contain hydrocarbons may cause an enlargement and a color change, from blue to orange-yellow, to the test flame. Continued heating of the sample above the ambient temperature can result in significant burning of vapors outside the cup and can be a potential fire hazard. Discontinue testing if this occurs.

5. INTERFERENCES

5.1. Samples that contain hydrocarbons cause a significant enlargement and color change, from blue to orange-yellow, of the test flame. This is not a halo effect and the sample will not flash. This phenomenon should not be treated as a flash.

5.2. If the test flame is extinguished, it will not ignite the vapors in the cup and the gas from the test flam will then enter the cup, possibly influencing the test result.

5.3. When the ignition source is a test flame, the application of the test flame may cause a blue halo or an enlarged flame prior to the actual flash point. This is not a flash and will be ignored.

5.4. Cleaning solvents left in the cup may influence results.

6. EQUIPMENT AND SUPPLIES

6.1. Pensky-Martens Flash Point Tester, Semi-automatic: Precision Scientific Model 32140 or equivalent.
6.2. Thermometer, Temperature range from 20-230°F: Traceable thermometer, Fisher Scientific cat. # 15-077-61 or equivalent.
6.3. Propane gas cylinder (Purchased locally).
6.4. Lighter.

7. REAGENTS AND STANDARDS

7.1. <u>p-Xylene: </u>Fisher cat. # 05082-500 or equivalent.
7.2. <u>Acetone (When soap and water do not entirely clean the vessel): </u>J. T Baker cat. # 9254-03 or equivalent.

8. SAMPLE HANDLING AND PRESERVATION

8.1. Samples should be collected in a glass or plastic container that is not gas permeable.
8.2. Samples should not be taken from a container that is less than 50% full.
8.3. Sample volume for each test is at least 75ml.
8.4. Samples should be cooled to 0-6 °C until analysis.
8.5. Caution should be used to avoid the loss of volatile material.

9. PROCEDURE

9.1. If placing the flash point apparatus in the hood, use a draft shield and turn the exhaust fan off.
9.2. Fill out the bench sheet (FWC34-02).
9.3. Fill the brass test cup with at least 75ml of sample to be tested.
9.4. Turn on stirrer, but only connect the stirrer to the cup if the sample is liquid.
9.5. Light the test flame. Adjust the flame to 3.2-4-8 mm in diameter. Gas pressure of the ignition source should not exceed 3 kPa.
9.6. Lower the test flame into the cup and see if it ignites.

9.7. If the sample does not ignite, turn the heater on so that the temperature increases approximately 10°F/minute.

9.8. Apply the test flame when the sample is 40° below the expected flash point temperature. If the flash point is unknown, start at room temperature (60±10°).

9.9. Continue applying the test flame every 5° F for 1 second in the cup until a flash is observe, or 140°F is passed. If the flashpoint is unknown, apply test flame every 2° F.

9.10. If a flash is seen, discard the sample and reanalyze a fresh portion of the sample. Begin applying the flame at 41±9° F below the temperature of the initial flash point.

9.11. Report flash point as the temperature where the flash was seen, or if no flash was seen before exceeding 140°, >140°F.

9.12. After the sample cup and apparatus have cooled, remove the cup and dispose of the sample.

9.13. Clean the cup with soap and water. Use the acetone to remove any residual sample and dry the cup thoroughly.

9.14. Sample results are then hand entered into LIMS.

10. CALCULATIONS AND DATA ANALYSIS AND REDUCTION

10.1. Accuracy: LCS

$$\text{\% Recovery} = \frac{\text{measured value}}{\text{true value}} \times 100$$

10.2. Precision- Relative percent difference (RPD):

$$\text{\% RPD} = \frac{\text{(sample conc. – duplicate con.)}}{\text{(sample conc. + dup.conc./2)}} \times 100$$

11. METHOD PERFORMANCE

11.1. Certified standard solutions and chemicals, properly used instrumentation, and analyst experience and expertise are critical elements in producing accurate results. Standards and instrument performance are continually checked by analyzing external performance test samples provided by appropriately accredited agencies. Internal blind spikes are also utilized to check analyst performance.

11.2. Initial demonstration of capability (IDC) is required for all analysts to ensure acceptable method performance. An analyst must demonstrate initial precision and accuracy through the analysis of 4-5 laboratory control spikes for each matrix and sample type. After analysis, the analyst calculated the average recovery (AR) and the relative standard deviation (RSD) of the recoveries for each analyte. In the absence of specific criteria found in the EPA methods or project specific limits, the default criteria of 80-120% recovery and 25% RSD are used until internal limits are generated.

12. QUALITY CONTROL

12.1. This SOP is designed to follow a variety of different projects and programs requirements. Figure 2 is designed to illustrate the control steps and provisions required to adequately produce acceptable data.

12.2. Contract Specific Sample Analysis: For certain samples, limits are specified by the QAPP (Quality Assurance Project Plan) associated with a given project. For these samples follow the limits specified in the QAPP for that project.

12.3. Program Specific Limits: Samples analyzed under the guidance of certain programs; such as the Department of Defense Quality Systems Manual (DoD /QSM) or Louisville Chemistry Guidance (LCG), require their own specified limits. For these samples follow the limits specified in the manuals for that program.

13. DATA ASSESSMENT/ACCEPTANCE CRITERIA FOR QC MEASURES

13.1. When the analysis of an analytical batch or sequence has been completed, the data is processed and prepared for reporting. The analyst will review the data to ensure QC is acceptable and that exceedances are addressed. Acceptable data is then entered into LIMS.

13.2. After data has been entered into LIMS, it is reviewed by the analyst for accuracy and completeness. See checklist (FWC34-01) for data review guidance.

13.3. Once the analyst has reviewed and approved the data, it is given to a peer or supervisor for review.

13.4. After the second reviewer approves the data, the reviewer sends the data to “validated” status in LIMS.

13.5. A paper hard copy of the data is then filed or archived. The package includes the checklist, the prep batch, a copy of the bench sheet, the LIMS run log, and verification of calibration data.

14. CORRECTIVE ACTIONS FOR OUT OF CONTROL DATA

14.1. When data is out of control, a number of corrective actions may need implementing. If the nonconformities involving failing QC within the analytical sequence batch, then reanalysis of samples may eliminate any out of control data. If the out of control data is the result of instrument malfunctions, then maintenance or repair of the downed instrument followed by reanalysis of the affected data may correct the problem. If sample matrix affect or contamination is the reason for poor data, the instrument may need cleaning and decontamination. In all cases, when out of control data presents itself, the appropriate corrective measures need to be enacted to eliminate unusable data. The Quality Control Requirements chart (Figure 2) can be used as a guide as to which corrective actions should be taken for different QC-type failures or nonconformities.

15. CONTINGENCIES FOR HANDLING OUT OF CONTROL OR UNACCEPTABLE DATA

15.1. Due to limited sample volume, expiration of hold times, downed instrumentation, and analyst error, the sample data may be out of control or unacceptable to report. Since these potential instances can arise, contingency plans need to be in place to prevent and/ or minimize their effect on data.

15.1.1. The first thing addressed is prevention of producing unacceptable data. When limited sample volume is the issue, the analyst should determine if splitting the sample into lesser volumes or weight is an option. To avoid sample hold time issues, the analyst’s first responsibility is to plan accordingly. The analyst is responsible for budgeting enough time for sample analysis, so if a problem arises reanalysis is an option. Analyst error is prevented by a second analyst confirmation and validation. If the initial analyst makes an analysis error or inadvertently reports unacceptable data, the second analyst is responsible for finding and/or correcting those errors.

15.1.2. When out of control or unacceptable data is produced and it is too late for corrective measures, a number of actions can be taken. The first and foremost is alerting the client and/ responsible parties. In some instances, more samples can be made available or re-sampling can occur, so it is important to alert the appropriate personnel as soon as possible.

15.1.2.1. If the out of control data affects only specific analytes, it is important to let the appropriate person(s) know in case his or her site assessment is based on a specific target analyte list.

15.1.2.2. In all instances, if results are reported from data that is out of control or unacceptable, that data should be qualified accordingly. Once the client has been notified and he or she instructs the project manager to report the data, flag the data indicating what type of nonconformity has occurred.

15.1.2.3. Out of control data is still retained by the laboratory and filed and archived along with acceptable data. The file folder should be labeled as such, indicating that the data is out of control.

15.1.2.4. A non-conformance/ corrective action report (CAR) form must be filled out whenever these types of events occur. The information on the report includes the problem encountered, planned corrective actions, and corrective action follow-up. The form is then discussed with and signed by the analyst, the client representative, the QA officer, and the laboratory manager. The purpose of the form is to document problems in order to eliminate the possibility of repeating nonconformance and to ensure that the proper corrective actions are employed.

16. DATA RECORDS MANAGEMENT

16.1. Records are stored for a minimum of 5 years in accordance with the Quality Manual.

16.2. See SOP QA 003 for specifics on document control.

17. WASTE MANAGEMENT

17.1. Samples are routinely held up to six weeks from analysis date before they enter the waste stream. Waste disposal of samples and standards follows the procedures documented in the Laboratory Waste Disposal SOP (Quality Assurance Section, SOP No. FO-8, Rev. 4)

18. REFERENCES

18.1. CT Laboratories Quality Manual, current revision

18.2. Department of Defense, Quality Systems Manual for Environmental Laboratories, Version 5.0, July 2013 or most recent revision.

18.3. National Environmental Laboratory Accreditation Conference (NELAC), 2003 NELAC Standard Chapters 1 to 6, EPA/600/R-04/003, June 5, 2003 or most recent version.
18.4. ISO. 2005. General requirements for the competence of testing and calibration laboratories. ISO17025.
18.5. Test Methods for Evalutaing Solid Waste, EPA SW-846 Method 1010 3rd Edition Rev 0, November 2004.
18.6. Precision Scientific Pensky-Martens Flash Point Tester Semi-Automatic Manual

19. FIGURES

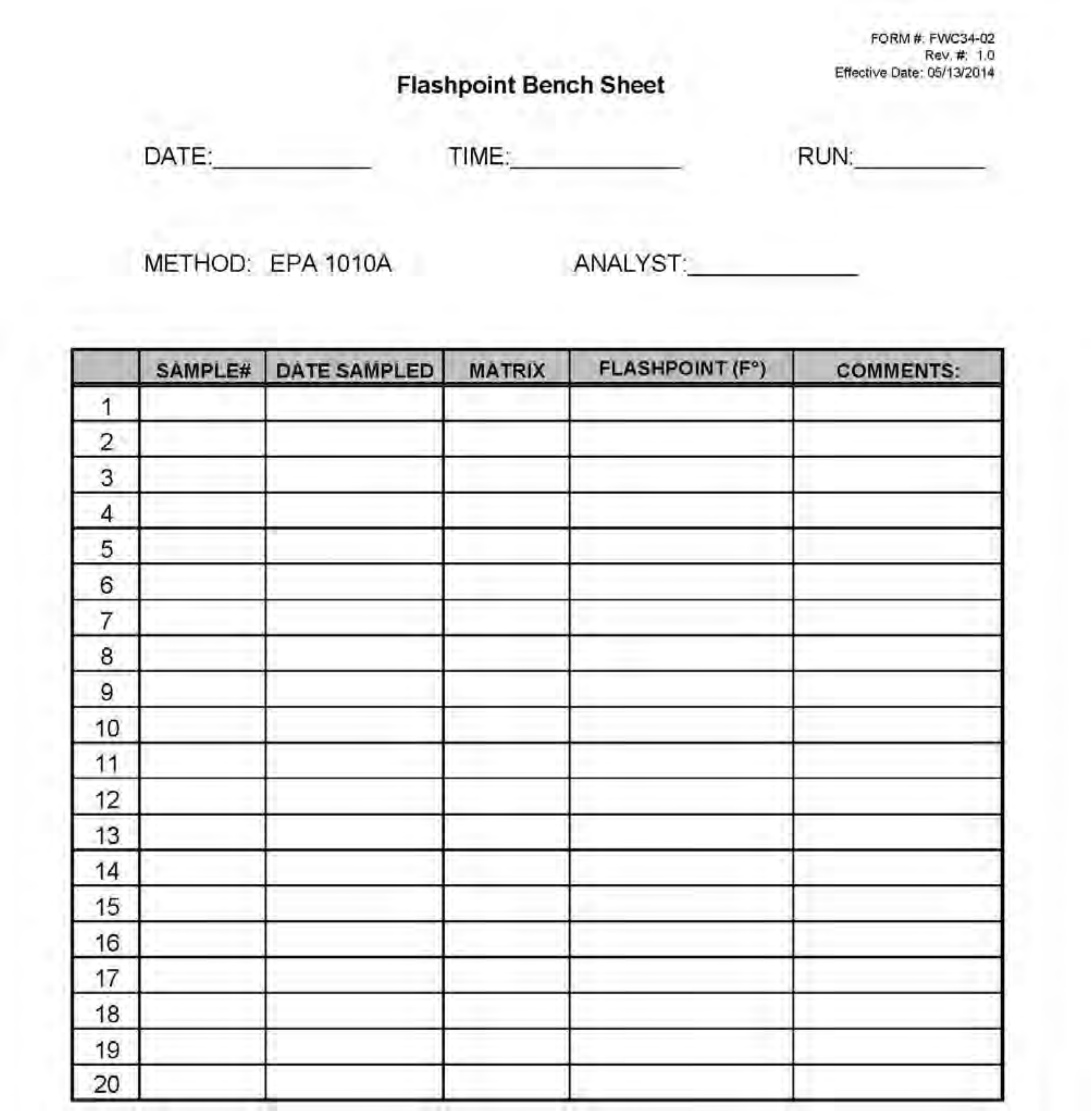
19.1. FWC34-01 Flashpoint Data Validation Checklist (Example)

LIMS #:	Method: Flashpoint EPA Method 1010			
Analysis Date	Analyst / Data Interpreter	Independent Reviewer	Date of Review	Approved
				Yes … No

Instructions: Complete one checklist per *analytical run*. Enter the appropriate response for each question. Each "No" response requires an explanation in the Comments section, and may require the initiation of a Nonconformance Report.

Requirement:	Acceptance Criteria	Analyst Review		Independent Review		Comments: (indicate reference to an attachment if necessary)
		Yes	No	Yes	No	
1. Was LCS within acceptable limits?	77.9-81.9 F					
2. Were duplicates analyzed at the appropriate frequency	1 every 20 samples of the same matrix					
3. Were the duplicates within acceptable limits?	Differ by ≤ 5 F					
2. Are all samples on the job lists accounted for?	---					

19.2. **FWC34-02 Flashpoint Bench Sheet (Example)**



FORM #: FWC34-02
Rev. #: 1.0
Effective Date: 05/13/2014

Flashpoint Bench Sheet

DATE:____________ TIME:____________ RUN:__________

METHOD: EPA 1010A ANALYST:____________

	SAMPLE#	DATE SAMPLED	MATRIX	FLASHPOINT (F°)	COMMENTS:
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					

19.3. Summary of Quality Control Requirements

Procedure	Frequency of Procedure	Acceptance Criteria	Corrective Action if Unacceptable
Capability demonstration sample (IDC)	Four (4) prepared samples analyzed one time prior to any sample analyses	In-house determined criteria for LCS recovery and precision	Repeat until acceptable
Sample duplicate (DUP)	One (1) per analytical batch per matrix or contract/projects specific. DOD-QSM: One (1) per ten samples	In-house derived limits Default: RPD ≤ 25% if analytes > RL or contract/project specific	Investigate problem, if system precision is in control qualify results, if system precision is out of control reanalyze entire batch or flag results with the appropriate qualifier
Lab Control Standard (LCS)	One (1) per analytical batch	%R: within in-house limits or contract/project specific DOD-QSM not to exceed %R: 80-120%	Reanalyze, if still unacceptable and the failure can not be determined to be restricted to the LCS, all associated samples must be analyzed

19.4 Description of Change Table

Revision Number	Description of Changes	Date
02	Document changed to incorporated administrative requirements of ISO 17025 and QSM 5.0. Descriptions of changes have not been tracked in previous versions of this document.	04/02/2014

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METHOD 1311

TOXICITY CHARACTERISTIC LEACHING PROCEDURE

1.0 SCOPE AND APPLICATION

1.1 The TCLP is designed to determine the mobility of both organic and inorganic analytes present in liquid, solid, and multiphasic wastes.

1.2 If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.

1.3 If an analysis of any one of the liquid fractions of the TCLP extract indicates that a regulated compound is present at such high concentrations that, even after accounting for dilution from the other fractions of the extract, the concentration would be above the regulatory level for that compound, then the waste is hazardous and it is not necessary to analyze the remaining fractions of the extract.

1.4 If an analysis of extract obtained using a bottle extractor shows that the concentration of any regulated volatile analyte exceeds the regulatory level for that compound, then the waste is hazardous and extraction using the ZHE is not necessary. However, extract from a bottle extractor cannot be used to demonstrate that the concentration of volatile compounds is below the regulatory level.

2.0 SUMMARY OF METHOD

2.1 For liquid wastes (i.e., those containing less than 0.5% dry solid material), the waste, after filtration through a 0.6 to 0.8 µm glass fiber filter, is defined as the TCLP extract.

2.2 For wastes containing greater than or equal to 0.5% solids, the liquid, if any, is separated from the solid phase and stored for later analysis; the particle size of the solid phase is reduced, if necessary. The solid phase is extracted with an amount of extraction fluid equal to 20 times the weight of the solid phase. The extraction fluid employed is a function of the alkalinity of the solid phase of the waste. A special extractor vessel is used when testing for volatile analytes (see Table 1 for a list of volatile compounds). Following extraction, the liquid extract is separated from the solid phase by filtration through a 0.6 to 0.8 µm glass fiber filter.

2.3 If compatible (i.e., multiple phases will not form on combination), the initial liquid phase of the waste is added to the liquid extract, and these are analyzed together. If incompatible, the liquids are analyzed separately and the results are mathematically combined to yield a volume-weighted average concentration.

3.0 INTERFERENCES

3.1 Potential interferences that may be encountered during analysis are discussed in the individual analytical methods.

4.0 APPARATUS AND MATERIALS

4.1 Agitation apparatus: The agitation apparatus must be capable of rotating the extraction vessel in an end-over-end fashion (see Figure 1) at 30 ± 2 rpm. Suitable devices known to EPA are identified in Table 2.

4.2 Extraction Vessels

4.2.1 Zero-Headspace Extraction Vessel (ZHE). This device is for use only when the waste is being tested for the mobility of volatile analytes (i.e., those listed in Table 1). The ZHE (depicted in Figure 2) allows for liquid/solid separation within the device, and effectively precludes headspace. This type of vessel allows for initial liquid/solid separation, extraction, and final extract filtration without opening the vessel (see Section 4.3.1). The vessels shall have an internal volume of 500-600 mL, and be equipped to accommodate a 90-110 mm filter. The devices contain VITON®[1] O-rings which should be replaced frequently. Suitable ZHE devices known to EPA are identified in Table 3.

For the ZHE to be acceptable for use, the piston within the ZHE should be able to be moved with approximately 15 psi or less. If it takes more pressure to move the piston, the O-rings in the device should be replaced. If this does not solve the problem, the ZHE is unacceptable for TCLP analyses and the manufacturer should be contacted.

The ZHE should be checked for leaks after every extraction. If the device contains a built-in pressure gauge, pressurize the device to 50 psi, allow it to stand unattended for 1 hour, and recheck the pressure. If the device does not have a built-in pressure gauge, pressurize the device to 50 psi, submerge it in water, and check for the presence of air bubbles escaping from any of the fittings. If pressure is lost, check all fittings and inspect and replace O-rings, if necessary. Retest the device. If leakage problems cannot be solved, the manufacturer should be contacted.

Some ZHEs use gas pressure to actuate the ZHE piston, while others use mechanical pressure (see Table 3). Whereas the volatiles procedure (see Section 7.3) refers to pounds per square inch (psi), for the mechanically actuated piston, the pressure applied is measured in torque-inch-pounds. Refer to the manufacturer's instructions as to the proper conversion.

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[1] VITON® is a trademark of Du Pont.

4.2.2 Bottle Extraction Vessel. When the waste is being evaluated using the nonvolatile extraction, a jar with sufficient capacity to hold the sample and the extraction fluid is needed. Headspace is allowed in this vessel.

The extraction bottles may be constructed from various materials, depending on the analytes to be analyzed and the nature of the waste (see Section 4.3.3). It is recommended that borosilicate glass bottles be used instead of other types of glass, especially when inorganics are of concern. Plastic bottles, other than polytetrafluoroethylene, shall not be used if organics are to be investigated. Bottles are available from a number of laboratory suppliers. When this type of extraction vessel is used, the filtration device discussed in Section 4.3.2 is used for initial liquid/solid separation and final extract filtration.

4.3 Filtration Devices: It is recommended that all filtrations be performed in a hood.

4.3.1 Zero-Headspace Extractor Vessel (ZHE): When the waste is evaluated for volatiles, the zero-headspace extraction vessel described in Section 4.2.1 is used for filtration. The device shall be capable of supporting and keeping in place the glass fiber filter and be able to withstand the pressure needed to accomplish separation (50 psi).

NOTE: When it is suspected that the glass fiber filter has been ruptured, an in-line glass fiber filter may be used to filter the material within the ZHE.

4.3.2 Filter Holder: When the waste is evaluated for other than volatile analytes, any filter holder capable of supporting a glass fiber filter and able to withstand the pressure needed to accomplish separation may be used. Suitable filter holders range from simple vacuum units to relatively complex systems capable of exerting pressures of up to 50 psi or more. The type of filter holder used depends on the properties of the material to be filtered (see Section 4.3.3). These devices shall have a minimum internal volume of 300 mL and be equipped to accommodate a minimum filter size of 47 mm (filter holders having an internal capacity of 1.5 L or greater, and equipped to accommodate a 142 mm diameter filter, are recommended). Vacuum filtration can only be used for wastes with low solids content (<10%) and for highly granular, liquid-containing wastes. All other types of wastes should be filtered using positive pressure filtration. Suitable filter holders known to EPA are shown in Table 4.

4.3.3 Materials of Construction: Extraction vessels and filtration devices shall be made of inert materials which will not leach or absorb waste components. Glass, polytetrafluoroethylene (PTFE), or type 316 stainless steel equipment may be used when evaluating the mobility of both organic and inorganic components. Devices made of high density polyethylene (HDPE), polypropylene (PP), or polyvinyl chloride (PVC) may be used only when evaluating the mobility of metals. Borosilicate glass bottles are recommended for use over other types of glass bottles, especially when inorganics are analytes of concern.

4.4 Filters: Filters shall be made of borosilicate glass fiber, shall contain no binder materials, and shall have an effective pore size of 0.6 to 0.8 µm, or equivalent. Filters known to EPA which meet these specifications are identified in Table 5. Pre-filters must not be used. When evaluating the mobility of metals, filters shall be acid-washed prior to use by rinsing with 1N nitric acid followed by three consecutive rinses with deionized distilled water (a minimum of 1 L per rinse is recommended). Glass fiber filters are fragile and should be handled with care.

4.5 pH Meters: The meter should be accurate to ± 0.05 units at 25 °C.

4.6 ZHE Extract Collection Devices: TEDLAR®[2] bags or glass, stainless steel or PTFE gas-tight syringes are used to collect the initial liquid phase and the final extract of the waste when using the ZHE device. The devices listed are recommended for use under the following conditions:

4.6.1 If a waste contains an aqueous liquid phase or if a waste does not contain a significant amount of nonaqueous liquid (i.e., <1% of total waste), the TEDLAR® bag or a 600 mL syringe should be used to collect and combine the initial liquid and solid extract.

4.6.2 If a waste contains a significant amount of nonaqueous liquid in the initial liquid phase (i.e., >1% of total waste), the syringe or the TEDLAR® bag may be used for both the initial solid/liquid separation and the final extract filtration. However, analysts should use one or the other, not both.

4.6.3 If the waste contains no initial liquid phase (is 100% solid) or has no significant solid phase (is 100% liquid), either the TEDLAR® bag or the syringe may be used. If the syringe is used, discard the first 5 mL of liquid expressed from the device. The remaining aliquots are used for analysis.

4.7 ZHE Extraction Fluid Transfer Devices: Any device capable of transferring the extraction fluid into the ZHE without changing the nature of the extraction fluid is acceptable (e.g., a positive displacement or peristaltic pump, a gas tight syringe, pressure filtration unit (see Section 4.3.2), or other ZHE device).

4.8 Laboratory Balance: Any laboratory balance accurate to within ± 0.01 grams may be used (all weight measurements are to be within ± 0.1 grams).

4.9 Beaker or Erlenmeyer flask, glass, 500 mL.

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[2] TEDLAR® is a registered trademark of Du Pont.

4.10 Watchglass, appropriate diameter to cover beaker or Erlenmeyer flask.

4.11 Magnetic stirrer.

5.0 REAGENTS

5.1 Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Reagent Water. Reagent water is defined as water in which an interferant is not observed at or above the method's detection limit of the analyte (s) of interest. For nonvolatile extractions, ASTM Type II water or equivalent meets the definition of reagent water. For volatile extractions, it is recommended that reagent water be generated by any of the following methods. Reagent water should be monitored periodically for impurities.

5.2.1 Reagent water for volatile extractions may be generated by passing tap water through a carbon filter bed containing about 500 grams of activated carbon (Calgon Corp., Filtrasorb-300 or equivalent).

5.2.2 A water purification system (Millipore Super-Q or equivalent) may also be used to generate reagent water for volatile extractions.

5.2.3 Reagent water for volatile extractions may also be prepared by boiling water for 15 minutes. Subsequently, while maintaining the water temperature at 90 ± 5 degrees C, bubble a contaminant-free inert gas (e.g. nitrogen) through the water for 1 hour. While still hot, transfer the water to a narrow mouth screw-cap bottle under zero-headspace and seal with a Teflon-lined septum and cap.

5.3 Hydrochloric acid (1N), HCl, made from ACS reagent grade.

5.4 Nitric acid (1N), HNO_3, made from ACS reagent grade.

5.5 Sodium hydroxide (1N), NaOH, made from ACS reagent grade.

5.6 Glacial acetic acid, CH_3CH_2OOH, ACS reagent grade.

5.7 Extraction fluid.

5.7.1 Extraction fluid # 1: Add 5.7 mL glacial CH_3CH_2OOH to 500 mL of reagent water (See Section 5.2), add 64.3 mL of 1N NaOH, and dilute to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 4.93 ± 0.05.

5.7.2 Extraction fluid # 2: Dilute 5.7 mL glacial CH_3CH_2OOH with reagent water (See Section 5.2) to a volume of 1 liter. When correctly prepared, the pH of this fluid will be 2.88 ± 0.05.

NOTE: These extraction fluids should be monitored frequently for impurities. The pH should be checked prior to use to ensure that these fluids are made up accurately. If impurities are found or the pH is not within the above specifications, the fluid shall be discarded and fresh extraction fluid prepared.

5.8 Analytical standards shall be prepared according to the appropriate analytical method.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 All samples shall be collected using an appropriate sampling plan.

6.2 The TCLP may place requirements on the minimal size of the field sample, depending upon the physical state or states of the waste and the analytes of concern. An aliquot is needed for preliminary evaluation of which extraction fluid is to be used for the nonvolatile analyte extraction procedure. Another aliquot may be needed to actually conduct the nonvolatile extraction (see Section 1.4 concerning the use of this extract for volatile organics). If volatile organics are of concern, another aliquot may be needed. Quality control measures may require additional aliquots. Further, it is always wise to collect more sample just in case something goes wrong with the initial attempt to conduct the test.

6.3 Preservatives shall not be added to samples before extraction.

6.4 Samples may be refrigerated unless refrigeration results in irreversible physical change to the waste. If precipitation occurs, the entire sample (including precipitate) should be extracted.

6.5 When the waste is to be evaluated for volatile analytes, care shall be taken to minimize the loss of volatiles. Samples shall be collected and stored in a manner intended to prevent the loss of volatile analytes (e.g., samples should be collected in Teflon-lined septum capped vials and stored at 4 °C. Samples should be opened only immediately prior to extraction).

6.6 TCLP extracts should be prepared for analysis and analyzed as soon as possible following extraction. Extracts or portions of extracts for metallic analyte determinations must be acidified with nitric acid to a pH < 2, unless precipitation occurs (see Section 7.2.14 if precipitation occurs). Extracts should be preserved for other analytes according to the guidance given in the individual analysis methods. Extracts or portions of extracts for organic analyte determinations shall not be allowed to come into contact with the atmosphere (i.e., no headspace) to prevent losses. See Section 8.0 (QA requirements) for acceptable sample and extract holding times.

7.0 PROCEDURE

7.1 Preliminary Evaluations

Perform preliminary TCLP evaluations on a minimum 100 gram aliquot of waste. This aliquot may not actually undergo TCLP extraction. These preliminary evaluations include: (1) determination of the percent solids (Section 7.1.1); (2) determination of whether the waste contains insignificant solids and is, therefore, its own extract after filtration (Section 7.1.2); (3) determination of whether the solid portion of the waste requires particle size reduction (Section 7.1.3); and (4) determination of which of the two extraction fluids are to be used for the nonvolatile TCLP extraction of the waste (Section 7.1.4).

7.1.1 Preliminary determination of percent solids: Percent solids is defined as that fraction of a waste sample (as a percentage of the total sample) from which no liquid may be forced out by an applied pressure, as described below.

7.1.1.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solids) proceed to Section 7.1.3.

7.1.1.2 If the sample is liquid or multiphasic, liquid/solid separation to make a preliminary determination of percent solids is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.1.1.3 through 7.1.1.9.

7.1.1.3 Pre-weigh the filter and the container that will receive the filtrate.

7.1.1.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure.

7.1.1.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight.

7.1.1.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Centrifugation is to be used only as an aid to filtration. If used, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.1.1.7 Quantitatively transfer the waste sample to the filter holder (liquid and solid phases). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.1.1.5 to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within any 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.1.1.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.1.1.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.1.1.9 Determine the weight of the liquid phase by subtracting the weight of the filtrate container (see Section 7.1.1.3) from the total weight of the filtrate-filled container. Determine the weight of the solid phase of the waste sample by subtracting the weight of the liquid phase from the weight of the total waste sample, as determined in Section 7.1.1.5 or 7.1.1.7.

Record the weight of the liquid and solid phases. Calculate the percent solids as follows:

$$\text{Percent solids} = \frac{\text{Weight of solid (Section 7.1.1.9)}}{\text{Total weight of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2 If the percent solids determined in Section 7.1.1.9 is equal to or greater than 0.5%, then proceed either to Section 7.1.3 to determine whether the solid material requires particle size reduction or to Section 7.1.2.1 if it is noticed that a small amount of the filtrate is entrained in wetting of the filter. If the percent solids determined in Section 7.1.1.9 is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed and to Section 7.3 with a fresh portion of the waste if the volatile TCLP is to be performed.

7.1.2.1 Remove the solid phase and filter from the filtration apparatus.

7.1.2.2 Dry the filter and solid phase at 100 ± 20 °C until two successive weighing yield the same value within ± 1%. Record the final weight.

NOTE: Caution should be taken to ensure that the subject solid will not flash upon heating. It is recommended that the drying oven be vented to a hood or other appropriate device.

7.1.2.3 Calculate the percent dry solids as follows:

$$\text{Percent dry solids} = \frac{\text{(Wt. of dry waste + filter) - tared wt. of filter}}{\text{Initial wt. of waste (Section 7.1.1.5 or 7.1.1.7)}} \times 100$$

7.1.2.4 If the percent dry solids is less than 0.5%, then proceed to Section 7.2.9 if the nonvolatile TCLP is to be performed, and to Section 7.3 if the volatile TCLP is to be performed. If the percent dry solids is greater than or equal to 0.5%, and if the nonvolatile TCLP is to be performed, return to the beginning of this Section (7.1) and, with a fresh portion of waste, determine whether particle size reduction is necessary (Section 7.1.3) and determine the appropriate extraction fluid (Section 7.1.4). If only the volatile TCLP is to be performed, see the note in Section 7.1.4.

7.1.3 Determination of whether the waste requires particle size reduction (particle size is reduced during this step): Using the solid portion of the waste, evaluate the solid for particle size. Particle size reduction is required, unless the solid has a surface area per gram of material equal to or greater than 3.1 cm^2, or is smaller than 1 cm in its narrowest dimension (i.e., is capable of passing through a 9.5 mm (0.375 inch) standard sieve). If the surface area is smaller or the particle size larger than described above, prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described above. If the solids are prepared for organic volatiles extraction, special precautions must be taken (see Section 7.3.6).

NOTE: Surface area criteria are meant for filamentous (e.g., paper, cloth, and similar) waste materials. Actual measurement of surface area is not required, nor is it recommended. For materials that do not obviously meet the criteria, sample specific methods would need to be developed and employed to measure the surface area. Such methodology is currently not available.

7.1.4 Determination of appropriate extraction fluid: If the solid content of the waste is greater than or equal to 0.5% and if the sample will be extracted for nonvolatile constituents (Section 7.2), determine the appropriate fluid (Section 5.7) for the nonvolatiles extraction as follows:

NOTE: TCLP extraction for volatile constituents uses only extraction fluid #1 (Section 5.7.1). Therefore, if TCLP extraction for nonvolatiles is not required, proceed to Section 7.3.

7.1.4.1 Weigh out a small subsample of the solid phase of the waste, reduce the solid (if necessary) to a particle size of approximately 1 mm in diameter or less, and transfer 5.0 grams of the solid phase of the waste to a 500 mL beaker or Erlenmeyer flask.

7.1.4.2 Add 96.5 mL of reagent water to the beaker, cover with a watchglass, and stir vigorously for 5 minutes using a magnetic stirrer. Measure and record the pH. If the pH is <5.0, use extraction fluid #1. Proceed to Section 7.2.

7.1.4.3 If the pH from Section 7.1.4.2 is >5.0, add 3.5 mL 1N HCl, slurry briefly, cover with a watchglass, heat to 50 °C, and hold at 50 °C for 10 minutes.

7.1.4.4 Let the solution cool to room temperature and record the pH. If the pH is <5.0, use extraction fluid #1. If the pH is >5.0, use extraction fluid #2. Proceed to Section 7.2.

7.1.5 If the aliquot of the waste used for the preliminary evaluation (Sections 7.1.1 - 7.1.4) was determined to be 100% solid at Section 7.1.1.1, then it can be used for the Section 7.2 extraction (assuming at least 100 grams remain), and the Section 7.3 extraction (assuming at least 25 grams remain). If the aliquot was subjected to the procedure in Section 7.1.1.7, then another aliquot shall be used for the volatile extraction procedure in Section 7.3. The aliquot of the waste subjected to the procedure in Section 7.1.1.7 might be appropriate for use for the Section 7.2 extraction if an adequate amount of solid (as determined by Section 7.1.1.9) was obtained. The amount of solid necessary is dependent upon whether a sufficient amount of extract will be produced to support the analyses. If an adequate amount of solid remains, proceed to Section 7.2.10 of the nonvolatile TCLP extraction.

7.2 Procedure When Volatiles are not Involved

A minimum sample size of 100 grams (solid and liquid phases) is recommended. In some cases, a larger sample size may be appropriate, depending on the solids content of the waste sample (percent solids, See Section 7.1.1), whether the initial liquid phase of the waste will be miscible with the aqueous extract of the solid, and whether inorganics, semivolatile organics, pesticides, and herbicides are all analytes of concern. Enough solids should be generated for extraction such that the volume of TCLP extract will be sufficient to support all of the analyses required. If the amount of extract generated by a single TCLP extraction will not be sufficient to perform all of the analyses, more than one extraction may be performed and the extracts from each combined and aliquoted for analysis.

7.2.1 If the waste will obviously yield no liquid when subjected to pressure filtration (i.e., is 100% solid, see Section 7.1.1), weigh out a subsample of the waste (100 gram minimum) and proceed to Section 7.2.9.

7.2.2 If the sample is liquid or multiphasic, liquid/solid separation is required. This involves the filtration device described in Section 4.3.2 and is outlined in Sections 7.2.3 to 7.2.8.

7.2.3 Pre-weigh the container that will receive the filtrate.

7.2.4 Assemble the filter holder and filter following the manufacturer's instructions. Place the filter on the support screen and secure. Acid wash the filter if evaluating the mobility of metals (see Section 4.4).

NOTE: Acid washed filters may be used for all nonvolatile extractions even when metals are not of concern.

7.2.5 Weigh out a subsample of the waste (100 gram minimum) and record the weight. If the waste contains <0.5% dry solids (Section 7.1.2), the liquid portion of the waste, after filtration, is defined as the TCLP extract. Therefore, enough of the sample should be filtered so that the amount of filtered liquid will support all of the analyses required of the TCLP extract. For wastes containing >0.5% dry solids (Sections 7.1.1 or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size (100 gram minimum) for filtration. Enough solids should be generated by filtration to support the analyses to be performed on the TCLP extract.

7.2.6 Allow slurries to stand to permit the solid phase to settle. Wastes that settle slowly may be centrifuged prior to filtration. Use centrifugation only as an aid to filtration. If the waste is centrifuged, the liquid should be decanted and filtered followed by filtration of the solid portion of the waste through the same filtration system.

7.2.7 Quantitatively transfer the waste sample (liquid and solid phases) to the filter holder (see Section 4.3.2). Spread the waste sample evenly over the surface of the filter. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering.

NOTE: If waste material (>1% of the original sample weight) has obviously adhered to the container used to transfer the sample to the filtration apparatus, determine the weight of this residue and subtract it from the sample weight determined in Section 7.2.5, to determine the weight of the waste sample that will be filtered.

Gradually apply vacuum or gentle pressure of 1-10 psi, until air or pressurizing gas moves through the filter. If this point is not reached under 10 psi, and if no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if the pressurizing gas has not moved through the filter, and if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When the pressurizing gas begins to move through the filter, or when the liquid flow has ceased at 50 psi (i.e., filtration does not result in any additional filtrate within a 2 minute period), stop the filtration.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.2.8 The material in the filter holder is defined as the solid phase of the waste, and the filtrate is defined as the liquid phase. Weigh the filtrate. The liquid phase may now be either analyzed (See Section 7.2.12) or stored at 4 °C until time of analysis.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying vacuum or pressure filtration, as outlined in Section 7.2.7, this material may not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the extraction as a solid. Do not replace the original filter with a fresh filter under any circumstances. Use only one filter.

7.2.9 If the waste contains <0.5% dry solids (see Section 7.1.2), proceed to Section 7.2.13. If the waste contains >0.5% dry solids (see Section 7.1.1 or 7.1.2), and if particle size reduction of the solid was needed in Section 7.1.3, proceed to Section 7.2.10. If the waste as received passes a 9.5 mm sieve, quantitatively transfer the solid material into the extractor bottle along with the filter used to separate the initial liquid from the solid phase, and proceed to Section 7.2.11.

7.2.10 Prepare the solid portion of the waste for extraction by crushing, cutting, or grinding the waste to a surface area or particle size as described in Section 7.1.3. When the surface area or particle size has been appropriately altered, quantitatively transfer the solid material into an extractor bottle. Include the filter used to separate the initial liquid from the solid phase.

NOTE: Sieving of the waste is not normally required. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended. If sieving is necessary, a Teflon coated sieve should be used to avoid contamination of the sample.

7.2.11 Determine the amount of extraction fluid to add to the extractor vessel as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.2.5 or 7.2.7)}}{100}$$

Slowly add this amount of appropriate extraction fluid (see Section 7.1.4) to the extractor vessel. Close the extractor bottle tightly (it is recommended that Teflon tape be used to ensure a tight seal), secure in rotary agitation device, and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (i.e., temperature of room in which extraction takes place) shall be maintained at 23 ± 2 °C during the extraction period.

NOTE: As agitation continues, pressure may build up within the extractor bottle for some types of wastes (e.g., limed or calcium carbonate containing waste may evolve gases such as carbon dioxide). To relieve excess pressure, the extractor bottle may be periodically opened (e.g., after 15 minutes, 30 minutes, and 1 hour) and vented into a hood.

7.2.12 Following the 18 ± 2 hour extraction, separate the material in the extractor vessel into its component liquid and solid phases by filtering through a new glass fiber filter, as outlined in Section 7.2.7. For final filtration of the TCLP extract, the glass fiber filter may be changed, if necessary, to facilitate filtration. Filter(s) shall be acid-washed (see Section 4.4) if evaluating the mobility of metals.

7.2.13 Prepare the TCLP extract as follows:

7.2.13.1 If the waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.2.12 is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.2 If compatible (e.g., multiple phases will not result on combination), combine the filtered liquid resulting from Section 7.2.12 with the initial liquid phase of the waste obtained in Section 7.2.7. This combined liquid is defined as the TCLP extract. Proceed to Section 7.2.14.

7.2.13.3 If the initial liquid phase of the waste, as obtained from Section 7.2.7, is not or may not be compatible with the filtered liquid resulting from Section 7.2.12, do not combine these liquids. Analyze these liquids, collectively defined as the TCLP extract, and combine the results mathematically, as described in Section 7.2.14.

7.2.14 Following collection of the TCLP extract, the pH of the extract should be recorded. Immediately aliquot and preserve the extract for analysis. Metals aliquots must be acidified with nitric acid to pH <2. If precipitation is observed upon addition of nitric acid to a small aliquot of the extract, then the remaining portion of the extract for metals analyses shall not be acidified and the extract shall be analyzed as soon as possible. All other aliquots must be stored under refrigeration (4 °C) until analyzed. The TCLP extract shall be prepared and analyzed according to appropriate analytical methods. TCLP extracts to be analyzed for metals shall be acid digested except in those instances where digestion causes loss of metallic analytes. If an analysis of the undigested extract shows that the concentration of any regulated metallic analyte exceeds the regulatory level, then the waste is hazardous and digestion of the extract is not necessary. However, data on undigested extracts alone cannot be used to demonstrate that the waste is not hazardous. If the individual phases are to be analyzed separately, determine the volume of the individual phases (to ± 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phase (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.2.15 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

7.3 Procedure When Volatiles are Involved

Use the ZHE device to obtain TCLP extract for analysis of volatile compounds only. Extract resulting from the use of the ZHE shall not be used to evaluate the mobility of nonvolatile analytes (e.g., metals, pesticides, etc.).

The ZHE device has approximately a 500 mL internal capacity. The ZHE can thus accommodate a maximum of 25 grams of solid (defined as that fraction of a sample from which no additional liquid may be forced out by an applied pressure of 50 psi), due to the need to add an amount of extraction fluid equal to 20 times the weight of the solid phase.

Charge the ZHE with sample only once and do not open the device until the final extract (of the solid) has been collected. Repeated filling of the ZHE to obtain 25 grams of solid is not permitted.

Do not allow the waste, the initial liquid phase, or the extract to be exposed to the atmosphere for any more time than is absolutely necessary. Any manipulation of these materials should be done when cold (4 °C) to minimize loss of volatiles.

7.3.1 Pre-weigh the (evacuated) filtrate collection container (See Section 4.6) and set aside. If using a TEDLAR® bag, express all liquid from the ZHE device into the bag, whether for the initial or final liquid/solid separation, and take an aliquot from the liquid in the bag for analysis. The containers listed in Section 4.6 are recommended for use under the conditions stated in Sections 4.6.1 - 4.6.3.

7.3.2 Place the ZHE piston within the body of the ZHE (it may be helpful first to moisten the piston O-rings slightly with extraction fluid). Adjust the piston within the ZHE body to a height that will minimize the distance the piston will have to move once the ZHE is charged with sample (based upon sample size requirements determined from Section 7.3, Section 7.1.1 and/or 7.1.2). Secure the gas inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange (top flange) aside.

7.3.3 If the waste is 100% solid (see Section 7.1.1), weigh out a subsample (25 gram maximum) of the waste, record weight, and proceed to Section 7.3.5.

7.3.4 If the waste contains < 0.5% dry solids (Section 7.1.2), the liquid portion of waste, after filtration, is defined as the TCLP extract. Filter enough of the sample so that the amount of filtered liquid will support all of the volatile analyses required. For wastes containing $\geq$ 0.5% dry solids (Sections 7.1.1 and/or 7.1.2), use the percent solids information obtained in Section 7.1.1 to determine the optimum sample size to charge into the ZHE. The recommended sample size is as follows:

7.3.4.1 For wastes containing < 5% solids (see Section 7.1.1), weigh out a 500 gram subsample of waste and record the weight.

7.3.4.2 For wastes containing $\geq$ 5% solids (see Section 7.1.1), determine the amount of waste to charge into the ZHE as follows:

$$\text{Weight of waste to charge ZHE} = \frac{25}{\text{percent solids (Section 7.1.1)}} \times 100$$

Weigh out a subsample of the waste of the appropriate size and record the weight.

7.3.5 If particle size reduction of the solid portion of the waste was required in Section 7.1.3, proceed to Section 7.3.6. If particle size reduction was not required in Section 7.1.3, proceed to Section 7.3.7.

7.3.6 Prepare the waste for extraction by crushing, cutting, or grinding the solid portion of the waste to a surface area or particle size as described in Section 7.1.3. Wastes and appropriate reduction equipment should be refrigerated, if possible, to 4 °C prior to particle size reduction. The means used to effect particle size reduction must not generate heat in and of itself. If reduction of the solid phase of the waste is necessary, exposure of the waste to the atmosphere should be avoided to the extent possible.

NOTE: Sieving of the waste is not recommended due to the possibility that volatiles may be lost. The use of an appropriately graduated ruler is recommended as an acceptable alternative. Surface area requirements are meant for filamentous (e.g., paper, cloth) and similar waste materials. Actual measurement of surface area is not recommended.

When the surface area or particle size has been appropriately altered, proceed to Section 7.3.7.

7.3.7 Waste slurries need not be allowed to stand to permit the solid phase to settle. Do not centrifuge wastes prior to filtration.

7.3.8 Quantitatively transfer the entire sample (liquid and solid phases) quickly to the ZHE. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with the manufacturer's instructions. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Do not attach the extract collection device to the top plate.

NOTE: If waste material (>1% of original sample weight) has obviously adhered to the container used to transfer the sample to the ZHE, determine the weight of this residue and subtract it from the sample weight determined in Section 7.3.4 to determine the weight of the waste sample that will be filtered.

Attach a gas line to the gas inlet/outlet valve (bottom flange) and, with the liquid inlet/outlet valve (top flange) open, begin applying gentle pressure of 1-10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue pressure. If filtration of the waste at 4 °C reduces the amount of expressed liquid over what would be expressed at room temperature, then allow the sample to warm up to room temperature in the device before filtering. If the waste is 100% solid (see Section 7.1.1), slowly increase the pressure to a maximum of 50 psi to force most of the headspace out of the device and proceed to Section 7.3.12.

7.3.9 Attach the evacuated pre-weighed filtrate collection container to the liquid inlet/outlet valve and open the valve. Begin applying gentle pressure of 1-10 psi to force the liquid phase of the sample into the filtrate collection container. If no additional liquid has passed through the filter in any 2 minute interval, slowly increase the pressure in 10 psi increments to a maximum of 50 psi. After each incremental increase of 10 psi, if no additional liquid has passed through the filter in any 2 minute interval, proceed to the next 10 psi increment. When liquid flow has ceased such that continued pressure filtration at 50 psi does not result in any additional filtrate within a 2 minute period, stop the filtration. Close the liquid inlet/outlet valve, discontinue pressure to the piston, and disconnect and weigh the filtrate collection container.

NOTE: Instantaneous application of high pressure can degrade the glass fiber filter and may cause premature plugging.

7.3.10 The material in the ZHE is defined as the solid phase of the waste and the filtrate is defined as the liquid phase.

NOTE: Some wastes, such as oily wastes and some paint wastes, will obviously contain some material that appears to be a liquid. Even after applying pressure filtration, this material will not filter. If this is the case, the material within the filtration device is defined as a solid and is carried through the TCLP extraction as a solid.

If the original waste contained <0.5% dry solids (see Section 7.1.2), this filtrate is defined as the TCLP extract and is analyzed directly. Proceed to Section 7.3.15.

7.3.11 The liquid phase may now be either analyzed immediately (See Sections 7.3.13 through 7.3.15) or stored at 4 °C under minimal headspace conditions until time of analysis. Determine the weight of extraction fluid #1 to add to the ZHE as follows:

$$\text{Weight of extraction fluid} = \frac{20 \times \text{percent solids (Section 7.1.1)} \times \text{weight of waste filtered (Section 7.3.4 or 7.3.8)}}{100}$$

7.3.12 The following Sections detail how to add the appropriate amount of extraction fluid to the solid material within the ZHE and agitation of the ZHE vessel. Extraction fluid #1 is used in all cases (See Section 5.7).

7.3.12.1 With the ZHE in the vertical position, attach a line from the extraction fluid reservoir to the liquid inlet/outlet valve. The line used shall contain fresh extraction fluid and should be preflushed with fluid to eliminate any air pockets in the line. Release gas pressure on the ZHE piston (from the gas inlet/outlet valve), open the liquid inlet/outlet valve, and begin transferring extraction fluid (by pumping or similar means) into the ZHE. Continue pumping extraction fluid into the ZHE until the appropriate amount of fluid has been introduced into the device.

7.3.12.2 After the extraction fluid has been added, immediately close the liquid inlet/outlet valve and disconnect the extraction fluid line. Check the ZHE to ensure that all valves are in their closed positions. Manually rotate the device in an end-over-end fashion 2 or 3 times. Reposition the ZHE in the vertical position with the liquid inlet/outlet valve on top. Pressurize the ZHE to 5-10 psi (if necessary) and slowly open the liquid inlet/outlet valve to bleed out any headspace (into a hood) that may have been introduced due to the addition of extraction fluid. This bleeding shall be done quickly and shall be stopped at the first appearance of liquid from the valve. Re-pressurize the ZHE with 5-10 psi and check all ZHE fittings to ensure that they are closed.

7.3.12.3 Place the ZHE in the rotary agitation apparatus (if it is not already there) and rotate at 30 ± 2 rpm for 18 ± 2 hours. Ambient temperature (_i.e._, temperature of room in which extraction occurs) shall be maintained at 23 ± 2 °C during agitation.

7.3.13 Following the 18 ± 2 hour agitation period, check the pressure behind the ZHE piston by quickly opening and closing the gas inlet/outlet valve and noting the escape of gas. If the pressure has not been maintained (_i.e._, no gas release observed), the device is leaking. Check the ZHE for leaking as specified in Section 4.2.1, and perform the extraction again with a new sample of waste. If the pressure within the device has been maintained, the material in the extractor vessel is once again separated into its component liquid and solid phases. If the waste contained an initial liquid phase, the liquid may be filtered directly into the same filtrate collection container (_i.e._, TEDLAR® bag) holding the initial liquid phase of the waste. A separate filtrate collection container must be used if combining would create multiple phases, or there is not enough volume left within the filtrate collection container. Filter through the glass fiber filter, using the ZHE device as discussed in Section 7.3.9. All extract shall be filtered and collected if the

TEDLAR® bag is used, if the extract is multiphasic, or if the waste contained an initial liquid phase (see Sections 4.6 and 7.3.1).

NOTE: An in-line glass fiber filter may be used to filter the material within the ZHE if it is suspected that the glass fiber filter has been ruptured.

7.3.14 If the original waste contained no initial liquid phase, the filtered liquid material obtained from Section 7.3.13 is defined as the TCLP extract. If the waste contained an initial liquid phase, the filtered liquid material obtained from Section 7.3.13 and the initial liquid phase (Section 7.3.9) are collectively defined as the TCLP extract.

7.3.15 Following collection of the TCLP extract, immediately prepare the extract for analysis and store with minimal headspace at 4 °C until analyzed. Analyze the TCLP extract according to the appropriate analytical methods. If the individual phases are to be analyzed separately (i.e., are not miscible), determine the volume of the individual phases (to 0.5%), conduct the appropriate analyses, and combine the results mathematically by using a simple volume-weighted average:

$$\text{Final Analyte Concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

where:

V_1 = The volume of the first phases (L).
C_1 = The concentration of the analyte of concern in the first phase (mg/L).
V_2 = The volume of the second phase (L).
C_2 = The concentration of the analyte of concern in the second phase (mg/L).

7.3.16 Compare the analyte concentrations in the TCLP extract with the levels identified in the appropriate regulations. Refer to Section 8.0 for quality assurance requirements.

8.0 QUALITY ASSURANCE

8.1 A minimum of one blank (using the same extraction fluid as used for the samples) must be analyzed for every 20 extractions that have been conducted in an extraction vessel.

8.2 A matrix spike shall be performed for each waste type (e.g., wastewater treatment sludge, contaminated soil, etc.) unless the result exceeds the regulatory level and the data are being used solely to demonstrate that the waste property exceeds the regulatory level. A minimum of one matrix spike must be analyzed for each analytical batch. As a minimum, follow the matrix spike addition guidance provided in each analytical method.

8.2.1 Matrix spikes are to be added after filtration of the TCLP extract and before preservation. Matrix spikes should not be added prior to TCLP extraction of the sample.

8.2.2 In most cases, matrix spikes should be added at a concentration equivalent to the corresponding regulatory level. If the analyte concentration is less than one half the regulatory level, the spike concentration may be as low as one half of the analyte concentration, but may not be not less than five times the method detection limit. In order to avoid differences in matrix effects, the matrix spikes must be added to the same nominal volume of TCLP extract as that which was analyzed for the unspiked sample.

8.2.3 The purpose of the matrix spike is to monitor the performance of the analytical methods used, and to determine whether matrix interferences exist. Use of other internal calibration methods, modification of the analytical methods, or use of alternate analytical methods may be needed to accurately measure the analyte concentration in the TCLP extract when the recovery of the matrix spike is below the expected analytical method performance.

8.2.4 Matrix spike recoveries are calculated by the following formula:

$$\%R\ (\%\text{Recovery}) = 100\ (X_s - X_u)/K$$

where:
X_s = measured value for the spiked sample,
X_u = measured value for the unspiked sample, and
K = known value of the spike in the sample.

8.3 All quality control measures described in the appropriate analytical methods shall be followed.

8.4 The use of internal calibration quantitation methods shall be employed for a metallic contaminant if: (1) Recovery of the contaminant from the TCLP extract is not at least 50% and the concentration does not exceed the regulatory level, and (2) The concentration of the contaminant measured in the extract is within 20% of the appropriate regulatory level.

8.4.1. The method of standard additions shall be employed as the internal calibration quantitation method for each metallic contaminant.

8.4.2 The method of standard additions requires preparing calibration standards in the sample matrix rather than reagent water or blank solution. It requires taking four identical aliquots of the solution and adding known amounts of standard to three of these aliquots. The forth aliquot is the unknown. Preferably, the first addition should be prepared so that the resulting concentration is approximately 50% of the expected concentration of the sample. The second and third additions should be prepared so that the concentrations are approximately 100% and

150% of the expected concentration of the sample. All four aliquots are maintained at the same final volume by adding reagent water or a blank solution, and may need dilution adjustment to maintain the signals in the linear range of the instrument technique. All four aliquots are analyzed.

8.4.3 Prepare a plot, or subject data to linear regression, of instrument signals or external-calibration-derived concentrations as the dependant variable (y-axis) versus concentrations of the additions of standard as the independent variable (x-axis). Solve for the intercept of the abscissa (the independent variable, x-axis) which is the concentration in the unknown.

8.4.4 Alternately, subtract the instrumental signal or external-calibration-derived concentration of the unknown (unspiked) sample from the instrumental signals or external-calibration-derived concentrations of the standard additions. Plot or subject to linear regression of the corrected instrument signals or external-calibration-derived concentrations as the dependant variable versus the independent variable. Derive concentrations for unknowns using the internal calibration curve as if it were an external calibration curve.

8.5 Samples must undergo TCLP extraction within the following time periods:

SAMPLE MAXIMUM HOLDING TIMES [DAYS]

	From: Field collection To: TCLP extraction	From: TCLP extraction To: Preparative extraction	From: Preparative extraction To: Determinative analysis	Total elapsed time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except mercury	180	NA	180	360

NA = Not applicable

If sample holding times are exceeded, the values obtained will be considered minimal concentrations. Exceeding the holding time is not acceptable in establishing that a waste does not exceed the regulatory level. Exceeding the holding time will not invalidate characterization if the waste exceeds the regulatory level.

9.0 METHOD PERFORMANCE

9.1 Ruggedness. Two ruggedness studies have been performed to determine the effect of various perturbations on specific elements of the TCLP protocol. Ruggedness testing determines the sensitivity of small procedural variations which might be expected to occur during routine laboratory application.

9.1.1 Metals - The following conditions were used when leaching a waste for metals analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Extraction time	16 hours vs. 18 hours
Headspace	20% vs. 60%
Buffer #2 acidity	190 meq vs. 210 meq
Acid-washed filters	yes vs. no
Filter type	0.7 µm glass fiber vs. 0.45 µm vs. polycarbonate
Bottle type	borosilicate vs. flint glass

Of the seven method variations examined, acidity of the extraction fluid had the greatest impact on the results. Four of 13 metals from an API separator sludge/electroplating waste (API/EW) mixture and two of three metals from an ammonia lime still bottom waste were extracted at higher levels by the more acidic buffer. Because of the sensitivity to pH changes, the method requires that the extraction fluids be prepared so that the final pH is within ± 0.05 units as specified.

9.1.2 Volatile Organic Compounds - The following conditions were used when leaching a waste for VOC analysis:

Varying Conditions	
Liquid/Solid ratio	19:1 vs. 21:1
Headspace	0% vs. 5%
Buffer #1 acidity	60 meq vs. 80 meq
Method of storing extract	Syringe vs. Tedlar ® bag
Aliquotting	yes vs. no
Pressure behind piston	0 psi vs. 20 psi

None of the parameters had a significant effect on the results of the ruggedness test.

9.2 Precision. Many TCLP precision (reproducibility) studies have been performed, and have shown that, in general, the precision of the TCLP is comparable to or exceeds that of the EP toxicity test and that method precision is adequate. One of the more significant contributions to poor precision appears to be related to sample homogeneity and inter-laboratory variation (due to the nature of waste materials).

9.2.1 Metals - The results of a multi-laboratory study are shown in Table 6, and indicate that a single analysis of a waste may not be adequate for waste characterization and identification requirements.

9.2.2 Semi-Volatile Organic Compounds - The results of two studies are shown in Tables 7 and 8. Single laboratory precision was excellent with greater than 90 percent of the results exhibiting an RSD less than 25 percent. Over 85 percent of all individual compounds in the multi-laboratory study fell in the RSD range of 20 - 120 percent. Both studies concluded that the TCLP provides adequate precision. It was also determined that the high acetate content of the extraction fluid did not present problems (i.e., column degradation of the gas chromatograph) for the analytical conditions used.

9.2.3 Volatile Organic Compounds - Eleven laboratories participated in a collaborative study of the use of the ZHE with two waste types which were fortified with a mixture of VOCs. The results of the collaborative study are shown in Table 9. Precision results for VOCs tend to occur over a considerable range. However, the range and mean RSD compared very closely to the same collaborative study metals results in Table 6. Blackburn and Show concluded that at the 95% level of significance: 1) recoveries among laboratories were statistically similar, 2) recoveries did not vary significantly between the two sample types, and 3) each laboratory showed the same pattern of recovery for each of the two samples.

10.0 REFERENCES

1. Blackburn, W.B. and Show, I. "Collaborative Study of the Toxicity Characteristics Leaching Procedure (TCLP)." Draft Final Report, Contract No. 68-03-1958, S-Cubed, November 1986.

2. Newcomer, L.R., Blackburn, W.B., Kimmell, T.A. "Performance of the Toxicity Characteristic Leaching Procedure." Wilson Laboratories, S-Cubed, U.S. EPA, December 1986.

3. Williams, L.R., Francis, C.W.; Maskarinec, M.P., Taylor D.R., and Rothman, N. "Single-Laboratory Evaluation of Mobility Procedure for Solid Waste." EMSL, ORNL, S-Cubed, ENSECO.

Table 1.
Volatile Analytes [1,2]

Compound	CAS No.
Acetone	67-64-1
Benzene	71-43-2
n-Butyl alcohol	71-36-3
Carbon disulfide	75-15-0
Carbon tetrachloride	56-23-5
Chlorobenzene	108-90-7
Chloroform	67-66-3
1,2-Dichloroethane	107-06-2
1,1-Dichloroethylene	75-35-4
Ethyl acetate	141-78-6
Ethyl benzene	100-41-4
Ethyl ether	60-29-7
Isobutanol	78-83-1
Methanol	67-56-1
Methylene chloride	75-09-2
Methyl ethyl ketone	78-93-3
Methyl isobutyl ketone	108-10-1
Tetrachloroethylene	127-18-4
Toluene	108-88-3
1,1,1,-Trichloroethane	71-55-6
Trichloroethylene	79-01-6
Trichlorofluoromethane	75-69-4
1,1,2-Trichloro-1,2,2-trifluoroethane	76-13-1
Vinyl chloride	75-01-4
Xylene	1330-20-7

[1] When testing for any or all of these analytes, the zero-headspace extractor vessel shall be used instead of the bottle extractor.

[2] Benzene, carbon tetrachloride, chlorobenzene, chloroform, 1,2-dichloroethane, 1,1-dichloroethylene, methyl ethyl ketone, tetrachloroethylene, and vinyl chloride are toxicity characteristic constituents.

Table 2.
Suitable Rotary Agitation Apparatus[1]

Company	Location	Model No.
Analytical Testing and Consulting Services, Inc.	Warrington, PA (215) 343-4490	4-vessel extractor (DC20S) 8-vessel extractor (DC20) 12-vessel extractor (DC20B) 24-vessel extractor (DC24C)
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	2-vessel (3740-2-BRE) 4-vessel (3740-4-BRE) 6-vessel (3740-6-BRE) 8-vessel (3740-8-BRE) 12-vessel (3740-12-BRE) 24-vessel (3740-24-BRE)
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	8-vessel (08-00-00) 4-vessel (04-00-00)
IRA Machine Shop and Laboratory	Santurce, PR (809) 752-4004	8-vessel (011001)
Lars Lande Manufacturing	Whitmore Lake, MI (313) 449-4116	10-vessel (10VRE) 5-vessel (5VRE) 6-vessel (6VRE)
Millipore Corp.	Bedford, MA (800) 225-3384	4-ZHE or 4 2-liter bottle extractor (YT310RAHW)

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[1] Any device that rotates the extraction vessel in an end-over-end fashion at 30 ± 2 rpm is acceptable.

Table 3.
Suitable Zero-Headspace Extractor Vessels[1]

Company	Location	Model No.
Analytical Testing & Consulting Services, Inc.	Warrington, PA (215) 343-4490	C102, Mechanical Pressure Device
Associated Design and Manufacturing Company	Alexandria, VA (703) 549-5999	3745-ZHE, Gas Pressure Device
Lars Lande Manufacturing[2]	Whitmore Lake, MI (313) 449-4116	ZHE-11, Gas Pressure Device
Millipore Corporation	Bedford, MA (800) 225-3384	YT30090HW, Gas Pressure Device
Environmental Machine and Design, Inc.	Lynchburg, VA (804) 845-6424	VOLA-TOX1, Gas Pressure Device
Gelman Science	Ann Arbor, MI (800) 521-1520	15400 Gas Pressure Device

[1] Any device that meets the specifications listed in Section 4.2.1 of the method is suitable.

[2] This device uses a 110 mm filter.

Table 4.
Suitable Filter Holders[1]

Company	Location	Model/ Catalogue No.	Size
Nucleopore Corporation	Pleasanton, CA (800) 882-7711	425910 410400	142 mm 47 mm
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	302400 311400	142 mm 47 mm
Millipore Corporation	Bedford, MA (800) 225-3384	YT30142HW XX1004700	142 mm 47 mm

[1] Any device capable of separating the liquid from the solid phase of the waste is suitable, providing that it is chemically compatible with the waste and the constituents to be analyzed. Plastic devices (not listed above) may be used when only inorganic analytes are of concern. The 142 mm size filter holder is recommended.

Table 5.
Suitable Filter Media [1]

Company	Location	Model	Pore Size (μm)
Millipore Corporation	Bedford, MA (800) 225-3384	AP40	0.7
Nucleopore Corporation	Pleasanton, CA (415) 463-2530	211625	0.7
Whatman Laboratory Products, Inc.	Clifton, NJ (201) 773-5800	GFF	0.7
Micro Filtration Systems	Dublin, CA (800) 334-7132 (415) 828-6010	GF75	0.7
Gelman Science	Ann Arbor, MI (800) 521-1520	66256 (90mm) 66257 (142mm)	0.7

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[1] Any filter that meets the specifications in Section 4.4 of the Method is suitable.

Table 6. Multi-Laboratory TCLP Metals, Precision

Waste	Extraction Fluid	Metal	$\bar{X}$	S	%RSD
Ammonia	#1	Cadmium	0.053	0.031	60
Lime Still	#2		0.023	0.017	76
Bottoms	#1	Chromium	0.015	0.0014	93
	#2		0.0032	0.0037	118
	#1	Lead	0.0030	0.0027	90
	#2		0.0032	0.0028	87
API/EW	#1	Cadmium	0.0046	0.0028	61
Mixture	#2		0.0005	0.0004	77
	#1	Chromium	0.0561	0.0227	40
	#2		0.105	0.018	17
	#1	Lead	0.0031	0.0031	100
	#2		0.0124	0.0136	110
Fossil	#1	Cadmium	0.080	0.069	86
Fuel Fly	#2		0.093	0.067	72
Ash	#1	Chromium	0.017	0.014	85
	#2		0.070	0.040	57
	#1	Lead	0.0087	0.0074	85
	#2		0.0457	0.0083	18
				%RSD Range = 17 - 118 Mean %RSD = 74	

NOTE: $\bar{X}$ = Mean results from 6 - 12 different laboratories
Units = mg/L
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 7. Single-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime Still Bottoms	Phenol	#1	19000	2230	11.6
		#2	19400	929	4.8
	2-Methylphenol	#1	2000	297	14.9
		#2	1860	52.9	2.8
	4-Methylphenol	#1	7940	1380	17.4
		#2	7490	200	2.7
	2,4-Dimethylphenol	#1	321	46.8	14.6
		#2	307	45.8	14.9
	Naphthalene	#1	3920	413	10.5
		#2	3827	176	4.6
	2-Methylnaphthalene	#1	290	44.8	15.5
		#2	273	19.3	7.1
	Dibenzofuran	#1	187	22.7	12.1
		#2	187	7.2	3.9
	Acenaphthylene	#1	703	89.2	12.7
		#2	663	20.1	3.0
	Fluorene	#1	151	17.6	11.7
		#2	156	2.1	1.3
	Phenanthrene	#1	241	22.7	9.4
		#2	243	7.9	3.3
	Anthracene	#1	33.2	6.19	18.6
		#2	34.6	1.55	4.5
	Fluoranthrene	#1	25.3	1.8	7.1
		#2	26.0	1.8	7.1
API/EW Mixture	Phenol	#1	40.7	13.5	33.0
		#2	19.0	1.76	9.3
	2,4-Dimethylphenol	#1	33.0	9.35	28.3
		#2	43.3	8.61	19.9
	Naphthalene	#1	185	29.4	15.8
		#2	165	24.8	15.0
	2-Methylnaphthalene	#1	265	61.2	23.1
		#2	200	18.9	9.5

%RSD Range = 1 - 33
Mean %RSD = 12

NOTE: Units = µg/L
Extractions were performed in triplicate
All results were at least 2x the detection limit
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

Table 8. Multi-Laboratory Semi-Volatiles, Precision

Waste	Compound	Extraction Fluid	$\bar{X}$	S	%RSD
Ammonia Lime	BNAs	#1	10043	7680	76.5
Still Bottoms (A)		#2	10376	6552	63.1
API/EW	BNAs	#1	1624	675	41.6
Mixture (B)		#2	2074	1463	70.5
Fossil Fuel	BNAs	#1	750	175	23.4
Fly Ash (C)		#2	739	342	46.3
				Mean %RSD = 54	

NOTE: Units = µg/L
X = Mean results from 3 - 10 labs
Extraction Fluid #1 = pH 4.9
#2 = pH 2.9

%RSD Range for Individual Compounds

A, #1	0 - 113
A, #2	28 - 108
B, #1	20 - 156
B, #2	49 - 128
C, #1	36 - 143
C, #2	61 - 164

Table 9. Multi-Laboratory (11 Labs) VOCs, Precision

Waste	Compound	$\bar{X}$	S	%RSD
Mine Tailings	Vinyl chloride	6.36	6.36	100
	Methylene chloride	12.1	11.8	98
	Carbon disulfide	5.57	2.83	51
	1,1-Dichloroethene	21.9	27.7	127
	1,1-Dichloroethane	31.4	25.4	81
	Chloroform	46.6	29.2	63
	1,2-Dichloroethane	47.8	33.6	70
	2-Butanone	43.5	36.9	85
	1,1,1-Trichloroethane	20.9	20.9	100
	Carbon tetrachloride	12.0	8.2	68
	Trichloroethene	24.7	21.2	86
	1,1,2-Trichloroethene	19.6	10.9	56
	Benzene	37.9	28.7	76
	1,1,2,2-Tetrachloroethane	34.9	25.6	73
	Toluene	29.3	11.2	38
	Chlorobenzene	35.6	19.3	54
	Ethylbenzene	4.27	2.80	66
	Trichlorofluoromethane	3.82	4.40	115
	Acrylonitrile	76.7	110.8	144
Ammonia Lime Still Bottoms	Vinyl chloride	5.00	4.71	94
	Methylene chloride	14.3	13.1	92
	Carbon disulfide	3.37	2.07	61
	1,1-Dichloroethene	52.1	38.8	75
	1,1-Dichloroethane	52.8	25.6	49
	Chloroform	64.7	28.4	44
	1,2-Dichloroethane	43.1	31.5	73
	2-Butanone	59.0	39.6	67
	1,1,1-Trichloroethane	53.6	40.9	76
	Carbon tetrachloride	7.10	6.1	86
	Trichloroethene	57.3	34.2	60
	1,1,2-Trichloroethene	6.7	4.7	70
	Benzene	61.3	26.8	44
	1,1,2,2-Tetrachloroethane	3.16	2.1	66
	Toluene	69.0	18.5	27
	Chlorobenzene	71.8	12.0	17
	Ethylbenzene	3.70	2.2	58
	Trichlorofluoromethane	4.05	4.8	119
	Acrylonitrile	29.4	34.8	118

%RSD Range = 17 - 144
Mean %RSD = 75

NOTE: Units = µg/L

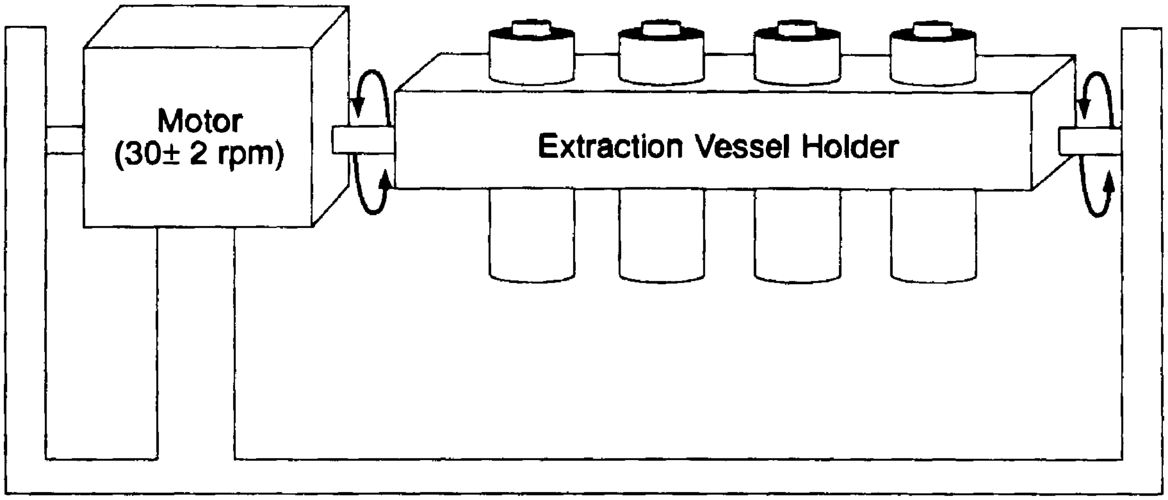


Figure 1. Rotary Agitation Apparatus

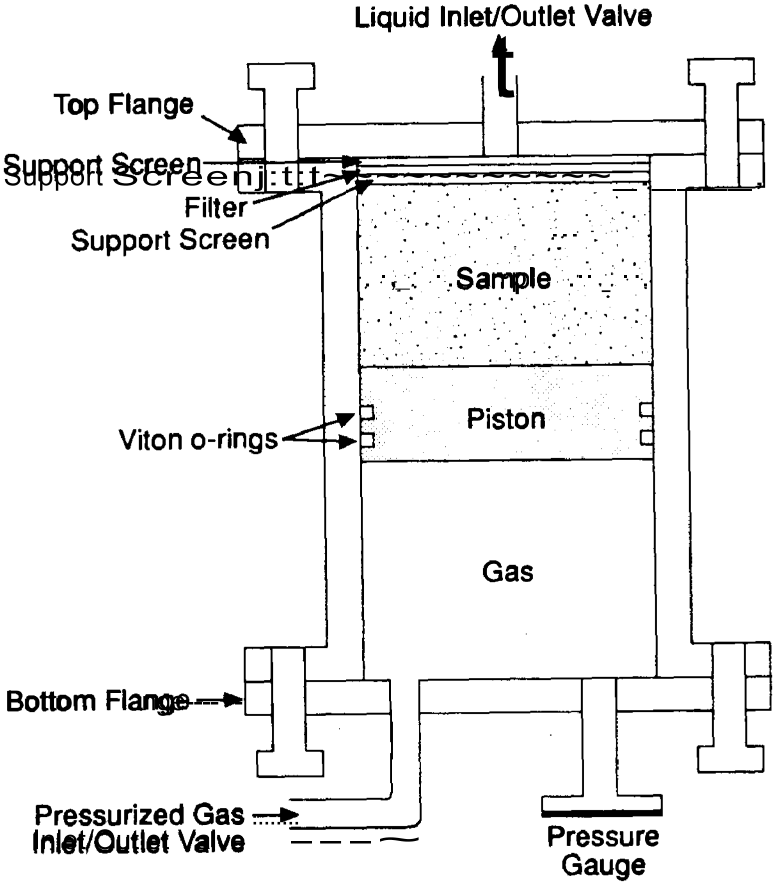
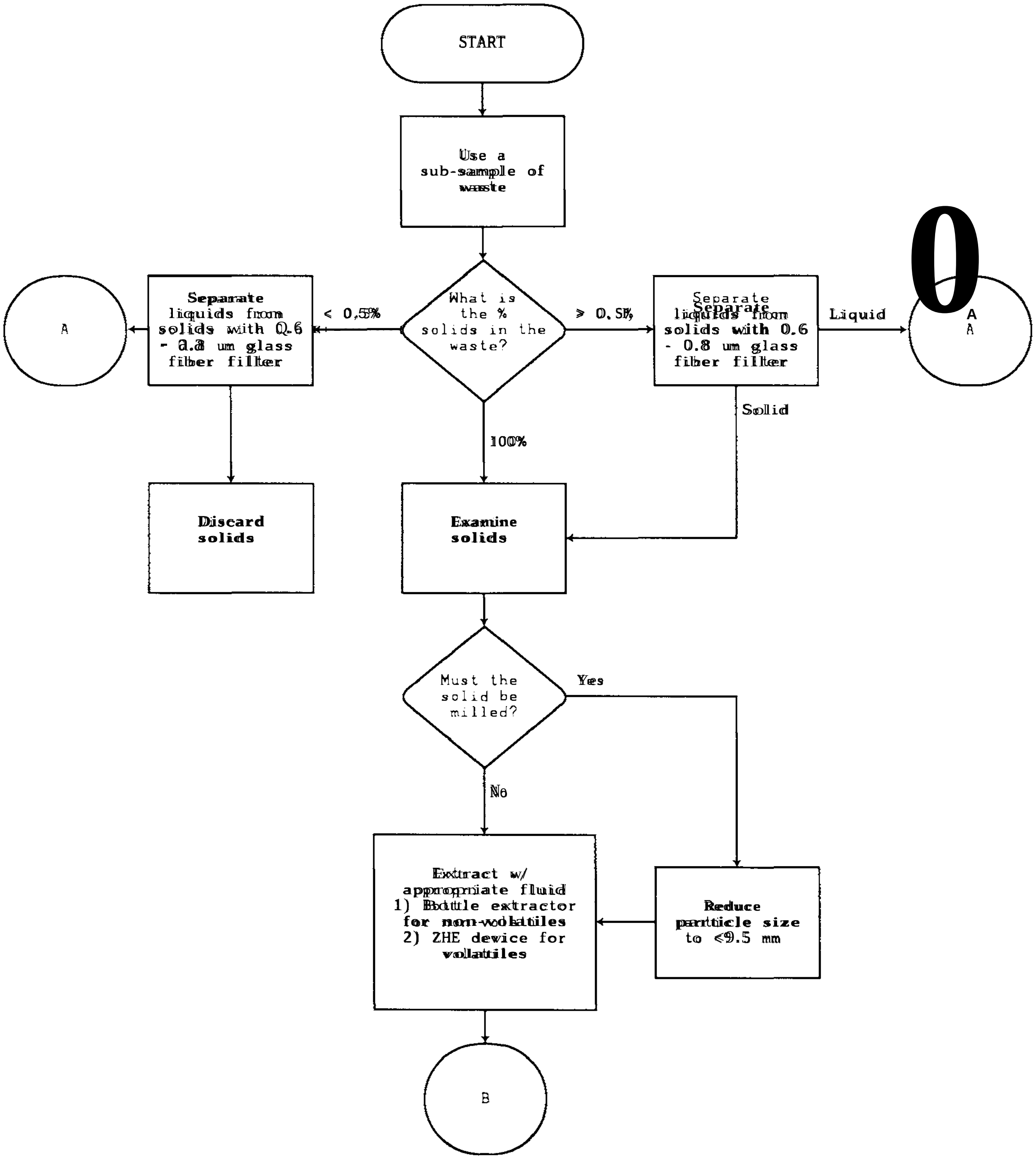


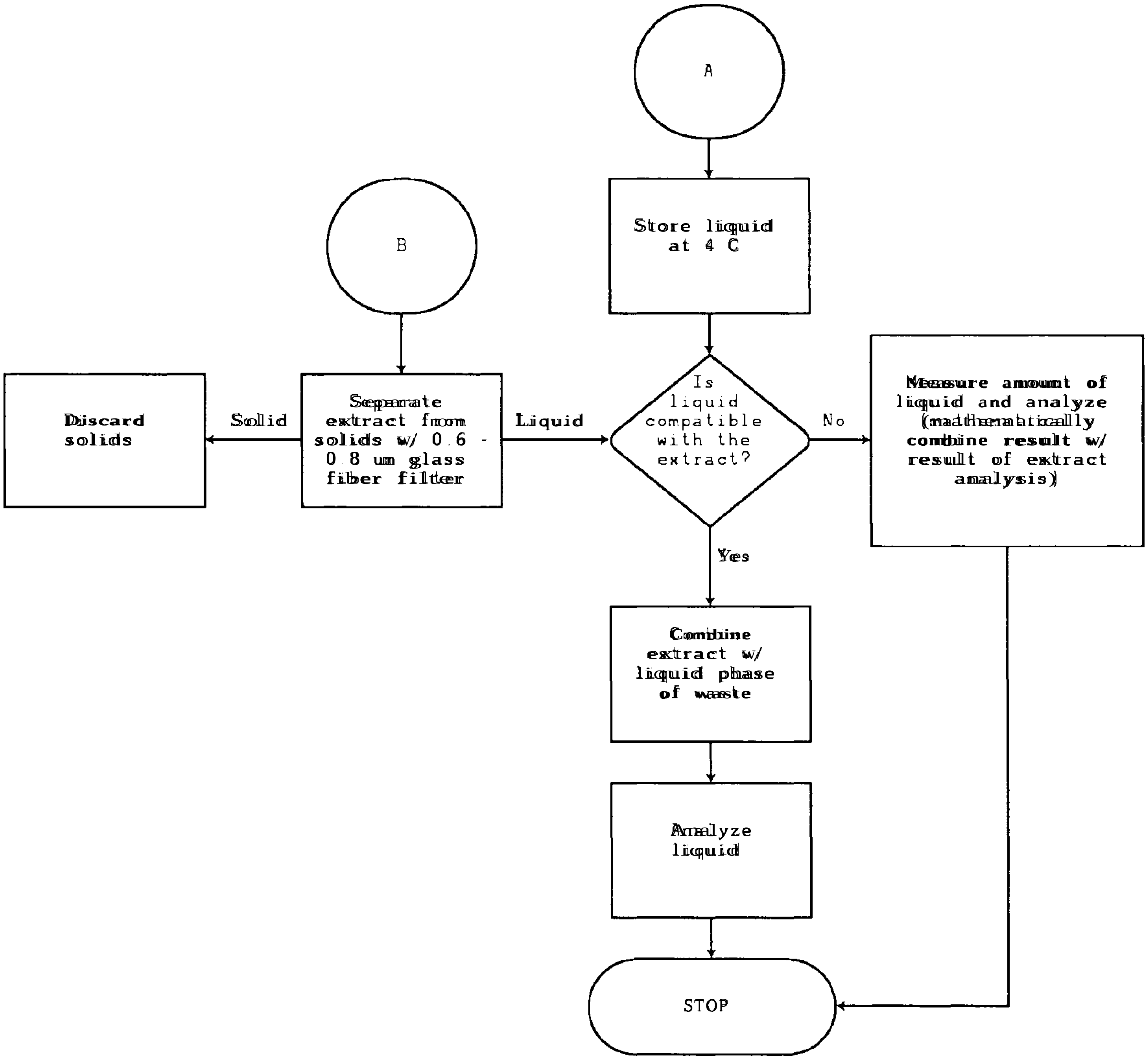
Figure 2. Zero-Headspace Extractor (ZHE)

METHOD 1311

TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



TOXICITY CHARACTERISTIC LEACHATE PROCEDURE



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RCRA Waste Sampling Draft Technical Guidance

Planning, Implementation, and Assessment

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Planning, Implementation, and Assessment

Office of Solid Waste
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LIST OF ACRONYMS

AL	Action Level
ASTM	American Society for Testing and Materials
BDAT	Best Demonstrated Available Technology
BIF	Boiler and Industrial Furnace
CERCLA	Comprehensive, Environmental Response, Compensation & Liability Act
CFR	Code of Federal Regulations
DOT	Department of Transportation
DQA	Data Quality Assessment
DQO	Data Quality Objective
EA	Exposure area
FR	Federal Register
HWIR	Hazardous Waste Identification Rule (waste)
IATA	International Air Transport Association
ICR	Ignitability, Corrosivity, and Reactivity
IDW	Investigation-derived waste
LCL	Lower confidence limit
LDR	Land Disposal Restrictions
ORD	Office of Research and Development
OSHA	Occupational Safety and Health Administration
OSW	Office of Solid Waste
PBMS	Performance-based measurement system
ppm	Parts per million
QAD	Quality Assurance Division
QAPP	Quality Assurance Project Plan
QA/QC	Quality Assurance/Quality Control
RCRA	Resource Conservation and Recovery Act
RT	Regulatory Threshold
SOP	Standard operating procedure
SWMU	Solid waste management unit
TC	Toxicity Characteristic
TCLP	Toxicity Characteristic Leaching Procedure
TSDF	Treatment, storage, or disposal facility
UCL	Upper confidence limit
USEPA	U.S. Environmental Protection Agency (we, us, our, EPA, the Agency)
UTS	Universal Treatment Standard
VOC	Volatile organic compound
WAP	Waste analysis plan

RCRA WASTE SAMPLING
DRAFT TECHNICAL GUIDANCE

1 INTRODUCTION

1.1 What Will I Find in This Guidance Document?

You'll find recommended procedures for sampling solid waste under the Resource Conservation and Recovery Act (RCRA). The regulated and regulatory communities can use this guidance to develop sampling plans to determine if (1) a solid waste exhibits any of the characteristics of a hazardous waste[1], (2) a hazardous waste is prohibited from land disposal, and (3) a numeric treatment standard has been met. You also can use information in this document along with that found in other guidance documents to meet other sampling objectives such as site characterization under the RCRA corrective action program.

This guidance document steps you through the three phases of the sampling and analysis process shown in Figure 1: planning, implementation, and assessment. Planning involves "asking the right questions." Using a systematic planning process such as the Data Quality Objectives (DQO) Process helps you do so. DQOs are the specifications you need to develop a plan for your project such as a quality assurance project plan (QAPP) or a waste analysis plan (WAP). Implementation involves using the field sampling procedures and analytical methods specified in the plan and taking measures to control error that might be introduced along the way. Assessment is the final stage in which you evaluate the results of the study in terms of the original objectives and make decisions regarding management or treatment of the waste.

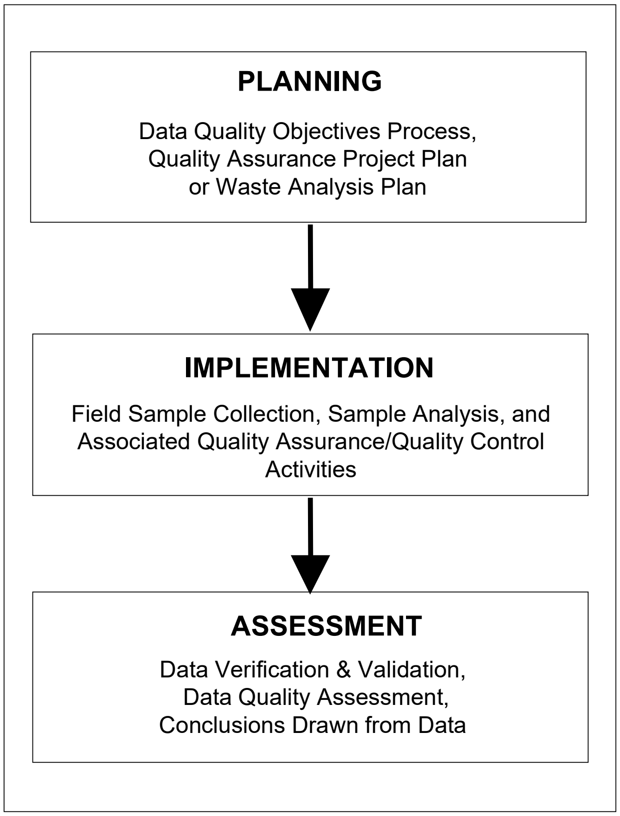

Figure 1. QA Planning and the Data Life Cycle (after USEPA 1998a).

1.2 Who Can Use This Guidance Document?

Any person who generates, treats, stores, or disposes of solid and hazardous waste and conducts sampling and analysis under RCRA can use the information in this guidance document.

---

[1] If a solid waste is not excluded from regulation under 40 CFR 261, then a generator must determine whether the waste exhibits any of the characteristics of hazardous waste. A generator may determine if a waste exhibits a characteristic either by testing the waste or applying knowledge of the waste, the raw materials, and the processes used in its generation.

For the development of a technically sound sampling and project plan, seek competent advice during the initial stages of project design. This is particularly true in the early developmental stages of a sampling plan when planners need to understand basic statistical concepts, how to establish objectives, and how the results of the project will be evaluated.

This document is a practical guide, and many examples are included throughout the text to demonstrate how to apply the guidance. In addition, we have included a comprehensive glossary of terms in Appendix A to help you with any unfamiliar terminology. We encourage you to review other documents referenced in the text, especially those related to the areas of sampling theory and practice and the statistical analysis of environmental data.

1.3 Does This Guidance Document Replace Other Guidance?

EPA prepared this guidance document to update technical information contained in other sources of EPA guidance such as Chapter Nine “Sampling Plan” found in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods,* EPA publication *SW-846* (1986a). This draft guidance document does not replace SW-846 Chapter Nine, nor does it create, amend, or otherwise alter any regulation. Since publication of SW-846 Chapter Nine, EPA has published a substantial body of additional sampling and statistical guidance documents that support waste and site characterization under both RCRA and the Comprehensive, Environmental Response, Compensation & Liability Act (CERCLA) or “Superfund.” Most of these guidance documents, which focus on specific Agency regulations or program initiatives, should continue to be used, as appropriate. Relevant EPA guidance documents, other references, and resources are identified in Appendix B and throughout this document.

In addition to RCRA program-specific guidance documents issued by EPA’s Office of Solid Waste (OSW), EPA’s Office of Environmental Information's Quality Staff has developed policy for quality assurance, guidance documents and software tools, and provides training and outreach. For example, the Quality Staff have issued guidance on the following key topic areas:

- The data quality objectives process (USEPA 2000a, 2000b, and 2001a)
- Preparation of quality assurance project plans (USEPA 1998a and 2001b) and sampling plans (2000c)
- Verification and validation of environmental data (USEPA 2001c)
- Data quality assessment (USEPA 2000d).

Information about EPA’s Quality System and QA procedures and policies can be found on the World Wide Web at http://www.epa.gov/quality/.

If you require additional information, you should review these documents and others cited in this document. In the future, EPA may issue additional supplemental guidance supporting other regulatory initiatives.

Finally, other organizations including EPA Regions, States, the American Society for Testing and Materials (ASTM), the Department of Defense (e.g., the Air Force Center for Environmental

Excellence), and the Department of Energy have developed a wide range of relevant guidance and methods. Consult these resources for further assistance, as necessary.

1.4 How Is This Document Organized?

As previously indicated in Figure 1, this guidance document covers the three components of a sampling and analysis program: planning, implementation, and assessment. Even though the process is pictured in a linear format, in practice a sampling program should include feedback between the various components. You should review and analyze data as collected so you can determine whether the data satisfy the objectives of the study and if the approach or objectives need to be revised or refined, and so you can make reasoned and intelligent decisions.

The remaining sections of this guidance document address specific topics pertaining to various components of a sampling program. These sections include the following:

> ***Section 2 - Summary of RCRA Regulatory Drivers for Waste Sampling and Analysis*** – This section identifies and summarizes the major RCRA programs that specify some sort of sampling and testing to determine if a waste is a hazardous waste, to determine if a hazardous waste treatment standard is attained, and other determinations.
>
> ***Section 3 - Fundamental Statistical Concepts*** -- This section provides an overview of fundamental statistical concepts and how the sample analysis results can be used to classify a waste or determine its status under RCRA. The section serves as a refresher to those familiar with basic statistics. In those cases where you require more advanced techniques, seek the assistance of a professional environmental statistician. Detailed guidance on the selection and use of statistical methods is provided in Section 8 and Appendix F.
>
> ***Section 4 - Planning Your Project Using the DQO Process*** -- The first phase of sampling involves development of DQOs using the DQO Process or a similar structured systematic planning process. The DQOs provide statements about the expectations and requirements of the data user (such as the decision maker).
>
> ***Section 5 - Optimizing the Design for Obtaining the Data*** -- This section describes how to link the results of the DQO Process with the development of the QAPP. You optimize the sampling design to control sampling errors within acceptable limits and minimize costs while continuing to meet the sampling objectives. You document the output of the DQO Process in a QAPP, WAP, or similar planning document. Here is where you translate the data requirements into measurement performance specifications and QA/QC procedures.
>
> ***Section 6 - Controlling Variability and Bias in Sampling*** -- In this section, we recognize that random variability and bias (collectively known as "error") in sampling account for a significant portion of the total error in the sampling and analysis process – far outweighing typical analytical error. To address this concern, the section describes the sources of error in sampling and offers some strategies for minimizing those errors.

Section 7 - Implementation: Selecting Equipment and Conducting Sampling -- In this section, we describe the steps for selecting sampling equipment based on the physical and chemical characteristics of the media to be sampled and the type of RCRA unit or location from which the samples will be obtained. The section provides guidance on field sampling activities, such as documentation, chain-of-custody procedures, decontamination, and sample packaging and shipping. Finally, guidance is provided on sample homogenization (or mixing), splitting, and subsampling.

Section 8 - Assessment: Analyzing and Interpreting Data -- Once you have obtained the data in accordance with the elements of the QAPP or WAP, you should evaluate the data to determine whether you have satisfied the DQOs. Section 8 describes the data quality assessment (DQA) process and the statistical analysis of waste-sampling data.

Appendix A - Glossary of Terms -- This appendix comprises a glossary of terms that are used in this document.

Appendix B - Summary of RCRA Regulatory Drivers for Conducting Waste Sampling and Analysis -- An overview of the RCRA regulatory requirements and other citations related to waste sampling and testing is provided in this appendix.

Appendix C - Strategies for Sampling Heterogeneous Wastes -- The heterogeneity of a waste or media plays an important role in how you collect and handle samples and what type of sampling design you use. This appendix provides a supplemental discussion of large-scale heterogeneity of waste and its impact on waste-sampling strategies. Various types of large-scale heterogeneity are identified and techniques are described for stratifying a waste stream based on heterogeneity. Stratified sampling can be a cost-effective approach for sampling and analysis of heterogeneous wastes.

Appendix D - A Quantitative Approach for Controlling Fundamental Error -- The mass of a sample can influence our ability to obtain reproducible analytical results. This appendix provides an approach for determining the appropriate mass of a sample of particulate material using information about the size and shape of the particles.

Appendix E - Sampling Devices -- This appendix provides descriptions of recommended sampling devices. For each type of sampling device, information is provided in a uniform format that includes a brief description of the device and its use, advantages and limitations of the device, and a figure to indicate the general design of the device. Each summary also identifies sources of other guidance on each device, particularly any relevant ASTM standards.

Appendix F - Statistical Methods -- This appendix provides statistical guidance for the analysis of data generated in support of a waste-testing program under RCRA.

Appendix G - Statistical Tables -- A series of statistical tables needed to perform the statistical tests used in this guidance document are presented here.

Appendix H - Statistical Software -- A list of statistical software and “freeware” (no-cost software) that you might find useful in implementing the statistical methods outlined in this guidance document is contained in this appendix, as are Internet addresses at which you can download no-cost software.

Appendix I - Examples of Planning, Implementation, and Assessment for RCRA Waste Sampling -- Two hypothetical examples of how to apply the planning, implementation, and assessment guidance provided in this guidance document are provided here.

Appendix J - Summaries of ASTM Standards -- This appendix provides summaries of ASTM standards related to waste sampling and referenced in this document.

2 SUMMARY OF RCRA REGULATORY DRIVERS FOR WASTE SAMPLING AND ANALYSIS

2.1 Background

Through RCRA, Congress provided EPA with the framework to develop regulatory programs for the management of solid and hazardous waste. The provisions of RCRA Subtitle C establish the criteria for identifying hazardous waste and managing it from its point of generation to ultimate disposal. EPA's regulations set out in 40 CFR Parts 260 to 279 are the primary source for the requirements of the hazardous waste program. These regulations were developed over a period of 25 years. While EPA's approach for developing individual regulations may have evolved over this period, the current RCRA statute and codified regulations remain the standard for determining compliance.

Many of the RCRA regulations either *require* the waste handler to conduct sampling and analysis, or they include provisions under which sampling and analysis can be performed at the discretion of the waste handler. If the regulations require sampling and analysis of a waste or environmental media, then any regulatory requirements for conducting the sampling and analysis and for evaluating the results must be followed. Regardless of whether there are regulatory requirements to conduct sampling, some waste handlers may wish to conduct a sampling program that allows them to quantify any uncertainties associated with their waste classification decisions. The information in this document can be used to aid in the planning and implementation of such a sampling program.

Some RCRA regulations *do not* specify sampling and analysis requirements and/or *do not* specify how the sample analysis results should be evaluated. In many cases, this is because EPA realized that the type, quantity, and quality of data needed should be specified on a site-specific basis, such as in the waste analysis plan of a permitted facility. In those situations, you can use the guidance in this document to help you plan and implement the sampling and analysis program, evaluate the sample analysis results against the regulatory standards, and quantify the level of uncertainty associated with the decisions.

This section identifies the major RCRA programs that specify some sort of sampling and testing to determine if a waste is a hazardous waste, to determine if a hazardous waste treatment standard is attained, or to meet other objectives such as site characterization. Table 1 provides a listing of these major RCRA programs that may require waste sampling and testing as part of their implementation. Appendix B provides a more detailed listing of the regulatory citations, the applicable RCRA standards, requirements for demonstrating attainment or compliance with the standards, and relevant USEPA guidance documents.

Prior to conducting a waste sampling and testing program to comply with RCRA, review the specific regulations in detail. Consult the latest 40 CFR, related *Federal Register* notices, and EPA's World Wide Web site (www.epa.gov) for new or revised regulations. In addition, because some states have requirements that differ from EPA regulations and guidance, we recommend that you consult with a representative from your State if your State is authorized to implement the regulation.

Table 1. Major RCRA Program Areas Involving Waste Sampling and Analysis [1]

40 CFR Citation	***Program Description***
	Hazardous Waste Identification
§ 261.3(a)(2)(v)	Used oil rebuttable presumption (also Part 279, Subparts B, E, F and G standards for the management of used oil)
§ 261.3(c)(2)(ii)(C)	Generic exclusion levels for K061, K062, and F006 nonwastewater HTMR residues
§ 261.21	Characteristic of Ignitability
§ 261.22	Characteristic of Corrosivity
§ 261.23	Characteristic of Reactivity
§ 261.24	Toxicity Characteristic
§ 261.38(c)(8)	Exclusion of Comparable Fuels from the Definition of Solid and Hazardous Waste
Part 261, Appendix I	Representative Sampling Methods
Mixed Hazardous Waste	Joint EPA-NRC sampling guidance. See November 20, 1997 *Federal Register* (62 *FR* 62079)
	Land Disposal Restriction Program
§ 268.6	Petitions to Allow Land Disposal of a Waste Prohibited Under Subpart C of Part 268 (No-Migration Petition). Sampling and testing criteria are specified at § 268.6(b)(1) and (2).
§ 268.40	Land Disposal Restriction (LDR) concentration-level standards
§ 268.44	Land Disposal Restriction Treatability Variance
§ 268.49(c)(1)	Alternative LDR Treatment Standards for Contaminated Soil
	Other RCRA Programs and References
§ 260.10	Definitions (for Representative Sample)
Part 260, Subpart C	Rulemaking Petitions
Part 262, Subpart A	Generator Standards - General (including § 262.11 Hazardous Waste Determination)
Part 262, Subpart C	Pre-Transport Requirements
Part 264, Subpart A	Treatment, Storage, and Disposal Facility Standards - General
Parts 264/265, Subpart B	Treatment, Storage, and Disposal Facility Standards - General Facility Standards
Parts 264/265, Subpart F	Releases from Solid Waste Management Units (ground-water monitoring)
Parts 264/265, Subpart G	Closure and Post-Closure
Parts 264, Subpart I	Use and Management of Containers
Parts 264/265 - Subpart J	Tank Systems

1. Expanded descriptions of the programs listed in Table 1 are given in Appendix B.

Table 1. Major RCRA Program Areas Involving Waste Sampling and Analysis (continued)

40 CFR Citation	*Program Description*
Other RCRA Programs and References (continued)	
Parts 264/265 - Subpart M	Land Treatment
Part 264/265 - Subpart O	Incinerators
Part 264, Subpart S	Corrective Action for Solid Waste Management Units (including § 264.552 Corrective Action Management Units)
Parts 264/265 - Subparts AA/BB/CC	Air Emission Standards
Part 266 - Subpart H	Hazardous Waste Burned in Boiler and Industrial Furnaces (BIFs) (including § 266.112 Regulation of Residues)
Part 270 - Subpart B	Permit Application, Hazardous Waste Permitting
Part 270 - Subpart C	Conditions Applicable to All Permits
Part 270 - Subpart F	Special Forms of Permits
Part 273	Standards for Universal Waste Management
Part 279	Standards for the Management of Used Oil

2.2 Sampling For Regulatory Compliance

Many RCRA programs involve sampling and analysis of waste or environmental media by the regulated community. Sampling and analysis often is employed to make a hazardous waste determination (see Section 2.2.1), to determine if a waste is subject to treatment or, if so, has been adequately treated under the Land Disposal Restrictions program (see Section 2.2.2), or in responding to other RCRA programs that include routine monitoring, unit closure, or cleanup (see Section 2.2.3).

2.2.1 Making a Hazardous Waste Determination

Under RCRA, a hazardous waste is defined as a solid waste, or a combination of solid wastes which, because of its quantity, concentration, or physical, chemical, or infectious characteristics, may cause, or significantly contribute to an increase in mortality or an increase in serious irreversible or incapacitating reversible illness, or pose a substantial present or potential hazard to human health or the environment when improperly treated, stored, transported, disposed, or otherwise managed. The regulatory definition of a hazardous waste is found in 40 CFR § 261.3.

Solid wastes are defined by regulation as hazardous wastes in two ways. First, solid wastes are hazardous wastes if EPA lists them as hazardous wastes. The lists of hazardous wastes are found in 40 CFR Part 261, Subpart D. Second, EPA identifies the characteristics of a hazardous waste based on criteria in 40 CFR § 261.10. Accordingly, solid wastes are hazardous if they exhibit any of the following four characteristics of a hazardous waste: ignitability, corrosivity, reactivity, or toxicity (based on the results of the Toxicity Characteristic Leaching Procedure, or TCLP). Descriptions of the hazardous waste characteristics are found in 40 CFR Part 261, Subpart C.

Generators must conduct a hazardous waste determination according to the hierarchy specified in 40 CFR § 262.11. Persons who generate a solid waste first must determine if the solid waste is excluded from the definition of hazardous waste under the provisions of 40 CFR § 261.4. Once the generator determines that a solid waste is not excluded, then he/she must determine if the waste meets one or more of the hazardous waste listing descriptions and determine whether the waste is mixed with a hazardous waste, is derived from a listed hazardous waste, or contains a hazardous waste.

For purposes of compliance with 40 CFR Part 268, or if the solid waste is not a listed hazardous waste, the generator must determine if the waste exhibits a characteristic of a hazardous waste. This evaluation involves testing the waste *or* using knowledge of the process or materials used to produce the waste.

When a waste handler conducts testing to determine if the waste exhibits any of the four characteristics of a hazardous waste, he or she must obtain a representative sample (within the meaning of a representative sample given at § 260.10) using the applicable sampling method specified in Appendix I of Part 261 or alternative method (per § 261.20(c))[1] and test the waste for the hazardous waste characteristics of interest at § 261.21 through 261.24.

For the purposes of subpart 261, the identification of hazardous waste, the regulations state that a sample obtained using any of the applicable sampling methods specified in Appendix I of Part 261 to be a representative sample within the meaning of the Part 260 definition of representative sample. Since these sampling methods are not officially required, anyone desiring to use a different sampling method may do so without demonstrating the equivalency of that method under the procedures set forth in § 260.21. The user of an alternate sampling method must use a method that yields samples that "meet the definition of representative sample found in Part 260" (45 FR 33084 and 33108, May 18, 1990). Such methods should enable one to obtain samples that are equally representative as those specified in Appendix I of Part 261. The planning process and much of the information described in this guidance document may be helpful to someone regulated under Part 261 wishing to use an alternate sampling method. The guidance should be help full as well for purposes other than Part 261.

Certain states also may have requirements for identifying hazardous wastes in addition to those requirements specified by Federal regulations. States authorized to implement the RCRA or HSWA programs under Section 3006 of RCRA may promulgate regulations that are more stringent or broader in scope than Federal regulations.

2.2.2 Land Disposal Restrictions (LDR) Program

The LDR program regulations found at 40 CFR Part 268 require that a hazardous waste generator determine if the waste has to be treated before it can be land disposed. This is done by determining if the hazardous waste meets the applicable treatment standards at § 268.40, § 268.45, or §268.49. EPA expresses treatment standards either as required treatment technologies that must be applied to the waste or as contaminant concentration levels that must

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[1] Since the 40 CFR Part 261 Appendix I sampling methods are not formally adopted by the EPA Administrator, a person who desires to employ an alternative sampling method is not required to demonstrate the equivalency of his or her method under the procedures set forth in §§ 260.20 and 260.21 (see comment at § 261.20(c)).

be met. (Alternative LDR treatments standards have been promulgated for contaminated soil, debris, and lab packs.) Determining the need for waste treatment can be made by either of two ways: testing the waste or using knowledge of the waste (see § 268.7(a)).

If a hazardous waste generator is managing and treating prohibited waste or contaminated soil in tanks, containers, or containment buildings to meet the applicable treatment standard, then the generator must develop and follow a written waste analysis plan (WAP) in accordance with § 268.7(a)(5).

A hazardous waste treater must test their waste according to the frequency specified in their WAP as required by 40 CFR 264.13 (for permitted facilities) or 40 CFR 265.13 (for interim status facilities). See § 268.7(b).

If testing is performed, *no portion of the waste may exceed the applicable treatment standard*, otherwise, there is evidence that the standard is not met (see 63 FR 28567, March 26, 1998). Statistical variability is “built in” to the standards (USEPA 1991c). Wastes that do not meet treatment standards can not be land disposed unless EPA has granted a variance, extension, or exclusion (or the waste is managed in a "no-migration unit"). In addition to the disposal prohibition, there are prohibitions and limits in the LDR program regarding the dilution and storage of wastes. The program also requires tracking and recordkeeping to ensure proper management and safe land disposal of hazardous wastes.

General guidance on the LDR program can be found in *Land Disposal Restrictions: Summary of Requirements* (USEPA 2001d). Detailed guidance on preparing a waste analysis plan (WAP) under the LDR program can be found in *Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes - A Guidance Manual* (USEPA 1994a). Detailed guidance on measuring compliance with the alternative LDR treatment standards for contaminated soil can be found in *Guidance on Demonstrating Compliance With the Land Disposal Restrictions (LDR) Alternative Soil Treatment Standards* (USEPA 2002a).

2.2.3 Other RCRA Regulations and Programs That May Require Sampling and Testing

In addition to the RCRA hazardous waste identification regulations and the LDR regulations, EPA has promulgated other regulations and initiated other programs that may involve sampling and testing of solid waste and environmental media (such as ground water or soil). Program-specific EPA guidance should be consulted prior to implementing a sampling or monitoring program to respond to the requirements of these regulations or programs. For example, EPA has issued separate program-specific guidance on sampling to support preparation of a delisting petition, ground-water and unsaturated zone monitoring at regulated units, unit closure, corrective action for solid waste management units, and other programs. See also Appendix B of this document.

2.2.4 Enforcement Sampling and Analysis

The sampling and analysis conducted by a waste handler during the normal course of operating a waste management operation might be quite different than the sampling and analysis conducted by an enforcement agency. The primary reason is that the data quality objectives (DQOs) of the enforcement agency often may be legitimately different from those of a waste handler. Consider an example to illustrate this potential difference in approach: Many of

RCRA's standards were developed as concentrations that should not be exceeded (or equaled) or as characteristics that should not be exhibited for the waste or environmental media to comply with the standard. In the case of such a standard, the waste handler and enforcement officials might have very different objectives. An enforcement official, when conducting a compliance sampling inspection to evaluate a waste handler's compliance with a "do not exceed" standard, take only one sample. Such a sample may be purposively selected based on professional judgment. This is because all the enforcement official needs to observe – for example to determine that a waste is hazardous – is a single exceedance of the standard.

A waste handler, however, in responding to the same regulatory standard may want to ensure, with a specified level of confidence, that his or her waste concentrations are low enough so that it would be unlikely, for example, that an additional sample drawn from the waste would exceed the regulatory standard. In designing such an evaluation the waste handler could decide to take a sufficient number of samples in a manner that would allow evaluation of the results statistically to show, with the desired level of confidence, that there is a low probability that another randomly selected sample would exceed the standard.

An important component of the enforcement official's DQO is to "prove the positive." In other words, the enforcement official is trying to demonstrate whether the concentration of a specific constituent in some portion of the waste exceeds the "do not exceed" regulatory level. The "prove the positive" objective combined with the "do not exceed" standard only requires a single observation above the regulatory level in order to draw a valid conclusion that at least some of the waste exceeds the level of concern.

The Agency has made it clear that in "proving the positive," the enforcement agency's DQOs may not require low detection limits, high analyte recoveries, or high degrees of precision:

> *"If a sample possesses the property of interest, or contains the constituent at a high enough level relative to the regulatory threshold, then the population from which the sample was drawn must also possess the property of interest or contain that constituent. Depending on the degree to which the property of interest is exceeded, testing of samples which represent all aspects of the waste or other material may not be necessary to prove that the waste is subject to regulation"* (see 55 FR 4440, "Hazardous Waste Management System: Testing and Monitoring Activities," February 8, 1990).

A waste handler may have a different objective when characterizing his or her waste. Instead, the waste handler may wish to "prove the negative." While proving the negative in absolute terms is not realistic, the waste handler may try to demonstrate with a desired level of confidence that the vast majority of his or her waste is well below the standard such that another sample or samples taken from the waste would not likely exceed the regulatory standard. The Agency also has spoken to the need for sound sampling designs and proper quality control when one is trying to "prove the negative:"

> *"The sampling strategy for these situations (proving the negative) should be thorough enough to insure that one does not conclude a waste is nonhazardous when, in fact, it is hazardous. For example, one needs to take enough samples so that one does not miss areas of high concentration in an otherwise clean material. Samples must be handled so that properties do not change and*

> *contaminants are not lost. The analytical methods must be quantitative, and regulatory detection limits must be met and documented"* (see 55 FR 4440, "Hazardous Waste Management System: Testing and Monitoring Activities," February 8, 1990).

"Proving the negative" can be a more demanding objective for the waste handler in terms of the sampling strategy and resources than that faced by the enforcement official. To address this objective the waste handler could use the advice in this or similar guidance documents. In doing so, the waste handler should establish objectives using a systematic planning process, design a sampling and analysis plan based on the objectives, collect and analyze the appropriate number of samples, and use the information from the sample analysis results for decision-making.

The distinction between a sampling strategy designed to "prove the negative" versus one designed to "prove the positive" also has been supported in a recent judicial ruling. In *United States v. Allen Elias* (9th Cir. 2001) the Government used a limited number of samples to prove that hazardous waste was improperly managed and disposed. The court affirmed that additional sampling by the Government was not necessary to "prove the positive."

3 FUNDAMENTAL STATISTICAL CONCEPTS

Throughout the life cycle of a waste-testing program, the tools of statistics often are employed -- in planning, implementation, and assessment. For example, in the planning phase, you may state certain project objectives quantitatively and use statistical terminology. Designing and implementing a sampling plan requires an understanding of error and uncertainty. Statistical techniques can be used to describe and evaluate the data and to support decisions regarding the regulatory status of a waste or contaminated media, attainment of treatment or cleanup goals, or whether there has been a release to the environment. Because statistical concepts may be used throughout the sampling and analysis program, an understanding of basic statistical concepts and terminology is important.

While statistical methods can be valuable in designing and implementing a scientifically sound waste-sampling program, their use should not be a substitute for knowledge of the waste or as a substitute for common sense. Not every problem can, or necessarily must, be evaluated using probabilistic techniques. Qualitative expressions of decision confidence through the exercise of professional judgment (such as a "weight of evidence" approach) may well be sufficient, and in some cases may be the only option available (Crumbling 2001).

Do the RCRA regulations require statistical sampling?

Some RCRA regulations *require* the use of statistical tests (e.g., to determine if there has been a release to ground water from a waste management unit under 40 CFR Subpart F), whereas, other RCRA regulations *do not* require the use of statistical tests (such as those for determining if a solid waste is or is not a hazardous waste or determining compliance with LDR treatment standards). Even where there is no regulatory obligation to conduct sampling or apply statistical tests to evaluate sampling results, statistical methods can be useful in interpreting data and managing uncertainty associated with waste classification decisions.

If the objective of the sampling program is to make a hazardous waste determination, the regulations allow that a single representative sample is sufficient to classify a waste as hazardous. If a representative sample is found to have the properties set forth for the corrosivity, ignitability, reactivity, or toxicity characteristics, then the waste is hazardous. The regulations do not address directly what is a sufficient number of samples to classify a solid waste as *nonhazardous*. However, for a petition to reclassify (delist) a listed hazardous waste, which includes a determination that the listed hazardous waste is not a characteristic hazardous waste (a "nonhazardous" classification), the regulations provide that at least four representative samples sufficient to represent the variability or uniformity of the waste must be tested (40 CFR 260.22). This approach is not necessarily based on any statistical method but reflects concepts of proving the negative and proving the positive (see also Section 2.2.4).

Even if you have no formal training in statistics, you probably are familiar with basic statistical concepts and how samples are used to make inferences about the population from which the samples were drawn. For example, the news media frequently cite the results of surveys that make generalized conclusions about public opinion based on interviews with a relatively small proportion of the population. These results, however, are only *estimates* because no matter how carefully a survey is done, if repeated over and over in an identical manner, the answer will be a little different each time. There always will be some random sampling variation because it is not possible to survey every member of a population. There also will be measurement and estimation errors because of mistakes made in how data are obtained and interpreted. Responsible pollsters report this as their "margin of error" along with the findings of the survey

(Edmondson 1996).

Similar to surveys of human populations, waste characterization studies can be designed in such a way that a population can be identified, samples can be collected, and the uncertainty in the results can be reported.

The following sections provide a brief overview of the statistical concepts used in this guidance. Four general topics are described:

- Populations, samples, and distributions (Section 3.1)
- Measures of central tendency, variability, and relative standing (Section 3.2)
- Precision and bias (Section 3.3)
- Using sample analysis results to classify a waste or determine its status under RCRA (Section 3.4).

Guidance on selecting and using statistical methods for evaluating data is given in Section 8.2 and Appendix F of this document. Statistical tables are given in Appendix G. Additional statistical guidance can be found in *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d) and other references cited.

3.1 Populations, Samples, and Distributions

A “population” consists of all the waste or media whose characteristics are to be studied and estimated. A set of observations, known as a statistical sample, is a portion of the population that is studied in order to learn about the whole population. Sampling is necessary when a study of the entire population would be too expensive or physically impossible.

Inferences about the population are made from samples selected from the population. For example, the sample mean (or average) is a consistent estimator of the population mean. In general, estimates made from samples tend to more closely approximate the true population parameter as the number of samples increases. The precision of these inferences depends on the theoretical sampling distribution of the statistic that would occur if the sampling process were repeated over and over using the same sampling design and number of samples.

3.1.1 Populations and Decision Units

A “population” is the *entire* selection of interest for study. Populations can have *spatial* boundaries, which define the physical area to be studied, and *temporal* boundaries, which describe the time interval the study will represent. The definition of the population can be subjective, defined by regulation or permit condition, or based on risks to human health and the environment. In all cases, however, the population needs to be finite and have well-defined, unambiguous physical and/or temporal boundaries. The physical boundary defines the size, shape, orientation, and location of the waste or media about which a decision will be made.

For a large population of waste or media, you may wish to subdivide the population into smaller units about which decisions can be made, rather than attempt to characterize the entire population. These units are called “decision units,” and they may represent a single type of waste at the point of waste generation, a waste from a single batch operation, waste generated over a specified time, or a volume of waste or contaminated media (such as soil) subject to characterization, removal, and/or treatment. The concept of a decision unit is similar to an “exposure unit” (Neptune, et al. 1990, Blacker and Goodman 1994a and 1994b, Myers 1997), or “exposure area” (USEPA 1992a and 1996a) in EPA’s Superfund program in which risk-based decisions consider the mass or area of the waste or media. A decision unit also is analogous to a “remediation unit” as described in EPA’s *Data Quality Objective Process for Superfund* (USEPA 1993a).

When using samples to determine whether a solid waste is a hazardous waste, that determination must be made at the **point of generation** (i.e., when the waste becomes a solid waste).

Hypothetical examples of populations or decision units that might be encountered in the context of RCRA waste characterization follow:

- Filter cake being placed in a 25-cubic-yard roll-off bin at the point of waste generation
- Waste water contained in a 55-gallon drum
- Liquid waste flowing from the point of generation during a specified time interval
- A block of soil (e.g., 10-feet-by-10-feet square, 6-inches deep) within a solid waste management unit (SWMU).

In some situations, it will be appropriate to define two separate populations for comparison to each other. For example, in monitoring a land-based waste management unit to determine if there has been a release to the subsurface at statistically significant levels above background, it is necessary to establish two populations: (1) a background population and (2) an exposed (or downgradient) population in the soil, pore-water, or ground-water system.

In situations in which the boundaries of the waste or contamination are not obvious or cannot be defined in advance (such as the case of contaminated soil *in situ*, as opposed to excavated soil in a pile), the investigator is interested in the *location* of the contamination as well as the concentration information. Such a sampling objective is best addressed by spatial analysis, for example, by using geostatistical methods (See also Section 3.4.4).

3.1.2 Samples and Measurements

Samples are portions of the population. Using information from a set of samples (such as measurements of chemical concentrations) and the tools of inductive statistics, inferences can be made about the population. The validity of the inferences depends on how closely the samples represent the physical and chemical properties of the population of interest.

In this document, we use the word “sample” in several different ways. To avoid confusion, definitions of terms follow:

> **Sample:** A portion of material that is taken from a larger quantity for the purpose of estimating properties or composition of the larger quantity (from ASTM D 6233-98).
>
> **Statistical sample:** A set of samples or measurements selected by probabilistic means (i.e., by using some form of randomness).

We sometimes refer to a “set of samples” to indicate more than one individual sample that may or may not have been obtained by probabilistic means.

Outside the fields of waste management and environmental sciences, the concept of a sample or “sampling unit” is fairly straightforward. For example, a pollster measures the opinions of individual human beings, or the QC engineer measures the diameter of individual ball bearings. It is easy to see that the measurement and the sampling unit correspond; however, in sampling waste or environmental media, *what is the appropriate “portion” that should be in a sampling unit*? The answer to this question requires consideration of the heterogeneities of the sample media and the dimension of the sampling problem (in other words, are you sampling over time or sampling over space?). The information can be used to define the appropriate *size, shape, and orientation* of the sample. The size, shape, and orientation of a sample are known as the **sample support**, and the sample support will affect the measurement value obtained from the sample.

As shown in Figure 2, after a sample of a certain *size, shape, and orientation* is obtained in the field (as the primary sample), it is handled, transported, and prepared for analysis. At each stage, changes can occur in the sample (such as the gain or loss of constituents, changes in the particle size distribution, etc.). These changes accumulate as errors throughout the sampling process such that measurements made on relatively small analytical samples (often less than 1 gram) may no longer “represent” the population of interest. Because sampling and analysis results may be relied upon to make decisions about a waste or media, it is important to understand the sources of the errors introduced at each stage of sampling and take steps to minimize or control those errors. In doing so, samples will be sufficiently “representative” of the population from which they are obtained.

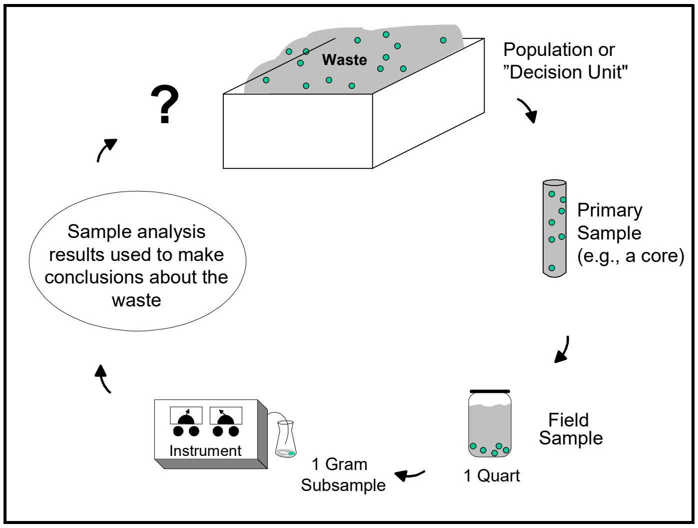

Figure 2. Very small analytical samples are used to make decisions about much larger volumes (modified after Myers 1997).

The RCRA solid waste regulations at 40 CFR §260.10 define a **representative sample** as:

> “a sample of a universe or whole (e.g., waste pile, lagoon, ground water) which can be expected to exhibit the average properties of the universe or whole."

RCRA implementors, at a minimum, must use this definition when a representative sample is called for by the regulations. Various other definitions of a representative sample have been developed by other organizations. For example, ASTM in their consensus standard D 6044-96 defines a representative sample as “a sample collected in such a manner that it reflects one or more characteristics of interest (as defined by the project objectives) of a population from which it was collected" (ASTM D 6044). A detailed discussion of representativeness also is given in *Guidance on Data Quality Indicators* (USEPA 2001e).

3.1.3 Distributions

Because the concentration of constituents of concern will not be the same for every individual sample, there must be a *distribution* of concentrations among the population. Understanding the distributional characteristics of a data set is an important first step in data analysis.

If we have a sufficient number of samples selected from a population, a picture of the distribution of the sample data can be represented in the form of a **histogram**. A histogram, which offers a simple graphical representation of the shape of the distribution of data, can be constructed by dividing the data range into units or “bins” (usually of equal width), counting the number of points within each unit, and displaying the data as the height or area within a bar graph. Figure 3 is an example of a histogram made using analysis results for total lead in 11 samples of No. 2 fuel oil (data set from USEPA 1998b). Guidance on constructing histograms can be found in EPA’s *Guidance for Data Quality Assessment, EPA QA/G*-9 (USEPA 2000d).

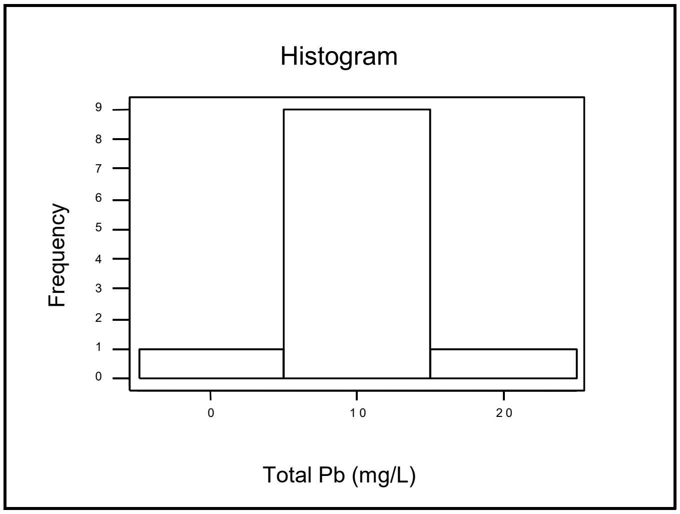

Figure 3. Histogram representing the distribution of total lead (Pb) in 11 samples of No. 2 fuel oil (USEPA 1998b).

With a sufficiently large number of samples, the bars of the histogram could be “blended together” to form a curve known as a probability density function (PDF). Figure 4 shows two probability density functions you might encounter: Figure 4(a) is a **normal distribution** with its familiar symmetrical mound-shape. Figure 4(b) is a **lognormal distribution** in which the natural log-transformed values exhibit a normal distribution. A lognormal distribution indicates that a relatively small proportion of the population includes some relatively large values.

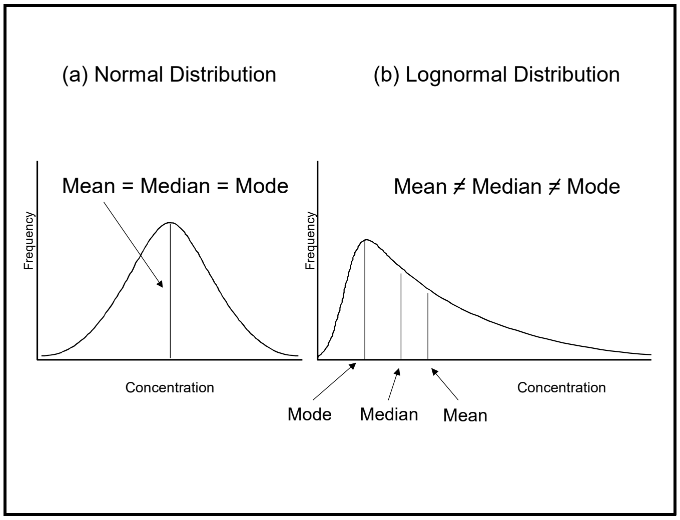

Figure 4. Examples of two distributions: (a) normal distribution and (b) lognormal distribution

Many of the tools used in statistics are based on the assumption that the data are normally distributed, can be transformed to a normal scale, or can be treated as if they are approximately normal. The assumption of a normal distribution often can be made without significantly increasing the risk of making a “wrong” decision. Of course, the normal and lognormal distributions are *assumed* models that only approximate the underlying population distribution.

Another distribution of interest is known as the **binomial distribution**. The binomial distribution can be used when the sample analysis results are interpreted as either “fail” or “pass” (e.g., a sample analysis result either exceeds a regulatory standard or does not exceed the standard).

In some cases, you may not be able to “fit” the data to any particular distributional model. In these situations, we recommend you consider using a “distribution-free” or “nonparametric” statistical method (see Section 8.2).

A simple but extremely useful graphical test for normality is to graph the data as a **probability plot**. In a probability plot, the vertical axis has a probability scale and the horizontal axis has a data scale. In general, if the data plot as a straight line, there is a qualitative indication of normality. If the natural logarithms of the data plot as a straight line, there is an indication of lognormality.

Figure 5 provides an example of a normal probability plot created from the same data used to generate the histogram in Figure 3. Guidance on constructing probability plots can be found in EPA’s *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d).

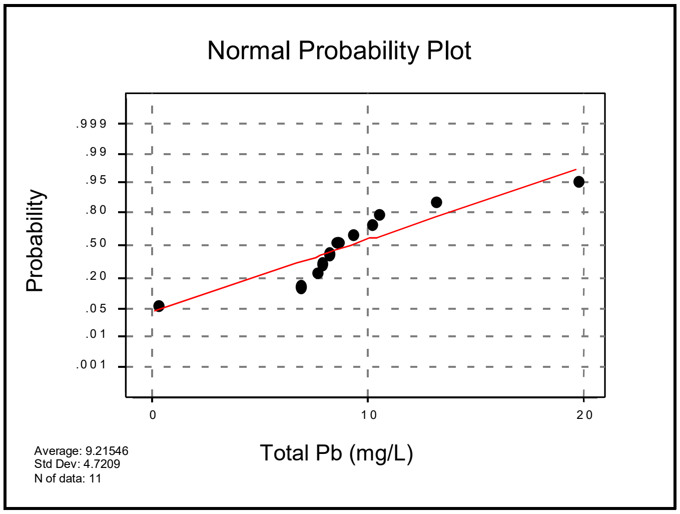

Figure 5. Normal probability plot

Section 8 (Assessment: Analyzing and Interpreting Data) provides guidance on checking the distribution of data sets and provides strategies for handling sample data exhibiting a non-normal distribution.

3.2 Measures of Central Tendency, Variability, and Relative Standing

In addition to graphical techniques for summarizing and describing data sets, numerical methods can be used. Numerical methods can be used to describe the central tendency of the set of measurements, the variability or spread of the data, and the relative standing or relative location of a measurement within a data set.

3.2.1 Measures of Central Tendency

The average or **mean** often is used as a measure of central tendency. The mean of a set of quantitative data is equal to the sum of the measurements divided by the number of measurements contained in the data set. Other measures of central tendency include the

median (the midpoint of an ordered data set in which half the values are below the median and half are above) and the **mode** (the value that occurs most often in the distribution). For distributions that are not symmetrical, the median and the mean do not coincide. The mean for a lognormal distribution, for instance, will exceed its median (see Figure 4(b)).

The true **population mean**, μ ("mu"), is the average of the true measurements (e.g., of the constituent concentration) made over all possible samples. The population mean is never known because we cannot measure all the members of a population (or all possible samples). We can, however, *estimate* the population mean by taking random samples from the population. The average of measurements taken on random samples is called the **sample mean**. The sample mean is denoted by the symbol $\bar{x}$ ("x-bar") and calculated by summing the value obtained from each random sample (x_i) and dividing by the number of samples (n):

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$$ **Equation 1**

Box 1 provides an example calculation of the sample mean.

Box 1. Example Calculation of the Sample Mean

Using Equation 1 and the following four data points in parts per million (ppm): 86, 90, 98, and 104, the following is an example of computing the sample mean.

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i = \frac{86+90+98+104}{4} = 95ppm$$

Therefore, the sample mean is 95 ppm.

3.2.2 Measures of Variability

Random variation in the population is described by "dispersion" parameters -- the **population variance** (σ^2) and the **population standard deviation** (σ). Because we cannot measure all possible samples that comprise the population, the values for σ^2 and σ are unknown. The variance, however, can be *estimated* from a statistical sample of the population by the **sample variance**:

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2$$ **Equation 2**

The variance calculated from the samples is known as the **sample variance** (s^2) and it includes random variation in the population as well as random variation that can be introduced by sample collection and handling, sample transport, and sample preparation and analysis. The sample variance is an estimate of the variance that one would obtain if the entire set of all possible samples in the population were measured using the same measurement process as is being employed for the n samples. If there were no sample handling or measurement error, this sample variance (s^2) would estimate the population variance (σ^2).

The **population standard deviation** (σ) is estimated by s, the **sample standard deviation**:

$$s = \sqrt{s^2}$$ **Equation 3**

Box 2 provides an example calculation of the sample variance and sample standard deviation.

Box 2. Example Calculations of Sample Variance and Standard Deviation

Using Equation 2 and the data points in Box 1, the following is an example calculation of the sample variance:

$$s^2 = \frac{\left[(86-94.5)^2 + (90-94.5)^2 + (98-94.5)^2 + (104-94.5)^2\right]}{4-1} = \frac{195}{3} = 65$$

Using Equation 3, the sample standard deviation is then calculated as follows:

$$s = \sqrt{s^2} = 8.1$$

The standard deviation is used to measure the variability in a data set. For a normal distribution, we know the following (see Figure 6):

- Approximately 68 percent of measurements will fall within ± 1 standard deviation of the mean
- Approximately 95 percent of the measurements will fall within ± 2 standard deviations of the mean
- Almost all (99.74 percent) of the measurements will fall within ± 3 standard deviations of the mean.

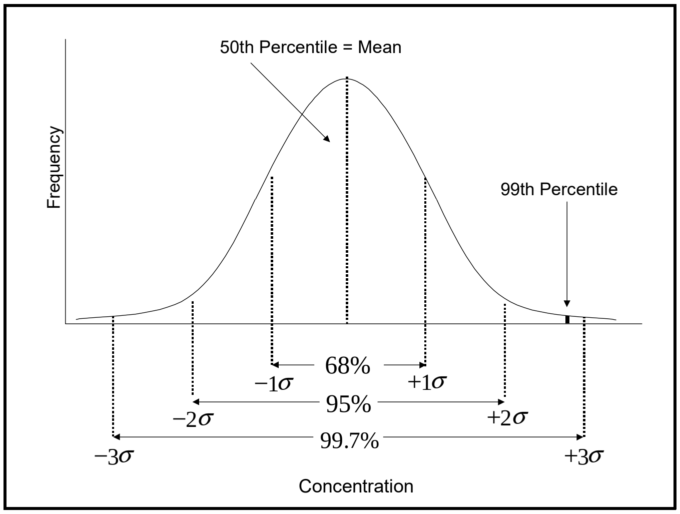

Figure 6. Percentage of values falling within 1, 2, and 3 standard deviations of the mean of a normal distribution. The figure also shows the relationship between the mean, the 50th percentile, and the 99th percentile in a normal distribution.

Estimates of the standard deviation, combined with the assumption of a normal distribution, allow us to make quantitative statements about the spread of the data. The larger the spread in the data, the less certainty we have in estimates or decisions made from the data. As discussed in the following section, a small spread in the data offers more certainty in estimates and decisions made from the data.

Because $\bar{x}$ is an estimate of a population parameter based on a statistical sample, we expect its value to be different each time a new set of samples is drawn from the population. The means calculated from repeated statistical samples also form a distribution. The estimate of the standard deviation of the sampling distribution of means is called the **standard error**.

The standard error of the mean ($s_{\bar{x}}$) is estimated by:

$$s_{\bar{x}} = \frac{s}{\sqrt{n}} \qquad \textbf{Equation 4}$$

The standard error is used in equations to calculate the appropriate number of samples to estimate the mean with specified confidence (see Section 5.4), and it is used in statistical tests to make inferences about $\bar{x}$ (see Appendix F).

3.2.3 Measures of Relative Standing

In addition to measures of central tendency and variability to describe data, we also may be interested in describing the relative standing or location of a particular measurement within a data set. One such measure of interest is the **percentile** ranking. A population percentile represents the percentage of elements of a population having values less than a specified value. Mathematically, for a set of n measurements the $p\text{th}$ percentile (or quantile) is a number such that $p\%$ of the measurements fall below the $p\text{th}$ percentile, and $(100 - p)\%$ fall above it. For example, if a measurement is located at the 99th percentile in a data set, it means that 99 percent of measurements are less than that measurement, and 1 percent are above. In other words, almost the *entire* distribution lies below the value representing the 99th percentile. Figure 6 depicts the relationship between the mean, the 50th percentile, and the 99th percentile in a normal distribution.

Just like the mean and the median, a percentile is a population parameter that must be estimated from the sample data. As indicated in Figure 6, for a normal distribution a "point estimate" of a percentile ($\hat{x}_p$) can be obtained using the sample mean ($\bar{x}$) and the sample standard deviation (s) by:

$$\hat{x}_p = \bar{x} + z_p s \qquad \textbf{Equation 5}$$

where z_p is the $p\text{th}$ quantile of the standard normal distribution. (Values of z_p that correspond to values of p can be obtained from the last row of Table G-1 in Appendix G). A probability plot (see Figure 5) offers another method of estimating normal percentiles. See EPA's *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d) for guidance on constructing probability plots and estimating percentiles.

3.3 Precision and Bias

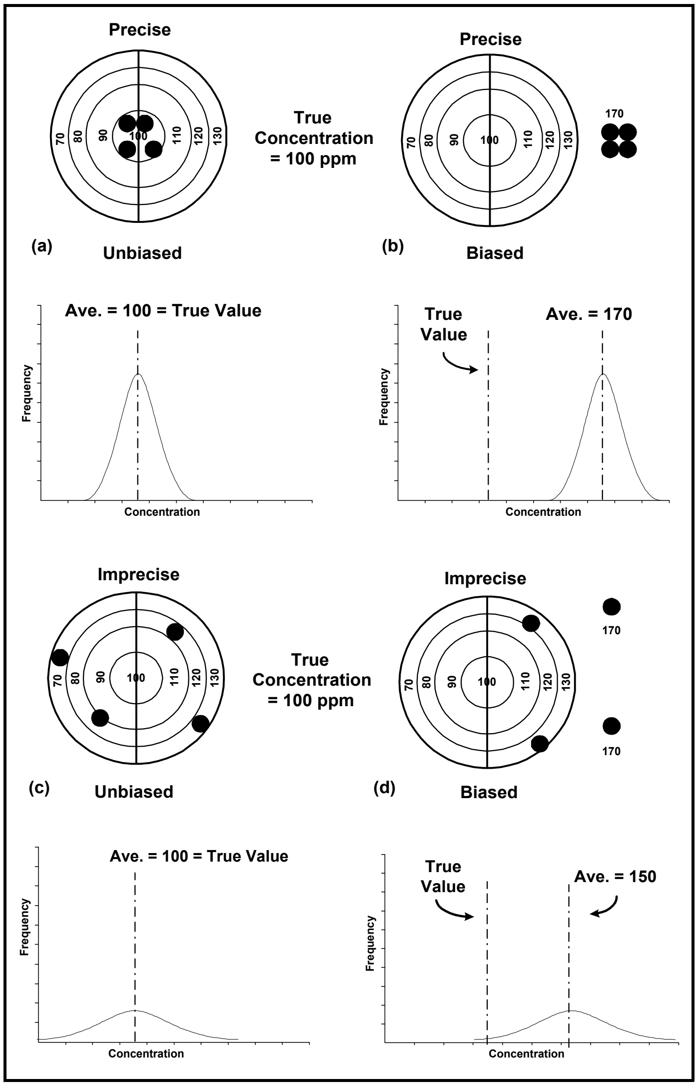

Figure 7. Shots at a target illustrate precision and bias (modified after Jessen 1978).

The representativeness of a statistical sample (that is, a set of samples) can be described in terms of **precision** and **bias**. Precision is a measurement of the *closeness of agreement* between repeated measurements. Bias is the systematic or consistent over- or underestimation of the true value (Myers 1997, USEPA 2000d).

The analogy of a target often is used to illustrate the concepts of precision and bias. In Figure 7, the center of each target represents the true (but unknown) average concentration in a batch of waste. The “shots” in targets (a) through (d) represent measurement results from samples taken to estimate the true concentration. The figure also can be used to illustrate precision and bias associated with measurement processes within a laboratory in which the same sample is analyzed multiple times (for example, four times).

Figure 7(a) indicates high precision and low bias in the sampling and analysis results. Generally, high precision and minimal bias are required when one or more chemical constituents in a solid waste are present at concentrations close to the applicable regulatory threshold or action level. Note that each of the measurements in Figure 7(a) is in close agreement with the true value. These measurements can be described as having high **accuracy**.

If the sampling and measurement process is very precise but suffers from bias (such as use of an incorrect sampling procedure or contamination of an analytical instrument), the situation could be as pictured in Figure 7(b) in which the repeated measurements are close to one another but not close to the true value. In fact, the data express a significant 70 percent bias that might go undetected if the true value is not known.

The opposite situation is depicted in Figure 7(c), where the data show low precision (that is, high dispersion around the mean) but are unbiased because the samples lack any systematic error and the average of the measurements reflects the true average concentration. Precision in sampling can be improved by increasing the number of samples, increasing the volume (mass) of each sample, or by employing a composite sampling strategies. Note, however, that relatively imprecise results can be tolerated if the contaminants of concern occur at levels either far below or far above their applicable thresholds.

Figure 7(d) depicts the situation where the sampling and analytical process suffers from both imprecision and bias. In both Figures 7(b) and (d), the bias will result in an incorrect estimate of the true concentration, even if innumerable samples are collected and analyzed to control the impact of imprecision (i.e., bias will not “cancel out” with increasing numbers of samples).

There are several types and causes of bias, including sampling bias, analytical bias, and statistical bias:

> **Sampling Bias:** There are three potential sources of sampling bias: (1) Bias can be introduced in the field and the laboratory through the improper selection and use of devices for sampling and subsampling. Bias related to sampling tools can be minimized by ensuring all of the material of interest for the study is accessible by the sampling tool. (2) Bias can be introduced through improper design of the sampling plan. Improper sampling design can cause parts of the population of interest to be over- or under-sampled, thereby causing the estimated values to be systematically shifted away from the true values. Bias related to sampling design can be minimized by ensuring the sampling protocol is impartial so there is an equal chance for each part of the waste to be included in the sample over both the spatial and temporal boundaries defined for the study. (3) Bias can be introduced in sampling due to the loss or addition of contaminants during sampling and sample handling. This bias can be controlled using sampling devices made of materials that do not sorb or leach constituents of concern, and by use of careful decontamination and sample handling procedures. For example, agitation or homogenization of samples can cause a loss of volatile constituents, thereby indicating a concentration of volatiles lower than the true value. Proper decontamination of sampling equipment between sample locations or the use of disposable devices, and the use of appropriate sample containers and preservatives also can control bias in field sampling.
>
> **Analytical Bias:** Analytical (or measurement) bias is a systematic error caused by instrument contamination, calibration drift, or by numerous other causes, such as extraction inefficiency by the solvent, matrix effect, and losses during shipping and handling.
>
> **Statistical Bias:** After the sample data have been obtained, statistics are used to estimate population parameters using the sample data. Statistical bias can occur in two situations: (1) when the assumptions made about the sampling distribution are not consistent with the underlying population distribution, or (2) when the statistical estimator itself is biased.

Returning to Figure 7, note that each target has an associated frequency distribution curve. Frequency curves are made by plotting a concentration value versus the frequency of occurrence of that concentration. The curves show that as precision decreases (i.e., the variance σ^2 increases), the curve flattens out and an increasing number of measurements are found further away from the average (figures c and d). More precise measurements result in steeper curves (figures a and b) with the majority of measurements relatively closer to the average value in normally distributed data. The greater the bias (figures b and d) the further the average of the measurements is shifted away from the true value. The smaller the bias (figures a and c) the closer the average of the samples is to the true average.

Representative samples are obtained by controlling (at acceptable levels) random variability (σ^2) and systematic error (or bias) in sampling and analysis. Quality control procedures and samples are used to estimate the precision and bias of sampling and analytical results.

3.4 Using Sample Analysis Results to Classify a Waste or to Determine Its Status Under RCRA

If samples are used to classify a waste or determine its regulatory status, then the sampling approach (including the number and type of samples) must meet the requirements specified by the regulations. Regardless of whether or not the regulations specify sampling requirements or the use of a statistical test, the Agency encourages waste handlers to use a systematic planning process such as the DQO Process to set objectives for the type, quantity, and quality of data needed to ensure with some known level of assurance that the regulatory standards are achieved.

After consideration of the objectives identified in the planning process, careful implementation of the sampling plan, and review of the analytical results, you can use the sample analysis results to classify a waste or make other decisions regarding the status of the waste under RCRA. The approach you select to obtain and evaluate the results will be highly dependent on the regulatory requirements (see Section 2 and Appendix B) and the data quality objectives (see Section 4 and Section 5).

The following sections provide a conceptual overview of how you can use sample analysis results to classify a waste or determine its status under RCRA. Guidance is provided on the following topics:

- Using an *average* to measure compliance with a fixed standard (Section 3.4.1)
- Using the *maximum* sample analysis result or an upper *percentile* to measure compliance with a fixed standard (Section 3.4.2)

There are other approaches you might use to evaluate sample analysis results, including tests that compare two populations, such as “downgradient” to “background” (see Section 3.4.3), and analysis of spatial patterns of contamination (see Section 3.4.4).

Detailed statistical guidance, including the necessary statistical equations, is provided in Section 8.2 and Appendix F.

3.4.1 Using an Average To Determine Whether a Waste or Media Meets the Applicable Standard

The arithmetic average (or mean) is a common parameter used to determine whether the concentration of a constituent in a waste or media is below a fixed standard. The mean often is used in cases in which a long-term (chronic) exposure scenario is assumed (USEPA 1992c) or where some average condition is of interest.

Because of the uncertainty associated with estimating the true mean concentration, a **confidence interval on the mean** is used to define the upper and lower limits that bracket the true mean with a known level of confidence. If the **upper confidence limit** (UCL) on the mean is less than the fixed standard, then we can conclude the true average is below the standard with a known amount of confidence. As an alternative to using a statistical interval to draw conclusions from the data, you could use hypothesis testing as described in EPA's *Guidance for the Data Quality Objectives Process, EPA QA/G-4* (USEPA 2000b) *and Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d).

Confidence intervals are calculated using the sample analysis results. Figure 8 shows what is expected to happen when ten different sets of samples are drawn from the same waste and a confidence interval for the mean is calculated for each set of samples. The true (but unknown) mean (μ) – shown as a vertical line – does not change, but the positions of the *sample means* ($\bar{x}$) and confidence intervals (shown as the horizontal lines) do change. For most of the sampling events, the confidence interval contains the true mean, but sometimes it does not. In this particular example, we expect 8 out of 10 intervals to contain the true mean, so we call this an "80-percent confidence interval on the mean." In practice, you only have one set of data from one sampling event, not ten. Note that an equal degree of uncertainty is associated with the parameter of interest being located outside each of the two interval endpoints. Consequently, the confidence interval employed in this example is, for all practical purposes, a 90-percent interval. We will refer to this as a "one-sided 90-percent confidence limit on the mean." Of course, other levels of confidence could be used, such as a 95-percent confidence limit.

The *width* of the confidence interval (defined by the upper and lower confidence limits) is an indicator of the precision of the estimate of the parameter of interest. Generally, one can improve precision (i.e., reduce the standard error, $s/\sqrt{n}$) by taking more samples, increasing the physical size of each

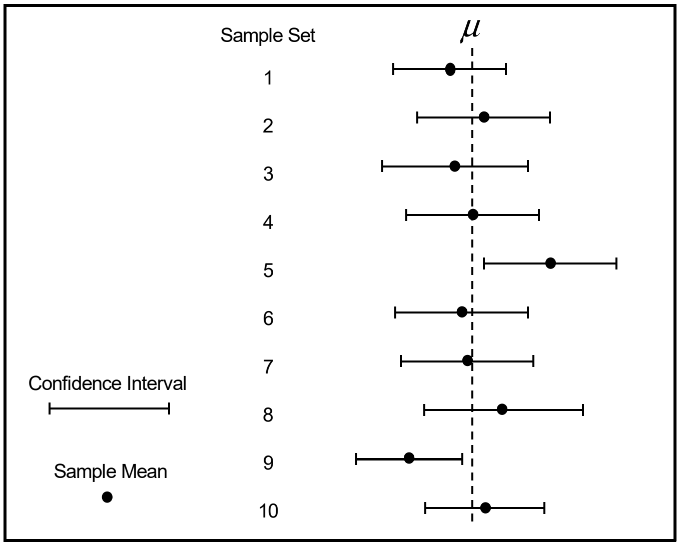

Figure 8. 80-percent confidence intervals calculated from 10 equal-sized sets of samples drawn at random from the same waste stream

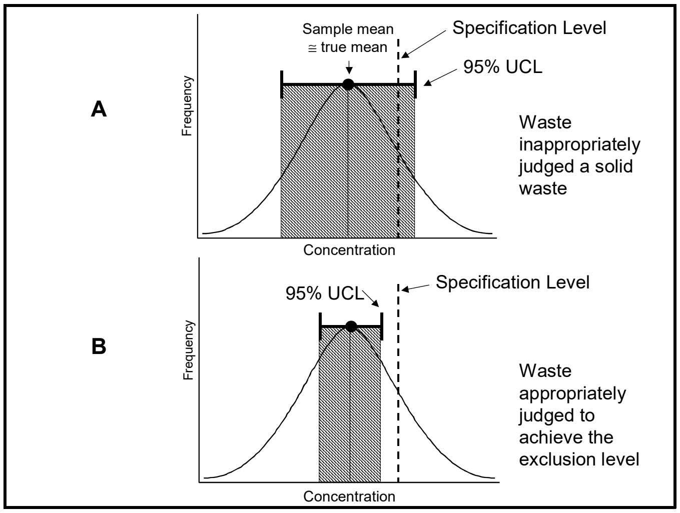

Figure 9. Example of how sampling precision could impact a waste exclusion demonstration under 40 CFR 261.38. Due to imprecision (A), the waste is inappropriately judged a solid waste. With more precise results (B), the entire confidence interval lies below the specification level, and the waste is appropriately judged eligible for the comparable fuels exclusion.

sample (i.e., increasing the sample support), and by minimizing random variability introduced in the sampling and measurement processes.

For example, Figure 9 shows how sampling precision can affect the ability to claim an exclusion from the definition of solid waste under the comparable fuels regulations at 40 CFR 261.38. In Figure 9 “A,” the sampling results are unbiased, but they are not sufficiently precise. In fact, the imprecision causes the confidence intervals to “straddle” the specification level; thus, there is not *statistically significant* evidence that the mean is below the standard. Imprecision can be caused by the heterogeneity of the material sampled, by random errors in the field and laboratory, and by too few samples. In Figure 9 “B,” the results also are unbiased, but significant improvement in precision is observed (e.g., because more or larger samples were analyzed and errors were kept within acceptable limits), allowing us to conclude that the mean is indeed below the specification level.

Detailed guidance on the calculation of confidence limits for the mean can be found in Appendix F of this document.

3.4.2 Using a Proportion or Percentile To Determine Whether a Waste or Media Meets an Applicable Standard

Under RCRA, some regulatory thresholds are defined as concentration values that cannot be exceeded (e.g., the RCRA LDR program concentration-based treatment standards for hazardous waste specified at § 268.40 and § 268.48), concentration values that cannot be equaled or exceeded (e.g., the Toxicity Characteristic maximum concentration levels specified at § 261.24), or waste properties that cannot be exhibited (e.g., ignitability per § 261.21, corrosivity per § 261.22, or reactivity per § 261.23) for the waste to comply with the regulatory standard.

To demonstrate compliance with such a standard using sampling, it is necessary to consider the waste or site (whose boundaries are defined as a decision unit) as a population of discrete sample units (of a defined size, shape, and orientation). Ideally, none of these sample units may exceed the standard or exhibit the properties of concern for the waste or site to be in compliance with the standard. However, since it is not possible to know the status of all portions of a waste or site, samples must be used to infer - using statistical methods - what proportion or percentage of the waste complies, or does not comply, with the standard. Generally, few if any samples drawn from the population of interest may exceed the regulatory standard or exhibit the property of concern to demonstrate with reasonable confidence that a high proportion or percentage of the population complies with the standard.

Two simple methods for measuring whether a specified proportion or percentile of a waste or media meets an applicable standard are described in the following sections:

- Using an upper confidence limit on a percentile to classify a waste or media (Section 3.4.2.1), and
- Using a simple exceedance rule method to classify a waste or media (Section 3.4.2.2).

3.4.2.1 *Using a Confidence Limit on a Percentile to Classify a Waste or Media*

A percentile is a population parameter. We cannot know the true value of that parameter, but we can *estimate* it from a statistical sample drawn from the population by using a **confidence interval for a percentile**. If the **upper confidence limit (UCL) on the upper percentile** is below the fixed standard, then there is statistically significant evidence that the specified proportion of the waste or media attains the standard (see Figure 10). If the UCL on the upper percentile exceeds the standard (but all sample analysis results are below the standard), then the waste or media still could be judged in compliance with the standard; however, you would not have the specified degree of confidence that the specified proportion of the waste or media complies with the standard (see also the exceedance rule method, Section 3.4.2.2).

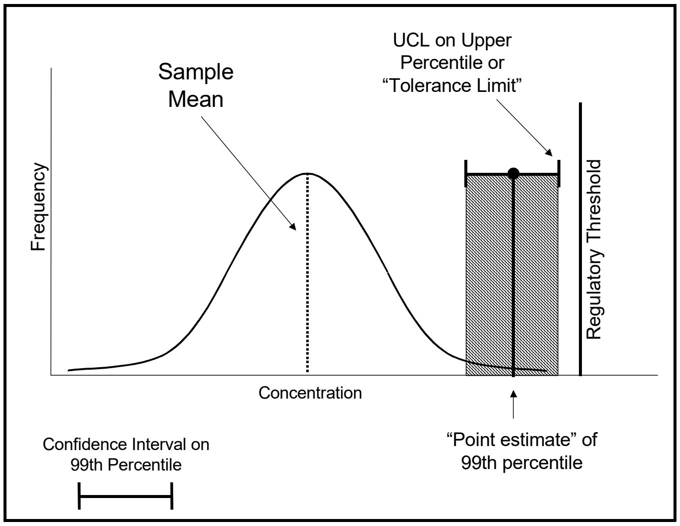

Figure 10. For a high percentile (e.g., the 99th percentile) to be less than an applicable standard, the mean concentration must be well below the standard.

Detailed guidance on the calculation of confidence limits for percentiles can be found in Section 8.2 and Appendix F of this document. Methods also are given in Conover (1999), Gilbert (1987, page 136), Hahn and Meeker (1991), and USEPA (1989a). A possible alternative to using a confidence limit on a percentile is the use of the "one-sample test for proportions" (see Section 3.2.2.1 of USEPA 2000d).

3.4.2.2 *Using a Simple Exceedance Rule Method To Classify a Waste*

One of the most straightforward methods for determining whether a given proportion or percentage of a waste (that is, all possible samples of a given sample support) complies with an applicable standard is to use a simple exceedance rule. To apply the method, simply obtain a number of samples and require that zero or few sample analysis results be allowed to exceed the applicable standard or possess the property (or "attribute") of interest. The method (also known as "inspection by attributes") is from a class of methods known as acceptance sampling plans (Schilling 1982, ASQ 1988 and 1993, and DoD 1996). One simple form of the exceedance rule, sometimes used by regulatory enforcement agencies, specifies zero exceedances in a set of samples. This method can be used to classify a waste (i.e., determine if it exhibits the characteristics of ignitability, corrosivity, reactivity[1], or toxicity) or to determine its status under RCRA (that is, to determine if the waste is prohibited from land disposal or if it attains an LDR treatment standard).

The method is attractive because it is simple (e.g., because sample analysis results are

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[1] EPA uses a narrative criteria to define most reactive wastes, and waste handlers should use their knowledge to determine if a waste is sufficiently reactive to be regulated.

recorded as either “pass” or “fail” and statistical tables can be used instead of equations), it does not require an assumption about the form of the underlying distribution, and it can be used when a large proportion of the data are reported as less than a quantitation limit. Furthermore, the method has statistical properties that allow the waste handler to have a known level of confidence that at least a given proportion of the waste complies with the standard. One potential drawback of using an exceedance rule is that with a small number of samples, you might not be able to conclude with high confidence that a high proportion of the waste complies with the applicable standard (unless you have sufficient knowledge of the waste indicating there is little variability in concentrations or properties). That is, with a small number of samples, there is little statistical power: an unacceptably large proportion of the waste or site could exceed the standard or exhibit the property even though no such exceedances or properties were observed in the samples. Increasing the number of samples will improve the statistical performance.

As a practical matter, it is suggested that you scale the statistical performance and acceptance requirements (and thus, the number of samples) to the size of the lot or batch of waste of interest. For example, when large and/or very heterogeneous volumes of waste are the subject of the study, decision-makers may require high confidence that a high proportion of the waste meets the applicable standard. A relatively large number of samples will be required to satisfy these criteria if the exceedance rule is used. On the other hand, decision-makers may choose to relax the statistical performance criteria when characterizing a small volume of waste (or a very homogeneous waste) and thus fewer samples would be needed.

Detailed guidance on the use of an exceedance rule is provided in Section 5.5.2 and in Appendix F, Section F.3.2, of this document. The exceedance rule method also is described in *Methods for Evaluating the Attainment of Cleanup Standards. Volume 1: Soils and Solid Media* (USEPA 1989a, Section 7.4).

3.4.3 Comparing Two Populations

Some environmental studies do not involve testing compliance against a fixed standard but require comparison of two separate data. This type of analysis is common for detecting releases to ground water at waste management units such as landfills and surface impoundments, detecting releases to soil and the unsaturated zone at land treatment units, or determining if site contamination is distinguishable from natural background concentrations. In these situations, the operator must compare “on site” or “downgradient” concentrations to “background.”

For example, at a new land-based waste management unit (such as a new landfill), we expect the concentrations in a set of samples from downgradient locations to be similar to a set of samples from background locations. If a statistically significant change in downgradient conditions is detected, then there may be evidence of a release to the environment. Statistical methods called *two-sample tests* can be used to make such comparisons (they are called two-sample tests because two *sets* of samples are used). A two-sample test also could be used to measure changes in constituent concentrations in a waste or soil “before” treatment and “after” treatment to assess the effectiveness of the treatment process (see USEPA 2002a).

For detailed guidance on the use of two-sample tests, see EPA’s G-9 guidance (USEPA 2000d) and EPA’s guidance on the statistical analysis of ground-water monitoring data (USEPA 1989b and 1992b).

Note that detecting a release to the environment may not necessarily involve use of a statistical test and may not even involve sampling. For example, observation of a broken dike at a surface impoundment may indicate that a release has occurred.

3.4.4 Estimating Spatial Patterns

Under some circumstances, a site investigator may wish to determine the location of a contaminant in the environment as well as its concentration. Knowledge of spatial trends or patterns may be of particular value when conducting risk assessments or locating areas for clean-up or removal under the RCRA Corrective Action program. Estimation of spatial patterns is best addressed by geostatistics or other spatial data analysis methods.

Geostatistical models are based on the notion that elements of the population that are close together in space and/or time exhibit an identifiable relationship or positive correlation with one another. Geostatistical techniques attempt to recognize and describe the pattern of spatial dependence and then account for this pattern when generating statistical estimates. On the other hand, “classical” methods assume that members of a population are not correlated (USEPA 1997a).

While a full treatment of spatial analysis and geostatistics is beyond the scope of this guidance, certain techniques recommended in the guidance require consideration of spatial differences. For example, you may need to consider whether there are any spatial correlations in a waste or site when selecting a sampling design. There are some relatively simple graphical techniques that can be used to explore possible spatial patterns or relationships in data. For example, posting plots or spatial contour maps can be generated manually or via software (e.g., see EPA’s Geo-EAS software described in Appendix H). Interested readers can find a more comprehensive explanation of spatial statistics in texts such as Myers (1997), Isaaks and Srivastava (1989), Journel (1988), USEPA (1991a, 1997a), or consult a professional environmental statistician or geostatistician.

4 PLANNING YOUR PROJECT USING THE DQO PROCESS

To be successful, a waste-testing program must yield data of the type and quality necessary to achieve the particular purpose of the program. This is accomplished through correct, focused, and well-documented sampling, testing, and data evaluation activities. In each case, a clear understanding of the program objectives and thorough planning of the effort are essential for a successful, cost-effective waste-testing program.

Each program design is unique because of the many possible variables in waste sampling and analysis such as regulatory requirements, waste and facility-specific characteristics, and objectives for the type and quantity of data to be provided. Nonetheless, a systematic planning process such as the Data Quality Objectives (DQO) Process, which takes these variables into account, can be used to guide planning efforts. EPA recommends using the DQO Process when data are being used to select between two opposing conditions, such as determining compliance with a standard.

The DQO Process yields qualitative and quantitative statements that:

- Clarify the study objectives
- Define the type, quantity, and quality of required data
- Determine the most appropriate conditions from which to collect the samples
- Specify the amount of uncertainty you are willing to accept in the results
- Specify how the data will be used to test a decision rule.

The outputs of the DQO Process are used to define the quality control requirements for sampling, analysis, and data assessment. These requirements are then incorporated into a QAPP, WAP, or other similar planning document.

The DQO Process comprises seven planning steps depicted in Figure 11. The figure shows one of the most important features of the process: its iterative nature. You don't have to "get it right the first time." You can use existing information to establish DQOs. If the initial design is not feasible, then you can iterate through one or more of the earlier planning steps to identify a sampling design that will meet the budget and generate data that are adequate for the decision. This way, you can evaluate sampling designs and related costs *in advance* before significant time and resources are expended to collect and analyze samples.

In a practical sense, the DQO Process offers a structured approach to "begin with the end in

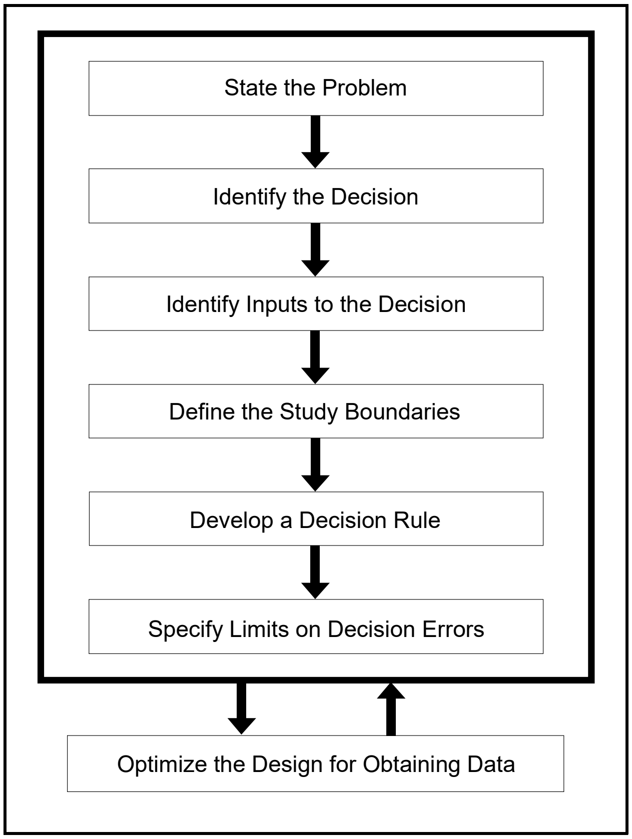

Figure 11. The seven steps of the DQO Process (from USEPA 2000b)

mind." It is a framework for asking the right questions and using the answers to develop and implement a cost-effective plan for data collection. The DQO Process does not necessarily proceed in a linear fashion or involve rigid procedures; rather, it is a thought process to enable you to get useful information in a cost-effective manner.

Systematic Planning and the DQO Process: EPA References and Software

Guidance for the Data Quality Objectives Process, EPA QA/G-4, August 2000, EPA/600/R-96/055. Provides guidance on how to perform the DQO Process.

Data Quality Objectives Decision Error Feasibility Trials Software (DEFT) - User's Guide, EPA QA/G-4D, September 2001, EPA/240/B-01/007 (User's Guide and Software). PC-based software for determining the feasibility of data quality objectives defined using the DQO Process.

Guidance for the Data Quality Objectives Process for Hazardous Waste Sites, EPA QA/G-4HW, January 2000, EPA/600/R-00/007. Provides guidance on applying the DQO Process to hazardous waste site investigations.

Failure to establish DQOs before implementing field and laboratory activities can cause difficulties in the form of inefficiencies, increased or unnecessary costs, or the generation of unusable data. For example, if the limit of quantitation for sample analysis is greater than the Action Level, then the data will not be useable for its intended purpose; or, if you do not collect enough samples, then you may not be able to draw conclusions with the desired level of confidence.

When properly used, the DQO Process:

- Provides a good way to document the key activities and decisions necessary to address the problem and to communicate the approach to others.
- Involves key decision makers, other data users, and technical experts in the planning process before data collection begins which helps lead to a consensus prior to beginning the project and makes it easier to change plans when circumstances warrant because involved parties share common understandings, goals, and objectives.
- Develops a consensus approach to limiting decision errors that strikes a balance between the cost of an incorrect decision and the cost of reducing or eliminating the possible mistake.
- Saves money by greatly reducing the tendency to collect unneeded data by encouraging the decision makers to focus on data that support only the decision(s) necessary to solve the problem(s). When used with a broader perspective in mind, however, the DQO Process may help identify opportunities to consolidate multiple tasks and improve the efficiency of the data collection effort.[1]

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[1] In some cases, it might be appropriate and cost-effective to collect data beyond that required to support a near-term decision. For example, if a drill rig is mobilized to collect deep soil samples to determine the need for remediation, it would be cost-effective to also collect relatively low-cost data (such as geotechnical parameters, total organic carbon, moisture content, etc.) needed by engineers to design the remedy. Otherwise, unnecessary costs might be incurred to remobilize a drill rig to obtain data that could have been obtained in the initial effort.

The remainder of this section addresses how the DQO Process can be applied to RCRA waste-characterization studies. While the discussion is based on EPA's *G-4* guidance (USEPA 2000b), some steps have been modified or simplified to allow for flexibility in their use. Keep in mind that not all projects or decisions (such as a hazardous waste determination) will require the full level of activities described in this section, but the logic applies nonetheless. In fact, EPA encourages use of a "graded approach" to quality assurance. A graded approach bases the level of management and QA/QC activities on the intended use of the results and the degree of confidence needed in their quality (USEPA 2001f).

4.1 Step 1: State the Problem

Before developing a data gathering program, the first step is to state the problem or determine what question or questions are to be answered by the study. For many waste characterization or monitoring programs the questions are spelled out in the applicable regulations; however, in some cases, determining the actual problem or question to be answered may be more complex. As part of this step, perform the four activities described in the following sections.

DQO Step 1: State the Problem

Purpose
To define the problem so that the focus of the study will be unambiguous.

Activities
- Identify members of the planning team.
- Identify the primary decision maker(s).
- Develop a concise description of the problem.
- Determine resources – budget, personnel, and schedule.

4.1.1 Identify Members of the Planning Team

The planning team comprises personnel representing all phases of the project and may include stakeholders, decision makers, technical project managers, samplers, chemists, process engineers, QA/QC managers, statisticians, risk assessors, community leaders, grass roots organizations, and other data users.

4.1.2 Identify the Primary Decision Maker

Identify the primary decision maker(s) or state the process by which the decision will be made (for example, by consensus).

4.1.3 Develop a Concise Description of the Problem

Develop a problem description to provide background information on the fundamental issue to be addressed by the study. For RCRA waste-related studies, the "problem" could involve determining one of the following: (1) if a solid waste should be classified as a hazardous waste, (2) if a hazardous waste is prohibited from land disposal, (3) if a treated hazardous waste attains the applicable treatment standard, (4) if a cleanup goal has been attained, or (5) if hazardous constituents have migrated from a waste management unit.

Summarize existing information into a "conceptual model" or conceptual site model (CSM) including previous sampling information, preliminary estimates of summary statistics such as the mean and standard deviation, process descriptions and materials used, and any spatial and temporal boundaries of the waste or study area that can be defined. A CSM is a three-dimensional “picture” of site conditions at a discrete point in time (a snapshot) that conveys what is known or suspected about the facility, releases, release mechanisms, contaminant fate and transport, exposure pathways, potential receptors, and risks. The CSM does not have to be based on a mathematical or computer model, although these tools often help to visualize current information and predict future conditions. The CSM should be documented by written descriptions of site conditions and supported by maps, cross sections, analytical data, site diagrams that illustrate actual or potential receptors, and any other descriptive, graphical, or tabular illustrations necessary to present site conditions.

4.1.4 Specify Available Resources and Relevant Deadlines

Identify available financial and human resources, identify deadlines established by permits or regulations, and establish a schedule. Allow time for developing acceptance and performance criteria, preparing planning documents (such as a QAPP, sampling plan, and/or WAP), collecting and analyzing samples, and interpreting and reporting data.

4.2 Step 2: Identify the Decision

The goal of this step is to define the questions that the study will attempt to answer and identify what actions may be taken based on the outcome of the study. As part of this step, perform the four activities described in the following sections.

DQO Step 2: Identify the Decision

Purpose
To define what specific decisions need to be made or what questions need to be answered.

Activities
- Identify the principal study question.
- Define the alternative actions that could result from resolution of the principal study question.
- Develop a decision statement.
- Organize multiple decisions.

4.2.1 Identify the Principal Study Question

Based on the problem identified in Step 1, identify the study question and state it as specifically as possible. This is an important step because the manner in which you frame the study question can influence whether sampling is even appropriate, and if so, how you will evaluate the results. Here are some examples of study questions that might be posed in a RCRA-related waste study:

- Does the filter cake from the filter press exhibit the TC at its point of generation?
- Does the treated waste meet the universal treatment standard (UTS) for land disposal under 40 CFR 268?
- Has the soil remediation at the SWMU attained the cleanup goal for benzene?
- Have hazardous constituents migrated from the land treatment unit to the underlying soil at concentrations significantly greater than background concentrations?
- Are radioactive and hazardous wastes colocated, producing a mixed waste management scenario?

Before conducting a waste-sampling and testing program to comply with RCRA, you should review the specific regulatory requirements in 40 CFR in detail and consult with staff from your EPA region or the representative from your State (if your State is authorized to implement the regulation).

4.2.2 Define the Alternative Actions That Could Result from Resolution of the Principal Study Question

Generally, two courses of action will result from the outcome of the study. One that involves action, such as deciding to classify a solid waste as a hazardous waste, and one that requires an alternative action, such as deciding to classify a solid waste as a nonhazardous solid waste.[2]

4.2.3 Develop a Decision Statement

In performing this activity, simply combine the principal study question and the alternative actions into a "decision statement." For example, you may wish to determine whether a waste exhibits a hazardous waste characteristic. The decision statement should be in writing (for example, in the QAPP) and agreed upon by the planning team. This approach will help avoid misunderstandings later in the process.

4.2.4 Organize Multiple Decisions

If several separate decisions statements must be defined to address the problem, then you should list them and identify the sequence in which they should be resolved. For example, if you classify a solid waste as a nonhazardous waste, then you will need to make a waste management decision. Options might include land disposal (e.g., in an industrial landfill or a municipal solid waste landfill), recycling, or some other use. You might find it helpful to document the decision resolution sequence and relationships in a diagram or flowchart.

4.3 Step 3: Identify Inputs to the Decision

In most cases, it will be necessary to collect data or new information to resolve the decision statement. To identify the type and source of this information, perform the activities outlined in the following four sections.

DQO Step 3: Identify Inputs to the Decision

Purpose
To identify data or other information required to resolve the decision statement.

Activities
- Identify the information required to resolve the decision statement.
- Determine the sources of information.
- Identify information needed to establish the Action Level.
- Identify sampling and analysis methods that can meet the data requirements.

4.3.1 Identify the Information Required

For RCRA-related waste studies, information requirements typically will

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[2] Testing alone might not be sufficient to determine if a solid waste is hazardous waste. You also should apply knowledge of the waste generation process to determine if the solid waste is a hazardous waste under 40 CFR 261.

include samples to be collected, variables to be measured (such as total concentrations, TCLP results, or results of tests for other characteristics, such as reactivity, ignitability, and corrosivity), the units of measure (such as mg/L), the form of the data (such as on a dry weight basis), and waste generation or process knowledge.

4.3.2 Determine the Sources of Information

Identify and list the sources of information needed and qualitatively evaluate the usefulness of the data. Existing information, such as analytical data, can be very valuable. It can help you calculate the appropriate number of new samples needed (if any) and reduce the need to collect new data (see also Section 5.4).

4.3.3 Identify Information Needed To Establish the Action Level

The Action Level is the threshold value that provides the criterion for choosing between alternative actions. Under RCRA, there are several types of Action Levels.

The first type of Action Level is a fixed standard or regulatory threshold (RT) usually specified as a *concentration* of a hazardous constituent (e.g., in mg/L). Examples of regulatory thresholds that are Action Levels in the RCRA regulations include the TC Regulatory Levels at 40 CFR 261.24 and the Land Disposal Restrictions (LDR) numeric treatment standards at 40 CFR 268.40.

Another criterion for choosing between alternative actions is defined by the *property* of a waste. Three such properties are defined in the RCRA regulations: ignitability (§ 261.21), corrosivity (§ 261.22), and reactivity (§ 261.23). The results of test methods used to determine if a waste is ignitable, corrosive, or reactive are interpreted as either “pass” or “fail” -- i.e., the waste either has the property or it does not. Note that a concentration measurement, such as a TCLP sample analysis result, also can be interpreted as either “pass” or “fail” based on whether the value is less than or greater than a specified threshold.

A third criterion for choosing between alternative actions involves making a comparison between constituent concentrations at different times or locations to determine if there has been a change in process or environmental conditions over time. In these situations, you need to determine if the two sets of data are different relative to each other rather than checking for compliance with a fixed standard.

Finally, an Action Level can represent a proportion of the population having (or not having) some characteristic. For example, while it might be desirable to have all portions of a waste or site comply with a standard, it would be more practical to test whether some high proportion (e.g., 0.95) of units of a given size, shape, and orientation comply with the standard. In such a case, the Action Level could be set at 0.95.

For more information on identifying the Action Level, see Section 2 (RCRA regulatory drivers for waste sampling and testing), the RCRA regulations in 40 CFR, ASTM Standard D 6250 (*Standard Practice for Derivation of Decision Point and Confidence Limit for Statistical Testing of Mean Concentration in Waste Management Decisions*), or consult with your State or EPA Regional staff.

4.3.4 Confirm That Sampling and Analytical Methods Exist That Can Provide the Required Environmental Measurements

Identify and evaluate candidate sampling and analytical methods capable of yielding the required environmental measurements. You will need to revisit this step during Step 7 of the DQO Process ("Optimize the Design for Obtaining the Data") after the quantity and quality of the necessary data are fully defined. In evaluating sampling methods, consider the medium to be sampled and analyzed, the location of the sampling points, and the size, shape and orientation of each sample (see also Section 6, "Controlling Variability and Bias in Sampling" and Section 7, "Implementation: Selecting Equipment and Conducting Sampling").

In evaluating analytical methods, choose the appropriate candidate methods for sample analyses based on the sample matrix and the analytes to be determined.

Guidance on the selection of analytical methods can be found in Chapter Two of SW-846 ("Choosing the Correct Procedure"). Up-to-date information on analytical methods can be found at SW-846 "On Line" at http://www.epa.gov/epaoswer/hazwaste/test/main.htm.

4.4 Step 4: Define the Study Boundaries

DQO Step 4: Define the Study Boundaries

Purpose
To define the spatial and temporal boundaries that are covered by the decision statement.

Activities
- Define the target population of interest.
- Define the "sample support"
- Define the spatial boundaries that clarify what the data must represent.
- Define the time frame for collecting data and making the decision.
- Identify any practical constraints on data collection.
- Determine the smallest subpopulation, area, volume, or time for which separate decisions must be made.

In this step of the DQO Process, you should identify the target population of interest and specify the spatial and temporal features of that population that are pertinent for decision making.

To define the study boundaries, perform the activities described in the following five sections.

4.4.1 Define the Target Population of Interest

It is important for you to clearly define the target population to be sampled. Ideally, the target population coincides with the population to be sampled (Cochran 1977) – that is, the target population should represent the total collection of all possible sampling units that could be drawn. Note that the "units" that make up the population are defined operationally based on their size, shape, orientation, and handling (i.e., the "sample support").[3] The sampling unit definition must be considered when defining the target population because any changes in the definition can affect the population characteristics. See Section 6.3.1 for guidance on establishing the appropriate size (mass) of a sample, and see Section 6.3.2 for guidance on

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[3] The physical size (expressed as mass or volume), shape, and orientation of a sample is known as the *sample support*. Sample support plays an important role in characterizing waste or environmental media and in minimizing variability caused by the sampling process. The concept of *support* is discussed in greater detail in Section 6.2.3.

establishing the appropriate shape and orientation of sample.

Define the target population in terms of sampling units, the decision-making volume, and the location of that volume.

Sampling at the **point of generation** is *required* by regulation when determining the regulatory status of a waste. See 55 FR 11804, March 29, 1990, and 55 FR 22652, June 1, 1990.

4.4.2 Define the Spatial Boundaries

If sampling at the point of waste generation (i.e., *before* the waste is placed in a container or transport unit), then the sampling problem could involve collecting samples of a moving stream of material, such as from a conveyor, discharge pipe, or as poured into a container or tank. If so, then physical features such as the width of the flow or discharge and the rate of flow or discharge will be of interest for defining the spatial boundary of the problem.

If the sampling problem involves collecting samples from a waste storage unit or transport container, then the spatial boundaries can be defined by some physical feature, such as volume, length, width, height, etc. The spatial boundaries of most waste storage units or containers can be defined easily. Examples of these units follow:

- Container such as a drum or a roll-off box
- Tank
- Surface Impoundment
- Staging Pile
- Waste Pile
- Containment Building.

In other cases, the spatial boundary could be one or more geographic areas, such as areas representing “background” and “downgradient” conditions at a land treatment unit. Another example is a SWMU area that has been subject to remediation where the objective is verify that the cleanup goal has been achieved over a specified area or volume at the SWMU. If the study requires characterization of subsurface soils and ground water, then consult other guidance (for example, see USEPA 1989a, 1989b, 1991d, 1992a, 1993c, and 1996b).

To help the planning team visualize the boundary, it may be helpful to prepare a drawing, map, or other graphical image of the spatial boundaries, including a scale and orientation (e.g., a north arrow). If appropriate and consistent with the intended use of the information, maps also should identify relevant surface features (such as buildings, structures, surface water bodies, topography, etc.) and known subsurface features (pipes, utilities, wells, etc.).

If samples of waste will be taken at the point of generation (e.g., when the waste becomes a solid waste), the location of that point should be defined in this step of the DQO Process.

4.4.3 Define the Temporal Boundary of the Problem

A temporal boundary could be defined by a permit or regulation (such as the waste generated per day) or operationally (such as the waste generated per “batch” or truck load). You should determine the time frame to which the decision applies and when to collect the data. In some cases, different time intervals might be established to represent different populations (e.g., in the case where there is a process change over time that affects the character of the waste).

Waste characteristics or chemistry, such as the presence of volatile constituents, also could influence the time frame within which samples are collected. For example, volatilization could occur over time.

4.4.4 Identify Any Practical Constraints on Data Collection

Identify any constraints or obstacles that could potentially interfere with the full implementation of the data collection design. Examples of practical constraints include physical access to a sampling location, unfavorable weather conditions, worker health and safety concerns, limitations of available sampling devices, and availability of the waste (e.g., as might be the case for wastes generated from batch processes) that could affect the schedule or timing of sample collection.

4.4.5 Define the Scale of Decision Making

Define the smallest, most appropriate subsets of the population (sub-populations), waste, or media to be characterized based on spatial or temporal boundaries. The boundaries will define the unit of waste or media about which a decision will be made. The unit is known as the **decision unit**.

When defining the decision unit, the consequences of making a decision error should be carefully considered. The consequences of making incorrect decisions (Step 6) are associated with the size, location, and shape of the decision unit. For example, if a decision, based on the data collected, results in a large volume of waste being classified as nonhazardous, when in fact a portion of the waste exhibits a hazardous waste characteristic (e.g., due to the presence of a “hot spot”), then the waste generator could potentially be found in violation of RCRA . To limit risk of managing hazardous waste with nonhazardous waste, the waste handler should consider dividing the waste stream into smaller decision units – such as the volume of waste that would be placed into an individual container to be shipped for disposal – and make a separate waste classification decision regarding each decision unit.

The planning team may establish decision units based on several considerations:

- **Risk** – The scale of the decision making could be defined based on an exposure scenario. For example, if the objective is to evaluate exposures via direct contact with surface soil, each decision unit could be defined based on the geographic area over which an individual is assumed to move randomly across over time. In EPA’s Superfund program, such a unit is known as an “exposure area” or EA (USEPA 1992c and 1996f). An example of an EA from EPA’s *Soil Screening Guidance: User’s Guide* (USEPA 1996f) is the top 2 centimeters of soil across a 0.5-acre area. In this example, the EA is the size of a suburban residential lot and the depth represents soil of the greatest concern for incidental ingestion of soil, dermal contact, and inhalation of fugitive dust.

  If evaluation of a decision unit or EA for the purpose of making a cleanup decision finds that cleanup is needed, then the same decision unit or EA should be used when evaluating whether the cleanup standard has been attained. Furthermore, the size, shape, and orientation (the "sample support") of the samples used to determine that cleanup was necessary should be the same for samples used to determine whether the cleanup standard is met (though this last condition is not strictly necessary when the parameter of interest is the mean).

- **Operational Considerations** – The scale of the decision unit could be defined based on operational considerations, such as the need to characterize each "batch" of waste after it has been treated or the need to characterize each drum as it is being filled at the point of waste generation. As a practical matter, the scale for the decision making often is defined by the spatial boundaries – for example as defined by a container such as a drum, roll-off box, truck load, etc. or the time required to fill the container.

- **Other** – The possibility of "hot spots" (areas of high concentration of a contaminant) may be apparent to the planning team from the history of the facility. In cases where previous knowledge (or planning team judgment) includes identification of areas that have a higher potential for contamination, a scale may be developed to specifically represent these areas.

Additional information and considerations on defining the scale of the decision making can be found in *Guidance for the Data Quality Objectives Process for Hazardous Waste Site Operations EPA QA/G-4HW* (USEPA 2000a) and *Guidance for the Data Quality Objectives Process EPA QA/G-4* (USEPA 2000b).

4.5 Step 5: Develop a Decision Rule

A statement must be developed that combines the parameter of interest and the Action Levels with the DQO outputs already developed. The combination of these three elements forms the decision rule and summarizes what attributes the decision maker wants to study and how the information will assist in solving the central problem. To develop the decision rule, perform the activities described in the following three sections:

4.5.1 Specify the Parameter of Interest

DQO Step 5: Develop a Decision Rule

Purpose

To define the parameter of interest, specify the Action Level and integrate previous DQO outputs into a single statement that describes a logical basis for choosing among alternative actions; i.e., define how the data will be used to make a decision.

Activities

- Specify the parameter of interest (mean, median, percentile).
- Specify the Action Level for the study.
- Develop a decision rule.

A statistical "parameter" is a descriptive measure of a population such as the population mean, median, or a percentile (see also Section 3.2). See Table 2.

Some of the RCRA regulations specify the parameter of interest. For example, the comparable fuels sampling and analysis requirements at 40 CFR 261.38(c)(8)(iii)(A) specify the *mean* as the parameter of interest, and the ground-water monitoring requirements at 40 CFR 264.97 specify the parameter of interest for each statistical test. Other RCRA regulations do not specify the parameter of interest, however, you can select a parameter based on what the Action Level is intended to represent. In general, if an Action Level is based on long-term average health effects, the parameter of interest could be the population mean (USEPA 1992a). If the Action Level represents a value that should never (or rarely) be exceeded, then the parameter of interest could be an upper population percentile, which can serve as a reasonable approximation of the *maximum* value.

If the objective of the study does not involve estimation of a parameter or testing a hypothesis, then specification of a parameter is not necessary.

Table 2. Population Parameters and Their Applicability to a Decision Rule

Parameter	***Definition***	***Appropriate Conditions for Use***
Mean	Average	Estimate central tendency: Comparison of middle part of population to an Action Level.
Median	Middle observation of the distribution; 50th percentile; half of data are above and below	May be preferred to estimate central tendency if the population contains many values that are less than the limit of quantitation. The median is not a good choice if more than 50% of the population is less than the limit of quantitation because a true median does not exist in this case. The median is not influenced by the extremes of the contaminant distribution.
Percentile	Specified percent of sample that is equal to or below the given value	For cases where it is necessary to demonstrate that, at most, only a small portion of a population could exceed the Action Level. Sometimes selected if the decision rule is being developed for a chemical that can cause acute health effects. Also useful when a large part of the population contains values less than the detection limit.

4.5.2 Specify the Action Level for the Study

You should specify an Action Level or concentration limit that would cause the decision maker to choose between alternative actions. Examples of Action Levels follow:

- Comparable/syngas fuel constituent specification levels specified at § 261.38
- Land disposal restrictions concentration level treatment standards at § 268.40 and § 268.48
- Risk-based cleanup levels specified in a permit as part of a corrective action
- "Pass" or "fail" thresholds for tests for ignitability, corrosivity, reactivity[4], and toxicity.

Also, be sure the detection or quantitation limits for the analytical methods identified in DQO Step 3 (Section 4.3) are below the Action Level, if possible.

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[4] EPA uses a narrative criteria to define most reactive wastes, and waste handlers should use their knowledge to determine if a waste is sufficiently reactive to be regulated.

If your objective is to compare “onsite” to “background” to determine if there is a statistically significant increase above background (as would be the case for monitoring releases from a land treatment unit under § 264.278), you will not need to specify an Action Level; rather, the Action Level is implicitly defined by the background concentration levels and the variability in the data. A summary of methods for determining background concentrations in soil can be found in USEPA 1995a. Methods for determining background concentrations in ground water can be found in USEPA 1989b and 1992b.

Finally, note that some studies will not require specification of a regulatory or risk-based Action Level. For example, if the objective may be to identify the existence of a release, samples could be obtained to verify the *presence or absence* of a spill, leak, or other discharge to the environment. Identifying a potential release also could include observation of abandoned or discarded barrels, containers, and other closed receptacles containing hazardous wastes or constituents (see 61 FR No. 85, page 19442).

4.5.3 Develop a Decision Rule

After you have completed the above activities, you can construct a decision rule by combining the selected population parameter and the Action Level with the scale of the decision making (from DQO Process Step 4) and the alternative action (from DQO Step 2). Decision rules are expressed as “if (criterion)..., then (action)....” A hypothetical example follows:

> “If the true 95th percentile of all possible 100-gram samples of the waste being placed in the 20-cubic yard container is less than 5.0 mg/L TCLP lead, then the solid waste will be classified as nonhazardous waste. Otherwise, the solid waste will be classified as a RCRA hazardous waste.”

Note that this is a functional decision rule based on an ideal condition (i.e., knowledge of the true concentration that equals the 95th percentile of all possible sample analysis results). It also identifies the boundary of the study by specifying the sample unit (100-gram samples in accordance with the TCLP) and the size of the decision unit. It does *not*, however, specify the amount of uncertainty the decision maker is willing to accept in the estimate. You specify that in the next step.

4.6 Step 6: Specify Limits on Decision Errors

Because samples represent only a portion of the population, the information available to make decisions will be incomplete; hence, *decision errors* sometimes will be made. Decision errors occur because decisions are made using *estimates* of the parameter of interest, rather than the true (and unknown) value. In fact, if you repeatedly sampled and analyzed a waste over and over in an identical manner the results would be a little different each time (see Figure 8 in Section 3). This variability

Step 6: Specify Limits on Decision Errors

Purpose
To specify the decision maker’s tolerable limits on decision error.

Activities
- Identify potential sources of variability and bias in the sampling and measurement processes (see Section 6)
- Determine the possible range on the parameter of interest.
- Choose the null hypothesis.
- Consider the consequences of making an incorrect decision.
- Specify a range of values where the consequences are minor (the “gray region”)
- Specify an acceptable probability of making a decision error.

in the results is caused by the non-homogeneity of the waste or media, slight differences in how the samples of the waste were collected and handled, variability in the analysis process, and the fact that only a small portion of the waste is usually ever sampled and tested. (See Section 6.1 for a more detailed discussion of sources of variability and bias in sampling). For example, if you conduct sampling and analysis of a solid waste and classify it as “nonhazardous” based on the results, when in fact it *is* a hazardous waste, you will have made a wrong decision or *decision error*. Alternatively, if you classify a solid waste as hazardous, when in fact it is nonhazardous, you also will have made a *decision error*.

There are two types of decision error. A “Type I” or “false rejection” decision error occurs if you reject the null hypothesis when it is true. (The “null hypothesis” is simply the situation presumed to be true or the “working assumption”.) A “Type II” or “false acceptance” decision error occurs if you accept the null hypothesis when it is false.[5]

Table 3 summarizes the four possible situations that might arise when a hypothesis is tested. The two possible true conditions correspond to the two columns of the table: the null hypothesis or “baseline assumption” is either true or the alternative is true. The two kinds of decisions are shown in the body of the table. Either you decide the baseline is true, or you decide the alternative is true. Associated with these two decisions are the two types of risk – the risk of making a Type I (false rejection) error (denoted by α) and the risk of making a Type II (false acceptance) error (denoted by β). You can improve your chances of making correct decisions by reducing α and β (which often requires more samples or a different sampling design) and by using field sampling techniques that minimize errors related to sampling collection and handling (see also Sections 6 and 7).

Table 3. Conclusions and Consequences for a Test of Hypotheses

		True Condition	
		Baseline is True	**Alternative is True**
Decision Based on Sample Data	Baseline is True	Correct Decision	Type II (false acceptance) error (probability β)
	Alternative is True	Type I (false rejection) error (probability α)	Correct Decision

For many sampling situations under RCRA, the most conservative (i.e., protective of the environment) approach is to presume that the constituent concentration in the waste or media exceeds the standard in the absence of strong evidence to the contrary.[6] For example, in

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[5] Statisticians sometimes refer to a Type I error as a “false positive,” and a Type II error as a “false negative.” The terms refer to decision errors made relative to a null hypothesis, and the terms may not necessarily have the same meaning as those used by chemists to describe analytical detection of a constituent when it is not really present (“false positive”) or failure to detect a constituent when it really *is* present (“false negative”).

[6] An exception to this assumption is found in “detection monitoring” and “compliance monitoring” in which underlying media (such as soil, pore water, or ground water) at a new waste management unit are presumed “clean” until a statistically significant increase above background is demonstrated (in the case of detection monitoring) or a statistically significant increase over a fixed standard is demonstrated (in the case of compliance or assessment monitoring).

testing a solid waste to determine if it exhibits the TC, the null hypothesis can be stated as follows: "the concentration is equal to or greater than the TC regulatory level." The alternative hypothesis is "the concentration is less than the TC regulatory level." After completion of the sampling and analysis phase, you conduct an assessment of the data. If your estimate of the parameter of interest is less than the threshold when the true value of the parameter exceeds the threshold, you will make a decision error (a Type I error). If the estimate of the parameter of interest is greater than the threshold when the true value is less than the threshold, you also will make an error (a Type II error) -- but one that has little potential adverse impacts to human health and the environment.

Note that during the planning phase and during sampling you will not know which kind of error you might make. Later, after a decision has been made, if you *rejected* the null hypothesis then you either made a Type I (false rejection) decision error or not; you could not have made a Type II (false acceptance) decision error. On the other hand, if you did not reject the null hypothesis, then you either made a Type II (false acceptance) error or not; you could not have made a Type I (false rejection) error. In either case, you will know which type of error you might have made and you will know the *probability* that the error was made.

In the RCRA program, EPA is concerned primarily with controlling errors having the most adverse consequences for human health and the environment. In the interest of protecting the environment and maintaining compliance with the regulations, there is an incentive on the part of the regulated entity to minimize the chance of a Type I decision error. The statistical methods recommended in this document emphasize controlling the Type I (false rejection) error rate and do not necessarily require specification of a Type II (false acceptance) error rate.

The question for the decision maker then becomes, what is the acceptable probability (or chance) of making a decision error? To answer this question, four activities are suggested. These activities are based on guidance found in *Guidance for the Data Quality Objectives Process QA/G-4* (USEPA 2000b) but have been tailored for more direct application to RCRA waste-related studies. The *Guidance for the Data Quality Objectives Process EPA QA/G-4* also provides detailed guidance on the use of a graphical construct called a Decision Performance Curve to represent the quality of a decision process.

4.6.1 Determine the Possible Range on the Parameter of Interest

Establish the possible range (maximum and minimum values) of the parameter of interest using data from a pilot study, existing data for a similar waste stream, or process knowledge (e.g., using a materials-balance approach). It is desirable, but not required, to have an estimate of the standard deviation as well.

4.6.2 Identify the Decision Errors and Choose the Null Hypothesis

Table 4 presents four examples of decision errors that could be made in a RCRA waste study. In the first three examples, the consequences of making a Type I error could include increased risk to human health and the environment or a potential enforcement action by a regulatory authority. The consequences of making a Type II error could include unnecessary financial and administrative resources required to manage the waste as hazardous (when, in fact, it is not) or continuing site cleanup activities when, in fact, the site is "clean."

Table 4. Examples of Possible Decision Errors in RCRA Waste Studies

Regulatory Requirement	***"Null Hypothesis" (baseline condition)***	***Possible Decision Errors***	
		Type I Error (α) "False Rejection"	***Type II Error (β) "False Acceptance"***
Example 1: Under 40 CFR 261.11, conduct sampling to determine if a solid waste is a hazardous waste by the TC.	The solid waste contains TC constituents at concentrations equal to or greater than their applicable regulatory levels (i.e., the solid waste is a hazardous waste).	Concluding the waste is not hazardous when, in fact, it is.	Deciding the waste is hazardous when, in fact, it is not.
Example 2: Under 40 CFR 268.7, conduct sampling and testing to certify that a hazardous waste has been treated so that concentrations of hazardous constituents meet the applicable LDR treatment standards.	The concentration of the hazardous constituents exceeds the treatment standard (i.e., the treatment standard has not been attained).	Concluding the treatment standard has been met when, in fact, it has not.	Concluding the treatment standard has not been met when, in fact, it has.
Example 3: Under 40 CFR 264.101 (and proposed Subpart S - Corrective Action at SWMUs), a permittee conducts testing to determine if a remediation at a SWMU has attained the risk-based cleanup standard specified in the permit.*	The mean concentration in the SWMU is greater than the risk-based cleanup standard (i.e., the site is contaminated).†	Concluding the site is "clean" when, in fact, it is contaminated.	Concluding the site is still contaminated when, in fact, it is "clean."
Example 4: Under 40 CFR 264.98(f), detection monitoring, monitor ground water at a regulated unit to determine if there is a statistically significant increase of contamination above background.	The level of contamination in each point of compliance well does not exceed background.	Concluding the contaminant concentration in a compliance well exceeds background when, in fact, it does not.	Concluding the contaminant concentration in a compliance well is similar to background when, in fact, it is higher.

* If the cleanup standard is based on "background" rather than a risk-based cleanup standard, then the hypotheses would be framed *in reverse* where the mean background and on-site concentrations are presumed equal unless there is strong evidence that the site concentrations are greater than background.

† A parameter other than the mean may be used to evaluate attainment of a cleanup standard (e.g., see USEPA 1989a).

In Example 4, however, the null hypothesis is framed *in reverse* of Examples 1 through 3. When conducting subsurface monitoring to detect contamination at a new unit (such as in detection monitoring in the RCRA ground-water monitoring program), the natural subsurface environment is presumed uncontaminated until statistically significant increases over the background concentrations are detected. Accordingly, the null hypothesis is framed such that the downgradient conditions are consistent with the background. In this case, EPA's emphasis on the protection of human health and the environment calls for minimizing the Type II error -- the mistake of judging downgradient concentrations the same as the background when, in fact, they are higher. Detailed guidance on detection and compliance monitoring can be found in *RCRA Ground-Water Monitoring: Draft Technical Guidance* (USEPA 1992c) and EPA's guidance on the statistical analysis of ground-water monitoring data at RCRA facilities (USEPA 1989b and 1992b).

4.6.3 Specify a Range of Possible Parameter Values Where the Consequences of a False Acceptance Decision Error are Relatively Minor (Gray Region)

The "gray region" is one component of the quantitative decision performance criteria the planning team establishes during the DQO Process to limit impractical and infeasible sample sizes. The gray region is a range of possible parameter values near the action level where it is "too close to call." This gray area is where the sample data tend toward rejecting the baseline condition, but the evidence (data statistics) is not sufficient to be overwhelming. In essence, the gray region is an area where it will not be feasible to control the false acceptance decision error limits to low levels because the high costs of sampling and analysis outweigh the potential consequences of choosing the wrong course of action.

In statistical language, the gray region is called the "minimum detectable difference" and is often expressed as the Greek letter delta (Δ). This value is an essential part of the calculations for determining the number of samples that need to be collected so that the decision maker may have confidence in the decision made based on the data collected.

The first boundary of the gray region is the Action Level. The other boundary of the gray region is established by evaluating the consequences of a false acceptance decision error over the range of possible parameter values in which this error may occur. This boundary corresponds to the parameter value at which the consequences of a false acceptance decision error are significant enough to have to set a limit on the probability of this error occurring. The gray region (or "area of uncertainty") establishes the minimum distance from the Action Level where the decision maker would like to begin to control false acceptance decision errors.

In general, the narrower the gray region, the greater the number of samples needed to meet the criteria because the area of uncertainty has been reduced.

The quality of the decision process, including the boundaries of the gray region, can be depicted graphically using a Decision Performance Goal Diagram (DPGD). Detailed guidance on the construction and use of DPGDs is given in EPA DQO guidance documents (e.g., USEPA 2000a and 2000b) and in *Data Quality Objectives Decision Error Feasibility Trials Software (DEFT) - User's Guide* (USEPA 2001a). Figure 12(a) and Figure 12(b) show how some of the key outputs of Step 6 of the DQO Process are depicted in a DPGD when the parameter of interest is the <u>mean</u> (Figure 12(a)) and a <u>percentile</u> (Figure 12(b) .

The DPGD given in Figure 12(a) shows how the boundaries of the gray region are set when the null hypothesis is established as "the true mean concentration exceeds the standard." Notice that the planning team has set the action level at 5 ppm and the other boundary of the gray region at 4 ppm. This implies that when the mean calculated from the sample data is less than 4 ppm (and the planning assumptions regarding variability hold true), then the data will be considered to provide "overwhelming evidence" that the true mean (unknown, of course) is below the action level.

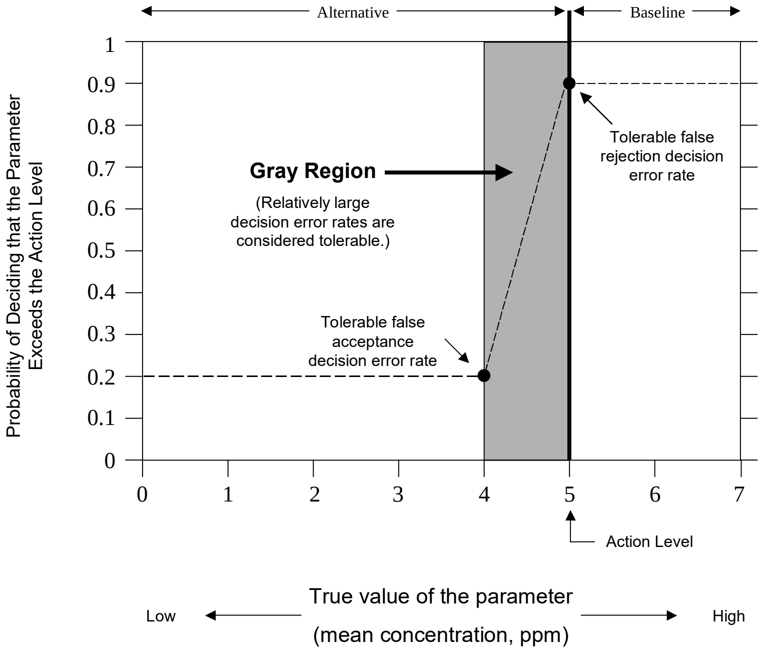

Figure 12(a). Decision Performance Goal Diagram where the mean is the parameter of interest. Null hypothesis (baseline condition): the true mean exceeds the action level.

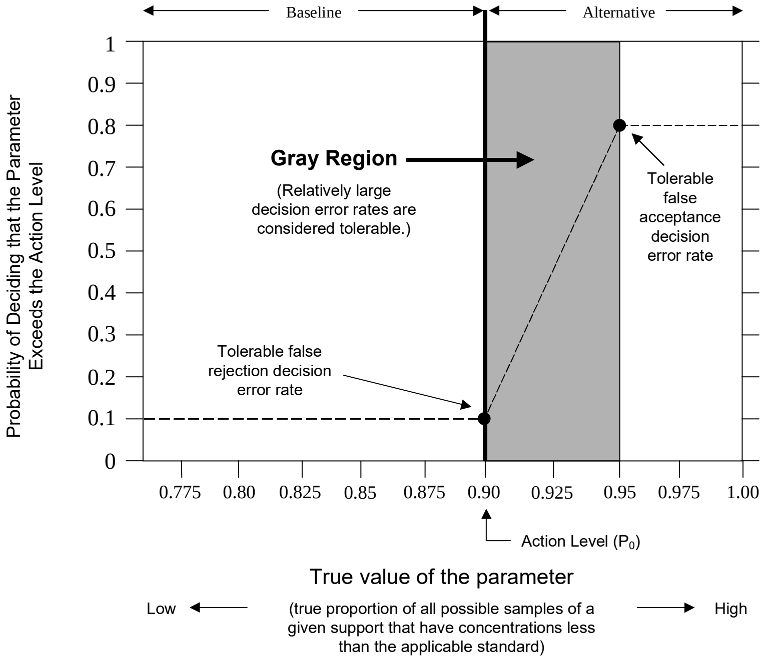

Figure 12(b). Decision Performance Goal Diagram where a percentile is the parameter of interest. Null hypothesis (baseline condition): true proportion -- of all possible samples of a given support that are less than the applicable standard -- is less than 0.90.

Now consider the DPGD given in Figure 12(b). The figure shows how the gray region is set when the null hypothesis is established as "the true proportion of samples below the concentration standard is less than 0.90." Notice in this example the planning team has set the action level at 0.90 and the other boundary of the gray region at 0.95. This implies that when the proportion of samples that comply with the standard is greater than 0.95, then the data will be considered to provide "overwhelming evidence" that the true proportion (unknown, of course) is greater than the action level of 0.90.

The term "samples" refers to all possible samples of a specified size, shape, and orientation (or **sample support**) drawn from the DQO decision unit. Sampling procedures and sample support can affect the measurement value obtained on individual samples and have a profound effect on the shape of the sampling distribution. Thus, the outcome of statistical procedures that examine characteristics of the upper tail of the distribution can be influenced by the sample support – more so than when the mean is the parameter of interest. Accordingly, when testing for a proportion, a complete statement of the null hypothesis should include specification of the sample support. See Sections 6.3.1 and 6.3.2 for guidance on establishing the appropriate sample support as part of the DQO Process.

4.6.4 Specify an Acceptable Probability of Making a Decision Error

You can never completely eliminate decision errors or even know when they have occurred, but you can quantify the probability of making such errors. In this activity, you establish the acceptable probability of making a decision error.

The Type I error rate (α) is a measure of the amount of "mistrust" you have in the conclusion (Myers 1997) and is also known as the **significance level** for a test. The flip side of this is the amount of faith or confidence you have in the conclusion. The **confidence level** is denoted mathematically as $1-\alpha$. As stated previously, the Type I error (the error of falsely rejecting the null hypothesis) is of greatest concern from the standpoint of environmental protection and regulatory compliance.

The probability of making a Type II error (the error of falsely accepting the null hypothesis) also can be specified. For example, if the sample data lead you to conclude that a waste does not qualify for the comparable fuels exclusion (40 CFR 261.38), when the true mean concentration in the waste is in fact below the applicable standard, then a Type II (false acceptance error) has been made. (Note that some of the statistical methods given in this document do not require specification of a Type II error rate).

As a general rule, the lower you set the probability of making a decision error, the greater the cost in terms of the number of samples required, time and personnel required for sampling and analysis, and financial resources required.

An acceptable probability level for making a decision error should be established by the planning team after consideration of the RCRA regulatory requirements, guidance from EPA or the implementing agency, the size (volume or weight) of the decision unit, and the consequences of making a decision error. In some cases, the RCRA regulations specify the Type I or Type II (or both) error rates that should be used. For example, when testing a waste to determine whether it qualifies for the comparable/syngas fuel exclusion under 40 CFR 261.38, the regulations *require* that the determination be made with a Type I error rate set at 5 percent (i.e., $\alpha = 0.05$).[7]

In other cases, the regulations do not specify any decision error limits. The planning team must specify the decision error limits based on their knowledge of the waste; impacts on costs, human health, and ecological conditions; and the potential consequences of making a decision error. For example, if the quantity of waste (that comprises a decision unit) is large and/or heterogeneous, then a waste handler may require high confidence (e.g., 95 or 99 percent) that a high proportion of the waste or media complies with the applicable standard. On the other hand, if the waste quantity is a relatively small (e.g., a drum) and sampling and measurement error can be minimized, then the waste handler may be willing to relax the confidence level required or simply use a nonstatistical (e.g., judgmental) sampling design and reduce the number of samples to be taken.

For additional guidance on controlling errors Section 6 and EPA's DQO guidance (USEPA 2000a and 2000b).

4.7 Outputs of the First Six Steps of the DQO Process

Table 5 provides a summary of the outputs of the first six steps of the DQO Process. Typically, this information will be incorporated into a QAPP, WAP, or other similar planning document (as described in Section 5.7). The DQOs can be simple and straight forward for simple projects and can be documented in just a few pages with little or no supporting data. For more complex projects, the DQOs can be more lengthy, and the supporting data may take up volumes. The team that will be optimizing the sample design(s) will need the information to support their plan development. The project manager and the individuals who assess the overall outcome of the project also will need the information to determine if the DQOs were achieved.

Keep in mind that the DQO Process is an iterative one; it might be necessary to return to earlier steps to modify inputs when new data become available or to change assumptions if achieving the original DQOs is not realistic or practicable.

The last step (Step 7) in the DQO Process is described in detail in the next section of this document. Example applications of the full DQO Process are presented in Appendix "I."

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[7] Under §261.38(c)(8)(iii)(A), a generator must demonstrate that "each constituent of concern is not present in the waste above the specification level at the 95% upper confidence limit around the mean."

Table 5. Summary of Outputs of the First Six Steps of the DQO Process

DQO Step	***Expected Outputs***
1. State the Problem	• List of members of the planning/scoping team and their role/expertise in the project. Identify individuals or organizations participating in the project (e.g. facility name) and discuss their roles, responsibilities, and organization. • A concise description of the problem. • Summary of available resources and relevant deadlines.
2. Identify the Decision	• A decision statement that links the principal study question to possible actions that will solve the problem or answer the question.
3. Identify Inputs to the Decision	• A list of informational inputs needed to resolve the decision statement, how the information will be used, sources of that information, and an indication of whether the information is available for will need to be obtained. • A list of environmental variables or characteristics that will be measured.
4. Define the Boundaries	• A detailed description of the spatial and temporal boundaries of the problem (i.e., define the population, each decision unit, and the sample support). • Options for stratifying the population under study. • Any practical constraints that may interfere with the study.
5. Develop a Decision Rule	• The parameter of interest that characterizes the population. • The Action Level or other method for testing the decision rule. • An “if ...then...” statement that defines the conditions that would cause the decision maker to choose among alternative actions.
6. Specify Limits on Decision Errors	• Potential variability and bias in the candidate sampling and measurement methods • The baseline condition (null hypothesis) • The boundaries of the gray region • The decision maker’s tolerable decision error rates based on a consideration of consequences of making an incorrect decision.

5 OPTIMIZING THE DESIGN FOR OBTAINING THE DATA

This section describes DQO Process Step 7, the last step in the DQO Process. The purpose of this step is to identify an optimal design for obtaining the data. An optimal sampling design is one that obtains the requisite information from the samples for the lowest cost and still satisfies the DQOs.

You can optimize the sampling design by performing five activities that are described in detail in this section. These activities are based on those described in *Guidance for the Data Quality Objectives Process EPA QA/G-4* (USEPA 2000b), but they have been modified to more specifically address RCRA waste-related studies.

In this final planning step, combine the data collection design information with the other outputs of the DQO Process and document the approach in a planning document such as a QAPP, WAP, or similar planning document. As part of this step, it may be necessary to work through Step 7 more than once after revisiting the first six steps of the DQO Process.

Step 7: Optimize the Design for Collecting the Data

Purpose
To identify a resource-effective data collection design for generating data that are expected to satisfy the DQOs.

Activities
- Review the outputs of the first six steps of the DQO Process (see Section 5.1).
- Consider various data collection design options, including sampling and analytical design alternatives (see Section 5.2), and composite sampling options (see Section 5.3).
- For each data collection design alternative, determine the appropriate number of samples (see Section 5.4 or 5.5).
- Select the most resource-effective design that satisfies all of the data needs for the least costs (see Section 5.6).
- Prepare a QAPP, WAP, or similar planning document as needed to satisfy the project and regulatory requirement (see Section 5.7).

5.1 Review the Outputs of the First Six Steps of the DQO Process

Each of the steps in the DQO Process has a series of outputs that include qualitative and quantitative information about the study. The outputs of the first six steps of the DQO Process, as described in Section 4, serve as inputs to DQO Step 7.

Review the existing information and DQO outputs (see Table 5). Determine if any data gaps exist and determine whether filling those gaps is critical to completion of the project. Data gaps can be filled by means of a “preliminary study” or “pilot study.” A preliminary study or pilot can include collection of samples to obtain preliminary estimates of the mean and standard deviation. In addition, a preliminary study can help you verify waste or site conditions, identify unexpected conditions or materials present, gain familiarization with the waste and facility operations, identify how the waste can be accessed, check and document the physical state of the material to be sampled, and identify potential health and safety hazards that may be present.

Review the potential sources of variability and bias (“error”) that might be introduced in the sampling design and measurement processes. See Section 6 for a discussion of sources of error in sampling and analysis.

5.2 Consider Data Collection Design Options

Data collection design incorporates two interdependent activities -- the sample collection design and analytical design.

> **Sampling Design:** In developing a sampling design, you consider various strategies for selecting the locations, times, and components for sampling, and you define appropriate sample support. Examples of sampling designs include simple random, stratified random, systematic, and judgmental sampling. In addition to sampling designs, make sure your organization has documented standard operation procedures (SOPs) that describe the steps to be followed when implementing a sampling activity (e.g., equipment preparation, sample collection, decontamination). For guidance on suggested content and format for SOPs, refer to *Guidance for the Preparing Standard Operating Procedures (SOPs) EPA QA/G-6* (USEPA 2001c). Sampling QA/QC activities also should be part of sampling design. Activities used to document, measure, and control data quality include project-specific quality controls (e.g., duplicate samples, equipment blanks, field blanks, and trip blanks) and the associated quality assessments (e.g., audits, reviews) and assurances (e.g., corrective actions, reports to management). These activities typically are documented in the QAPP (see Section 5.7 and USEPA 1998a).
>
> **Analytical Design:** In DQO Steps 3 and 5, an Action Level and candidate analytical methods were identified. The information should be used to develop analytical options in terms of cost, method performance, available turnaround times, and QA/QC requirements. The analytical options can be used as the basis for designing a performance-based cost-effective analytical plan (e.g., deciding between lower-cost field analytical methods and/or higher cost laboratory methods). Candidate laboratories should have adequate SOPs that describe the steps to be followed when implementing an analytical activity (e.g., sample receipt procedures, subsampling, sample preparation, cleanup, instrumental analysis, data generation and handling). If field analytical techniques are used, hard copies of the analytical methods or SOPs should be available in the field. Refer to Chapter Two of SW-846 for guidance on the selection of analytical methods.

The goal of this step is to find cost-effective design alternatives that balance the number of samples and the measurement performance, given the feasible choices for sample designs and measurement methods.

Sampling design is the “where, when, and how” component of the planning process. In the context of waste sampling under RCRA, there are two categories of sampling designs: (1) **probability** sampling and (2) **authoritative** (nonprobability) sampling. The choice of a sampling design should be made after consideration of the DQOs and the regulatory requirements.

Probability sampling refers to sampling designs in which all parts of the waste or media under study have a known probability of being included in the sample. In cases in which all parts of the waste or media are not accessible for sampling, the situation should be documented so its potential impacts can be addressed in the assessment phase. Probability samples can be of various types, but in some way, they all make use of randomization, which allows probability statements to be made about the quality of estimates derived from the resultant data.

Probability sampling designs provide the ability to reliably estimate variability, the reproducibility of the study (within limits), and the ability to make valid statistical inferences. Five types of probability sampling designs are described in Sections 5.2.1 through 5.2.5:

- Simple random sampling
- Stratified random sampling
- Systematic sampling
- Ranked set sampling
- Sequential sampling.

A strategy that can be used to improve the precision (reproducibility) of most sampling designs is **composite** sampling. Composite sampling is not a sampling design in and of itself, rather composite sampling is a *strategy* used as part of a probability sampling design or an authoritative sampling design. Composite sampling is discussed in Section 5.3.

Sampling Over Time or Space?

An important feature of probability sampling designs is that they can be applied along a line of time or in space (see Figure 13) or both (Gilbert 1987):

Time
Sampling designs applied over time can be described by a *one-dimensional* model that corresponds to flowing streams such as the following:

- Solid materials on a conveyor belt
- A liquid stream, pulp, or slurry moving in a pipe or from a discharge point (e.g., from the point of waste generation)
- Continuous elongated piles (Pitard 1993).

Space
For practical reasons, sampling of material over a *three-dimensional* space is best addressed as though the material consists of a series of overlapping *two-dimensional* planes of more-or-less uniform thickness (Pitard 1993, Gy 1998). This is the case for obtaining samples from units such as the following:

- Drums, tanks, or impoundments containing single or multi-phasic liquid wastes
- Roll-off bins, relatively flat piles, or other storage units
- Landfills, soil at a land treatment unit, or a SWMU.

One common misconception of probability sampling procedures is that these procedures preclude the use of important prior information. Indeed, just the opposite is true. An efficient sampling design is one that uses all available prior information to help design the study. Information obtained during DQO Step 3 (“Identify Inputs to the Decision”) and DQO Step 4 (“Define the Study Boundaries”) should prove useful at this stage. One of the activities suggested in DQO Step 4 is to segregate the waste stream or media into less heterogeneous subpopulations as a means of segregating variability. To determine if this activity is appropriate, it is critical to have an understanding of the various kinds of heterogeneity the constituent of concern exhibits within the waste or media (Pitard 1993). Making assumptions that a waste stream is homogeneous can result in serious sampling errors. In fact, some authors suggest the word “homogeneous” be removed from our sampling vocabulary (Pitard 1993, Myers 1997).

Table 6 provides a summary of sampling designs discussed in this guidance along with conditions for their use, their advantages, and their disadvantages. Figure 13 provides a graphical representation of the probability sampling designs described in this guidance. A number of other sampling designs are available that might perform better for your particular situation. Examples include cluster sampling and double sampling. If an alternative sampling design is required, review other publications such as Cochran (1977), Gilbert (1987), USEPA (2000c) and consult a professional statistician.

Table 6. Guidance for Selection of Sampling Designs

Sampling Design	*Appropriate Conditions for Use*	*Advantages*	*Limitations*
Probability Sampling			
Simple Random Sampling (Section 5.2.1)	Useful when the population of interest is relatively homogeneous (i.e., there are no major patterns or "hot spots" expected).	• Provides statistically unbiased estimates of the mean, proportions, and the variability. • Easy to understand and implement.	• Least preferred if patterns or trends are known to exist and are identifiable. • Localized clustering of sample points can occur by random chance.
Stratified Random Sampling (Section 5.2.2)	Most useful for estimating a parameter (e.g., the mean) of wastes exhibiting high heterogeneity (e.g., there are distinct portions or components of the waste with high and low constituent concentrations or characteristics).	• Ensures more uniform coverage of the entire target population. • Potential for achieving greater precision in estimates of the mean and variance. • May reduce costs over simple random and systematic sampling designs because fewer samples may be required. • Enables computation of reliable estimates for population subgroups of special interest.	• Requires some prior knowledge of the waste or media to define strata and to obtain a more precise estimate of the mean. • Statistical procedures for calculating the number of samples, the mean, and the variance are more complicated than for simple random sampling.
Systematic Sampling (Section 5.2.3)	Useful for estimating spatial patterns or trends over time.	• Preferred over simple random when sample locations are random within each systematic block or interval. • Practical and easy method for designating sample locations. • Ensures uniform coverage of site, unit, or process. • May be lower cost than simple random sampling because it is easier to implement.	• May be misleading if the sampling interval is aligned with the pattern of contamination, which could happen inadvertently if there is inadequate prior knowledge of the pattern of contamination. • Not truly random, but can be modified through use of the "random within blocks" design.

Table 6. Guidance for Selection of Sampling Designs (Continued)

Sampling Design	*Appropriate Conditions for Use*	*Advantages*	*Limitations*
Probability Sampling (continued)			
Ranked Set Sampling (Section 5.2.4)	• Useful for reducing the number of samples required. • Useful when the cost of analysis is much greater than the cost of collecting samples. • Inexpensive auxiliary variable (based on expert knowledge or measurement) is needed and can be used to rank randomly selected population units with respect to the variable of interest. • Useful if the ranking method has a strong relationship with accurate measurements.	• Can reduce analytical costs.	• Requires expert knowledge of waste or process or use of auxiliary quantitative measurements to rank population units.
Sequential Sampling (Section 5.2.5)	• Applicable when sampling and/or analysis are quite expensive, when information concerning sampling and/or measurement variability is lacking, when the waste and site characteristics of interest are stable over the time frame of the sampling effort, or when the objective of the sampling effort is to test a specific hypothesis. • May not be especially useful if multiple waste characteristics are of interest or if rapid decision making is necessary.	• Can reduce the number of samples required to make a decision. • Allows a decision to be made with less sampling if there is a large difference between the two populations or between the true value of the parameter of interest and the standard.	• If the concentration of the constituent of concern is only marginally different from the action level, sequential procedures will require an increasing number of samples approaching that required for other designs such as simple random or systematic sampling.

Table 6. Guidance for Selection of Sampling Designs (Continued)

Sampling Design	*Appropriate Conditions for Use*	*Advantages*	*Limitations*
Authoritative Sampling			
Judgmental (Section 5.2.6.1)	• Useful for generating rough estimates of the average concentration or typical property. • To obtain preliminary information about a waste stream or site to facilitate planning or to gain familiarity with the waste matrix for analytical purposes. • To assess the usefulness of samples drawn from a small portion of the waste or site. • To screen samples in the field to identify "hot" samples for subsequent analysis in a laboratory.	• Can be very efficient with sufficient knowledge of the site or waste generation process. • Easy to do and explain.	• The utility of the sampling design is highly dependent on expert knowledge of waste. • Nonprobability-based so inference to the general population is difficult. • Cannot determine reliable estimates of variability.
Biased (Section 5.2.6.2)	• Useful to estimate "worst-case" or "best-case" conditions (e.g., to identify the composition of a leak, spill, or waste of unknown composition).		

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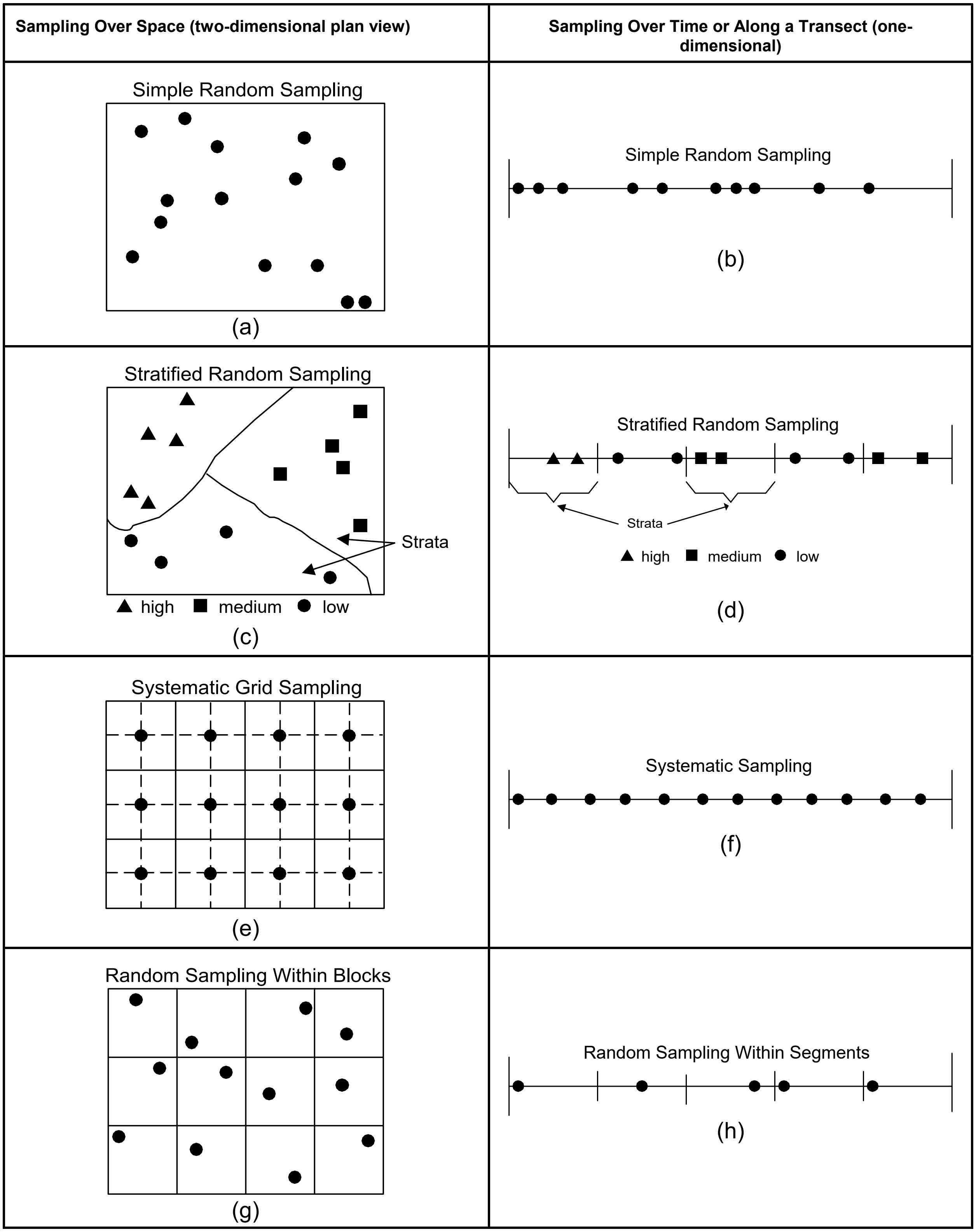

Figure 13. Probability sampling designs over space or along an interval (modified after Cochran 1977 and Gilbert 1987)

5.2.1 Simple Random Sampling

The simplest type of probability sampling is simple random sampling (without replacement), in which every possible sampling unit in the target population has an equal chance of being selected. Simple random samples, like the other samples, can be either samples in space (Figure 13(a)) or in time (Figure 13(b)) and are often appropriate at an early stage of an investigation in which little is known about nonrandom variation within the waste generation process or the site. All of the sampling units should have equal volume or mass, and ideally be of the same shape and orientation if applicable (i.e., they should have the same "sample support").

Box 3. Simple Random Sampling: Procedure

1. Divide the area of the study into N equal-size grids, intervals (if sampling over time), or other units. The spacing between adjacent sampling locations should be established in the DQOs, but the length should be measurable in the field with reasonable accuracy. The total number of possible sampling locations (N) should be much larger than n (the number of samples to be collected).*
2. Assign a series of consecutive numbers to each location between 1 and N.
3. Draw n integers between 1 and N from a random number table or use the random number function on a hand-held calculator (i.e., generate a random number between 0 and 1 and multiply the number by N).
4. Collect samples at each of the n locations or intervals.

* For additional guidance on calculating spacing between sampling locations, see *Methods for Evaluating the Attainment of Cleanup Standards, Volume I: Soil and Solid Media* (USEPA 1989a).

With a simple random sample, the term "random" should not be interpreted to mean haphazard; rather, it has the explicit meaning of equiprobable selection. Simple random samples are generally developed through use of a random number table (found in many statistical text books), a random number function on a hand-held calculator, or by a computer.

One possible disadvantage of pure random sampling is that localized clustering of sample points can occur. If this occurs, one option is to select a new random time or location for the sample. Spatial or temporal biases could result if unknown trends, patterns, or correlations are present. In such situations, stratified random sampling or systematic sampling are better options.

5.2.2 Stratified Random Sampling

In stratified random sampling, a heterogeneous unit, site, or process is divided into nonoverlapping groups called **strata**. Each stratum should be defined so that internally it is relatively homogeneous (that is, the variability within each stratum is less than the variability observed over the entire population) (Gilbert 1987). After each stratum is defined, then simple random sampling is used within each stratum (see Figure 13(c) and 15(d)). For very heterogeneous wastes, stratified random sampling can be used to obtain a more efficient estimate of the parameter of interest (such as the mean) than can be obtained from simple random sampling.

It is important to note that stratified random sampling, as described in this guidance, can be used when the objective is to make a decision about the *whole* population or decision unit. If the objective is to determine of a solid waste is a hazardous waste or to measure attainment of a treatment standard for a hazardous waste, then any obvious "hot spots" or high concentration wastes should be characterized separately from low concentration wastes to minimize mixing of hazardous waste with nonhazardous wastes and to prevent impermissible dilution (see also Appendix C). If the objective of the sampling effort is to identify nonrandom spatial patterns (for example, to create a map of contamination in shallow soils), then consider the use of a geostatistical technique to evaluate the site.

Box 4. Stratified Random Sampling: Procedure

1. Use prior knowledge of the waste stream or site to divide the target population into L nonoverlapping strata such that the variability within stratum is less than the variability of the entire population (for example, see Figure 13c and Figure 13d). The strata can represent area, volume, mass, or time intervals.
2. Assign a weight W_h to each hth stratum. The value of each W_h should be determined based on its relative importance to the data user, or it can be the proportion of the volume, mass, or area of the waste that is in stratum h .
3. Conduct random sampling within each stratum.

In stratified random sampling it is usually necessary to incorporate prior knowledge and professional judgment into a probabilistic sampling design. Generally, wastes or units that are “alike” or anticipated to be “alike” are placed together in the same stratum. Units that are contiguous in space (e.g., similar depths) or time are often grouped together into the same stratum, but characteristics other than spatial or temporal proximity can be employed. For example, you could stratify a waste based on particle size (such that relatively large pieces of contaminated debris are assigned to one stratum and unconsolidated fines assigned to a separate stratum). This is called *stratification by component*. See Appendix C of this guidance for additional information on stratification, especially as a strategy for sampling heterogeneous wastes, such as debris.

In stratified random sampling a decision must be made regarding the allocation of samples among strata. When chemical variation within each stratum is known, samples can be allocated among strata using *optimum allocation* in which more samples are allocated to strata that are large, more variable internally, or cheaper to sample (Cochran 1977, Gilbert 1987). An alternative is to use *proportional allocation*. In proportional allocation, the sampling effort in each stratum is directly proportional to the size (for example, the mass) of the stratum. See Section 5.4.2 for guidance on determining optimum and proportional allocation of samples to strata.

There are several advantages to stratified random sampling. Stratified random sampling:

- Ensures more uniform coverage of the entire target population
- Ensures that subareas that contribute to overall variability are included in the sample
- Achieves greater precision in certain estimation problems
- Generally will be more cost-effective than simple random sampling even when imperfect information is used to form the strata.

There are also some disadvantages to stratified random sampling. Stratified random sampling is slightly more difficult to implement in the field and statistical calculations for stratified sampling are more complex than for simple random sampling (e.g., due to the use of weighting factors and more complex equations for the appropriate number of samples).

5.2.3 Systematic Sampling

Box 5: Systematic Sampling: Procedure

Sampling Over Space

1. Determine the size of the area to be sampled.
2. Denote the surface area of the sample area by A.
3. Assuming a square grid is used, calculate the length of spacing between grid nodes (L)

$$L = \sqrt{\frac{A}{n}}$$

where n is the number of samples. The distance L should be rounded to the nearest unit that can be easily measured in the field.
4. To determine the sampling locations, randomly select an initial sampling point within the area to be sampled. Using this location as one intersection of two gridlines, construct gridlines parallel to the original grid and separated by distance L.
5. Collect samples *at* each grid node (line intersection) (see Figure 13e). Alternatively, randomly select a sampling point *within* each grid block (see Figure 13g).

Sampling Along a Line (e.g., Over Time)

1. Determine the start time and point and the total length of time (N) over which the samples will be collected.
2. Decide how many samples (n) will be collected over the sampling period.
3. Calculate a sampling interval where $k = \frac{N}{n}$.
4. Randomly select a start time and collect a sample every kth interval until n samples have been obtained (see Figure 13f). Alternatively, randomly select a sampling point *within* each interval (Figure 13h).

Systematic sampling entails taking samples at a preset interval of time or in space and using a randomly selected time or location as the first sampling point (Gilbert 1987).

Systematic sampling over space involves establishing a two-dimensional grid of the unit or waste under investigation (Figure 13(e)). The orientation of the grid is sometimes chosen randomly and various types of systematic samples are possible. For example, points may be arranged in a pattern of squares (rectangular grid sampling) or a pattern of equilateral triangles (triangular grid sampling). The result of either approach is a simple pattern of equally spaced points at which sampling is to be performed. As shown in Figure 13(f), systematic sampling also can be conducted along a transect (every five feet, for example), along time intervals (every hour, for example), or by flow or batches (every 10,000 gallons, for example) (King 1993).

The systematic sampling approach is attractive because it can be easily implemented in the field, but it has some limitations such as not being truly random. You can improve on this sampling design by using random sampling within each grid block (Figure 13(g)) or within each time interval (Figure 13(h)). This approach maintains the condition of equiprobability during the sampling event (Myers 1997) and can be considered a form of *stratified random sampling* in which each of the boundaries of the strata are arbitrarily defined (rather than using prior information) and only one random sample is taken per stratum (Gilbert 1987). This approach is advantageous because it avoids potential problems caused by cycles or trends.

Systematic sampling also is preferred when one of the objectives is to locate “hot spots” within a site or otherwise map the pattern of concentrations over an area (e.g., using geostatistical techniques). Even without using geostatistical methods, “hot spots” or other patterns could be identified by using a systematic design (see “ELIPGRID” software in Appendix H and Gilbert 1987, page 119). On the other hand, the systematic sampling design should be used with caution whenever there is a possibility of some type of cyclical pattern in the waste unit or process that might match the sampling frequency, especially processes being measured over time (such as discharges from a pipe or material on a conveyor).

Figure 14 illustrates the potential disadvantage of using systematic sampling when cyclic trends are present. When there is a cyclic trend in a waste generation process, using a uniform pattern of sampling points can result in samples with very unusual properties. The sets of points labeled "A" and "B" are systematic samples for which the sampling intervals are one period and one-half period, respectively. The points labeled "A" would result in a biased estimate of the mean but a sampling variance of zero. The points labeled "B" would result in an unbiased estimate of the mean with very small variance, even a zero variance if the starting point happened to be aligned exactly with the mean.

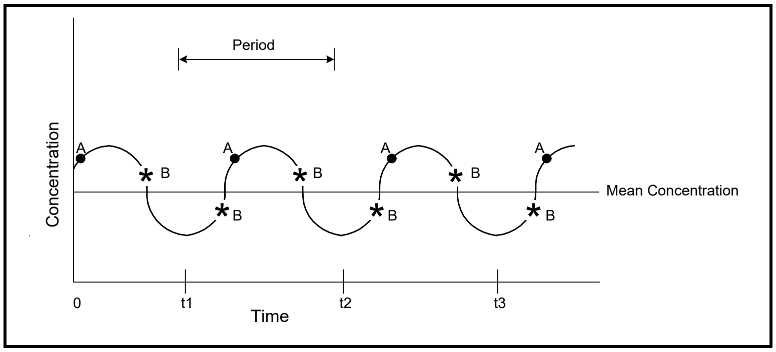

Figure 14. Potential pitfall of systematic sampling over time: cyclic trend combined with a systematic sampling design (after Cochran 1977 and Gilbert 1987)

5.2.4 Ranked Set Sampling

Ranked set sampling (RSS) (McIntyre 1952) can create a set of samples that at a minimum is equivalent to a simple random sample, but can be as much as two to three times more efficient than simple random sampling. This is because RSS uses the availability of expert knowledge or an inexpensive surrogate measurement or auxiliary variable that is correlated with the more expensive measurement of interest. The auxiliary variable can be a qualitative measure, such as visual inspection for color or an inexpensive quantitative (or semi-quantitative) measure that can be obtained from a field instrument such as a photoionization detector for volatile organics or an X-ray fluorescence analyzer for elemental analysis. RSS exploits this correlation to obtain a sample that is more representative of the population than would be obtained by random sampling, thereby leading to more precise estimates of the population parameters than random sampling. RSS is similar to other probabilistic sampling designs such as simple random sampling in that sampling points are identified and samples are collected. In RSS, however, only a subset of the samples are selected for analysis.

RSS consists of creating m groups, each of size m (for a total of "m x m" initial samples), then ranking the surrogate from largest to smallest within each group. One sample from each group is then selected according to a specified procedure and these m samples are analyzed for the more expensive measurement of interest (see Box 6 and Figure 15).

The true mean concentration of the characteristic of interest is estimated by the arithmetic sample mean of the measured samples (e.g., by Equation 1). The population variance and standard deviation also are estimated by the traditional equations (e.g., by Equations 2 and 3). For additional information on RSS, see USEPA 1995b, USEPA 2000c, and ASTM D 6582 *Standard Guide for Ranked Set Sampling: Efficient Estimation of a Mean Concentration in Environmental Sampling*.

Box 6. Ranked Set Sampling: Procedure

1. Identify some auxiliary characteristic by which samples can be ranked in order from lowest to highest (e.g., by use of a low-cost field screening method).
2. Randomly select $m \times m$ samples from the population (e.g., by using simple random sampling).
3. Arrange these samples into m sets of size m.
4. Within each set, rank the samples by using only the auxiliary information on the samples.
5. Select the samples to be analyzed as follows (see Figure 17):
   - In Set 1, select the sample with rank 1
   - In Set 2, select the sample with rank 2, etc ...
   - In Set m, select the unit with rank m.
6. Repeat Steps 1 through 5 for r cycles to obtain a total of $n = m \cdot r$ samples for analysis.

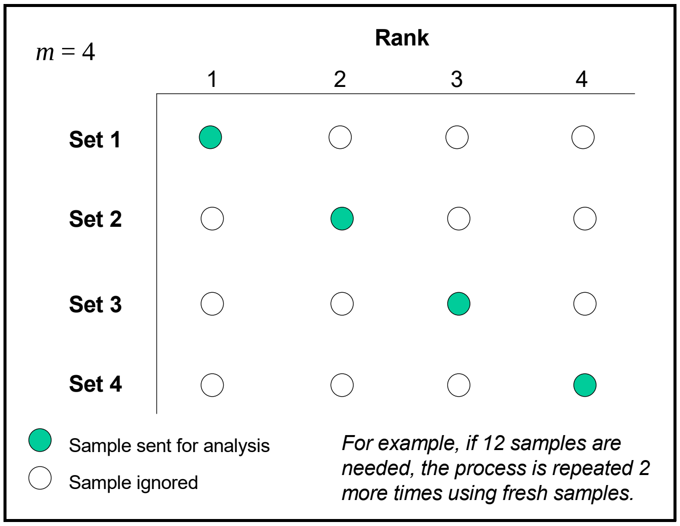

Figure 15. Ranked set sampling. After the samples are ranked in order from lowest to highest, a sample is selected for analysis from Set 1 with Rank 1, from Set 2 with Rank 2, etc.

5.2.5 Sequential Sampling

In sequential testing procedures (Wald 1973), sampling is performed by analyzing one (or more) sample(s) at a time until enough data have been collected to meet the statistical confidence level that the material does not exceed the critical level. The expected sample size, using this sequential procedure, can be approximately 30- to 60-percent lower than a corresponding fixed sample size test with the same power. The sequential procedure is especially helpful in situations in which the contamination is very high or very low relative to the action level. In these situations, the sequential procedure will quickly accumulate enough evidence to conclude that the waste or site either meets or fails to meet the standard.

Figure 16 shows how the procedure operates in a simple example for determining the mean concentration of a constituent of concern in soil. This particular example involves clean closure of a waste management unit, however, the approach could be used for other situations in which the mean is the parameter of interest. The procedure consists of analyzing groups of samples and calculating the mean and 80-percent confidence interval (or upper 90-percent confidence limit) for the mean after analysis of each group of samples. The horizontal axis represents the number of sample units evaluated. The vertical axis represents the concentration of the contaminant; plotted are the mean and 80-percent confidence interval after analysis of n samples. The AL, against which the sample is to be judged, is shown as a horizontal line.

The sampled units are analyzed first in a small lot (e.g., five samples). After each evaluation the mean and confidence interval on the mean are determined (point “a”). If the 90-percent UCL on the mean value stays above the critical value, AL, after successive increments are analyzed, the soil in the unit cannot be judged to attain the action level (point “b”). If the UCL goes below the critical value line, it may be concluded that the soil attains the standard. In the figure, the total number of samples is successively increased until the 90-percent UCL falls below the critical level (points “c” and “d”).

A sequential sampling approach also can be used to test a percentile against a standard. A detailed description of this method is given in Chapter 8 of *Methods for Evaluating the Attainment of Cleanup Standards Volume 1: Soil and Solid Media* (USEPA 1989a).

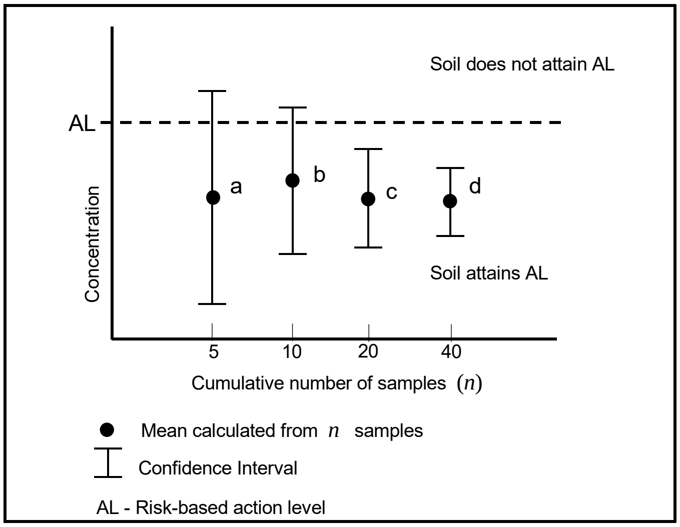

Figure 16. Example of sequential testing for determining if concentrations of a constituent of concern in soil at a closed waste management unit are below a risk-based action level (AL).

In sequential sampling, the number of samples is not fixed *a priori*; rather, a statistical test is performed after each analysis to arrive at one of three possible decisions: reject the hypothesis, accept the hypothesis, or perform another analysis. This strategy is applicable when sampling and/or analyses are quite expensive, when information concerning sampling and/or measurement variability is lacking, when the waste and site characteristics of interest are stable over the time frame of the sampling effort, or when the objective of the sampling effort is to test a specific hypothesis. It may not be especially useful if multiple waste characteristics are of interest or if rapid decision making is necessary.

In planning for a sequential sampling program, the following considerations are important:

- Pre-planning the effort between the field and laboratory, including developing a system of pre-planned paperwork and sample containers
- Arranging for a system of rapid delivery of samples to the laboratory
- Providing rapid turnaround in the laboratory
- Rapidly returning data to the planners, supervisors, and others responsible for decision making.

If the sequential sampling program is carried out using field methods (e.g., portable detectors), much of the inconvenience involved with shipping and return of results can be avoided.

5.2.6 Authoritative Sampling

Authoritative sampling is a nonstatistical sampling design because it does not assign an equal probability of being sampled to all portions of the population. This type of sampling should be considered only when the objectives of the investigation do not include the estimation of a population parameter. For example, authoritative sampling might be appropriate when the objective of a study is to identify specific locations of leaks, or when the study is focused solely on the sampling locations themselves. The validity of the data gathered with authoritative sampling is dependent on the knowledge of the sampler and, although valid data sometimes can be obtained, it is not recommended for the chemical characterization of wastes when the parameter of interest (such as the mean) is near the action level.

Authoritative sampling (also known as judgmental sampling, biased sampling, nonprobability sampling, nonstatistical sampling, purposive sampling, or subjective sampling) may be appropriate under circumstances such as the following:

- You need preliminary information about a waste stream or site to facilitate planning or to gain familiarity with the waste matrix for analytical purposes.
- You are conducting sampling for a RCRA Facility Assessment (RFA) to identify a potential or actual release to the environment.
- You have encountered a spill of an unknown chemical and need to determine the chemical makeup of the spilled material.
- You have access to only small portions of the population and judgment is applied to assess the usefulness of samples drawn from the small portion.
- You are screening samples in the field, using an appropriate field method, to identify “hot” samples for subsequent analysis in a laboratory.
- You are sampling to support case development for an enforcement agency or to “prove the positive” (see also Section 2.2.4).

With authoritative sampling, it is not possible to accurately estimate the population variance. Also, due to its subjective nature, the use of authoritative sampling by the regulated community to demonstrate compliance with regulatory standards generally is not advisable except in those cases in which a small volume of waste is in question or where the concentration is either well above or well below the regulatory threshold.

The ASTM recognizes two types of authoritative sampling: judgmental sampling and biased sampling (ASTM D 6311).

5.2.6.1 Judgmental Sampling

Judgmental sampling is a type of authoritative sampling. The goal of judgmental sampling is to use process or site knowledge to choose one or more sampling locations to represent the “average” concentration or “typical” property.

Judgmental sampling designs can be cost-effective *if* the people choosing the sampling locations have sufficient knowledge of the waste. If the people choosing the sampling locations intentionally distort the sampling by a prejudiced selection, or if their knowledge is wanting, judgmental sampling can lead to incorrect and sometimes very costly decisions. Accurate and useful data can be generated from judgmental sampling more easily if the population is relatively homogeneous and the existence of any strata and their boundaries is known.
The disadvantages of judgmental sampling designs follow:

- It can be difficult to demonstrate that prejudice was not employed in sampling location selection

- Variances calculated from judgmental samples may be poor estimates of the actual population variance

- Population statistics cannot be generated from the data due to the lack of randomness.

An example application of judgement sampling is given in Appendix C of *Guidance for the Data Quality Objectives Process for Hazardous Waste Site Operations* (USEPA 2000a).

5.2.6.2 Biased Sampling

Biased sampling is the type of authoritative sampling that intends not to estimate average concentrations or typical properties, but to estimate "worst" or "best" cases (ASTM D 6051-96). The term "biased," as used here, refers to the collection of samples with expected very high or very low concentrations. For example, a sample taken at the source of a release could serve as an estimate of the "worst-case" concentration found in the affected media. This information would be useful in identifying the constituent of concern and estimating the maximum level of contamination likely to be encountered during a cleanup.

At times, it may be helpful to employ a "best case" or both a "best-case" and "worst-case" biased sampling approach. For example, if there is a range of wastes and process knowledge can be used to identify the wastes likely to have the lowest and highest contamination levels, then these two extremes could be sampled to help define the extent of the problem.

Biased sampling, while having the ability to cost-effectively generate information, has similar disadvantages to that of judgmental sampling.

5.3 Composite Sampling

Composite sampling is a strategy in which multiple individual or "grab" samples (from different locations or times) are physically combined and mixed into a single sample so that a physical, rather than a mathematical, averaging takes place.[1] Figure 17 illustrates the concept of composite samples. For a well-formed composite, a single measured value should be similar to the mean of measurements of the individual components of the composite (Fabrizio, et al. 1995). Collection of multiple composite samples can provide improved sampling precision and reduce the total number of analyses required compared to noncomposite sampling. This strategy is sometimes employed to reduce analysis costs when analysis costs are large relative to sampling costs. The appropriateness of using composite sampling will be highly dependent on the DQOs (Myers 1997), the constituent of concern, and the regulatory requirements. *To realize the full benefits of composite sampling, field and laboratory personnel must carefully*

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[1] Some authors use the term "discrete sample" to refer to an individual sample that is used to form a composite sample. The RCRA regulations often use the term "grab sample." For the purpose of this guidance, the terms "discrete," "grab," and "individual" sample have the same meaning.

follow correct procedures for sample collection, mixing, and subsampling (see Sections 6 and 7).

5.3.1 Advantages and Limitations of Composite Sampling

A detailed discussion of the advantages and limitations of composite sampling is presented in the *Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities* (ASTM D 6051-96) and EPA's *Guidance for Choosing a Sampling Design for Environmental Data Collection, EPA QA/G-5S* (USEPA 2000c). Additional information on composite sampling can be found in Edland and van Belle (1994), Gilbert (1987), Garner, et al. (1988 and 1989), Jenkins, et al. (1996 and 1997), Myers (1997), and USEPA (1995c).

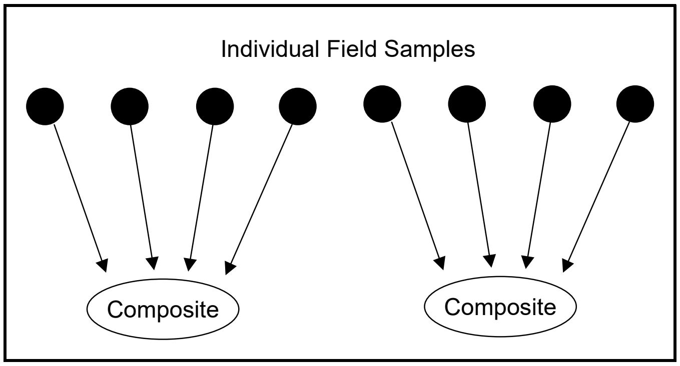

Figure 17. Forming composite samples from individual samples (from USEPA 1995c).

Advantages

Three principal advantages to using composite sampling (see ASTM D 6051-96) follow:

- It can improve the precision (i.e., reduce between-sample variance) of the estimate of the mean concentration of a constituent in a waste or media (see Section 5.3.5)
- It can reduce the cost of estimating a mean concentration, especially in cases in which analytical costs greatly exceed sampling costs or in which analytical capacity is limited
- A "local" composite sample, formed from several increments obtained from a localized area, is an effective way to increase the sample support, which reduces grouping and segregation errors (see also Section 6.2.2.2)
- It can be used to determine whether the concentration of a constituent in one or more individual samples used to form a composite might exceed a fixed standard (i.e., is there a "hot spot"?) (see Section 5.3.6).

Limitations

Composite sampling should not be used if the integrity of the individual sample values changes because of the physical mixing of samples (USEPA 1995c). The integrity of individual sample values could be affected by chemical precipitation, exsolvation, or volatilization during the pooling and mixing of samples. For example, volatile constituents can be lost upon mixing of samples or interactions can occur among sample constituents. In the case of volatile constituents, compositing of individual sample extracts within a laboratory environment may be a reasonable alternative to mixing individual samples as they are collected.

Listed below are some additional conditions under which compositing usually is *not* advantageous:

- When regulations *require* the use of discrete or grab samples. For example, compliance with the LDR numeric treatment standards for non-wastewaters typically is to be determined using "grab" samples rather than composite samples. Grab samples processed, analyzed, and evaluated individually normally reflect maximum process variability, and thus reasonably characterize the range of treatment system performance. Typically, grab samples are used to evaluate LDR non-wastewaters and composite samples are used to evaluate LDR wastewaters, except when evaluating wastewaters for metals (D004 through D011) for which grab samples are required [40 CFR 268.40(b)].

- When data users require specific data points to generate high-end estimates or to calculate upper percentiles

- When sampling costs are much greater than analytical costs

- When analytical imprecision outweighs sampling imprecision and population heterogeneity

- When individual samples are incompatible and may react when mixed

- When properties of discrete samples, such as pH or flash point, may change qualitatively upon mixing. (Compositing of individual samples from different locations to be tested for hazardous waste characteristic properties, such as corrosivity, reactivity, ignitability, and toxicity, is not recommended)

- When analytical holding times are too short to allow for analysis of individual samples, *if* testing of individual samples is required later (for example, to identify a "hot" sample) (see Section 5.3.6)

- When the sample matrix impedes correct homogenization and/or subsampling

- When there is a need to evaluate whether the concentrations of different contaminants are correlated in time or space.

5.3.2 Basic Approach To Composite Sampling

The basic approach to composite sampling involves the following steps:

- Identify the boundaries of the waste or unit. The boundaries may be spatial, temporal, or based on different components or strata in the waste (such as battery casings and soil)

- Conduct sampling in accordance with the selected sampling design and collect a set of n x g individual samples where g is the number of individual samples used to form each composite and n is the number of such composites

- Group either randomly or systematically the set of n x g individual samples into n composite samples and thoroughly mix and homogenize each composite sample

- Take one or more subsamples from each composite

- Analyze each subsample for the constituent(s) of concern.

The n composite samples can then be used to estimate the mean and variance (see Section 5.3.5) or identify "hot spots" in the waste (see Section 5.3.6).

5.3.3 Composite Sampling Designs

Composite sampling can be implemented as part of a statistical sampling design, such as simple random sampling and systematic sampling. The choice of a sampling design to use with compositing will depend upon the study objectives.

5.3.3.1 Simple Random Composite Sampling

Figure 18 shows how composite sampling can be integrated into a simple random sampling design. In this figure, the decision unit could represent any waste or media about which a decision must be made (such as a block of contaminated soil at a SWMU). Randomly positioned field samples are randomly grouped together into composite samples. The set of composite samples can then be used to estimate the mean and the variance.

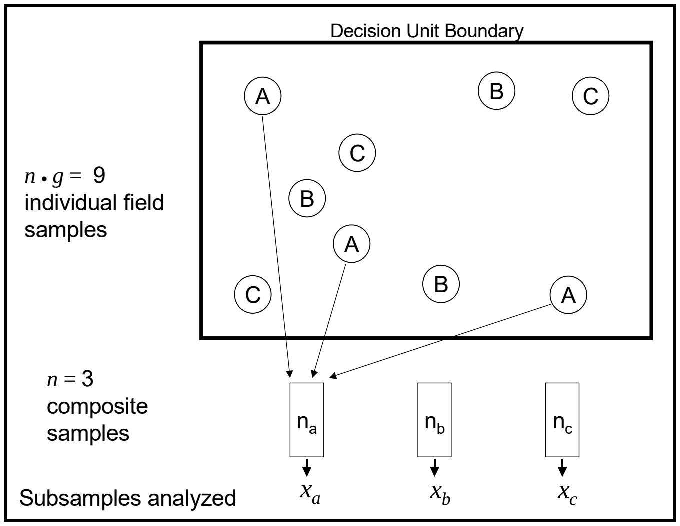

Figure 18. A basic approach to composite sampling. The figure shows how composite sampling can be integrated into a simple random sampling design. Random samples with the same letter are randomly grouped into composite samples to obtain an estimate of the unit-wide mean.

Because the compositing process is a mechanical way of averaging out variabilities in concentrations from location to location over a unit, the resulting concentration data should tend to be more normally distributed than individual samples (Exner, et al. 1985). This is especially advantageous because the assumption of many statistical tests is that the underlying data exhibit an approximately normal distribution.[2]

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[2] By the Central Limit Theorem (CLT), we expect composite samples to generate normally distributed data. The CLT states that if a population is repeatedly sampled, the means of all the sampling events will tend to form a normal distribution, regardless of the shape of the underlying distribution.

5.3.3.2 Systematic Composite Sampling

A systematic composite sampling design is shown in Figure 19. The design can be used to estimate the mean concentration because each composite sample is formed from field samples obtained across the entire unit. For example, each field sample collected at the "A" locations is pooled and mixed into one composite sample. The process is then repeated for the "B," "C," and "D" locations. The relative location of each individual field sample (such as "A") should be the same within each block.

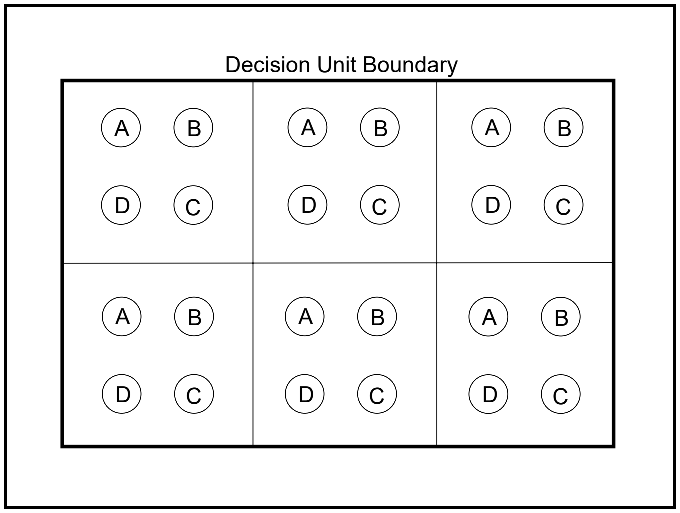

Figure 19. Systematic composite sampling across a unit or site. Samples with the same letter are pooled into composites.

This design is particularly advantageous because it is easy to implement and explain and it provides even coverage of the unit. Exner, et al. (1985) demonstrated how this design was used to make cleanup decisions for blocks of soil contaminated with tetrachlorodibenzo-p-dioxin.

A second type of systematic composite involves collecting and pooling samples from *within* grid blocks, time intervals, or batches of waste grouped together (see Figure 20).

If there is spatial correlation between the grid blocks, compositing within grids can be used to estimate block-to-block variability (Myers 1997) or improve the estimate of the mean within a block or interval (if multiple composite samples are collected within each block). In fact, compositing samples collected from localized areas is an effective means to control "short-range" (small-scale) heterogeneity (Pitard 1993). When this type of compositing is used on localized areas in lieu of "grab" sampling, it is an attractive option to improve representativeness of individual samples (Jenkins, et al. 1996).

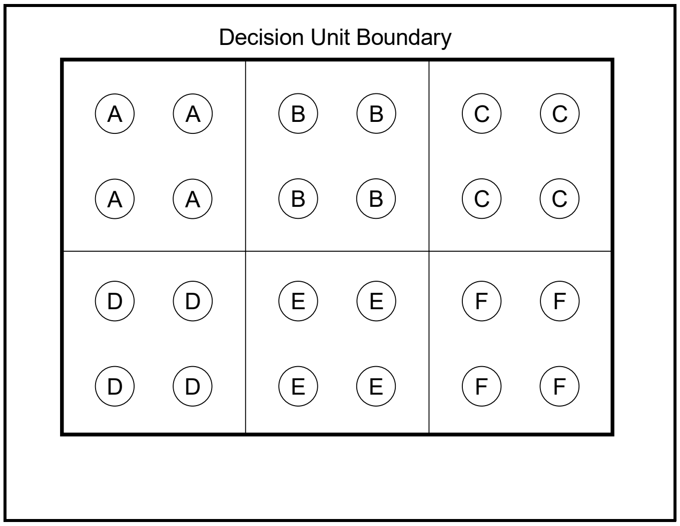

Figure 20. Systematic sampling within grid blocks or intervals. Samples with the same letter are pooled into a composite sample.

Systematic sampling within time intervals could be used in cases in which compositing occurs as part of sample collection (such as sampling of liquid effluent with an autosampling device into a single sample container over a specified time period).

If the individual field sample locations are independent (that is, they have no temporal or spatial correlation), then compositing within blocks can be an efficient strategy for estimating the population mean. If the assumption of sample independence cannot be supported, then an alternative design should be selected if the objective is to estimate the mean.

5.3.4 Practical Considerations for Composite Sampling

In creating composite samples from individual field samples, it is possible that a relatively large volume of material will need to be physically mixed at some point -- either in the field or in the laboratory. Thorough mixing is especially important when the individual samples exhibit a high degree of heterogeneity.

Once the individual samples are mixed, one or more subsamples must be taken because the entire composite sample usually cannot be analyzed directly. A decision must be made as to *where* the individual samples will be combined into the composite samples. Because large samples (e.g., several kilograms or more) may pose increased difficulties to the field team for containerization and shipping and pose storage problems for the laboratory due to limited storage space, there may be a distinct advantage to performing mixing or homogenization in the field. There are, however, some disadvantages to forming the composite samples in the field. As pointed out by Mason (1992), the benefits of homogenization may be temporary because gravity induced segregation can occur during shipment of the samples. Unless homogenization (mixing), particle size reduction, and subsampling are carried out immediately prior to analysis, the benefits of these actions may be lost. Therefore, if practical, it may be best to leave the mixing and subsampling operations to laboratory personnel.

See Section 7.3 of this document and ASTM standards D 6051 and D 6323 for guidance on homogenization, particle size reduction, and subsampling.

5.3.5 Using Composite Sampling To Obtain a More Precise Estimate of the Mean

When analytical error is minor compared to sampling error, then composite sampling can be a resource-efficient mechanism for increasing the precision of estimates of the population mean. If composite sampling is to be used to estimate the mean with a specified level of confidence, then multiple composite samples can be used to estimate the mean and variance. Alternately, confidence limits can be constructed around the sample analysis result for a single composite sample if an estimate of the variance of the fundamental error is available (see Gy 1998, page 73).[3] See Section 6.2.2.1 for a discussion of fundamental error.

The population mean (μ) can be estimated from the analysis of n composite samples (each made from g individual samples). The population mean (μ) is estimated by the sample mean ($\bar{x}$) by

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i \qquad \textbf{Equation 6}$$

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[3] ASTM D 6051, *Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities*, also provides a procedure for estimating the precision of a single composite sample.

The sample variance (s^2) can then be calculated by

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2 \qquad \textbf{Equation 7}$$

Note that Equations 6 and 7 are the same as Equations 1 and 2, respectively, for the mean and variance. When the equations are used for composite sampling, x_i is the measurement value from a subsample taken from each n composite sample rather than each individual sample. Use of these equations assumes equal numbers of individual field samples (g) are used to form each composite, and equal numbers of subsamples are taken from each composite sample and analyzed. If these assumptions are not correct, an alternative approach described in Gilbert (1987, page 79) can be used.

By increasing the number of individual field samples (g) per composite sample, there will be a corresponding decrease in the standard error ($s_{\bar{x}}$), thus improving the precision of the estimate of the mean. Edland and van Belle (1994) show that by doubling the number of individual samples per composite (or laboratory) sample, the expected size of the confidence interval around the mean decreases by a factor of $1/\sqrt{2}$, which is a 29-percent decrease in the expected width of the confidence interval. One of the key assumptions underlying the above discussion is that variances between the samples greatly exceed the random error variance of the analytical method (Garner, et al. 1988).

Williams, et al. (1989) demonstrated the benefits of using composite sampling to obtain a more precise estimate of the mean. One of their objectives was to study the efficiency of using composite sampling as compared to collecting individual samples for the purpose of estimating the mean concentration at a site. Five sites known to have radium contamination in shallow soils were extensively sampled. At each site, shallow soil samples were collected at approximately uniformly spaced points over the entire site. Three types of samples were taken: (1) individual 500-gram samples, (2) composite samples consisting of ten 50-gram aliquots uniformly spaced over the site, and (3) composite samples consisting of twenty 25-gram aliquots uniformly spaced over the site. The samples were measured for ^{226}Ra. The results indicated the individual samples yielded the least precision, even when more than twice as many individual samples were collected. Sixty-six individual samples produced a standard error of 1.35, while the thirty 10-aliquot composites and the thirty 20-aliquot composite samples produced standard errors of 0.76 and 0.51 respectively. The results demonstrate that composite sampling can produce more precise estimates of the mean with fewer analytical samples.

Box 7 provides an example of how a mean and variance can be estimated using composite sampling combined with systematic sampling.

Box 7. Example of How To Estimate the Mean and Variance Using Systematic Composite Sampling (Assume Samples Are Independent)

Under 40 CFR 261.38, a generator of hazardous waste-derived fuel is seeking an exclusion from the definition of solid and hazardous-waste. To prepare the one-time notice under 40 CFR 261.38(c), the generator requires information on the mean and variance of the concentrations of constituents of concern in the waste as generated. The generator elects to use composite samples to estimate the mean and variance of the nonvolatile constituents of concern.

Using a systematic sampling design, a composite sample is prepared by taking an individual (grab) sample at regular time intervals t_1 through t_4. The set of four grab samples are thoroughly mixed to form a composite, and one subsample is taken from each composite for analysis. The process is repeated until five composite samples are formed (see Figure 21). (*Note*: If the assumption of independent samples cannot be supported, then a simple random design should be used in which the 20 grab samples are randomly grouped to form the five composites).

The analytical results for one of the constituents of concern, in ppm, are summarized as follows for the composite samples (n_1 through n_5):
2.75, 3.71, 3.28, 1.95, and 5.10.

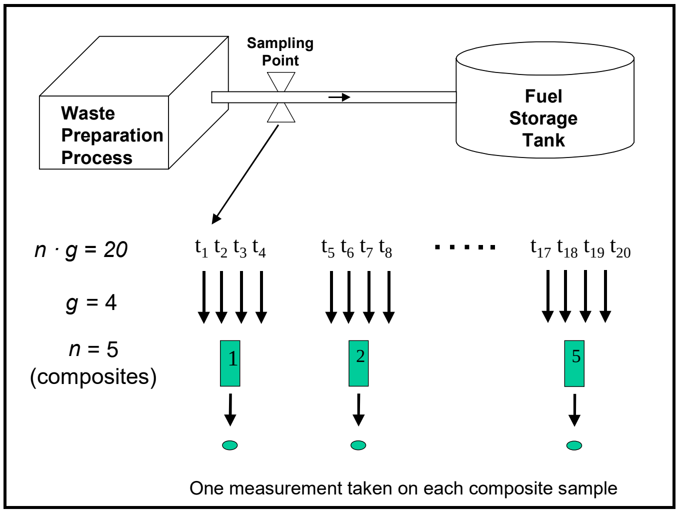

Figure 21. Example of systematic composite sampling

Using Equations 6 and 7 for the mean and variance of composite samples, the following results are obtained:

$$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i = \frac{16.79}{5} = 3.36\,ppm$$

$$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2 = \frac{1}{4}\left[0.3721 + 0.1225 + 0.0064 + 1.99 + 3.03\right] = 1.38$$

The standard error is obtained as follows:

$$s_{\bar{x}} = \frac{s}{\sqrt{n}} = \frac{1.17}{\sqrt{5}} = 0.52\,ppm$$

5.3.6 Using Composite Sampling To Locate Extreme Values or “Hot Spots”

One disadvantage of composite sampling is the possibility that one or more of the individual samples making up the composite could be “hot” (exceed a fixed standard), but remain undetected due to dilution that results from the pooling process. If the sampling objective is to determine if any one or more individual samples is “hot,” composite sampling can still be used.

A procedure for detecting hot spots using composite sampling is given below. The approach assumes the underlying distribution is normal and the composite samples were formed from equal-sized individual samples.

Let AL be some "action level" or regulatory threshold that cannot be exceeded in an individual sample. Note that AL must be large relative to the quantitation limit for the constituent of concern. For a measurement x_i from a composite sample formed from g individual samples, the following rules apply, assuming analytical and sampling error are negligible:

- If $x_i < \frac{AL}{g}$, then no single individual sample can be $> AL$
- If $x_i > AL$, then at least one *must*, and as many as all individual samples *may*, be $> AL$
- If $x_i > \frac{AL}{g}$, then at least one of the g individual samples must be $> AL$.

As a general rule, we can say that no more than $\frac{g \cdot x_i}{AL}$ individual samples can be $> AL$.

If one or more of the composites are "hot" (i.e., $> AL$), then it might be desirable to go back and analyze the individual samples used to form the composite. Consider saving splits of each individual field sampling so individual samples can be analyzed later, if needed.

If compositing is used to identify a hot spot, then the number of samples that make up the composite should be limited to avoid overall dilution below the analytical limit. It is possible for a composite sample to be diluted to a concentration below the quantitation limit if many of the individual samples have concentrations near zero and a single individual sample has a concentration just above the action level. Mason (1992) and Skalski and Thomas (1984) suggest the maximum number of identically sized individual samples (g) that can be used to form such a composite should not exceed the action level (AL) divided by the quantitation limit (QL). But the relationship of $g \leq AL / QL$ indicates that the theoretical maximum number of samples to form a composite can be quite high, especially given a very low quantitation limit. As a practical matter, the number of individual samples used to form a composite should be kept to a minimum (usually between 2 and 10).

An example of the above procedure, provided in Box 8, demonstrates how a "hot" drum can be identified through the analysis of just nine samples (five composites plus four individual analyses), resulting in considerable savings in analytical costs over analysis of individual samples from each of the 20 drums.

Box 8. How To Locate a "Hot Spot" Using Composite Sampling - Hypothetical Example

A secondary lead smelter produces a slag that under some operating conditions exhibits the Toxicity Characteristic (TC) for lead. At the point of generation, a grab sample of the slag is taken as the slag is placed in each drum. A composite sample is formed from the four grab samples representing a set of four drums per pallet. The process is repeated until five composite samples representing five sets of four drums (20 drums total) have been prepared (see Figure 22).

The generator needs to know if the waste in any single drum in a given set of four drums contains lead at a total concentration exceeding 100 ppm. If the waste in any single drum exceeds 100 ppm, then its maximum theoretical TCLP leachate concentration could exceed the regulatory limit of 5 mg/L. Waste in drums exceeding 100 ppm total lead will be tested using the TCLP to determine if the total leachable lead equals or exceeds the TC regulatory limit.

The sample analysis results for total lead are measured as follows (in ppm) in composite samples n_1 through n_5:
6, 9, 18, 20, and 45.

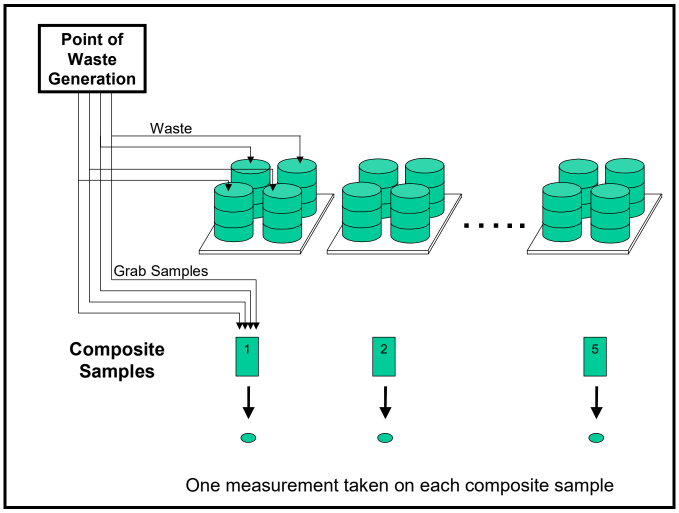

Figure 22. Composite sampling strategy for locating a "hot" drum

Using the approach for locating a "hot spot" in a composite sample, we observe that all of the composite samples except for n_5 are less than AL / g or 100 ppm/4 (i.e., 25 ppm). The result for n_5 (45 ppm) is greater than 25 ppm, indicating a potential exceedance of the TC regulatory level. A decision about the set of drums represented by n_5 can be made as follows:

No more than $\frac{g \cdot x_i}{AL}$ individual samples can be $> AL$, or no more than $\frac{(4)45\,ppm}{100\,ppm} = 1.8$ or 1 (round *down*) individual sample exceeds 100 ppm total lead.

We now know that it is possible that one of the four drums on the fifth palette exceeds 100 ppm, but we do not know *which* one. As a practical matter, analysis of all four of the individual samples should reveal the identity of the "hot" drum (if, indeed, one exists); however, the above process of elimination could be repeated on two new composite samples formed from samples taken from just the four drums in question.

5.4 Determining the Appropriate Number of Samples Needed To Estimate the Mean

This section provides guidance for determining the appropriate number of samples (n) needed to estimate the mean. The procedures can be used when the objective is to calculate a confidence limit on the mean. If the objective is to estimate a percentile, see Section 5.5.

To calculate the appropriate number of samples, it is necessary to assemble existing data identified in DQO Step 3 ("Identify Inputs to the Decision") and Step 6 ("Specify Limits on Decision Errors"). If the parameter of interest is the mean, you can calculate n using equations presented in the following sections or by using EPA's DEFT software (USEPA 2001a).

Alternative equations can be found in the statistical literature and guidance, including ASTM (Standard D 6311), Cochran (1977), Gilbert (1987), and USEPA (2000a, 2000b, and 2000d).

The equations presented here should yield the approximate minimum number of samples needed to estimate the mean within the precision and confidence levels established in the DQO Process; however, *it is prudent to collect a somewhat greater number of samples than indicated by the equations*.[4] This is recommended to protect against poor preliminary estimates of the mean and standard deviation, which could result in an underestimate of the appropriate number of samples to collect. For analytes with long holding times (e.g., 6 months), it may be possible to process and store extra samples appropriately until analysis of the initially identified samples is completed and it can be determined if analysis of the additional samples is warranted.

It is important to note that the sample size equations do not account for the number or type of control samples (or quality assessment samples) required to support the QC program associated with your project. Control samples may include blanks (e.g., trip, equipment, and laboratory), field duplicates, spikes, and other samples used throughout the data collection process. Refer to Chapter One of SW-846 for recommendations on the type and number of control samples needed to support your project. It is best to first determine how each type of control sample is to be used, then to determine the number of that type based on their use (van Ee, et al. 1990).

A key assumption for use of the sample size equations is that you have some prior estimate of the total study error, measured as the sample standard deviation (s) or sample variance (s^2). Since total study error includes variability associated with the sampling and measurement methods (see Section 6), it is important to understand the relative contributions that sampling and analysis activities make to the overall estimate of variability. Lack of prior information regarding population and measurement variability is one of the most frequently encountered difficulties in sampling. It quickly resembles a "chicken-and-the-egg" question for investigators – you need an estimate of the standard deviation to calculate how many samples you need, yet you cannot derive that estimate without any samples. To resolve this seemingly paradoxical question, two options are available:

Option 1. *Conduct a pilot study*. A pilot study (sometimes called an exploratory or preliminary study) is the preferred method for obtaining estimates of the mean and standard deviation, as well as other relevant information. The pilot study is simply phase one of a multi-phase sampling effort (Barth, et al. 1989). For some pilot studies, a relatively small number of samples (e.g., four or five or more) may provide a suitable *preliminary estimate* of the standard deviation.

Option 2. *Use data from a study of a similar site or waste stream*. In some cases, you might be able to use sampling and analysis data from another facility or similar operation that generates the same waste stream and uses the same process.

If neither of the above options can provide a suitable estimate of the standard deviation (s), a crude approximation of s still can be obtained using the following approach adopted from

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[4] One exception is when sequential sampling is used in which the number of samples is not fixed *a priori*; rather, the statistical test is performed after each round of sampling and analysis (see Section 5.2.5).

USEPA 1989a (page 6-6). The approximation is based on the *judgment* of a person knowledgeable of the waste and his or her *estimate* of the range within which constituent concentrations are likely to fall. Given a range of constituent concentrations in a waste, but lacking the individual data points, an *approximate* value for s may be computed by dividing the *range* (the estimated maximum concentration minus the minimum concentration) by 6, or $s \approx Range / 6$. This approximation method should be used only if no other alternative is available. The approach is based on the assumption that more than 99 percent of all normally distributed measurements will fall within three standard deviations of the mean; therefore, the length of this interval is $6s$.

5.4.1 Number of Samples to Estimate the Mean: Simple Random Sampling

In Step 6 of the DQO Process ("Specify Limits on Decision Errors"), you established the width of the gray region (Δ) and acceptable probabilities for making a decision error (α and β). Using this information, along with an estimate of the standard deviation (s), calculate the appropriate number of samples (n) for simple random sampling using

$$n = \frac{(z_{1-\alpha} + z_{1-\beta})^2 s^2}{\Delta^2} + \frac{z_{1-\alpha}^2}{2}$$

Equation 8

where

$z_{1-\alpha}$ = the pth quantile of the standard normal distribution (from the last row of Table G-1, Appendix G), where α is the probability of making a Type I set in DQO Step 6 (Section 4.6.4).

$z_{1-\beta}$ = the pth quantile of the standard normal distribution (from the last row of Table G-1, Appendix G), where β is the probability of making a Type II error set in DQO Step 6 (Section 4.6.4).

s = an estimate of the standard deviation.

Δ = the width of the gray region from DQO Step 6.

An example application of Equation 8 is presented in Box 9.

Two assumptions underlie the use of Equation 8. First, it is assumed that data are drawn from an approximately normal distribution. Second, it is assumed the data are uncorrelated. In correlated data, two or more samples taken close to each other (in time or in space) will have similar concentrations (Gilbert 1987). In situations in which spatial or temporal correlation is expected, some form of systematic sampling is preferred.

If the underlying population appears to exhibit a lognormal distribution, normal theory sample size equations (such as Equation 8) still can be used though they will tend to *underestimate* the minimum number of samples when the geometric standard deviation ($\exp(s_y)$) is low (e.g., ≤ 2). If the underlying distribution is known to be lognormal, the method given by Land (1971, 1975) and Gilbert (1987) for calculating confidence limits for a lognormal mean can be solved "in reverse" to obtain n. (A software tool for performing the calculation, MTCA*Stat* 3.0, is published by the Washington Department of Ecology. See Appendix H). Also, techniques described by Perez and Lefante (1996 and 1997) can be used to estimate the sample sizes needed to estimate the mean of a lognormal distribution. Otherwise, consult a professional statistician for assistance.

Box 9. Number of Samples Required to Estimate the Mean Using Simple Random Sampling: Hypothetical Example

Under 40 CFR 261.38, a generator of hazardous waste-derived fuel is seeking an exclusion from the definition of solid and hazardous-waste. To prepare the one-time notice under 40 CFR 261.38(c), the generator plans to conduct waste sampling and analysis to support the exclusion. The output of the first six steps of the DQO Process are summarized below:

Step 1: State the Problem: The planning team reviewed the applicable regulations, historical analyses, and process chemistry information. The problem is to determine whether Appendix VIII constituents present in the waste are at concentration levels less than those specified in Table 1 of §261.38.

Step 2: Identify the Decision: If the waste attains the specification levels, then it will be judged eligible for the exclusion from the definition of hazardous and solid waste.

Step 3: Identify Inputs to the Decision: Sample analysis results are required for a large number of constituents present in the waste, however, most constituents are believed to be present at concentrations well below the specification levels. Historically, benzene concentrations have been most variable, therefore, the planning team will estimate the number of samples required to determine if the specification level for benzene is attained.

Step 4: Define the Boundaries: The DQO decision unit is defined as the batch of waste generated over a one-week period. Samples will be taken as the waste exits the preparation process and prior to storage in a fuel tank (i.e., at the point of generation).

Step 5: Develop a Decision Rule: The RCRA regulations at 40 CFR 261.38(c)(8)(iii)(A) specify the mean as the parameter of interest. The "Action Level" for benzene is specified in Table 1 of §268.38 as 4,100 ppm. If the mean concentration of benzene within the DQO decision unit is less than or equal to 4,100 ppm, then the waste will be considered eligible for the exclusion (for benzene). Otherwise, the waste will not be eligible for the exclusion for benzene. (Note that the demonstration must be made for all Appendix VIII constituents known to be present in the waste).

Step 6: Specify Limits on Decision Errors: In the interest of being protective of the environment, the null hypothesis was established as "the mean concentration of benzene within the decision unit boundary exceeds 4,100 ppm," or H_o: mean (benzene) > 4,100 ppm. The boundaries of the gray region were set at the Action Level (4,100 ppm) and at a value less than the Action Level at 3000 ppm. The regulations at §261.38(c)(8)(iii)(A) specify a Type I (false rejection) error rate (α) of 0.05. The regulations do not specify a Type II (false acceptance) error rate (β), but the planning team deemed a false acceptance as of lesser concern than a false rejection, and set the false acceptance rate at 0.25. Sample analysis results from previous sampling and analyses indicate the standard deviation (s) of benzene concentrations is about 1,200 ppm.

What is the appropriate number of samples to collect and analyze for a simple random sampling design?

Solution: Using Equation 8 and the outputs of the first six steps of the DQO Process, the number of samples is determined as:

$$n = \frac{(z_{1-\alpha} + z_{1-\beta})^2 s^2}{\Delta^2} + \frac{z_{1-\alpha}^2}{2}$$

$$= \frac{(1.645 + 0.674)^2 (1200)^2}{(4100 - 3000)^2} + \frac{(1.645)^2}{2} = 7.75 \approx 8 \text{ (round up)}$$

where the values for $z_{1-\alpha}$ and $z_{1-\beta}$ are obtained from the last row of Table G-1 in Appendix G.

5.4.2 Number of Samples to Estimate the Mean: Stratified Random Sampling

An important aspect of a stratified random sampling plan is deciding how many samples to collect within each of the strata (Gilbert 1987). There are many ways to design a stratified random sampling plan; the development here makes the following assumptions (refer to Section 5.2.2 for a description of terms and symbols used below):

- Weights for each stratum (W_h) are known in advance. One possible way to assign weights to each stratum is to calculate the ratio between the waste volume classified as the $h\text{th}$ stratum and the total waste volume.
- The number of *possible* sample units (i.e., physical samples) of a certain physical size is much larger than the number of sample units that will be collected and analyzed. As a general guide, this assumption should be reasonable as long as the ratio between the stratum waste volume and the volume of the individual samples is at least 100. Otherwise, you may need to consider formulas that include the finite population correction (see Cochran 1977, page 24).
- The number of sample units to be collected and analyzed in each stratum, due to analytical costs and other considerations, generally will be fairly small.
- A preliminary estimate of variability (s_h^2) is available for each stratum. If this is not the case, one can use an estimate of the overall variability (s^2) as a substitute for the separate stratum estimates. By ignoring possible differences in the variance characteristics of separate strata, the sample size formulas given below may tend to underestimate the necessary number of samples for each strata (n_h).

Given a set of stratum weights and sample measurements in each stratum, the *overall* mean ($\bar{x}_{st}$) and *overall* standard error of the mean ($s_{\bar{x}_{st}}$) (i.e., for the entire waste under study) are computed as follows for a stratified random sample:

$$\bar{x}_{st} = \sum_{h=1}^{L} W_h \bar{x}_h$$ **Equation 9**

and

$$s_{\bar{x}_{st}} = \sqrt{\sum_{h=1}^{L} W_h^2 \frac{s_h^2}{n_h}}$$ **Equation 10**

Note that $\bar{x}_h$ and s_h^2 in these formulas represent the arithmetic mean and sample variance for the measurements taken within each stratum.

In general, there are two approaches for determining the number of samples to take when stratified random sampling is used: *optimal allocation* and *proportional allocation*.

5.4.2.1 Optimal Allocation

In optimal allocation, the number of samples assigned to a stratum (n_h) is proportional to the relative variability within each stratum and the relative cost of obtaining samples from each stratum. The number of samples can be determined to minimize the variance for a fixed cost or to minimize the cost for a prespecified variance.

Optimal allocation requires considerable advance knowledge about the relative variability within each stratum and the costs associated with obtaining samples from each stratum; therefore, we recommend the use of proportional allocation (see below) as an alternative. For more complex situations in which optimal allocation is preferred, consult a statistician or see Cochran (1977, page 96), Gilbert (1987, page 50), or USEPA (1989a (page 6-13)).

5.4.2.2 Proportional Allocation

In proportional allocation, the number of samples assigned to a stratum (n_h) is proportional to the stratum size, that is, $n_h = nW_h$. To determine the total number of samples (n) so that a true difference (Δ) between the mean waste concentration and the Action Level can be detected with Type I error rate α and Type II error rate β, use the following equation:

$$n = \frac{\left[t_{1-\alpha,df} + t_{1-\beta,df}\right]^2}{\Delta^2} \sum_{h=1}^{L} W_h s_h^2$$ **Equation 11**

To use this formula correctly, the degrees of freedom (df) connected with each t-quantile (from Table G-1, Appendix G) in the above equation must be computed as follows:

$$df = \left(\sum_{h=1}^{L} W_h s^2 \right)^2 \Bigg/ \sum_{h=1}^{L} \frac{W_h^2 s_h^4}{nW_h - 1}$$ **Equation 12**

Because the degrees of freedom also depend on n, the final number of samples must be computed iteratively. Then, once the final total number of samples is computed, the number of samples for each stratum is determined by multiplying the total number of samples by the stratum weight. An example of this approach is presented in Box 10.

If only an *overall* estimate of s^2 is available in the preliminary data, Equation 11 reduces to:

$$n = \frac{\left[t_{1-\alpha,df} + t_{1-\beta,df}\right]^2 s^2}{\Delta^2}$$ **Equation 13**

and Equation 12 reduces to

$$df = 1 \Bigg/ \sum_{h=1}^{L} \frac{W_h^2}{nW_h - 1}$$ **Equation 14**

Box 10. Number of Samples Required to Estimate the Mean Using Stratified Random Sampling – Proportional Allocation: Hypothetical Example

Under the RCRA Corrective Action program, a facility owner has conducted a cleanup of a solid waste management unit (SWMU) in which the contaminant of concern is benzene. The cleanup involved removal of all waste residues, contaminated subsoils, and structures. The facility owner needs to conduct sampling and analysis to confirm that the remaining soils comply with the cleanup standard.

Step 1: State the Problem: The planning team needs to confirm that soils remaining in place contain benzene at concentrations below the risk-based levels established by the authorized state as part of the cleanup.

Step 2: Identify the Decision: If the soils attain the cleanup standard, then the land will be used for industrial purposes. Otherwise, additional soil removal will be required.

Step 3: Identify Inputs to the Decision: A sampling program will be conducted, and sample analysis results for benzene will be used to make the cleanup attainment determination.

Step 4: Define the Boundaries: The DQO decision unit is the top 6 inches of soil within the boundary of the SWMU. Based on prior sample analysis results and field observations, two strata are identified: fine-grained soils in 20 percent of the unit ("Stratum 1"), and coarse-grained soils comprising the other 80 percent of the unit ("Stratum 2"). Based on the relative mass of the two strata, a weighting factor W_h is assigned to each $h\text{th}$ stratum such that $W_1 = 0.2$ and $W_2 = 0.8$.

Step 5: Develop a Decision Rule: The parameter of interest is established as the mean, and the Action Level for benzene is set at 1.5 mg/kg. If the mean concentration of benzene within the DQO decision unit is less than or equal to 1.5 mg/kg, then the unit will be considered "clean." Otherwise, another layer of soil will be removed.

Step 6: Specify Limits on Decision Errors: In the interest of being protective of the environment, the null hypothesis is established as "the mean concentration of benzene within the decision unit boundary exceeds 1.5 mg/kg," or Ho: mean (benzene) > 1.5 mg/kg. The boundaries of the gray region are set at the Action Level (1.5 mg/kg) and at a value less than the Action Level at 1.0 mg/kg. The Type I error rate (α) is set at 0.10 and the Type II error rate (β) is set at 0.25. Sample analysis results from $n = 8$ initial non-composite samples provided an estimate of the overall standard deviation of $s = 1.83$, and the standard deviations (s_h) within each $h\text{th}$ stratum of $s_1 = 2.5$ and $s_2 = 1.3$ (and $s_1^2 = 6.25$ and $s_2^2 = 1.69$).

What is the appropriate number of samples to collect and analyze for a stratified random sampling design?

Solution: Using Equation 12 for the degrees of freedom under proportional allocation:

$$df_1 = \left((0.2 \times 6.25) + (0.8 \times 1.69)\right)^2 \Big/ \left[\frac{(0.2 \times 6.25)^2}{8(0.2) - 1} + \frac{(0.8 \times 1.69)^2}{8(0.8) - 1}\right] = 2.3 \approx 2$$

Then, looking up the *t*-quantiles (from Table G-1, Appendix G) with 2 degree of freedom and taking $\Delta = 0.5$ (i.e., 1.5 ppm - 1.0 ppm), the total sample size (using Equation 12) works out to

$$n_1 = \frac{[1.886 + 0.816]^2}{(0.5)^2}\left((0.2 \times 6.25) + (0.8 \times 1.69)\right) = 76$$

Since the equations must be solved iteratively, recompute the formulas using $n = 76$. The same calculations give $df_2 = 48$ and $n_2 = 41$. After two more iterations, the sample size stabilizes at $n = 42$. Using the proportional allocation with $n = 42$ one should take 42(0.2) = 8.4 (round up to 9) measurements from the first stratum and 42(0.8) = 33.6 (round up to 34) measurements from the second stratum. Since four samples already were collected from each stratum, at least five additional random samples should be obtained from the first stratum and at least thirty additional random samples should be collected from the second stratum.

In the example in Box 10, stratified random sampling provides a more efficient and cost-effective design compared to simple random sampling of the same unit. If simple random sampling were used, a total of 52 samples would be required. With stratified random sampling, only 42 samples are required, thereby reducing sampling and analytical costs.

5.4.3 Number of Samples to Estimate the Mean: Systematic Sampling

Despite the attractiveness and ease of implementation of systematic sampling plans, whether via a fixed square, rectangular, or triangular grid, or through the use of systematic random sampling, methods for estimating the standard error of the mean are beyond the scope of this guidance (for example, see Cochran 1977) and often involve more advanced geostatistical techniques (for example, see Myers 1997). An alternate approach is to treat the set of systematic samples as though they were obtained using simple random sampling. Such an approach should provide reasonable results as long as there are no strong cyclical patterns, periodicities, or significant spatial correlations between adjacent sample locations. If such features are present or suspected to be present, consultation with a professional statistician is recommended.

By regarding the systematic sample as a simple random sample, one can simply use the algorithm and formulas for simple random sampling described in Section 5.4.1 (Equation 8) to estimate the necessary sample size. As with all the sampling designs described in this section, you should have a preliminary estimate of the sample variance before using the sample size equation.

5.4.4 Number of Samples to Estimate the Mean: Composite Sampling

In comparison to noncomposite sampling, composite sampling may have the effect of minimizing between-sample variation, thereby reducing somewhat the total number of composite samples that must be submitted for analysis.

The appropriate number of composite samples to be collected from a waste or media can be estimated by Equation 8 for simple random and systematic composite sampling. Equation 11 can be used when composite sampling will be implemented with a stratified random sampling design (using proportional allocation). Any preliminary or pilot study conducted to estimate the appropriate number of composite samples should be generated using the same compositing scheme planned for the confirmatory study. If the preliminary or pilot study data were generated using random "grab" samples rather than composites, then the sample variance (s^2) in the sample size equations should be replaced with s^2/g where g is the number of individual or grab samples used to form each composite (Edland and Van Belle 1994, page 45).

Additional guidance on the optimal number of samples required for composite sampling and the number of subsample aliquots required to achieve maximum precision for a fixed cost can be found in Edland and van Belle (1994, page 36 and page 44), Exner, et al. (1985, page 512), and Gilbert (1987, page 78).

5.5 Determining the Appropriate Number of Samples to Estimate A Percentile or Proportion

This section provides guidance for determining the appropriate number of samples (n) needed to estimate an upper percentile or proportion with a prespecified level of confidence. The approaches can be used when the objective is to determine whether the upper percentile is less than a concentration standard or whether a given proportion of the population or decision unit is less than a specified value.

Two methods for determining the appropriate number of samples are given below: (1) Section 5.5.1 provides a method based on the assumption that the population is large and the samples are drawn at random from the population, and (2) Section 5.5.2 provides a method with similar assumptions but only requires specification of the level of confidence required and the number of exceedances allowed (usually zero). For both methods, it is assumed that the measurements can be expressed as a binary variable – that is, that the sample analysis results can be interpreted as either in compliance with the applicable standard (“pass”) or not in compliance with the applicable standard (“fail”).

5.5.1 Number of Samples To Test a Proportion: Simple Random or Systematic Sampling

This section provides a method for determining the appropriate number of samples when the objective is to test whether a proportion or percentile of a population complies with an applicable standard. A population proportion is the ratio of the number of elements of a population that has some specific characteristic to the total number of elements. A population percentile represents the percentage of elements of a population having values less than some value. The number of samples needed to test a proportion can be calculated using

$$n = \left[\frac{z_{1-\beta}\sqrt{GR(1-GR)} + z_{1-\alpha}\sqrt{AL(1-AL)}}{\Delta} \right]^2 \qquad \textbf{Equation 15}$$

where

α = false rejection error rate
β = false acceptance error rate
z_p = the $p\text{th}$ percentile of the standard normal distribution (from the last row of Table G-1 in Appendix G)
AL = the Action Level (e.g., the proportion of all possible samples of a given support that must comply with the standard)
GR = other bound of the gray region,
Δ = width of the gray region ($GR - AL$), and
n = the number of samples.

An example calculation of n using the approach described here is presented in Box 11.

Box 11. Example Calculation of the Appropriate Number of Samples Needed To Test a Proportion – Simple Random or Systematic Sampling

A facility is conducting a cleanup of soil contaminated with pentachlorophenol (PCP). Based on the results of a field test method, soil exceeding the risk-based cleanup level of 10 mg/kg total PCP will be excavated, classified as a solid or hazardous waste, and placed into roll-off boxes for subsequent disposal, or treatment (if needed) and disposal. The outputs of the first six steps of the DQO Process are summarized below.

Step 1: State the Problem: The project team needs to decide whether the soil being placed in each roll-off box is a RCRA hazardous or nonhazardous waste.

Step 2: Identify the Decision: If the excavated soil is hazardous, it will be treated to comply with the applicable LDR treatment standard and disposed as hazardous waste. If it is nonhazardous, then it will be disposed as solid waste in a permitted industrial waste landfill (as long as it is not mixed with a listed hazardous waste).

Step 3: Identify Inputs to the Decision: The team requires sample analysis results for TCLP PCP to determine compliance with the RCRA TC regulatory threshold of 100 mg/L.

Step 4: Define the Boundaries: The DQO "decision unit" for each hazardous waste determination is defined as a roll-off box of contaminated soil. The "support" of each sample is in part defined by SW-846 Method 1311 (TCLP) as a minimum mass of 100-grams with a maximum particle size of 9.5 mm. Samples will be obtained as the soil is excavated and placed in the roll-off box (i.e., at the point of generation).

Step 5: Develop a Decision Rule: The project team wants to ensure with reasonable confidence that little or no portions of the soil in the roll-off box are hazardous waste. The parameter of interest is then defined as the 90th percentile. If the 90th percentile concentration of PCP is less than 100 mg/L TCLP, then the waste will be classified as nonhazardous. Otherwise, it will be considered hazardous.

Step 6: Specify Limits on Decision Errors: The team establishes the null hypothesis (H_o) as the "true proportion (P) of the waste that complies with the standard is less than 0.90," or H_o: $P < 0.90$. The false rejection error rate (α) is set at 0.10. The false acceptance error rate (β) is set at 0.30. The Action Level (AL) is 0.90, and the other boundary of the gray region (GR) is set at 0.99.

How many samples are required?

Solution: Using Equation 15 and the outputs of the first six steps of the DQO Process, the number of samples (n) is determined as:

$$= \left[\frac{0.524\sqrt{0.99(1-0.99)} + 1.282\sqrt{0.90(1-0.90)}}{0.99-0.90} \right]^2 = 23.5 \approx 24$$

where the values for $z_{1-\alpha}$ and $z_{1-\beta}$ are obtained from the last row of Table G-1 in Appendix G.

5.5.2 Number of Samples When Using a Simple Exceedance Rule

If a simple exceedance rule is used (see Section 3.4.2.2), then it is possible to estimate the number of samples required to achieve a prespecified level of confidence that a given fraction of the waste or site has a constituent concentration less than the standard or does not exhibit a characteristic or property of concern. The approach is based on the minimum sample size required to determine a nonparametric (distribution-free) one-sided confidence bound on a percentile (Hahn and Meeker 1991 and USEPA 1989a).

If the exceedance rule specifies no exceedance of the standard in any sample, then the number of samples that must achieve the standard can be obtained from Table G-3a in Appendix G. The table is based on the expression:

$$n = \log(\alpha)/\log(p) \qquad \textbf{Equation 16}$$

where alpha (α) is the probability of a Type I error and p is the proportion of the waste or site that must comply with the standard. Alternatively, the equation can be rearranged so that statistical performance ($1-\alpha$) can determined for a fixed number of samples:

$$(1-\alpha) = 1 - p^n \qquad \textbf{Equation 17}$$

Notice that the method does not require specification of the other bound of the gray region, nor does it require specification of a Type II (false acceptance) error rate (β).

If the decision rule allows *one* exceedance of the standard in a set of samples, then the number of samples required can be obtained from Table G-3b in Appendix G.

An example application of the above equations is presented in Box 12. See also Appendix F, Section F.3.2.

Box 12. Example Calculation of Number of Samples Needed When a Simple Exceedance Rule Is Used – Simple Random or Systematic Sampling

What is the minimum number of samples required (with no exceedance of the standard in any of the samples) to determine with at least 90-percent confidence ($1-\alpha = 0.90$) that at least 90 percent of all possible samples from the waste (as defined by the DQO decision unit) are less than the applicable standard?

From Table G-3a, we find that for $1 - a = 0.90$ and $p = 0.90$ that 22 samples are required. Alternately, using Equation 16, we find

$$n = \frac{\log(\alpha)}{\log(p)} = \frac{\log(0.10)}{\log(0.90)} = \frac{-1}{-0.0457} = 21.8 \approx 22$$

If only 11 samples were analyzed (with no exceedance of the standard in any of the samples), what level of confidence can we have that at least 90 percent of all possible samples are less than the standard? Using Equation 17, we find

$$(1-\alpha) = 1 - p^n = 1 - 0.90^{11} = 1 - 0.3138 = 0.6862$$

Rounding *down*, we can say with at least 68 percent confidence that at least 90 percent of all possible samples would be less than the applicable standard.

5.6 Selecting the Most Resource-Effective Design

For additional guidance on selecting the most resource-efficient design, see ASTM standard D 6311-98, *Standard Guide for Generation of Environmental Data Related to Waste Management Activities: Selection and Optimization of Sampling Design*.

If more than one sampling design option is under consideration, evaluate the various designs based on their cost and the ability to achieve the data quality and regulatory objectives. Choose the design that provides the best balance between the expected cost and the ability to meet the objectives. To improve the balance between meeting your cost objectives and achieving the DQOs, it might be necessary to modify either the budget or the DQOs. As can be seen from the sample size equations in Section 5.4 and 5.5, there is an interrelationship between the appropriate number of samples and the desired level of confidence, expected variability (both population and measurement variability), and the width of the gray region. To reduce costs (i.e., decrease the number of samples required), several options are available:

- Decrease the confidence level for the test
- Increase the width of the "gray region" (not recommended if the parameter of interest is near the Action Level)
- Divide the population into smaller less heterogeneous decision units, or use a stratified sampling design in which the population is broken down into parts that are internally less heterogeneous
- Employ composite sampling (if non-volatile constituents are of interest and if allowed by the regulations).

Note that seemingly minor modifications to the sampling design using one or more of the above strategies may result in major increases or decreases in the number of samples needed.

When estimating costs, be sure to include the costs for labor, travel and lodging (if necessary), expendable items (such as personal protective gear, sample containers, preservatives, etc.), preparation of a health and safety plan, sample and equipment shipping, sample analysis, assessment, and reporting. Some sampling plans (such as composite sampling) may require fewer analyses and associated analytical costs, but might require more time to implement and not achieve the project objectives. EPA's *Data Quality Objectives Decision Error Feasibility Trials Software (DEFT)* (USEPA 2001a) is one tool available that makes the process of selecting the most resource effective design easier.

5.7 Preparing a QAPP or WAP

In this activity, the outputs of the DQO Process and the sampling design are combined in a planning document such as a QAPP or WAP. The Agency has developed detailed guidance on how to prepare a QAPP (see USEPA 1998a) or WAP (see USEPA 1994a). The minimum requirements for a WAP are specified at 40 CFR §264.13. The following discussion is focused on the elements of a QAPP; however, the information can be used to help develop a WAP.

The QAPP is a critical planning document for any environmental data collection operation because it documents project activities including how QA and QC activities will be implemented during the life cycle of a project. The QAPP is the "blueprint" for identifying how the quality system of the organization performing the work is reflected in a particular project and in associated technical goals. QA is a system of management activities designed to ensure that data produced by the operation will be of the type and quality needed and expected by the data user. QA, acknowledged to be a management function emphasizing systems and policies, aids the collection of data of needed and expected quality appropriate to support management decisions in a resource-efficient manner.

Additional EPA Guidance on Preparing a QAPP or WAP

- Chapter One, SW-846
- *EPA Requirements for Quality Assurance Project Plans, EPA QA/R*-5 (replaces QAMS-005/80) (USEPA 2001b)
- *EPA Guidance for Quality Assurance Project Plans, EPA QA/G*-5 (EPA/600/R-98/018) (USEPA 1998a)
- *Guidance for Choosing a Sampling Design for Environmental Data Collection, EPA QA/G-5S - Peer Review Draft* (EPA QA/G-5S) (USEPA 2000c)
- *Waste Analysis at Facilities That Generate, Treat, Store, And Dispose Of Hazardous Wastes, a Guidance Manual* (USEPA 1994a)

The activities addressed in the QAPP cover the entire project life cycle, integrating elements of the planning, implementation, and assessment phases. If the DQOs are documented (e.g., in a memo or report format), include the DQO document as an attachment to the QAPP to help document the technical basis for the project and to document any agreements made between stakeholders.

As recommended in EPA QA/G-5 (USEPA 1998a), a QAPP is composed of four sections of project-related information called "groups," which are subdivided into specific detailed "elements." The elements and groups are summarized in the following subsections.

5.7.1 Project Management

The QAPP (or WAP) is prepared after completion of the DQO Process. Much of the following guidance related to project management can be excerpted from the outputs of the DQO Process.

The following group of QAPP elements covers the general areas of project management, project history and objectives, and roles and responsibilities of the participants. The following elements ensure that the project's goals are clearly stated, that all participants understand the goals and the approach to be used, and that project planning is documented:

- Title and approval sheet
- Table of contents and document control format
- Distribution list
- Project/task organization and schedule (from DQO Step 1)
- Problem definition/background (from DQO Step 1)
- Project/task description (from DQO Step 1)
- Quality objectives and criteria for measurement data (DQO Step 3)

- Special training requirements/certification
- Documentation and records.

For some projects, it will be necessary to include the names and qualifications of the person(s) who will obtain the samples (e.g., as required under 40 CFR §261.38(c)(7) in connection with testing for the comparable fuels exclusion).

5.7.2 Measurement/Data Acquisition

This group of QAPP elements covers all aspects of measurement system design and implementation, ensuring that appropriate methods for sampling, analysis, data handling, and QC are employed and thoroughly documented. Apart from the sample design step (DQO Step 7), the following information should be included in the QAPP or incorporated by reference:

- Sampling process design/experimental design (DQO Steps 5 and 7)
- Sampling methods and SOPs
- Sample handling and chain-of-custody requirements
- Analytical methods and SOPs (DQO Step 3)
- QC requirements;
- Instrument/equipment testing, inspection, and maintenance requirements
- Instrument calibration and frequency
- Inspection/acceptance requirements for supplies and consumables
- Data acquisition requirements (non-direct measurements)
- Data management.

For some projects, under various circumstances it may be appropriate to include hard copies of the SOPs in the QAPP, rather than incorporate the information by reference. For example, under the performance-based measurement system (PBMS) approach, alternative sampling and analytical methods can be used. Such methods can be reviewed and used more readily if actual copies of the SOPs are included in the QAPP. Hard copies of SOPs also are critically important when field analytical techniques are used. Field personnel must have detailed instructions available to ensure that the methods are followed. If it is discovered that deviation from an SOP is required due to site-specific circumstances, the deviations can be documented more easily if hard copies of the SOPs are available in the field with QAPP.

5.7.3 Assessment/Oversight

The purpose of assessment is to ensure that the QAPP is implemented as prescribed. The elements below address the activities for assessing the effectiveness of the implementation of the project and the associated QA/QC activities:

- Assessments and response actions
- Reports to management.

5.7.4 Data Validation and Usability

Implementation of these elements ensures that the data conform to the specified criteria, thus enabling reconciliation with the project’s objectives. The following elements cover QA activities that occur after the data collection phase of the project has been completed:

- Data review, verification, and validation requirements
- Verification and validation methods
- Reconciliation with DQOs.

5.7.5 Data Assessment

Historically, the focus of most QAPPs has been on analytical methods, sampling, data handling, and quality control. Little attention has been paid to data assessment and interpretation. We recommend that the QAPP address the data assessment steps that will be followed after data verification and validation. While it may not be possible to specify the statistical test to be used in advance of data generation, the statistical objective (identified in the DQO Process) should be stated along with general procedures that will be used to test distributional assumptions and select statistical tests. EPA's *Guidance for Data Quality Assessment* (USEPA 2000d) suggests the following five-step methodology (see also Section 8 for a similar methodology):

1. Review the DQOs
2. Conduct a preliminary data review
3. Select the statistical test
4. Verify the assumptions of the test
5. Draw conclusions from the Data.

The degree to which each QAPP element should be addressed will be dependent on the specific project and can range from "not applicable" to extensive documentation. The final decision on the specific need for these elements for project-specific QAPPs will be made by the regulatory agency. Documents prepared prior to the QAPP (e.g., SOPs, test plans, and sampling plans) can be appended or, in some cases, incorporated by reference.

6 CONTROLLING VARIABILITY AND BIAS IN SAMPLING

The DQO Process allows you to identify the problem to be solved, set specific goals and objectives, establish probability levels for making incorrect decisions, and develop a resource-efficient data collection and analysis plan. While most of the sampling designs suggested in this guidance incorporate some form of randomness so that unbiased estimates can be obtained from the data, there are other equally important considerations (Myers 1997). Sampling and analysis activities must also include use of correct devices and procedures to minimize or control random variability and biases (collectively known as "error") that can be introduced in field sampling, sample transport, subsampling, sample preparation, and analysis. Sampling error can lead to incorrect conclusions irrespective of the quality of the analytical measurements and the appropriateness of the statistical methods used to evaluate the data.

This section is organized into three subsections which respond to these questions:

1. What are the sources of error in sampling (Section 6.1)?
2. What is sampling theory (Section 6.2)?
3. How can you reduce or otherwise control sampling error in the field and laboratory (Section 6.3)?

6.1 Sources of Random Variability and Bias in Sampling

In conducting sampling, we are interested in obtaining an estimate of a population parameter (such as the mean, median, or a percentile); but an estimate of a parameter made from measurements of samples always will include some random variability (or variances) and bias (or a systematic shift away from the true value) due primarily to (1) the inherent variability of the waste or media (the "between-sampling-unit variability") and (2) imprecision in the methods used to collect and analyze the samples (the "within-sampling-unit variability") (USEPA 2001e).

Errors caused by the sample collection process can be much greater than the preparation, analytical, and data handling errors (van Ee, et al. 1990, Crockett, et al 1996) and can dominate the overall uncertainty associated with a characterization study (Jenkins, et al. 1996 and 1997). In fact, analytical errors are usually well-characterized, well-understood, and well-controlled by laboratory QA/QC, whereas sampling and sample handling errors are not usually well-characterized, well-understood, or well-controlled (Shefsky 1997). Because sampling error contributes to overall error, it is important for field and laboratory personnel to understand the sources of sampling errors and to take measures to control them in field sampling.

The two components of error -- random variability and bias -- are independent. This concept is demonstrated in the "target" diagram (see Figure 7 in Section 2), in which random variability (expressed as the variance, σ^2) refers to the "degree of clustering" and bias ($\mu - \bar{x}$) relates to the "amount of offset from the center of the target" (Myers 1997).

Random variability and bias occur at each stage of sampling. Variability occurs due to the **heterogeneity** of the material sampled and random variations in the sampling and sample handling procedures. In addition, bias can be introduced at each stage by the sampling device (or the manner in which it is used), sample handling and transport, subsampling, and analysis.

While it is common practice to calculate the variability of sample analysis results "after the fact," it is more difficult to identify the sources and potential impacts of systematic sampling bias. As discussed in more detail below, it usually is best to understand the potential sources of error "up front" and take measures to minimize them when planning and implementing the sampling and analysis program.

Even though random variability and bias are independent, they are related quantitatively (see Figure 23). Errors expressed as the variance can be added together to estimate overall or "total study error." Biases can be added together to estimate overall bias (though sampling bias is difficult to measure in practice). Conceptually, the sum of all the variances can be added to the sum of all biases (which is then squared) and expressed as the (**mean square error** $MSE(\bar{x})$) which provides a quantitative way of measuring the degree of *representativeness* of the samples. In practice, it is not necessary to try to calculate mean square error, however, we suggest you understand the sources and impacts of variability and bias so you can take steps to control them in sampling and improve the representativeness of the samples. (See Sections 5.2.4 and 5.2.5 of EPA's *Guidance for Data Quality Assessment, EPA QA/G-9 - QA00 Update* (USEPA 2000d) for a more detailed discussion of how to address measurement variability and bias in the sampling design).

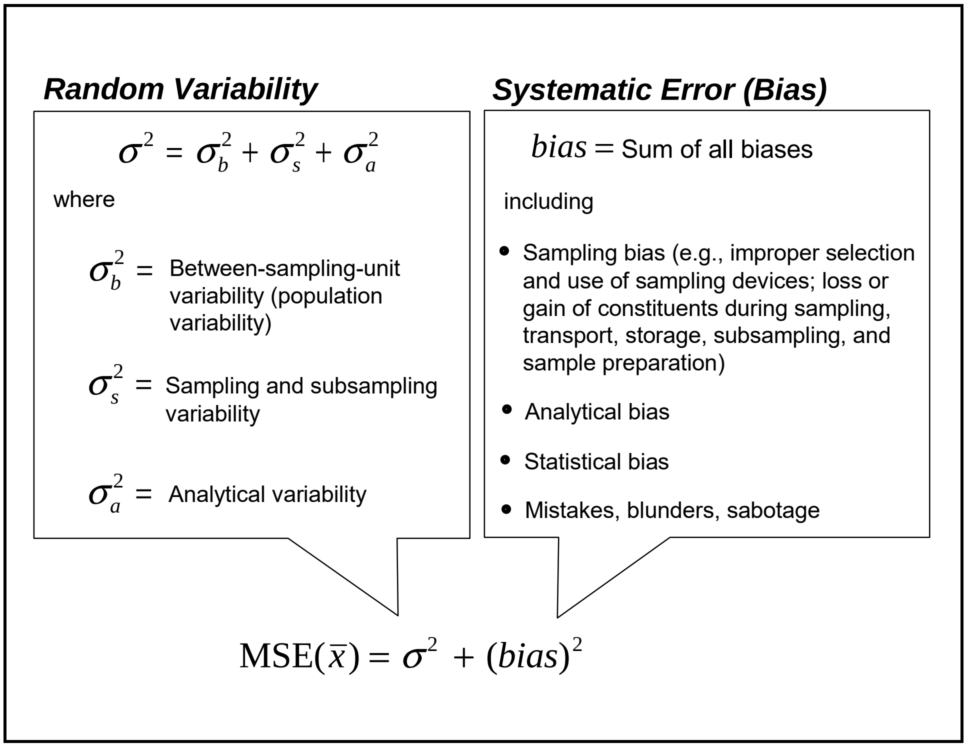

Figure 23. Components of error and the additivity of variances and biases in sampling and analysis

The relatively new science of sampling theory and practice (Myers 1997) provides a technically based approach for addressing sampling errors (see Section 6.2). Sampling theory recognizes that sampling errors arise from or are related to the size and distribution of particles in the waste, the weight of the sample, the shape and orientation of the sampling device, the manner in which the sample is collected, sample handling, and the manner in which subsampling is performed within the laboratory. Sampling theory applies to particulate solids, liquids, and mixtures of solids and liquids. Understanding sampling theory does not allow us to completely eliminate sampling and analytical errors, but sampling theory does allow us to identify the sources and magnitudes of sampling errors so we can take steps to minimize those that are the largest. In doing so, samples will be more precise and unbiased (i.e., more "representative"), thus reducing the number of samples required (lowering costs) and improving our ability to achieve the decision error rate specified in the DQOs.

6.2 Overview of Sampling Theory

A number of environmental scientists have recognized a set of sampling theories developed by Dr. Pierre Gy (Gy 1982 and 1998) and others (Ingamells and Switzer 1973; Ingamells 1974; Ingamells and Pitard 1986; Pitard 1989; and Visman 1969) as one set of tools for improving sampling. These researchers have studied the sources of sampling error (particularly in the sampling of particulate matter) and developed techniques for quantifying the amount of error that can be introduced by the physical sampling process. The theories were originally developed in support of mineral exploration and mining and more recently were adopted by EPA for soil sampling (van Ee, et al. 1990; Mason 1992). Under some conditions, however, the theories can be applied to waste sampling as a means for improving the efficiency of the sampling and analysis process (Ramsey, et al. 1989).

As discussed in the context of this guidance, Gy's theories focus on minimizing error during the physical collection of a sample of solid and liquid media and should not be confused with the statistical sampling designs such as simple random, stratified random, etc. discussed in Section 5. Both sampling theory and sampling design, however, are critical elements in sampling: Gy's theories facilitate collection of "correct" individual samples, while statistical sampling designs allow us to conduct statistical analyses and make conclusions about the larger mass of waste or environmental media (i.e., the decision unit).

The following three subsections describe key aspects of sampling theory including heterogeneity, sampling errors, and the concept of sample support. The descriptions are mostly qualitative and intended to provided the reader with an appreciation for the types and complexities of sampling error. Detailed descriptions of the development and application of sampling theory can be found in *Sampling for Analytical Purposes* (Gy 1998), *Geostatistical Error Management* (Myers 1997), *Pierre Gy's Sampling Theory and Sampling Practice* (Pitard 1993), and in EPA's guidance document *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (Mason 1992).

6.2.1 Heterogeneity

One of the underlying principles of sampling theory is that the medium to be sampled is not uniform in its composition or in the distribution of constituents in the medium, rather, it is **heterogeneous**. Heterogeneity causes the sampling errors.

Appropriate treatment of heterogeneity in sampling depends on the scale of observation. Large-scale variations in a waste stream or site affect where and when we take samples. Small-scale variations in a waste or media affect the size, shape, and orientation of individual field samples and laboratory subsamples. Gy's theory identifies three major types of heterogeneity: (1) short-range (or small-scale) heterogeneity, (2) long-range (or large-scale) heterogeneity, and (3) periodic heterogeneity:

> **Short-range heterogeneity** refers to properties of the waste at the sample level or in the immediate vicinity of a sample location. Two other types of heterogeneity are found within short-range heterogeneity: one reflected by differences in the *composition* between individual particles, the other having to do with the *distribution* of those particles in the waste. Composition heterogeneity (also known as *constitution heterogeneity*) is constant and cannot be altered except by particle size reduction (e.g., grinding or crushing the material). The distribution heterogeneity plays an important role in sampling because particles can separate into groups. Distribution heterogeneity can be increased (e.g., by gravitational segregation of particles or liquids) and can be reduced by homogenization (mixing) or by taking many small increments to form a sample.
>
> **Large-scale heterogeneity** reflects local trends and plays an important role in deciding whether to divide the population into smaller internally homogenous decision units or to use a stratified sampling design. See Appendix C for a detailed description of large-scale heterogeneity.
>
> **Periodic heterogeneity**, another larger-scale phenomena, refers to cyclic phenomena found in flowing streams or discharges. Understanding periodic heterogeneity can aid in dividing a waste into separate waste streams or in establishing a stratified sampling design.

Forming a conceptual model of the heterogeneity of a waste will help you to determine how to address it in sampling.

6.2.2 Types of Sampling Error

Gy’s theory (see also Mason 1992, Pitard 1993, and Gy 1998) identifies a number of different types of error that can occur in sampling as a result of heterogeneity in the waste and failure to correctly define the appropriate shape and volume of material for inclusion in the sample. Understanding the types and sources of the errors is an important step toward *avoiding* them. In qualitative terms, these errors include the following:

- *Fundamental error*, which is caused by differences in the composition of individual particles in the waste
- Errors due to *segregation* and *grouping* of particles and the constituent associated with the particles
- Errors due to various types of trends including small-scale trends, large-scale trends, or cycles
- Errors due to defining (or *delimiting*) the sample space and *extracting* the sample from the defined area
- Errors due to *preparation* of the sample, including shipping and handling. [Note that the term “preparation,” as used here, describes all the activities that take

place after the primary sample is obtained in the field and includes sample containerization, preservation, handling, mixing, grinding, subsampling, and other preparative steps taken prior to analysis (such as the "sample preparation methods" as described in Chapters Three, Four, and Five of SW-846).]

Errors that can occur during sampling are described below.

6.2.2.1 Fundamental Error

The composition of a sample never perfectly matches the overall composition of the larger mass from which is was obtained because the mass of an individual sample is always less than the mass of the population and the population is never completely homogeneous. These conditions result in a sampling error known as **fundamental error**. The error is referred to as "fundamental" because it is an incompressible minimum sampling error that depends on the composition, shape, fragment size distribution, and chemical properties of the material, and it is not affected by homogenization or mixing. It arises when the constituent of interest is concentrated in constituent "nuggets" in a less concentrated matrix, especially when the constituent is present at a trace concentration level (e.g., less than 1 percent). This type of sampling error occurs even when the nuggets are mixed as well as possible in the matrix (so long as they are not dissolved). The fundamental error is the only error that remains when the sampling operation is "perfect"; that is, when all parts of the sample are obtained in a probabilistic manner and each part is independent.

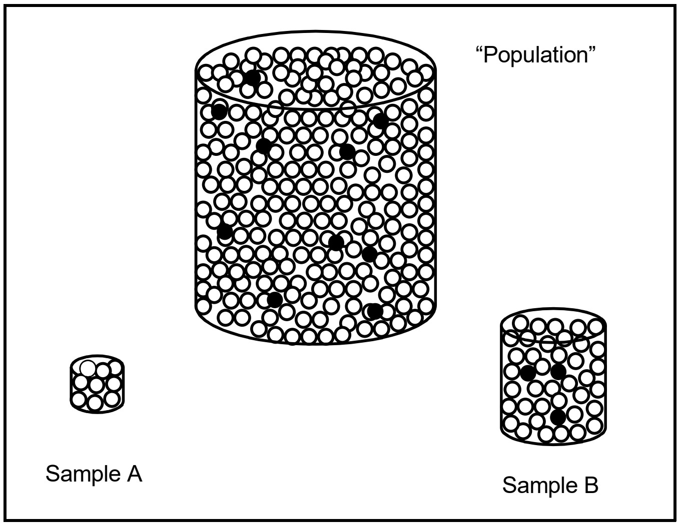

Figure 24. Effects of sample size on fundamental error. Small samples such as "A" cause the constituent of interest to be under-represented in most samples and over-represented in a small proportion of samples. Larger samples such as "B" more closely reflect the parent population.

As a conceptual example of fundamental error, consider a container filled with many white marbles and a few black marbles that have been mixed together well (Figure 24). If a small sample comprising only a few marbles is picked at random, there is a high probability they would all be white (Sample "A" in Figure 24) and a small chance that one or more would be black. As the sample size becomes larger, the distribution in the sample will reflect more and more closely the parent population (Sample "B" in Figure 24). The situation is similar in a waste that contains rare highly concentrated "nuggets" of a constituent of concern. If a small sample is taken, it is possible, and even likely, that no nuggets of the constituent would be selected as part of the sample. This would lead to a major *underestimate* of the true parameter of interest. It also is possible with a small sample that a gross *overestimate* of the parameter of interest will occur if a nugget is included in the sample because the nugget would comprise a relatively large proportion of the analytical sample compared to the true population. To minimize fundamental error, the point is not to simply "fish" for a black marble (the contaminant), but to sample for all of the fragments and constituents such that the sample is a representation of the lot from which it is derived.

The fundamental error is never zero (unless the population is completely homogeneous or the entire population is submitted for analysis) and it never "cancels out." It can be controlled by taking larger physical samples; however, larger samples can be difficult to handle in the field and within the laboratory, and they may pose practical constraints due to increased space needed for storage. Furthermore, small samples (e.g., less than 1 gram) generally are required for analytical purposes. To preserve the character of a large sample in the small analytical sample, subsampling and particle size reduction strategies should be employed (see also Section 7.3).

6.2.2.2 Grouping and Segregation Error

Grouping and segregation results from the short-range heterogeneity within and around the area from which a sample is collected (i.e., the sampling location) and within the sample container. This small-scale heterogeneity is caused by the tendency for some particles to associate into groups of like particles due to gravitational separation, chemical partitioning, differing moisture content, magnetism, or electrostatic charge. Grouping and segregation of particles can lead to sampling bias.

Figure 25 depicts grouping of particles (at "A") and segregation of particles (at "B") within a sample location. The grouping of particles at location "A" could result from an affinity between like particles (for example, due to electrostatic forces). Analytical samples formed from just one group of particles would yield biased results.

The segregation of particles at location "B" could result from gravitation separation (e.g., during sample shipment). If the contaminant of interest was associated with only one class of particle (for example, only the black diamond shapes), then a sample collected from the top would result in a different concentration than a sample collected from the bottom, thus biasing the sample.

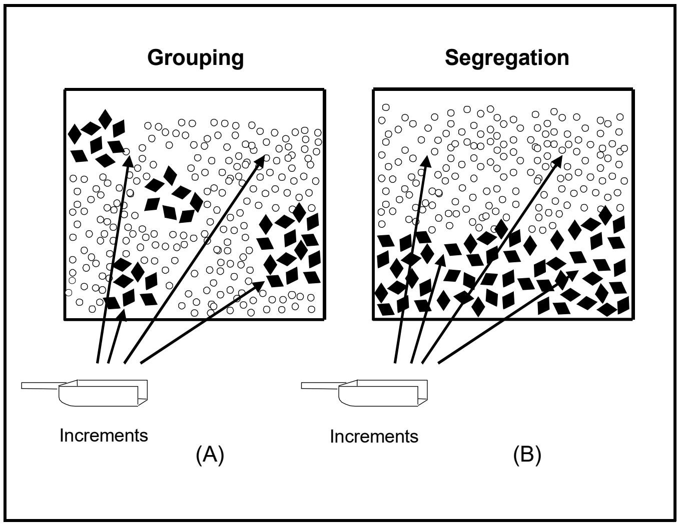

Figure 25. How grouping and segregation of particles can affect sampling results. Grouping and segregation error can be minimized by taking many small increments.

Grouping and segregation error can be minimized by properly homogenizing and splitting the sample. As an alternative, an individual sample can be formed by taking a number of increments (small portions of media) in the immediate vicinity of the sampling location and combining them into the final collected sample.[1] Pitard (1993) suggests collecting between 10 and 25 increments as a means to control grouping and segregation error. These increments are then combined to form an individual sample to be submitted to the laboratory for analysis.

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[1] This approach should not be confused with composite sampling, in which individual samples from different times or locations are pooled and mixed into a single sample.

The approach of taking multiple increments to form a sample is not recommended when volatile constituents are of interest and may have practical limitations when sampling highly heterogeneous wastes or debris containing very large fragments.

6.2.2.3 Increment Delimitation Error

Increment delimitation error occurs when the shape of the sampling device excludes or discriminates against certain portions of the material to be sampled. For example, a sampling device that only samples the top portion of a liquid effluent as it is leaves a discharge pipe (leaving a portion of the flow unsampled) causes increment delimitation error. This type of error is eliminated by choosing a sampling device capable of obtaining all of the flow for a fraction of the time (see also Sections 6.3.2 and 6.3.3).

6.2.2.4 Increment Extraction Error

Increment extraction error occurs when portions of the sample are lost or extraneous materials are included in the sample. For example, if the coring device is too small to accommodate a large fragment of waste, particles that should be in the sample might get pushed aside, causing sampling bias. Extraction error can be controlled through selection of devices designed to accommodate the physical characteristics of the waste.

6.2.2.5 Preparation Error

This error results from the incorrect preservation, handling, mixing, grinding, and subsampling that can result in loss, contamination, or altering of the sample such that it no longer is an accurate representation of the material being sampled. Proper choice and implementation of preparation methods controls this error.

6.2.3 The Concept of "Sample Support"

The weight, shape (length, width and height dimensions), and orientation of a sample describe the "sample support." The term "support" has been used in sampling and statistical literature in various ways, such as to describe the mass or volume of an "exposure unit" or "exposure area" in the Superfund program -- similar to the "decision unit" described in the DQO Process.

Conceptually, there is a continuum of support from the decision unit level (e.g., an exposure area of a waste site or a drum of solid waste) to the sample and subsample level down to the molecular level. Because it is not possible to submit the entire decision unit for analysis, samples must be submitted instead. For heterogeneous media, *the sample support will have a substantial effect on the reported measurement values*.

Measures can be taken to ensure adequate size, shape, and orientation of a sample:

- The appropriate *size* of a sample (either volume or mass) can be determined based on the relationship that exists between the particle size distribution and expected sampling error -- known as the fundamental error (see Section 6.2.2.1). In the DQO Process, you can define the amount of fundamental error that is acceptable (specified in terms of the standard deviation of the fundamental error) and estimate the volume required for field samples. The sampling tool should have dimensions three or more times larger than that of the diameter of the largest particles. Proper sizing of the sampling tool will help ensure that the particle size distribution of the sampled material is represented in the sample (see discussion at Section 6.3.1).

- The appropriate *shape and orientation* of the sample are determined by the sampling mode. For a one-dimensional waste (e.g., liquid flowing from a discharge pipe or solids on a conveyor belt), the correct or “ideal” sample is an undisturbed cross section delimited by two parallel planes (Pitard 1993, Gy 1998) (see discussion at Section 6.3.2.1). For three-dimensional waste forms (such as solids in a roll-off bin, piles, thick slabs, soil in drums, liquids in a tank, etc.), the sampling problem is best treated as a series of overlapping two-dimensional problems. The correct or ideal sample is an undisturbed core (Pitard 1993) that captures the entire thickness of the waste (see discussion at Section 6.3.2.2).

6.3 Practical Guidance for Reducing Sampling Error

This section describes steps that can be taken to control sampling error. While the details of sampling theory may appear complex and difficult to explain, in practice most sampling errors can be minimized by observing a few simple rules that, when used, can greatly improve the reliability of sampling results with little or no additional costs (Gy 1998):

- Determine the optimal mass of each field sample. For particulate solids, determine the appropriate sample weight based on the particle size distribution and characteristics, and consider any practical constraints (see Section 6.3.1). Also, determine additional amounts of the sampled material needed for split samples, for field and laboratory quality control purposes, or for archiving.

- Select the appropriate shape and orientation of the sample based on the sampling design model identified in DQO Step 7 (see Section 6.3.2).

- Select sampling devices and procedures that will minimize grouping and segregation errors and increment delimitation and increment extraction errors (see Sections 6.3.3 and 7.1).

Implement the sampling plan by obtaining the number of samples at the sampling locations and times specified in the sampling design selected in DQO Step 7, and take measures to minimize preparation errors during sample handling, subsampling, analysis, documentation, and reporting. When collecting samples for analysis for volatile organic constituents, special considerations are warranted to minimize bias due to loss of constituents (see Section 6.3.4).

Table 7 provides a summary of strategies that can be employed to minimize the various types of sampling error.

Table 7. Strategies for Minimizing Sampling Error

Type of Sampling Error	*Strategy To Minimize or Reduce Error*
Fundamental Error	• To reduce variability caused by fundamental error, increase the volume of the sample. • To reduce the volume of the sample and maintain low fundamental error, perform particle-size reduction followed by subsampling. • When volatile constituents are of interest, do not grind or mix the sample. Rather, take samples using a method that minimizes disturbances of the sample material (see also Section 6.3.4).
Grouping and Segregation Error	• To minimize grouping error, take many increments. • To minimize segregation error, homogenize the sample (but beware of techniques that promote segregation)
Increment Delimitation/Extraction Errors	• Select sampling devices that delimit and extract the sample so that all material that should be included in the sample is captured and retained by the device (Pitard 1993, Myers 1997). • For one-dimensional wastes (e.g., flowing streams or waste on a conveyor), the correct or “ideal” sample is an undisturbed cross section delimited by two parallel planes (Pitard 1993, Gy 1998). To obtain such a sample, use a device that can obtain “all of the flow for a fraction of the time” (Gy 1998) (see also Section 6.3.2.1). • For three-dimensional wastes (e.g., solids in a roll-off bin), the waste can be considered for practical purposes a series of overlapping two-dimensional wastes. The correct or “ideal” sample is an undisturbed vertical core (Pitard 1993, Gy 1998) that captures the full depth of interest.
Preparation Error	• Take steps to prevent contamination of the sample during field handling and shipment. Sample contamination can be checked through preparation and analysis of field quality control samples such as field blanks, trip blanks, and equipment rinsate blanks. • Prevent loss of volatile constituents through proper storage and handling. • Minimize chemical transformations via proper storage and chemical/physical preservation. • Take care to avoid unintentional mistakes when labeling sample containers, completing other documentation, and handling and weighing samples.

6.3.1 Determining the Optimal Mass of a Sample

As part of the DQO Process (Step 4 - Define the Boundaries), we recommend that you determine the appropriate size (i.e., the mass or volume), shape, and orientation of the primary field sample. For heterogeneous materials, the size, shape, and orientation of each field sample will affect the analytical result. To determine the optimal mass (or weight) of samples to be collected in the field, you should consider several key factors:

- The number and type of chemical and/or physical analyses to be performed on each sample, including extra volumes required for QA/QC. (For example, SW-846 Method 1311 (TCLP) specifies the minimum sample mass to be used for the extraction.)

- Practical constraints, such as the available volume of the material and the ability to collect, transport, and store the samples

- The characteristics of the matrix (such as particulate solid, sludge, liquid, debris, oily waste, etc.)
- Health and safety concerns (e.g., acutely toxic, corrosive, reactive, or ignitable wastes should be transported and handled in safe quantities)
- Availability of equipment and personnel to perform particle-size reduction (if needed) in the field rather than within a laboratory.

Often, the weight (or mass) of a field sample is determined by "whatever will fit into the jar." While this criterion may be adequate for some wastes or media, it can introduce serious biases – *especially in the case of sampling particulate solids*.

If a sample of particulate material is to be representative, then it needs to be representative of the largest particles of interest (Pitard 1993). This is relevant if the constituent of concern is not uniformly distributed across all the particle size fractions. To obtain a sample representative of the largest particles of interest, the sample must be of sufficient weight (or mass) to control the amount of fundamental error introduced during sampling.

If the constituent(s) of concern is uniformly distributed throughout all the particle size fractions, then determination of the optimal sample mass using Gy's approach will not improve the representativeness of the sample. Homogeneous or uniform distribution of contaminants among all particle sizes, however, is not a realistic assumption, especially for contaminated soils. In contaminated soils, concentrations of metals tend to be higher in the clay- and silt-size fractions and organic contaminants tend to be associated with organic matter and fines in the soil.

The following material provides a "rule of thumb" approach for determining the particle-size sample-weight relationship sufficient to maintain fundamental error (as measured by the standard deviation of the fundamental error) within desired limits. A detailed quantitative method is presented in Appendix D. Techniques for calculating the variance of the fundamental error also are presented in Mason (1992), Pitard (1993), Myers (1997), and Gy (1998).

The variance of the fundamental error (s_{FE}^2) is directly proportional to the size of the largest particle and inversely proportional to the mass of the sample.[2] To calculate the appropriate mass of the sample, Pitard (1989) proposed a "Quick Safety Rule" for use in environmental sampling based on a standard deviation of the fundamental error of 5 percent ($s_{FE} = \pm 5\%$):

$$\mathrm{M_S} \geq 10000 d^3$$

Equation 18

where $\mathrm{M_S}$ is the mass of the sample in grams (g) and d of the diameter of the largest particle in centimeters (cm).

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[2] In this section, we use the "relative variance" ($s^2 / \bar{x}^2$) and the "relative standard deviation" ($s / \bar{x}$). The values are dimensionless and are useful for comparing results from different experiments.

Alternatively, if we are willing to accept $s_{FE} = \pm 16\%$, we can use

$$\mathrm{M_S} \geq 1000 d^3 \qquad \textbf{Equation 19}$$

An important feature of the fundamental error is that it does not "cancel out." On the contrary, the variance of the fundamental error adds together at each stage of subsampling. As pointed out by Myers (1997), the fundamental error quickly can accumulate and exceed 50 percent, 100 percent, 200 percent, or greater unless it is controlled through particle-size reduction *at each stage of sampling and subsampling*. The variance, s^2_{FE} , calculated at each stage of subsampling and particle-size reduction, must be added together at the end to derive the total s^2_{FE} . A example of how the variances of the fundamental error can be added together is provided in Appendix D.

6.3.2 Obtaining the Correct Shape and Orientation of a Sample

When sampling heterogeneous materials, the shape and orientation of the sampling device can affect the composition of the resulting samples and facilitate or impede achievement of DQOs. The following two subsections provide guidance on selecting the appropriate shape and orientation of samples obtained from a moving stream of material and a stationary batch or unit of material.

6.3.2.1 Sampling of a Moving Stream of Material

In sampling a moving stream of material, such as solids, liquids, and multi-phase mixtures moving through a pipe, on a conveyor, etc., the material can be treated as a one-dimensional mass. That is, the material is assumed to be linear in time or space.

The correct or "ideal" sample is an undisturbed cross section delimited by two parallel planes (Pitard 1993, Gy 1998). The approach is depicted in Figure 26 in which all of the flow is collected for part of the time. In practice, the condition can be met by using "cross-stream" sampling devices positioned at the discharge of a conveyor, hose, duct, etc. (Pitard 1993). Alternatively, in sampling solids from a conveyor belt, a transverse cutter or flat scoop (with vertical sides) can be used to obtain a sample, preferably with the conveyor stopped (though this condition may not be practical for large industrial conveyors).

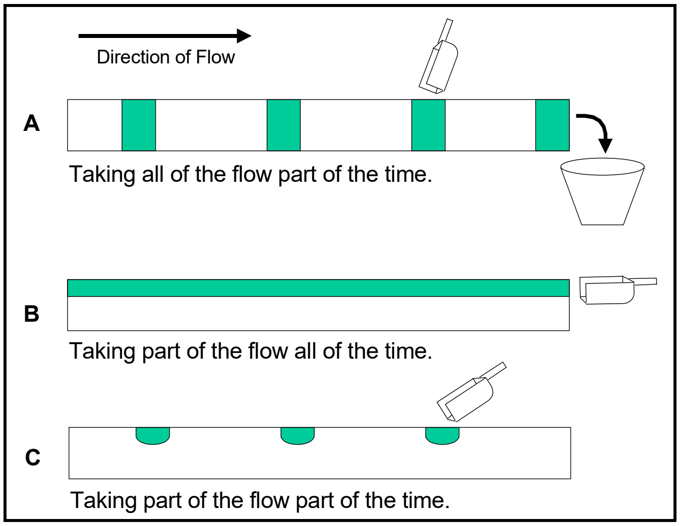


Figure 26. Three ways of obtaining a sample from a moving stream. "A" is correct. "B" and "C" will obtain biased samples unless the material is homogeneous (modified after Gy 1998).

For sampling of liquids, if the entire stream cannot be obtained for a fraction of the time (e.g., at the discharge point), then it may be necessary to introduce turbulence in the stream using baffles and to obtain a portion of the mixed stream part of the time (Pitard 1993).

6.3.2.2 Sampling of a Stationary Batch of Material

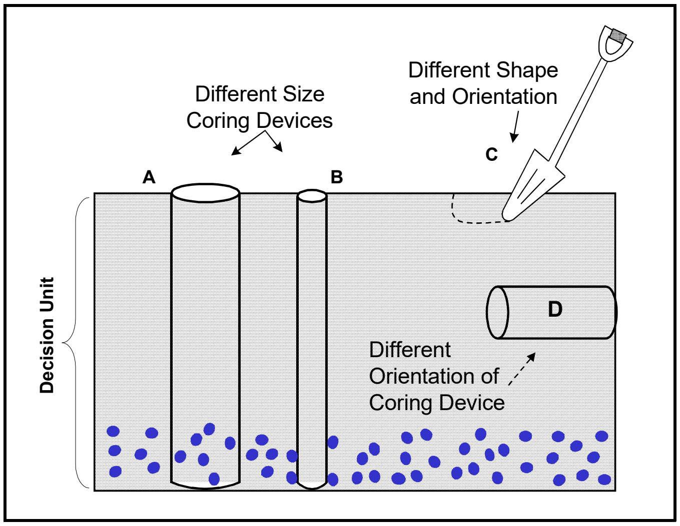


Figure 27. Sampling a three-dimensional waste by treating the sampling problem as a series of overlapping two-dimensional wastes. Only device "A" provides the correct size, shape, and orientation of the sample.

Sampling of a stationary batch of material, such as filter cake in a roll-off bin, soil in a drum, or liquid in a tank can be approached by viewing the three-dimensional space as a series of overlapping two-dimensional (i.e., relatively flat) masses in a horizontal plane. The correct or "ideal" sample of a is a core that obtains the full thickness of the material of interest.

For example, Figure 27 shows a bin of granular waste with fine grain material in the upper layer and larger fragments in the bottom layer. The entire batch of material is the "decision unit." Coring device "A" is correct: it is wide enough and long enough to include the largest fragments in the waste. Coring device "B" is too narrow. It either fails to capture the larger particles or simply pushes them out of the way (causing *increment delimitation error*). Device "C," a trowel or small shovel, can collect an adequate volume of sample, but it preferentially selects only the finer grained material near the top of the bin. Device "D" is the correct shape, but it is not in the correct orientation. Devices "B," "C," and "D" yield incorrect sample support.

6.3.3 Selecting Sampling Devices That Minimize Sampling Errors

As part of the project planning process, you should establish performance goals for the sampling devices to be used and understand the possible limitations of any candidate sampling devices or equipment. The performance goals can then be used to select specific sampling devices or technologies with a clear understanding of the limitations of those devices in the field. Detailed guidance on the selection of specific sampling devices is provided in Section 7 and Appendix E of this document.

6.3.3.1 General Performance Goals for Sampling Tools and Devices

Selection of the appropriate sampling device and sampling method will depend on the sampling objectives, the physical characteristics of the waste or media, the chemical constituents of concern, the sampling location, and practical concerns such as technology limitations and safety issues (see also Section 7). The following general performance goals apply to the selection of sampling devices for use in those situations in where it is desirable to control or otherwise minimize biases introduced by the sampling device:

- The device should not include or exclude portions of the waste that do not belong in the sample (in other words, the device should minimize delimitation and extraction errors).

- If volatile constituents are of interest, the device should obtain samples in an undisturbed state to minimize loss of volatile constituents.

- The device should be constructed of materials that will not alter analyte concentrations due to loss or gain of analytes via sorption, desorption, degradation, or corrosion.

- The device should retain the appropriate size (volume or mass) and shape of sample, and obtain it in the orientation appropriate for the sampling condition -- preferably in one pass.

Other considerations not related to performance follow:

- "Ease of use" of the sampling device under the conditions that will be encountered in the field. This includes the ease of shipping to and from the site, ease of deployment, and ease of decontamination.

- The degree of hazard associated with the deployment of one sampling device versus another (e.g., consider use of an extension pole instead of a boat to sample from a waste lagoon).

- Cost of the sampling device and of the labor (e.g., single vs. multiple operators) for its deployment (including training) and maintenance.

6.3.3.2 Use and Limitations of Common Devices

Unfortunately, many sampling devices in common use today lack the properties required to minimize certain types of sampling error. In fact, there are few devices available that satisfy all the general performance goals stated above. Pitard (1993), however, has identified a number of devices that can help minimize delimitation and extraction error (depending on the physical form of the waste to be sampled). These devices include:

- COLIWASA (or “composite liquid waste sampler”) -- for sampling free-flowing liquids in drums or containers

- Shelby tube or similar device -- for obtaining core samples of solids

- Kemmerer depth sampler -- for obtaining discrete samples of liquids

- Flat scoop (with vertical walls) -- for subsampling solids on a flat surface.

Some devices in common use that can cause delimitation and extraction errors include the following: auger, shovel, spoon, trowel, thief, and trier. In spite of the limitations of many conventional sampling devices, it is necessary to use them under some circumstances encountered in the field because there are few alternatives. When selecting a sampling tool, choose the one that will introduce the least sampling error. In cases in which no such tool exists, document the approach used and be aware of the types of errors likely introduced and their possible impact on the sampling results. To the extent possible and practicable, minimize sampling errors by applying the concepts presented in this chapter.

6.3.4 Special Considerations for Sampling Waste and Soils for Volatile Organic Compounds

In most contaminated soils and other solid waste materials, volatile organic compound (VOCs), when present, coexist in gaseous, liquid, and solid (sorbed) phases. Of particular concern with regard to the collection, handling, and storage of samples for VOC characterization is the retention of the gaseous component. This phase exhibits molecular diffusion coefficients that allow for the immediate loss of gas-phase VOCs from a freshly exposed surface and continued losses from well within a porous matrix. Furthermore, once the gaseous phase becomes depleted, nearly instantaneous volatilization from the liquid and sorbed phases occurs in an attempt to restore the temporal equilibrium that often exists, thereby allowing the impact of this loss mechanism to continue.

Another mechanism that can influence VOC concentrations in samples is biological degradation. In general, this loss mechanism is not expected to be as large a source of determinate error as volatilization. This premise is based on the observation that losses of an order of magnitude can occur on a time scale of minutes to hours due solely to diffusion and advection, whereas losses of a similar magnitude due to biological processes usually require days to weeks. Furthermore, under aerobic conditions, which is typical of most samples that are transported and stored, biological mechanisms favor the degradation of aromatic hydrocarbons over halogenated compounds. Therefore, besides the slower rate of analyte loss, biodegradation is compound selective.

To limit the influence of volatilization and biodegradation losses, which, if not addressed can biased results by one or more orders of magnitude, it is currently recommended that sample collection and preparation, however not necessarily preservation, follow one or the other of these two protocols:

- The immediate in-field transfer of a sample into a weighed volatile organic analysis vial that either contains VOC-free water so that a vapor partitioning (purge-and-trap or headspace) analysis can be performed without reopening or that contains methanol for analyte extraction in preparation for analysis, or

- The collection and up to 2-day storage of intact samples in airtight containers before initiating one of the aforementioned sample preparation procedures.

In both cases, samples should be held at 4±2 °C while being transported from the sampling location to the laboratory.

The *Standard Guide for Sampling Waste and Solids for Volatile Organics* (ASTM D 4547-98) is recommended reading for those unfamiliar with the many challenges associated with collecting and handling samples for VOC analysis.

7 IMPLEMENTATION: SELECTING EQUIPMENT AND CONDUCTING SAMPLING

This section provides guidance on selecting appropriate sampling tools and devices (Section 7.1), conducting field sampling activities (Section 7.2), and using sample homogenization, splitting, and subsampling techniques (Section 7.3).

7.1 Selecting Sampling Tools and Devices

The tools, devices, and methods used for sampling waste materials will vary with the form, consistency, and location of the waste materials to be sampled. As part of the DQO Process, you identify the location (type of unit or other source description) from which the samples will be obtained and the "dimension" of the sampling problem (such as "one-dimensional" or "two-dimensional"). In the DQO Process, you also specify the appropriate size, shape, orientation and other characteristics for each sample (called the "sample support"). In addition to the DQOs for the sample, you will identify performance goals for the sampling device. You may need a device that meets the following qualifications:

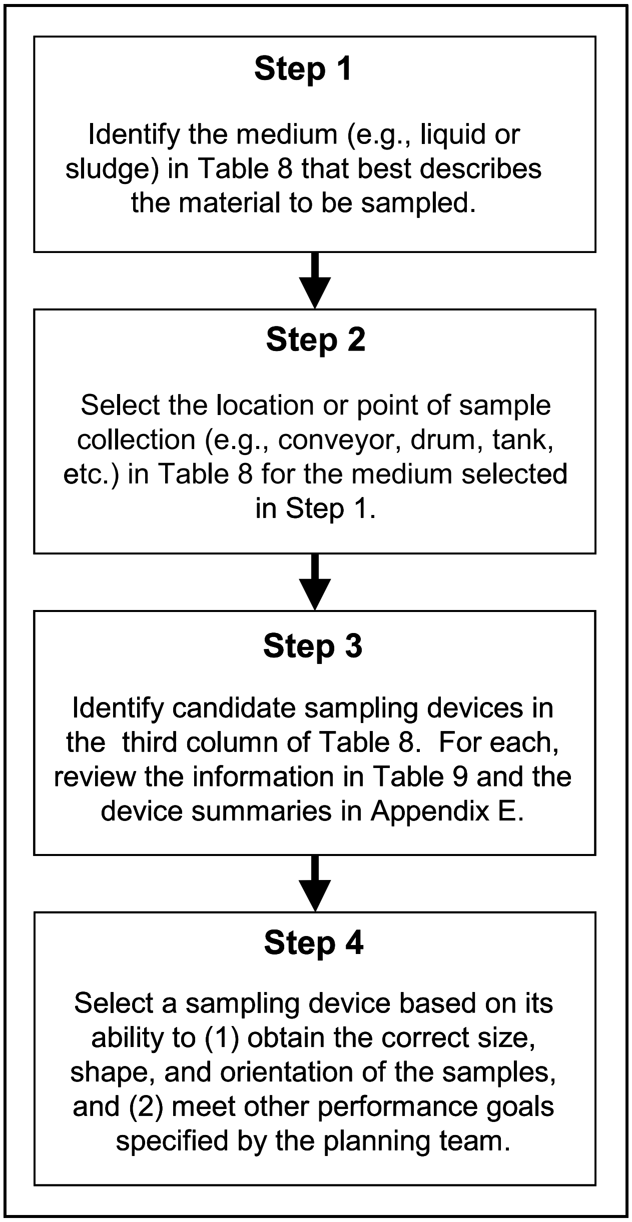
For additional guidance on the selection and use of sampling tools and devices, see:

- 40 CFR 261, Appendix I, Representative Sampling Methods
- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities* (ASTM D 6232)

- Minimizes delimitation and extraction errors so that it does not include material that should not be in the sample, nor exclude material that should be in the sample
- Provides a largely undisturbed sample (e.g., one that minimizes the loss of volatile constituents, if those are constituents of concern)
- Is constructed of materials that are compatible with the media and the constituents of concern (e.g., the materials of construction do not cause constituent loss or gain due to sorption, desorption, degradation, or corrosion)
- Is easy to use under the conditions of the sampling location, and the degree of health or safety risks to workers is minimal
- Is easy to decontaminate
- Is cost-effective during use and maintenance.

Unfortunately, few devices will satisfy all of the above goals for a given waste or medium and sampling design. When selecting a device, try first to choose one that will introduce the least sampling error and satisfy other performance criteria established by the planning team, within practical constraints.

Figure 28 summarizes the steps you can use to select an optimal device for obtaining samples.



Step 1

Identify the medium (e.g., liquid or sludge) in Table 8 that best describes the material to be sampled.

Step 2

Select the location or point of sample collection (e.g., conveyor, drum, tank, etc.) in Table 8 for the medium selected in Step 1.

Step 3

Identify candidate sampling devices in the third column of Table 8. For each, review the information in Table 9 and the device summaries in Appendix E.

Step 4

Select a sampling device based on its ability to (1) obtain the correct size, shape, and orientation of the samples, and (2) meet other performance goals specified by the planning team.

Figure 28. Steps for selecting a sampling device

Using the outputs from the DQO Process, a description of the medium to be sampled, and knowledge of the site or location of sample collection, Tables 8 and 9 (beginning on pages 109 and 115 respectively) can be used to quickly identify an appropriate sampling device. For most situations, the information in the tables will be sufficient to make an equipment selection; however, if you need additional guidance, review the more detailed information provided in Appendix E or refer to the references cited.

If desired, you can refer to the documents (such as ASTM standards) referenced by Table 8 for supplementary guidance specific to sampling a specific medium and site, or refer to those referenced by Table 9 for supplementary guidance on a device.[1] The contents of the ASTM standards are summarized in Appendix J. (For more information on ASTM or purchasing their publications, including the standards referenced in this chapter, contact ASTM at: ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959, or by telephone at 610-832-9585, via the World Wide Web at http://www.astm.org.)

In particular, we recommend that you review the guidance found in ASTM Standard D 6232, *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*. Most of the information on sampling devices found in this chapter and in Tables 8 and 9 came from that standard. As noted by the standard, it covers criteria that should be considered when selecting sampling equipment for collecting environmental and waste samples for waste management activities. It also describes many of the typical devices used during such sampling.

Because each sampling situation is unique, the guidance in this chapter may not adequately cover your specific sampling scenario. You may have to modify a part of the device or modify the device application to improve its performance or to facilitate sample collection. For

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[1] ASTM is a consensus standards development organization. Consistent with the provisions of the National Technology Transfer and Advancement Act of 1995 (NTTAA), Public Law 104-113, Section 12(d), which directs EPA to use voluntary consensus standards to the extent possible, this guidance supports the use of and provides references to ASTM standards applicable to waste sampling.

example, you might use a rope or an extension handle on a device to access a particular location within a waste management unit. In other cases, you may need auxiliary equipment that will increase the cost or complexity of sampling operation (such as a drill rig to drive a split barrel sampler or a power supply to run a pump). The physical state of the waste or design of the unit also may affect how the equipment is deployed. You should address such variations as part of your sampling plan and make sure that any modifications do not cause sampling bias.

Finally, other sampling devices not addressed in this chapter can and should be used if appropriate (e.g., if the device meets the performance goals and is more practical). New or innovative devices not discussed in this chapter also should be considered for use if they allow you to meet the sampling objectives in a more cost-effective manner. In other words, we encourage and recommend a performance-based approach for selecting sampling equipment.

7.1.1 Step 1: Identify the Waste Type or Medium to be Sampled

The first column of Table 8 (page 109) lists the media type or waste matrix commonly sampled under RCRA. These media may include liquids, sludges or slurries, various unconsolidated solids, consolidated solids and debris, soil, ground water, sediment, soil gas, and air. In general, the types of media describe the physical state of the material to be sampled. The physical characteristics of the waste or medium affect many aspects of sampling, including the volume of material required, selection of the appropriate sampling device, how the device is deployed, and the containers used for the samples. Table 10 provides an expanded description of the media listed in Table 8.

7.1.2 Step 2: Identify the Site or Point of Sample Collection

In the second column of Table 8, identify the site or point of sample collection that best describes where you plan to obtain the samples. The “site or point of sample collection” may include (1) the point at which the waste is generated (e.g., as the waste exits a pipe, moves along a conveyor, or is poured or placed into a container, tank, impoundment or other waste management unit); (2) the unit in which the waste is stored (such as a drum, collection hopper, tank, waste pile, surface impoundment, sack or bag) or transported (such as a drum, tanker truck, or roll-off box); or (3) the environmental medium to be sampled (such as surface soil, subsurface soil, ground water, surface water, soil gas, or air).

When testing a solid waste to determine if it should be characterized as a hazardous waste or to determine if the waste is restricted from land disposal, such a determination must be made at the **point of waste generation**.

7.1.2.1 Drums and Sacks or Bags

Drums and sacks or bags are portable containers used to store, handle, or transport waste materials and sometimes are used in waste disposal (e.g., drums in a landfill). “Drums” include metal drums and pails, plastic drums, or durable fiberboard paper drums or pails (USEPA 1994a). Drums and pails may contain nearly the full range of media -- liquids (single or multi-layered), sludges, slurries, or solids. Sacks or bags include less rigid portable containers and thus can contain only solids. The sampling approach (including number of samples, locations of samples, sampling device, depth of samples) for these containers will depend on the number of containers to be sampled, waste accessibility, physical and chemical characteristics of the waste, and component distribution within the containers.

Review ASTM Standards D 6063, *Guide for Sampling Drums and Similar Containers by Field Personnel*, and D 5679, *Practice for Sampling Consolidated Solids in Drums or Similar Containers*, for more information on the sampling of drums and sacks or bags. Other useful guidance on sampling drums includes "Drum Sampling" (USEPA 1994b), issued by EPA's Environmental Response Team.

7.1.2.2 Surface Impoundments

Surface impoundments include natural depressions, manmade excavations, or diked areas that contain an accumulation of liquids or wastes containing free liquids and solids. Examples of surface impoundments are ponds, lagoons, and holding, storage, settling, and aeration pits (USEPA 1994a). The appropriate sampling device for sampling a surface impoundment will depend on accessibility of the waste, the type and number of phases of the waste, the depth, and chemical and physical characteristics of the waste.

7.1.2.3 Tanks

A tank is defined at § 260.10 as a stationary device, designed to contain an accumulation of hazardous waste which is constructed primarily of non-earthen materials which provide structural support. A container is defined at § 260.10 as a portable device, in which a material is stored, transported, treated, disposed of, or otherwise handled. The distinction that a tank is not a container is important because the regulations at 261.7 set forth conditions to distinguish whether hazardous waste in a container is subject to regulation. Nevertheless, for the purpose of selecting an appropriate sampling device, the term "tank" as used in Table 8 could include other units such as tank trucks and tanker cars even though they are portable devices.

The selection of equipment for sampling the pipes and sampling ports of a tank system is covered separately under those categories. The equipment used to sample a pipe or spigot can be very different from that used to sample an open tank.

Tanks usually contain liquids (single or multi-layered), sludges, or slurries. In addition, suspended solids or sediments may have settled in the bottom of the tank. When sampling from a tank, one typically considers how to acquire a sufficient number of samples from different locations (including depths) to adequately represent the entire content of the tank.

Waste accessibility and component distribution will affect the sampling strategy and equipment selection. In addition to discharge valves near the bottom, most tanks have hatches or other openings at the top. It is usually desirable to collect samples via a hatch or opening at the top of the tank because of the potential of waste stratification in the tank (USEPA 1996b). In an open tank, the size of the tank may restrict sampling to the perimeter of the tank. Usually, the most appropriate type of sampling equipment for tanks depends on the design of the tanks and the media contained within the tank.

You can find additional guidance on sampling tanks in "Tank Sampling" (USEPA 1994c), issued by the EPA's Environmental Response Team.

7.1.2.4 Pipes, Point Source Discharges, or Sampling Ports

For the purpose of this guidance, pipes or point source discharges include moving streams of sludge or slurry discharging from a pipe opening, sluice, or other discharge point (such as the point of waste generation). Sampling ports include controlled liquid discharge points that were installed for the purpose of sampling, such as may be found on tank systems, a tank truck, or leachate collection systems at waste piles or landfills.

A dipper also is used to sample liquids from a sampling port. Typically, it is passed through the stream in one sweeping motion so that it is filled in one pass. In that instance, the size of the dipper beaker should be related to the stream flow rate. If the cross-sectional area of the stream is too large, more than one pass may be necessary to obtain a sample (USEPA 1993b). Besides the use of a dipper or other typical sampling devices, sometimes the sample container itself is used to sample a spigot or point source discharge. This eliminates the possibility of contaminating the sample with intermediate collection equipment, such as a dipper (USEPA 1996b).

See ASTM D 5013-89 *Standard Practices for Sampling Wastes from Pipes and Other Point Discharges* for more information on sampling at this location. Also see Gy (1998) and Pitard (1989, 1993).

7.1.2.5 Storage Bins, Roll-Off Boxes, or Collection Hoppers

Discharges of unconsolidated solids from a process, such as filter cakes, often fall from the process into a collection hopper or other type of open-topped storage container. Sometimes the waste materials are combined into large a storage bin, such as a roll-off box or collection hopper. A storage bin also may be used to collect consolidated solids, such as construction debris. The waste can be sampled either as it is placed in the container or after a certain period of accumulation, depending on the technical and regulatory objectives of the sampling program.

7.1.2.6 Waste Piles

Waste piles include the non-containerized accumulation of solid and nonflowing waste material on land. The size of waste piles can range from small heaps to large aggregates of wastes. Liners may underlie a waste pile, thereby preventing direct contact with the soil. As with other scenarios, waste accessibility and heterogeneity will be key factors in the sampling design and equipment selection. Besides the devices listed in this chapter, excavation equipment may be needed at first to properly sample large piles. Waste piles may present unique sample delimitation problems (Pitard 1993 and Myers 1997), and special considerations related to sampling design may be necessary (such as the need to flatten the pile).

We recommend a review of ASTM Standard D 6009, *Guide for Sampling Waste Piles* for more information. Another source of information on sampling waste piles is "Waste Pile Sampling" (USEPA 1994d), issued by EPA’s Environmental Response Team.

7.1.2.7 Conveyors

Solid process discharges are sometimes sampled from conveyors such as conveyor belts or screw conveyors. Conveyor belts are open moving platforms used to transport material between locations. Solid or semi-solid wastes on a conveyor belt can be sampled with a flat scoop or similar device (see also Section 6.3.2.1). Screw conveyors usually are enclosed systems that require access via a sampling port, or they can be sampled at a discharge point. See also ASTM D 5013 and Gy (1998, pages 43 through 56).

7.1.2.8 Structures and Debris

This guidance assumes that the sampling of structure or debris typically will include the sampling of consolidated solids such as concrete, wood, or other structure debris. Appendix C provides supplemental guidance on developing a sampling strategy for such heterogeneous wastes. See also AFCEE (1995), Koski, et al. (1991), Rupp (1990), USEPA and USDOE (1992), and ASTM Standard D 5956, *Standard Guide For Sampling Strategies for Heterogeneous Wastes*.

7.1.2.9 Surface or Subsurface Soil

Selection of equipment for sampling soil is based on the depth of sampling, the grain-size distribution, physical characteristics of the soil, and the chemical parameters of interest (such as the need to analyze the samples for volatiles). Your sampling strategy should specify the depth and interval (e.g., “0 to 6 inches below ground surface”) of interest for the soil samples.

Simple manual techniques and equipment can be used for surface or shallow depth sampling. To obtain samples of soil from greater depths, powered equipment (e.g., power augers or drill rigs) will be required; however, those are not used for actual sample collection, but are used solely to gain easier access to the required sample depth (USEPA 1996b). Once at the depth, surface sampling devices may be used.

ASTM has developed many informative standards on the sampling of soil, including D 4700, *Standard Guide for Soil Sampling from the Vadose Zone*, and D 4220, *Standard Practices for Preserving and Transporting Soil Samples*. In addition, see EPA-published guidance such as *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (Mason 1992) and *Description and Sampling of Contaminated Soils - A Field Pocket Guide* (USEPA 1991b).

7.1.3 Step 3: Consider Device-Specific Factors

After you identify the medium and site of sample collection, refer to the third column of Table 8 for the list of candidate sampling devices. We listed common devices that are appropriate for the given media and site. Next, refer to the information in Table 9 for each of the candidate devices to select the most appropriate one for your sampling effort.

Table 9 provides device-specific information to help you choose the appropriate device based on the study objective and the DQOs established for volume (size), shape, depth, and orientation of the sample, and sample type (discrete or composite, surface or at depth).

For easy reference, the devices are listed alphabetically in Table 9. Appendix E contains a summary description of key features of each device and sources for other information. Under the third column in Table 9, “Other Device-Specific Guidance,” we have identified some of those sources, especially relevant ASTM standards (see summaries of ASTM standards in Appendix J).

7.1.3.1 Sample Type

The column “Sample Type” Table 9 identifies whether the device can sample at surface only, shallow or at a deeper profile (depth), and whether the device can obtain a discrete sample or a composite sample. For example, a COLIWASA or drum thief can be used to sample a container that is 3-feet deep, but a Kemmerer sampler may be required to sample the much deeper depth of an impoundment. We also identify in this column whether the device collects a undisturbed or disturbed solid sample. Also, the actual depth capacity may depend on the design of the device. Some devices can be modified or varied to collect at different depths or locations in a material. You should refer to the device summary in Appendix E if you need specifics regarding the sampling depth available for a given device.

7.1.3.2 Sample Volume

The column for volume in Table 9 identifies the range of sample volume, in liters, that the device can obtain. It may be possible to increase or decrease this value through modification of the device. During the planning process, you should determine the correct volume of sample needed. Volume is one of the components of sample “support” (that is, the size, shape, and orientation of the sample).

7.1.3.3 Other Device-Specific Considerations

The last column of Table 9 notes other considerations for device selection. The comments focus on those factors that may cause error to be introduced or that might increase the time or cost of sampling. For some devices, the column includes comments on how easy the equipment is to use, such as whether it needs a power source or is heavy, and whether it can be decontaminated easily. The table also mentions whether the device is appropriate for samples requiring the analysis of volatile organic constituents and any other important considerations regarding analyte and device compatibility. The equipment should be constructed of materials that are compatible with the waste and not susceptible to reactions that might alter or bias the physical or chemical characteristics of the sample of the waste.

7.1.4 Step 4: Select the Sampling Device

Select the sampling device based on its ability to (1) obtain the correct size, shape, and orientation of the samples (see Sections 6.3.1 and 6.3.2) and (2) meet any other performance criteria specified by the planning team in the DQO Process (see Section 6.3.3.1). In addition, samples to be analyzed for volatile organic constituents should be obtained using a sampling technique that will minimize the loss of constituents and obtain a sample volume required for the analytical method (see Section 6.3.4).

Table 8. Device Selection Guide -- Media and Site of Sample Collection

Media (See Section 7.1.1)	***Site or Point of Sample Collection (See Section 7.1.2)***	***Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)***	***Other Related Guidance***
Liquids, no distinct layer of interest Examples: Containerized spent solvents, leachates or other liquids discharged from a pipe or spigot	Drum	COLIWASA Dipper Drum thief Liquid grab sampler Peristaltic pump Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler Valved drum sampler	ASTM D 5743 ASTM D 6063 EPA/ERT SOP 2009 (USEPA 1994b)
	Surface impoundment	Automatic sampler Bacon bomb Bailer Bladder pump Centrifugal sub-pump Dipper Displacement pump Kemmerer sampler Liquid grab sampler Peristaltic pump Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler	ASTM D 6538 USEPA (1984, 1985, and 1989c)
	Tank	Bacon bomb Bailer COLIWASA Dipper Drum thief Kemmerer sampler Liquid grab sampler Peristaltic pump Plunger type sampler Settleable solids profiler Submersible pump Swing jar sampler Syringe sampler	ASTM D 6063 ASTM D 5743 EPA/ERT SOP 2010 (USEPA 1994c)

* Copies of EPA/ERT SOPs are available on the Internet at http://www.ert.org/

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Media (See Section 7.1.1)	*Site or Point of Sample Collection (See Section 7.1.2)*	*Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)*	*Other Related Guidance*
Liquids, no distinct layer of interest (continued)	Pipe, point source discharge	Automatic sampler Bladder pump Centrifugal submersible pump Dipper Displacement pump Liquid grab sampler Plunger type sampler Sample container Swing jar sampler	ASTM D 5013 ASTM D 5743 ASTM D 6538 Gy 1998
	Sampling port (e.g., spigot)	Beaker, bucket, sample container Swing jar sampler	Gy 1998
Liquids, multi-layered, with one or more distinct layers of interest Examples: Non-aqueous phase liquids (NAPLs) in a tank; mixtures of antifreeze in a tank.	Drum	COLIWASA Discrete level sampler Drum thief Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler Valved drum sampler	ASTM D 6063
	Surface impoundment	Automatic sampler Bacon bomb Bailer (point source bailer) Bladder pump Centrifugal submersible pump Discrete level sampler Displacement pump Peristaltic pump Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler	ASTM D 6538 USEPA (1989c)
	Tank	COLIWASA Centrifugal submersible pump Bacon bomb Bailer Discrete level sampler Peristaltic pump Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler Valved drum sampler	ASTM D 6063 ASTM D 5743 EPA/ERT SOP 2010 (USEPA 1994c)

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Media (See Section 7.1.1)	***Site or Point of Sample Collection (See Section 7.1.2)***	***Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)***	***Other Related Guidance***
Sludges, slurries, and solid-liquid suspensions Examples: Paint sludge, electroplating sludge, and ash and water slurry.	Drum	COLIWASA Dipper Liquid grab sampler Plunger type sampler Settleable solids profiler Swing jar sampler Syringe sampler	ASTM D 6063
	Tank	COLIWASA Dipper Lidded sludge/water sampler Liquid grab sampler Plunger type sampler Ponar dredge Settleable solids profiler Swing jar sampler Syringe sampler	ASTM D 6063 EPA/ERT 2010 (USEPA 1994c)
	Surface impoundment	Dipper Lidded sludge/water sampler Liquid grab sampler Peristaltic pump Plunger type sampler Ponar dredge Settleable solids profiler Swing jar sampler	USEPA (1989c)
	Pipe or conveyor	Dipper or bucket Scoop/trowel/shovel Swing jar sampler	ASTM D 5013
Granular solids – unconsolidated Examples: Filter press cake, powders, excavated (*ex situ*) soil, incinerator ash	Drum	Bucket auger Coring type sampler (w/valve) Miniature core sampler Modified syringe sampler Trier Scoop/trowel/shovel	ASTM D 5680 ASTM D 6063 EPA/ERT SOP 2009 (USEPA 1994b)
	Sack or bag	Concentric tube thief Miniature core sampler Modified syringe sampler Scoop/trowel/shovel Trier	ASTM D 5680 ASTM D 6063

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Media (See Section 7.1.1)	***Site or Point of Sample Collection (See Section 7.1.2)***	***Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)***	***Other Related Guidance***
Granular solids – unconsolidated (continued)	Storage bin, roll-off box, or collection hopper	Bucket auger Concentric tube thief Coring type sampler (w/valve) Miniature core sampler Modified syringe sampler Scoop/trowel Trier	ASTM D 5680 ASTM D 6063
	Waste pile	Bucket auger Concentric tube thief Coring type sampler (w/valve) Miniature core sampler Modified syringe sampler Scoop/trowel/shovel Thin-walled tube Trier	ASTM D 6009 EPA/ERT SOP 2017 (USEPA 1994d)
	Pipe (e.g., vertical discharge from cyclone centrifuge or baghouse) or conveyor	Bucket, dipper, pan, or sample container Miniature core sampler Scoop/trowel/shovel Trier	ASTM D 5013 Gy (1998) Pitard (1993)
Other solids – unconsolidated Examples: Waste pellets, catalysts, or large-grained solids.	Drum	Bucket auger Scoop/trowel/shovel	ASTM D 5680 ASTM D 6063 EPA/ERT SOP 2009 (USEPA 1994b)
	Sack or bag	Bucket auger Scoop/trowel/shovel	ASTM D 5680 ASTM D 6063
	Storage bin, roll-off box, or collection hopper	Bucket auger Scoop/trowel/shovel	ASTM D 5680 ASTM D 6063
	Waste pile	Bucket auger Scoop/trowel/shovel Split barrel Thin-walled tube	ASTM D 6009 EPA/ERT SOP 2017 (USEPA 1994d)
	Conveyor	Scoop/trowel/shovel	ASTM D 5013 Gy (1998) Pitard (1993)

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Media (See Section 7.1.1)	***Site or Point of Sample Collection (See Section 7.1.2)***	***Candidate Devices (Listed Alphabetically. For Device-Specific Information, See Table 9)***	***Other Related Guidance***
Soil and other unconsolidated geologic material Examples: *In situ* soil at a land treatment unit or *in situ* soil at a SWMU	Surface	Bucket auger Concentric tube thief Coring type sampler Miniature core sampler Modified syringe sampler Penetrating probe sampler Scoop/trowel/shovel Thin-Walled Tube Trier	ASTM D 5730 ASTM E 1727 ASTM D 4700 EISOPQA Manual (USEPA 1996b)
	Subsurface	Bucket auger Coring type sampler Miniature core sampler Mod. syringe sampler Penetrating probe sampler Shovel/scoop/shovel Split barrel Thin-walled tube	ASTM D 4700 ASTM D 5730 ASTM D 6169 ASTM D 6282 USEPA (1996b) USEPA (1993c)
Solids – consolidated Examples: Concrete, wood, architectural debris*	Storage bin (e.g., roll-off box)	Penetrating probe sampler Rotating coring device	ASTM D 5679 ASTM D 5956 ASTM D 6063 USEPA and USDOE (1992)
	Waste pile	Penetrating probe sampler Rotating coring device Split barrel	ASTM D 6009 USEPA and USDOE (1992)
	Structure	Rotating coring device (See also Appendix C, Section C.5)	AFCEE (1995) Koski, et al (1991) USEPA and USDOE (1992)

* The term "debris" has a specific definition under 40 CFR 268.2(g) (Land Disposal Restrictions regulations) and includes "solid material exceeding a 60 mm particle size that is intended for disposal and that is a manufactured object; or plant or animal matter; or natural geologic material." § 268.2(g) also identifies materials that are not debris. In general, debris includes materials of either a large particle size or variation in the items present.

Table 8. Device Selection Guide -- Media and Site of Sample Collection (Continued)

Selected References for Sampling of Other Media	
Air Example: BIF emissions	Chapter Ten SW-846 EISOPQA Manual (USEPA 1996b)
Sediment Example: Surface impoundment sediment	*QA/QC Guidance for Sampling and Analysis of Sediments, Water, and Tissues for Dredged Material Evaluations* (USEPA 1995d) *Superfund Program Representative Sampling Guidance Volume 5; Water and Sediment, Part I – Surface Water and Sediment, Interim Final Guidance* (USEPA 1995e) Region 4 EISOPQA Manual (USEPA 1996b) *Sediment Sampling* (USEPA 1994e) ASTM D 4823; ASTM D 5387
Soil Gas or Vapor Examples: Soil, soil water, or gas in the vadose zone at a waste disposal site	*Subsurface Characterization and Monitoring Techniques - A Desk Reference Guide* (USEPA 1993c) ASTM *Standard Guide for Soil Gas Monitoring in the Vadose Zone* (ASTM D 5314) *Soil Gas Sampling* (USEPA 1996c)
Ground Water Example: Ground-water monitoring wells at a landfill	*RCRA Ground-Water Monitoring Draft Technical Guidance* (USEPA 1992c) *Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures* (Puls and Barcelona 1996) ASTM D4448-01 Standard Guide for Sampling Ground-Water Monitoring Wells ASTM D 5092-90 *Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers* ASTM D 6286-98 *Standard Guide for Selection of Drilling Methods for Environmental Site Characterization* ASTM D 6282 *Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations* ASTM D 6771-02 *Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations*

Table 9. Device Selection Guide – Device-Specific Factors

Sampling Device (Listed in Alphabetical Order)	***Description, Appendix E, Section No.***	***Other Device-Specific Guidance (in Addition to ASTM D 6232)***	***Sample Type***	***Volume (Liters per Pass)***	***Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)***
Automatic sampler	E.1.1	ASTM D 6538 EISOPQA Manual (USEPA 1996b)	Shallow (25 in.), discrete or composite	Unlimited	Auto samplers are available to collect samples for volatile organics analysis, provide a grab or composite sample, and may be unattended. Need power source/battery. Commonly used at waste water treatment plants. Must be knowledgeable of compatibility of waste and sampler components.
Bacon bomb	E.3.1	USEPA 1984 USEPA 1994c	Depth, discrete	0.1 to 0.5	For parameters that do not require a polytetrafluroethylene (PTFE) sampler. Recommended for sampling of lakes, ponds, large tanks, or lagoons. May be difficult to decontaminate and materials of construction may not be compatible with sample matrix.
Bailer	E.7.1	ASTM D 4448 USEPA 1992c USEPA 1994c	Depth, discrete	0.5 to 2.0	Bailers are not recommended for sampling ground water for trace constituent analysis due to sampling induced turbidity (USEPA 1992c and Puls and Barcelona 1996). Unable to collect samples from specific depths (unless a point-source bailer is used). Available in a variety of sizes as either reusable or single use devices. May be chemically incompatible with certain matrices unless constructed of resistant material.
Bladder pump	E.1.2	ASTM D 4448 USEPA 1992c USEPA 1996b	Depth, discrete	Unlimited	For purging or sampling of wells, surface impoundments, or point discharges. Contact parts are made of PTFE, PVC and stainless steel. Requires a power source, compressed gas, and a controller. Difficult to decontaminate (based on design). Suitable for samples requiring VOAs. May require a winch or reel.
Bucket auger	E.5.1	ASTM D 1452 ASTM D 4700 ASTM D 6063 Mason 1992 USEPA 1993c	Surface or depth, disturbed	0.2 to 1.0	Easy and quick for shallow subsurface samples but not recommended for VOAs. Requires considerable strength and labor and destroys soil horizons.

Table 9. Device Selection Guide – Device-Specific Factors (Continued)

Sampling Device (listed in alphabetical order)	*Description, Appendix E, Section*	*Other Device-Specific Guidance (in addition to ASTM D 6232)*	*Sample Type*	*Volume (Liters Per Pass)*	*Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)*
Centrifugal submersible pump	E.1.4	ASTM D 4448 ASTM D 4700 USEPA 1992c	Depth, discrete	Unlimited	For purging or sampling wells, surface impoundments, or point discharges. Contact parts are made of PTFE and stainless steel. Requires a power source. Adjustable flow rate and easy to decontaminate. Not compatible with liquids containing high percent solids. May require a winch or reel.
COLIWASA	E.6.1	ASTM D 5495 ASTM D 5743 ASTM D 6063 USEPA 1980	Shallow, composite	0.5 to 3.0	Reusable and single use models available. Inexpensive. Glass type devices may be difficult to decontaminate. Collects undisturbed sample. For mixed solid/liquid media will collect semi-liquid only. Not for high viscosity liquids.
Concentric tube thief	E.4.3	ASTM D 6063 USEPA 1994d	Surface, relatively undisturbed, selective	0.5 to 1.0	Recommended for powdered or granular materials or wastes in piles or in bags, drums or similar containers. Best used in dry, unconsolidated materials. Not suitable for sampling large particles due to narrow width of slot.
Coring type sampler (with or without valve)	E.4.6	ASTM D 4823 USEPA 1989c	Surface or depth, disturbed	0.2 to 1.5	Designed for wet soils and sludge. May be equipped with a plastic liner and caps. May be used for VOAs. Reusable and easy to decontaminate.
Dipper (or “pond sampler”)	E.7.2	ASTM D 5358 ASTM D 5013 USEPA 1980	Shallow, composite	0.5 to 1.0	For sampling liquids in surface impoundments. Inexpensive. Not appropriate for sampling stratified waste if discrete characterization needed.
Discrete level sampler	E.3.5		Depth, discrete	0.2 to 0.5	Easy to decontaminate. Obtains samples from a discrete interval. Limited by sample volume and liquids containing high solids. Can be used to store and transport sample.
Displacement pumps	E.1.5	ASTM D 4448	Depth, discrete	Unlimited	Can be used for purging or sampling of wells, impoundments, or point discharges. Contact parts are made of PVC, stainless steel, or PTFE to reduce risk of contamination when trace levels or organics are of interest. Requires a power source and a large gas source. May be difficult to decontaminate (piston displacement type). May require a winch or reel to deploy.

Table 9. Device Selection Guide – Device-Specific Factors (Continued)

Sampling Device (listed in alphabetical order)	*Description, Appendix E, Section*	*Other Device-Specific Guidance (in addition to ASTM D 6232)*	*Sample Type*	*Volume (Liters Per Pass)*	*Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)*
Drum thief	E.6.2	ASTM D 6063 ASTM D 5743 USEPA 1994b	Shallow, composite	0.1 to 0.5	Usually single use. If made of glass and reused, decontamination may be difficult. Limited by length of sampler, small volume of sample collected, and viscosity of fluids.
Kemmerer sampler	E.3.2		Depth, discrete	1.0 to 2.0	Recommended for lakes, ponds, large tanks or lagoons. May be difficult to decontaminate. Materials may not be compatible with sample matrix but all PTFE construction is available. Sample container exposed to media at other depths while being lowered to sample point.
Lidded sludge/water sampler	E.3.4		Discrete, composite	1.0	1-L sample jar placed into device (low risk of contamination). May sample at different depths and samples up to 40-percent solids. Equipment is heavy and limited to one bottle size.
Liquid grab sampler	E.7.3		Shallow, discrete, composite-suspended solids only	0.5 to 1.0	For sampling liquids or slurries. Can be capped and used to transport sample. Easy to use. May be lowered to specific depths. Compatibility with sample parameters is a concern.
Miniature core sampler	E.4.7	ASTM D 4547 ASTM D 6418	Discrete	0.01 to 0.05	Used to retrieve samples from surface soil, trench walls, or sub-samples from soil cores. O-rings on plunger and cap minimize loss of volatiles and allow device to be used to transport sample. Designed for single use. Cannot be used on gravel or rocky soils must avoid trapping air with samples.
Modified syringe sampler	E.4.8	ASTM D 4547	Discrete	0.01 to 0.05	Made by modifying a plastic, medical, single-use syringe. Used to collect a sample from a material surface or to sub-sample a core. The sample is transferred to a vial for transportation. Inexpensive. Must ensure device is clean and compatible with media to be sampled.

Table 9. Device Selection Guide – Device-Specific Factors (Continued)

Sampling Device (listed in alphabetical order)	*Description, Appendix E, Section*	*Other Device-Specific Guidance (in addition to ASTM D 6232)*	*Sample Type*	*Volume (Liters Per Pass)*	*Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)*
Penetrating probe sampler	E.4.1	USEPA 1993c	Discrete, undisturbed	0.2 to 2.0	Used to sample soil vapor, soil, and ground water (pushed or hydraulically driven). Versatile, make samples available for onsite analysis and reduces investigation derived waste. Limited by sample volume and composition of subsurface material.
Peristaltic pump	E.1.3	ASTM D 4448 ASTM D 6063 USEPA 1996b	Shallow, discrete or composite-suspended solids only	Unlimited	Possible to collect samples from multiple depths up to 25 feet. Decontamination of pump is not required and tubing is easy to replace. Can collect samples for purgeable organics with modified equipment, but may cause loss of VOAs.
Plunger type sampler	E.6.4	ASTM D 5743	Surface or depth, discrete	0.2 to Unlimited	Made of high-density polyethylene (HDPE) or PTFE with optional glass sampling tubes. Used to collect a vertical column of liquid. Either a reusable or single use device. Decontamination may be difficult (with glass tubes).
Ponar dredge	E.2.1	ASTM D 4387 ASTM D 4342 USEPA 1994e	Bottom surface, rocky or soft, disturbed	0.5 to 3.0	One of the most effective samplers for general use on all types of substrates (silt to granular material). May be difficult to repeatedly collect representative samples. May be heavy.
Rotating coring device	E.5.2	ASTM D 5679	Surface or depth, undisturbed	0.5 to 1.0	May obtain a core of consolidated solid. Requires power and water source and is difficult to operate. Sample integrity may be affected.
Scoop	E.7.5	ASTM D 5633 ASTM D 4700 ASTM D 6063	Surface, disturbed, selective	<0.1 to 0.6	Usually for surface soil and solid waste samples. Available in different materials and simple to obtain. May bias sample because of particle size. May exacerbate loss of VOCs.
Settleable solids profiler	E.6.5		Depth, composite-suspended solids only	1.3 to 4.0	Typically used at waste water treatment plants, waste settling ponds, and impoundments to measure and sample settleable solids. Easy to assemble, reusable and unbreakable under normal use. Not recommended for caustics or high viscosity materials.

Table 9. Device Selection Guide – Device-Specific Factors (Continued)

Sampling Device (listed in alphabetical order)	*Description, Appendix E, Section*	*Other Device-Specific Guidance (in addition to ASTM D 6232)*	*Sample Type*	*Volume (Liters Per Pass)*	*Comments (For Example: Effects on Matrix, Operational Considerations, Typical Uses)*
Shovel	E.7.5	ASTM D 4700	Surface, disturbed	1.0 to 5.0	Used to collect surface material or large samples from waste piles. Easy to decontaminate and rugged. Limited to surface use and may exacerbate the loss of samples for VOAs.
Split barrel sampler	E.4.2	ASTM D 1586 ASTM D 4700 ASTM D 6063	Discrete, undisturbed	0.5 to 30.0	May be driven manually, or mechanically by a drill rig with trained personnel. May collect a sample at depth. A liner may be used in the device to minimize disturbance or for samples requiring VOAs.
Swing jar sampler	E.7.4		Shallow, composite	0.5 to 1.0	Used to sample liquids, powders, or small solids at a distance up to 12 feet. Adaptable to different container sizes. Not suitable for discrete samples. Can sample a wide variety of locations.
Syringe sampler	E.3.3	ASTM D 5743 ASTM D 6063	Shallow, discrete, disturbed	0.2 to 0.5	Recommended for highly viscous liquids, sludges and tar-like substances. Easy to decontaminate. Obtains samples at discrete depths but limited to length of device. Waste must be viscous enough to stay in sampler.
Thin-walled tube	E.4.5	ASTM D 1587 ASTM D 4823 ASTM D 4700	Surface or depth, undisturbed	0.5 to 5.0	Useful for collecting an undisturbed sample (depends on extension). May require a catcher to retain soil samples. Inexpensive, easy to decontaminate. Samples for VOAs may be biased when sample is extruded.
Trier	E.4.4	ASTM D 5451 ASTM D 6063	Surface, relatively undisturbed, selective	0.1 to 0.5	Recommended for powdered or granular materials or wastes in piles or in bags, drums, or similar containers. Best for moist or sticky materials. Will introduce sampling bias when used to sample coarse-grained materials.
Trowel	E.7.5	ASTM D 5633 ASTM D 4700 ASTM D 6063	Surface, disturbed, selective	0.1 to 0.6	Usually for surface soil and solid waste samples. Available in different materials and simple to obtain. May bias sample because of particle size, and may exacerbate loss of VOAs.
Valved drum sampler	E.6.3		Shallow, composite	0.3 to 1.6	Used to collect a vertical column of liquid. Available in various materials for repeat or single use. High viscosity liquids may be difficult to sample.

Table 10. Descriptions of Media Listed in Table 8.

Media	***Description***	***Examples***
Liquids -- no distinct layer of interest	Liquids (aqueous or nonaqueous) that are or are not stratified and samples from discrete intervals are not of interest. Sampling devices for this medium do not need to be designed to collect liquids at discrete depths.	Containerized leachates or spent solvents; leachates or other liquids released from a spigot or discharged from a pipe.
Liquids -- one or more distinct layers of interest	Liquids (aqueous or nonaqueous) that are stratified with distinct layers and collection of samples from discrete intervals is of interest. Sampling devices for this media do need to be designed to collect liquids at discrete depths.	Mixtures of antifreeze and used oil; light or dense non-aqueous phase liquids and water in a container, such as a tank.
Sludges or slurries	Materials that are a mixture of liquids and solids and that may be viscous or oily. Includes materials with suspended solids.	Waste water treatment sludges from electroplating; slurry created by combining solid waste incinerator ash and water.
Granular solids, unconsolidated	Solids which are not cemented, or do not require significant pressure to separate into particles, and are comprised of relatively small particles or components.	Excavated (*ex situ*) soil in a staging pile; filter press cake; fresh cement kiln dust; incinerator ash.*
Other solids, unconsolidated	Solids with larger particles than those covered by granular solids. The sampling device needs to collect a larger diameter or volume of sample to accommodate the larger particles.	Waste pellets or catalysts.

* For EPA-published guidance on the sampling of incinerator ash, see *Guidance for the Sampling and Analysis of Municipal Waste Combustion Ash for the Toxicity Characteristic* (USEPA 1995f).

Table 10. Descriptions of Media Listed in Table 8 (Continued).

Media	***Description***	***Examples***
Soil (in-situ) and other unconsolidated geologic material	Soil in its original undisturbed location or other geologic material that does not require significant pressure to separate into particles. *In situ* soil sampling may be conducted at subsurface or surface depths. Surface soils generally are defined as soils between the ground surface and 6 to 12 inches below the ground surface (USEPA 1996b); however, the definition of surface soils in State programs may vary considerably from EPA's.	Subsurface soil at a land treatment unit; surface soil contaminated by a chemical spill on top of the ground or soil near a leak from an excavated underground storage tank.*
Solids, consolidated	Cemented or otherwise dense solids that require significant physical pressure to break apart into smaller parts.	Concrete, wood, and architectural debris.
Air	For the purpose of RCRA sampling, air includes emissions from stationary sources or indoor air.	Emissions from boilers and industrial furnaces (BIFs).**
Sediment	Settled, unconsolidated solids beneath a flowing or standing liquid layer.	Sediment in a surface water body.
Soil gas or vapor	Gas or vapor phase in the vadose zone. The vadose zone is the hydrogeological region extending from the soil surface to the top of the principal water table.	Soil gas overlying a waste disposal site.
Ground water	"Water below the land surface in a zone of saturation" (40 CFR 260.10). Water can also be present below the land surface in the unsaturated (vadose) zone.	Ground water in monitoring wells surrounding a hazardous waste landfill.***

* Detailed guidance on soil sampling can be found in *Preparation of Soil Sampling Protocols: Sampling Techniques and Strategies* (Mason 1992), which provides a discussion of the advantages and disadvantages of various sample collection methods for soil.

** See Chapter Ten of SW-846 for EPA-approved methods for sampling air under RCRA.

*** Detailed guidance on ground-water sampling can be found in *RCRA Ground-Water Monitoring -- Draft Technical Guidance* (USEPA 1992c), which updates technical information in Chapter Eleven of SW-846 (Rev. 0, Sept. 1986) and the *Technical Enforcement Guidance Document* (TEGD).

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7.2 Conducting Field Sampling Activities

This section provides guidance on performing field sampling activities that typically are performed during implementation of the sampling plan. Additional guidance can be found in *Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual* (USEPA 1994a), *Environmental Investigations Standard Operating Procedures and Quality Assurance Manual, U.S. EPA Region 4, May 1996* (USEPA 1996b), other USEPA guidance cited in the reference section of this chapter, and various ASTM standards summarized in Appendix J of this guidance. See also Appendix C of EPA's *Guidance for Quality Assurance Project Plans* (USEPA 1998a). The latter document includes extensive checklists, including the following:

- Sample handling, preparation, and analysis checklist
- QAPP review checklist
- Chain-of-custody checklist.

In this section, we provide guidance on the following topics:

- Sample containers (Section 7.2.1)
- Sample preservation and holding times (Section 7.2.2)
- Documentation of field activities (Section 7.2.3)
- Field quality control samples (Section 7.2.4)
- Sample identification and chain-of-custody procedures (Section 7.2.5)
- Decontamination of equipment and personnel (Section 7.2.6)
- Health and safety (Section 7.2.7)
- Sample packaging and shipping (Section 7.2.8).

7.2.1 Selecting Sample Containers

All samples should be placed in containers of a size and construction appropriate for the volume of material specified in the sampling plan and as appropriate for the requested analyses. If sufficient sample volume is not collected, the analysis of all requested parameters and complete quality control determinations may not be possible. In addition, minimum sample volumes may be required to control sampling errors (see Section 6). Chapters Two, Three, and Four of SW-846 identify the appropriate containers for RCRA-related analyses by SW-846 methods.

Chapters Two, Three, and Four of SW-846 identify some of the appropriate containers for RCRA-related analyses by SW-846 methods.

It is important to understand that a single "sample" may need to be apportioned to more than one container to satisfy the volume and preservation requirements specified by different categories of analytical methods. Furthermore, the analytical plan may require transport of portions of a sample to more than one laboratory.

Factors to consider when choosing containers are compatibility with the waste components, cost, resistance to breakage, and volume. Containers must not distort, rupture, or leak as a result of chemical reactions with constituents of waste samples. The containers must have adequate wall thickness to withstand handling during sample collection and transport. For analysis of non-volatile constituents, containers with wide mouths are often desirable to facilitate transfer of samples from the equipment. The containers must be large enough to contain the optimum sample volume specified in the DQO Process.

You should store samples containing light-sensitive organic constituents in amber glass bottles with Teflon®-lined lids. Polyethylene containers are not appropriate for use when the samples are to be analyzed for organic constituents because the plastics could contribute organic contaminants and potentially introduce bias. If liquid samples are to be submitted for analysis of volatile compounds, you must store the samples in air-tight containers with zero head space. You can store samples intended for metals and other inorganic constituent analyses in polyethylene containers with polyethylene-lined lids. We recommend that you consult with a chemist for further direction regarding chemical compatibility of available containers and the media to be sampled. We recommend that an extra supply of containers be available at the sampling location in case you want to collect more sample material than originally planned or you need to retain splits of each sample.[2]

Always use clean sample containers of an assured quality. For container cleaning procedures and additional container information, refer to the current iteration of *Specifications and Guidance for Contaminant-Free Sample Containers* (USEPA 1992d). You may wish to purchase pre-cleaned/quality assured bottles in lieu of cleaning your own bottles (USEPA 2001g).

7.2.2 Sample Preservation and Holding Times

Samples are preserved to minimize any chemical or physical changes that might occur between the time of sample collection and analysis. Preservation can be by physical means (e.g., kept at a certain temperature) or chemical means (e.g., with the addition of chemical preservatives). If a sample is not preserved properly, the levels of constituents of concern in the sample may be altered through chemical, biological, or photo-degradation, or by leaching, sorption, or other chemical or physical reactions within the sample container.

The appropriate method for preserving a sample will depend on the physical characteristics of the sample (such as soil, waste, water, etc.), the concentration of constituents in the sample, and the analysis to be performed on the sample. Addition of chemical preservatives may be required for samples to be analyzed for certain parameters. You should not chemically preserve highly concentrated samples. Samples with low concentrations, however, should be preserved. You should consult with a chemist at the laboratory regarding the addition of chemical preservatives and the possible impact on the concentration of constituents in the sample. Also, be aware that addition of some chemical preservatives to highly concentrated waste samples may result in a dangerous reaction.

Regardless of preservation measures, the concentrations of constituents within a sample can degrade over time. Therefore, you also should adhere to sample holding times (time from sample collection to analysis), particularly if the constituents of concern are volatiles in low concentrations. Analytical data generated outside of the specified holding times are considered to be minimum values only. You may use such data to demonstrate that a waste is hazardous

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[2] For example, when inspections are conducted under Section 3007 of RCRA (42 U.S.C. § 6927), and samples are obtained, EPA must provide a split sample to the facility, upon request.

where the value of a constituent-of-concern is above the regulatory threshold, but you cannot use the data to demonstrate that a waste is not hazardous. Exceeding a holding time when the results are above a decision level does not invalidate the data.

Appropriate sample preservation techniques and sample holding times for aqueous matrices are listed in Chapters Two, Three, and Four of SW-846. You should also consult the methods to be used during analysis of the sampled waste. In addition, *Standard Guide for Sampling Waste and Soil for Volatile Organic Compounds* (ASTM D 4547-98) provides information regarding the preservation of volatile organic levels in waste and soil samples.

7.2.3 Documentation of Field Activities

This section provides guidance on documenting field activities. Records of field activities should be legible, identifiable, retrievable and protected against damage, deterioration, and loss. You should record all documentation in waterproof, non-erasable ink. If you make an error in any of these documents, make corrections by crossing a single line through the error and entering the correct information adjacent to it. The corrections should then be initialed and dated. Stick-on labels of information should not be removable without evidence of the tampering. Do not put labels over previously recorded information.

Keep a dedicated logbook for each sampling project with the name of the project leader, team members, and project name written inside the front cover. Document all aspects of sample collection and handling in the logbook. Entries should be legible, accurate, and complete. The language should be factual and objective.

You also should include information regarding sample collection equipment (use and decontamination), field analytical equipment and the measurements, calculations and calibration data, the name of the person who collected the sample, sample numbers, sample location description and diagram or map, sample description, time of collection, climatic conditions, and observations of any unusual events. Document the collection of QC samples and any deviations from procedural documents, such as the QAPP and SOPs.

When videos, slides, or photographs are taken, you should number them to correspond to logbook entries. The name of the photographer, date, time, site location, and site description should be entered sequentially into the logbook as photos are taken. A series entry may be used for rapid aperture settings and shutter speeds for photographs taken within the normal automatic exposure range. Special lenses, films, filters, or other image enhancement techniques must be noted in the logbook. Chain-of-custody procedures for photoimages depend on the subject matter, type of film, and the processing it requires. Adequate logbook notations and receipts may be used to account for routine film processing. Once developed, the slides or photographic prints should be serially numbered corresponding to the logbook descriptions and labeled (USEPA 1992e).

7.2.4 Field Quality Control Samples

Quality control samples are collected during field studies to monitor the performance of sample collection and the risk of sampling bias or errors. Field QC samples could include the following:

Equipment blank: A rinse sample of the decontaminated sampling equipment using organic/analyte free water under field conditions to evaluate the effectiveness of equipment decontamination or to detect sample cross contamination.

Trip blank: A sample prepared prior to the sampling event and stored with the samples throughout the event. It is packaged for shipment with the samples and not opened until the shipment reaches the laboratory. The sample is used to identify any contamination that may be attributed to sample handling and shipment.

Field blank: A sample prepared in the field using organic/analyte free water to evaluate the potential for contamination by site contaminants not associated with the sample collected (e.g., airborne organic vapors)

Field split sample: Two or more representative portions taken from the same sample and submitted for analysis to different laboratories. Field split samples are used to estimate interlaboratory precision.

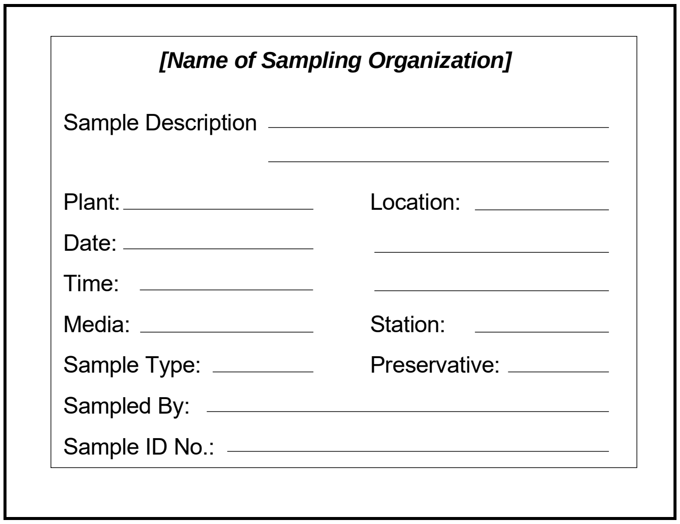
In addition to collecting field QC samples, other QC procedures include sample storage, handling, and documentation protocols. These procedures are covered separately in the following sections. In addition, Chapter One of SW-846, entitled "Quality Control", contains guidance regarding both field and laboratory QA/QC. We also recommend reviewing the following for information on field QA/QC:

- *EPA Guidance for Quality Assurance Project Plans* (USEPA 1998a)
- *Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Quality Assurance and Quality Control Planning and Implementation* (ASTM D 5283-92).

7.2.5 Sample Identification and Chain-of-Custody Procedures

You should identify samples for laboratory analysis with sample tags or labels. An example of a sample label is given in Figure 29. Typically, information on the sample label should include the sample identification code or number, date, time of collection, preservative used, media, location, initials of the sampler, and analysis requested. While not required, you may elect to seal each sample container with a custody seal (Figure 30).

You should use chain-of-custody procedures to record the custody of the samples. Chain-of-custody is the custody of samples from time of collection through shipment to analysis. A sample is in one's custody if:



[Name of Sampling Organization]

Sample Description ____________________

Plant: __________ Location: __________
Date: __________
Time: __________
Media: __________ Station: __________
Sample Type: __________ Preservative: __________
Sampled By: ____________________
Sample ID No.: ____________________

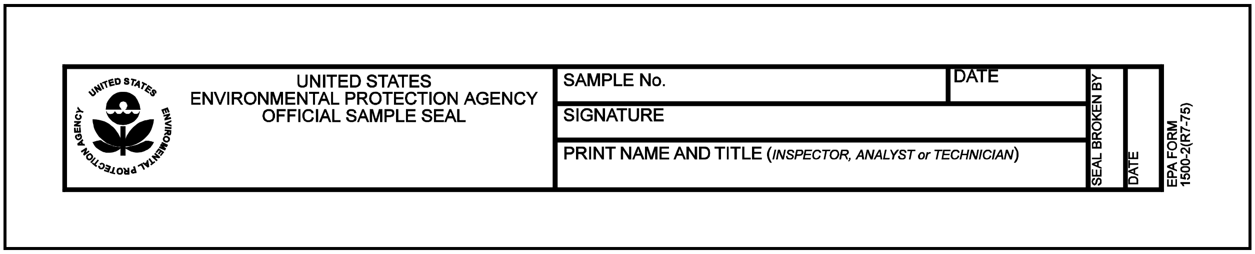
Figure 29. Sample label

- It is in the actual possession of an investigator

- It is in the view of an investigator, after being in their physical possession

- It is in the physical possession of an investigator, who secures it to prevent tampering

- It is placed in a designated secure area.



UNITED STATES
ENVIRONMENTAL PROTECTION AGENCY
OFFICIAL SAMPLE SEAL

SAMPLE No.

DATE

SIGNATURE

PRINT NAME AND TITLE (*INSPECTOR, ANALYST or TECHNICIAN*)

SEAL BROKEN BY

DATE

EPA FORM
1500-2(R7-75)

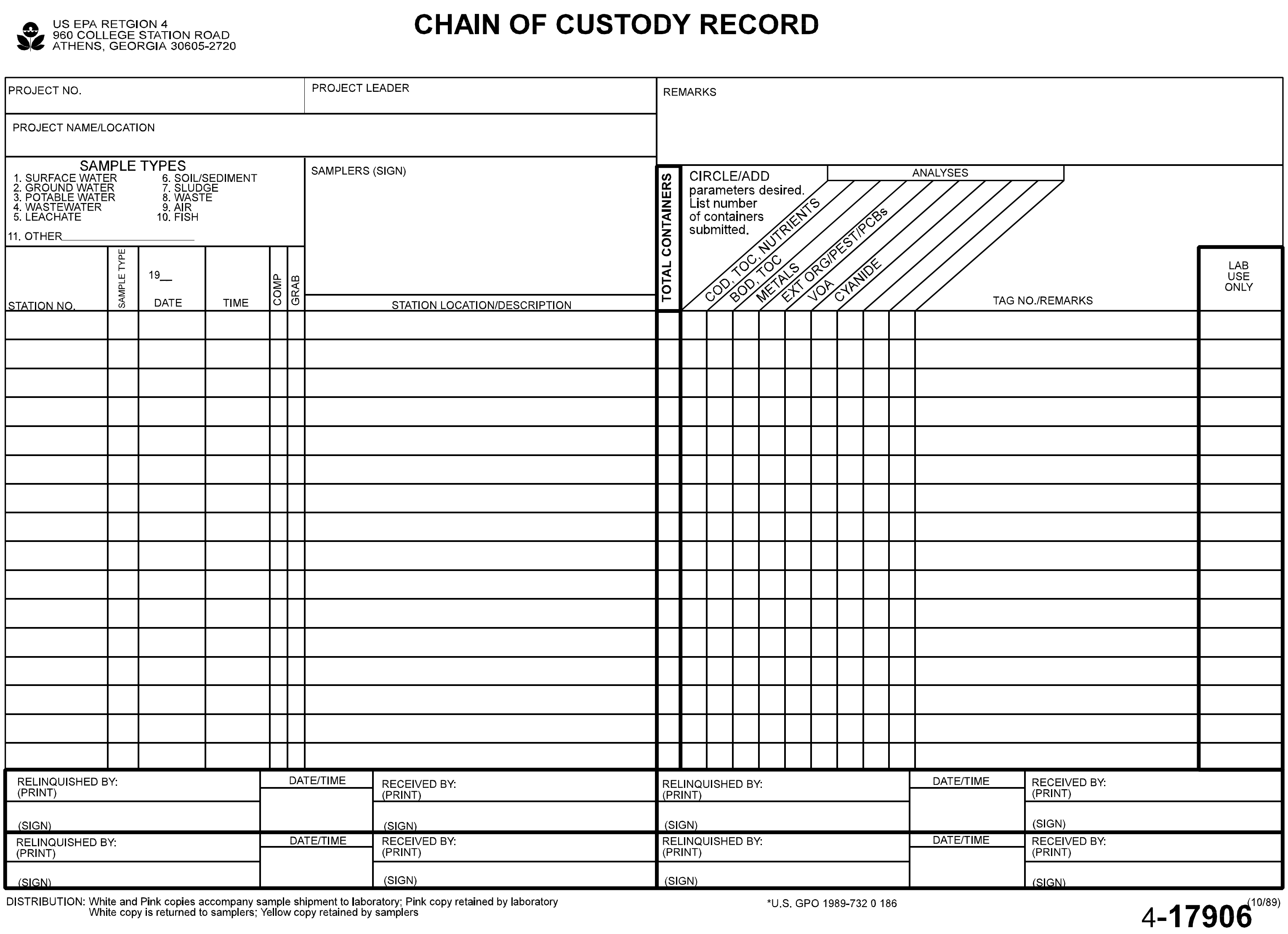
Figure 30. Custody seal

All sample sets should be accompanied by a chain-of-custody form. This record also serves as the sample logging mechanism for the laboratory sample custodian. Figure 31 illustrates the content of a chain-of-custody form. When the possession of samples is transferred, both the individual relinquishing the samples and the individual receiving the samples should sign, date, and note the time on the chain-of-custody document. If you use overnight shipping service to transport the samples, record the air bill number on the chain-of-custody form. This chain-of-custody record represents the official documentation for all transfers of the sample custody until the samples have arrived at the laboratory. The original form of the chain-of-custody record should accompany each shipment. A copy should be retained by a representative of the sampling team.

When sample custody is transferred between individuals, the samples or coolers containing the samples are sealed with a custody seal. This seal cannot be removed or broken without destruction of the seal, providing an indicator that custody has been terminated.

EPA's Superfund Program has developed software called *Field Operations and Records Management System (FORMS) II Lite*™ that automates the printing of sample documentation in the field, reduces time spent completing sample collection and transfer documentation, and facilitates electronic capture of data prior to and during field sampling activities. For information on *FORMS II Lite*™, see http://www.epa.gov/superfund/programs/clp/f2lite.htm.

For additional information on chain-of-custody procedures, we recommend ASTM D 4840, *Standard Guide for Sampling Chain-of-Custody Procedures*.



US EPA RETGION 4
960 COLLEGE STATION ROAD
ATHENS, GEORGIA 30605-2720

CHAIN OF CUSTODY RECORD

PROJECT NO.

PROJECT LEADER

REMARKS

PROJECT NAME/LOCATION

SAMPLE TYPES
1. SURFACE WATER
2. GROUND WATER
3. POTABLE WATER
4. WASTEWATER
5. LEACHATE
6. SOIL/SEDIMENT
7. SLUDGE
8. WASTE
9. AIR
10. FISH
11. OTHER________________________

SAMPLERS (SIGN)

CIRCLE/ADD parameters desired. List number of containers submitted.

ANALYSES

STATION NO.	SAMPLE TYPE	19__ DATE	TIME	COMP	GRAB	STATION LOCATION/DESCRIPTION	TOTAL CONTAINERS	COD, TOC, NUTRIENTS	BOD, TOC	METALS	EXT ORG/PEST/PCBs	VOA	CYANIDE				TAG NO./REMARKS	LAB USE ONLY

RELINQUISHED BY: (PRINT) (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) (SIGN)	RELINQUISHED BY: (PRINT) (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) (SIGN)
RELINQUISHED BY: (PRINT) (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) (SIGN)	RELINQUISHED BY: (PRINT) (SIGN)	DATE/TIME	RECEIVED BY: (PRINT) (SIGN)

DISTRIBUTION: White and Pink copies accompany sample shipment to laboratory; Pink copy retained by laboratory
White copy is returned to samplers; Yellow copy retained by samplers

*U.S. GPO 1989-732 0 186

4-**17906** (10/89)

Figure 31. Chain-of-custody form

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7.2.6 Decontamination of Equipment and Personnel

Decontamination of sampling equipment refers to the physical and chemical steps taken to remove any chemical or material contamination. Equipment decontamination helps prevent sampling bias. All equipment that comes in contact with the sampled material should be free of components that could influence (contaminate) the true physical or chemical composition of the material. Besides the equipment used to collect the samples, any containers or equipment used for sample compositing or for field subsampling should be free of contamination.

Equipment decontamination also prevents cross-contamination of samples when the equipment is used to collect more than one sample. Disposable equipment or the use of dedicated equipment provides the most effective means of avoiding cross-contamination; however, the use of such equipment is not always practical.

You should decontaminate equipment to a level that meets the minimum requirements for your data collection effort. Your decontamination steps (e.g., use of solvents versus use of only soap and water), therefore, should be selected based on the constituents present, their concentration levels in the waste or materials sampled, and their potential to introduce bias in the sample analysis results if not removed from the sampling equipment. You should describe the project-specific decontamination procedures in your planning document for the sampling effort. In addition, items used to clean the equipment, such as bottle brushes, should be free of contamination.

The following procedure is an example of one you could use to decontaminate a sampling device to be used for collecting samples for trace organic or inorganic constituent analyses (from USEPA 1996b):

1. Clean the device with tap water and soap, using a brush if necessary to remove particulate matter and surface films.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with analyte- or organic-free water.

4. Rinse thoroughly with solvent. Do not solvent-rinse PVC or plastic items.

5. Rinse thoroughly with organic/analyte free water, or allow equipment to dry completely.

6. Remove the equipment from the decontamination area. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

The specifications for the cleaning materials are as follows (you should justify and document the use of substitutes):

- "Soap" should be a phosphate-free laboratory detergent such as Liquinox®. It must be kept in clean plastic, metal, or glass containers until used and poured directly from the container when in use.

- "Solvent" should be pesticide-grade isopropanol. It must be stored in the unopened original containers until used. It may be applied using the low pressure nitrogen system fitted with a Teflon® nozzle, or using Teflon® squeeze bottles. For equipment highly contaminated with organics (such as oily waste), a laboratory-grade hexane may be a more suitable alternative to isopropanol.

- "Tap water" may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute. Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly from a hose or tap.

- "Analyte free water" (deionized water) is tap water treated by passing it through a standard deionizing resin column. At a minimum, it must contain no detectable heavy metals or other inorganic compounds as defined by a standard ICP (or equivalent) scan. It may be obtained by other methods as long as it meets the analytical criteria. Analyte free water must be stored in clean glass, stainless steel, or plastic containers that can be closed prior to use. It can be applied from plastic squeeze bottles.

- "Organic/analyte free water" is tap water that has been treated with activated carbon and deionizing units. A portable system to produce such water under field conditions is available. At a minimum, the water must meet the criteria of analyte free water and not contain detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined for a given set of analyses. Organic/analyte free water obtained by other methods is acceptable, as long as it meets the analytical criteria. It must be stored in clean glass, Teflon®, or stainless steel containers. It may be applied using Teflon® squeeze bottles or with the portable system.

Clean the field equipment prior to field use. Designate a decontamination zone at the site and, if necessary, construct a decontamination pad at a location free of surface contamination. You should collect wastewater from decontamination (e.g., via a sump or pit) and remove it frequently for appropriate treatment or disposal. The pad or area should not leak contaminated water into the surrounding environment. You also should collect solvent rinses for proper disposal.

You should always handle field-cleaned equipment in a manner that prevents recontamination. For example, after decontamination but prior to use, store the equipment in a location away from the cleaning area and in an area free of contaminants. If it is not immediately reused, you should cover it with plastic or aluminum foil to prevent recontamination.

Decontamination will generate a quantity of wastes called investigation derived waste (IDW). You should address the handling and disposal of IDW in your sampling plan. You must handle this material in accordance with whether it is nonhazardous or suspected of, or known to be, hazardous. You should minimize the generation of hazardous IDW and keep it separated from nonhazardous IDW. For example, you should control the volume of spent solvents during equipment decontamination by applying the minimum amount of liquid necessary and capturing it separately from the nonhazardous washwater. For additional guidance on handling IDW, see *Management of Investigation-Derived Wastes* (USEPA 1992f).

Decontamination of personnel and their protective gear also is often necessary during hazardous waste sampling. This important type of decontamination protects personnel from chemical exposure and prevents cross-contamination when personnel change locations. The level or degree of such decontamination will depend on site-specific considerations, such as the health hazards posed by exposure to the sampled waste. You should address these decontamination procedures in your health and safety plan.

For additional information regarding decontamination, see ASTM D 5088, *Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites*. Another source of additional information is "Sampling Equipment Decontamination" (USEPA 1994f), issued by EPA's Environmental Response Team.

7.2.7 Health and Safety Considerations

Regulations published by the Occupational Safety and Health Administration (OSHA) at 29 CFR Part 1910.120 govern workers at hazardous waste sites and include requirements for training, equipment, medical monitoring, and other practices. Many sampling activities covered by this guidance may require compliance with OSHA's health and safety regulations. Specific guidance on worker health and safety is beyond the scope of this chapter; however, development and use of a project-specific health and safety plan may be required. It is the responsibility of the sampling team leader and others in charge to ensure worker safety.

Some important health and safety considerations follow:

- Field personnel should be up-to-date in their health and safety training.

- Field personnel should have a medical examination at the initiation of sampling activities and routinely thereafter, as appropriate and as required by the OSHA regulations. Unscheduled examinations should be performed in the event of an accident or suspected exposure to hazardous materials.

- Staff also should be aware of the common routes of exposure at a site and be instructed in the proper use of safety equipment and protective clothing and equipment. Safe areas should be designated for washing, drinking, and eating.

- To minimize the impact of an emergency situation, field personnel should be aware of basic first aid and have immediate access to a first aid kit.

The guidance manual *Occupational Safety and Health Guidance Manual for Hazardous Waste Site Activities* (OSHA 1985, revised 1998) was jointly developed by the National Institute for Occupational Safety and Health (NIOSH), OSHA, the United States Coast Guard (USCG), and EPA. Its intended audience is those who are responsible for occupational safety and health programs at hazardous waste sites.

7.2.8 Sample Packaging and Shipping

During transport of waste samples, you should follow all State and Federal regulations governing environmental sample packaging and shipment and ship according to U.S. Department of Transportation (DOT) and International Air Transportation Association (IATA) regulations. Minimum guidelines for sample packaging and shipping procedures follow in the next subsections; however, the rules and regulations for sample packaging and shipping are complex, and for some samples and shipping situations the procedures outlined below may need to be exceeded.

7.2.8.1 Sample Packaging

You should package and label samples in an area free of contamination. You also should ship or transport samples to a laboratory within a time frame that meets recommended sample holding times for the respective analyses. Additional guidelines follow:

- Aqueous samples for inorganic analysis and volatile organic analysis may require chemical preservation. The specific preservation requirements will depend on the analytical method to be used.

- Make sure all lids/caps are tight and will not leak.

- Make sure sample labels are intact and covered with a piece of clear tape for protection.

- Enclose the sample container in a clear plastic bag and seal the bag. Make sure the sample labels are visible. If bubble wrap or other wrapping material will be placed around the labeled containers, write the sample number and fraction (e.g., "BLH01-VOCs") so that it is visible on the outside of the wrap, then place the wrapped container in a clear plastic bag and seal the bag.

- Make sure that all samples that need to be kept cold (4 ± 2 °C) have been thoroughly cooled before placing in packing material so that the packing material serves to insulate the cold. Change the ice prior to shipment as needed. Ideally, pack the cooled samples into shipping containers that have already been chilled. (Of course, these precautions are not necessary if none of the samples in the shipping container need to be kept cold.)

- Any soil/sediment samples suspected to be of medium/high concentration or containing dioxin must be enclosed in a metal can with a clipped or sealable lid (e.g., paint cans) to achieve double containment of those samples. Place suitable absorbent packing material around the sample container in the can. Make sure the sample is securely stored in a can and the lid is sealed. Label the outer metal container with the sample number and fraction of the sample inside.

- Use *clean* waterproof metal or hard plastic ice chests or coolers that are in good repair for shipping samples.

- Remove the inapplicable previous shipping labels. Make sure any drain plugs are shut. Seal plugs shut on the inside and outside with a suitable tape such as duct tape. Line the cooler with plastic (e.g., large heavy-duty garbage bag) before inserting samples.

- Ship samples at 4 ± 2 °C, place double-bagged ice on top of samples. Ice must be sealed in double plastic bags to prevent melting ice from soaking the packing material. Loose ice should not be poured into the cooler.

- Conduct an inventory of sample numbers, fractions, and containers when placing samples into the coolers. Check the inventory against the corresponding chain-of-custody form before sealing the cooler to make sure that all samples and containers are present.

- Pack the lined shipping containers with noncombustible absorbent packing material, such as vermiculite or rock wool. Place the packing material on the bottom of the shipping container (inside the plastic liner) and around sample bottles or metal cans to avoid breakage during shipment. Never use earth, ice, paper, or styrofoam to pack samples. Earth is a contaminant, melted ice may cause complications and allow the sample containers to bang together when the shipping container is moved, and styrofoam presents a disposal problem (it also may easily blow out of the shipping container at the site).

- For samples that need to be shipped at 4 ± 2ºC, place double-bagged ice on top of samples and fill remaining space with packing material. If sample bottles have been protected with packaging material such as bubble wrap, then some double-bagged ice or ice packs also may be placed between samples.

- Use tape to securely fasten the top of the plastic used to line the shipping container. It is a good idea to then place a completed custody seal around the top of the bag that contains the sample in case the outer seals placed across the cooler lid are inadvertently damaged during shipment.

- Enclose all sample documentation (i.e., chain-of-custody forms and cooler return shipping documents) in a waterproof plastic bag, and tape the bag to the underside of the cooler lid. This documentation should address all samples in the cooler, but not address samples in any other cooler.

- If more than one cooler is being used, place separate sample documentation in each cooler. Instructions for returning the cooler should be documented inside the cooler lid. Write a return name and address for the sample cooler on the inside of the cooler lid in permanent ink to ensure return of the cooler.

- Tape the cooler shut using strapping tape over the hinges. Place completed custody seals across the top and sides of the cooler lid so that lid cannot be opened without breaking the seal.

- Place clear tape over the seal to prevent inadvertent damage to the seal during shipment. Do not place clear tape over the seals in a manner that would allow the seals to be lifted off with the tape and then reaffixed without breaking the seal.

For additional detailed guidance on sample documentation, packaging, and shipping, we recommend the *Contract Laboratory Program (CLP) Guidance for Field Samplers - Draft Final* (USEPA 2001g).

7.2.8.2 Sample Shipping

In general, samples of drinking water, most ground waters and ambient surface waters, soil, sediment, treated waste waters, and other low concentration samples can be shipped as environmental samples; however, shipment of high concentration waste samples may require shipment as dangerous goods (not as “hazardous waste”). Note that RCRA regulations specifically exempt samples of hazardous waste from RCRA waste identification, manifest, permitting, and notification requirements (see 40 CFR §261.4(d)). The shipment of samples to and from a laboratory, however, must comply with U.S. DOT, U.S. Postal Service, or any other applicable shipping requirements. If a sample is a hazardous waste, once received at the laboratory, it must be managed as a hazardous waste.

In recent years, commercial overnight shipping services have adopted the regulations of the IATA for shipment of dangerous goods by air. The IATA Dangerous Goods Regulations contain all provisions mandated by the International Civil Aviation Organization and all rules universally agreed to by airlines to correctly package and safely transport dangerous goods by air. Contact IATA for a copy of the IATA Dangerous Goods Regulations and for assistance in locating suppliers of specialized packaging for dangerous goods.

> For information on shipping dangerous goods visit the International Air Transport Association (IATA) Dangerous Goods Information Online at http://www.iata.org/cargo/dg/index.htm or call 1-800-716-6326.

When shipping samples, perform the following activities:

- Clearly label the cooler and fill out appropriate shipping papers.
- Place return address labels clearly on the outside of the cooler.
- If more than one cooler is being shipped, mark each cooler as "1 of 2," "2 of 2," etc.
- Ship samples through a commercial carrier. Use appropriate packaging, mark and label packages, and fill out all required government and commercial carrier shipping papers according to DOT and IATA commercial carrier regulations.
- Ship all samples by overnight delivery in accordance with DOT and IATA regulations.

7.3 Using Sample Homogenization, Splitting, and Subsampling Techniques

7.3.1 Homogenization Techniques

The objective of homogenization (mixing) is to minimize grouping and segregation of particles so they are randomly distributed within the sample. While homogenization can reduce grouping and segregation of particles, it will not eliminate it and will not make the material "homogeneous." If homogenization is successful, subsamples of the homogenized material will show less variability than if the material was not homogenized. Homogenization, combined with a composite sampling strategy, can be an efficient method for improving the accuracy and precision in sampling of particulate material (Jenkins, et al. 1996). Homogenization can be applied to solids, liquids, slurries, and sludges.

Pitard (1993) recognizes two processes for homogenization:

> **Stationary processes** - in which the material is not mixed but is redistributed so that any correlation between the characteristics of individual fragments or particles is lost or minimized. An example of this process is the collection of many small increments to form an individual sample (ideally we would pick many individual particles at random to form the sample, but this is not possible).
>
> **Dynamic processes** - in which the material is mechanically mixed to remove or minimize correlation between the characteristics of the fragment or particle and its position within the sample. Examples of this process include mechanical mixing within a container and use of magnetic stirrers in a beaker.

Note that the benefits of homogenization may be temporary because gravity-induced segregation can occur during shipment, storage, and handling of samples. For this reason, consider carrying out homogenization (mixing) immediately prior to analysis.

Some homogenization techniques work better than others. The strengths and limitations of homogenization equipment and procedures (cone and quartering, riffle splitters, rotary splitters, multiple cone splitters, and V-blenders) have been reviewed in the literature by Pitard (1993), Schumacher, et al. (1991), ASTM (Standard D 6051-96), and others. The preferred techniques for use within the laboratory follow:

- Riffling (see also Section 7.3.2)
- Fractional shoveling (see also Section 7.3.2)
- Mechanical mixing
- Cone and quartering
- Magnetic stirrers (e.g., to homogenize the contents of an open beaker)
- V-blenders.

Fractional shoveling and mechanical mixing also can be used in the field. Note that some techniques for homogenization, such as riffling and fractional shoveling, also are used for splitting and subsampling. Note that Pitard (1993) discourages the use of "sheet mixing" (also called "mixing square") and vibratory spatulas because they tend to segregate particles of different density and size.

7.3.2 Sample Splitting

Splitting is employed when a field sample is significantly larger than the required analytical sample. The goal of splitting is to reduce the mass of the retained sample and obtain an aliquot of the field sample that reflects the average properties of the entire field sample. It is often necessary to repeat the splitting process a number of times to achieve a sufficient reduction in mass for analytical purposes.

Splitting can be used to generate a reduced mass aliquot that can be analyzed in its entirety or a much reduced and homogenized mass from which an analytical or subsample can be collected. ASTM's *Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities* (ASTM D 6323-98), lists and discusses a variety of splitting equipment (such as sectorial splitters and riffle splitters) and splitting procedures (such as cone and quartering and the alternate scoop method). Gerlach, et al. (2002) also evaluated sample splitting methods (riffle splitting, paper cone riffle splitting, fractional shoveling, coning and quartering, and grab sampling) and found that riffle splitting methods performed the best.

A simple alternative to riffle splitting a sample of solid media is a technique called "fractional shoveling." To perform fractional shoveling, deal out small increments from the larger sample in sequence into separate piles, randomly select one of the piles and retain it as the subsample (or retain more than one if a portion of the sample is to be "split" with another party and/or retained for archive purposes), and reject the others (see Figure 32).

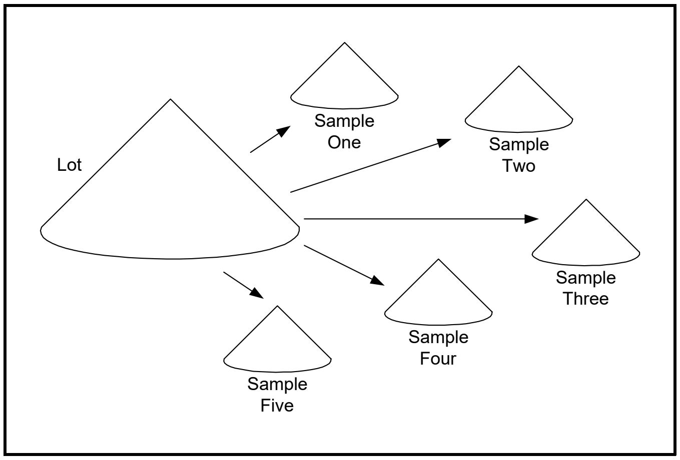

Figure 32. Fractional shoveling as a sample splitting method (after Pitard 1993)

7.3.3 Subsampling

The size of the sample submitted to the laboratory (either an individual sample or a composite) by field personnel typically far exceeds that required for analysis. Consequently, subsampling is needed. A subsample is defined as "a portion of material taken from a larger quantity for the purpose of estimating properties or the composition of the whole sample" (ASTM D 4547-98). Taking a subsample may be as simple as collecting the required mass from a larger mass, or it may involve one or more preparatory steps such as grinding, homogenization, and/or splitting of the larger mass prior to removal of the subsample.

Specific procedures for maintaining sample integrity (e.g., minimizing fundamental error) during splitting and subsampling operations typically are not addressed in quality assurance, sampling, or analytical plans, and error may be introduced unknowingly in subsampling and sample preparation. Many environmental laboratories do not have adequate SOPs for subsampling; therefore, it is important for the data users to provide the laboratory personnel clear instruction if any special subsampling or sample handling procedures are needed (such as instructions on mixing of the sample prior to analysis, removing particles greater than a certain size, analyzing phases separately, etc.). If proper subsampling procedures are not specified in planning documents, SOPs, or documents shipped with the samples, it may be difficult to assess the usability of the results.

The following sections provide general guidance on obtaining subsamples of liquids, mixtures of liquids and solids, and soils and solid media. For additional guidance and detailed procedures, see *Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities* (ASTM D 6051-96) and *Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities* (ASTM D 6323-98).

7.3.3.1 Subsampling Liquids

In the case of subsampling a liquid, special precautions may be warranted if the liquid contains suspended solids and/or the liquid comprises multiple liquid phases. In practice, samples may contain solids and/or separate phases that are subject to gravitational action (Gy 1998). Even a liquid that appears clear (absent of solids and without iridescence) may not be “homogeneous.”

Subsampling of liquids (containing solids and/or in multiple phases) can be addressed by using one or the other of two possible approaches:

- Mixing the sample such that all phases are homogenized, and then taking a subsample (using a pipette, for example)
- Allowing all of the phases to separate followed by subsampling and analysis of each phase separately.

Of course, the characteristics of the waste and the type of test must be considered. For example, mixing of multi-phasic wastes to be analyzed for volatiles should be avoided due to the potential loss of constituents. Some multi-phasic liquid wastes can form an emulsion when mixed. Others, in spite of mixing, will quickly separate back into distinct phases.

7.3.3.2 Subsampling Mixtures of Liquids and Solids

If the sample is a mixture of liquids and solids, subsampling usually requires that the phases be separated. The separate phases are then separately subsampled. Subsampling of the liquid phase can be accomplished as described above, while subsampling of the solid phase should be done according to sampling theory, as summarized below.

7.3.3.3 Subsampling Soils and Solid Media

To correctly subsample soil or solid media, use sampling tools and techniques that minimize delimitation and extraction error. If the particles in the sample are too coarse to maintain fundamental error within desired limits, it may be necessary to perform a series of steps of particle size reduction followed by subsampling (see Appendix D). If the field sample mass is equal to or less than the specified analytical size, the field sample can be analyzed in its entirety. If the mass of the field sample is greater than the specified analytical sample size, subsampling will be required.

One possible alternative to particle-size reduction prior to subsampling is to simply remove the coarse particles (e.g., via a sieve or visually) from the sample. This selective removal technique is *not* recommended in situations in which the larger particles contribute to the overall concentration of the constituent of concern in the waste. In other words, do not remove the large particles if the constituents of concern tend to be concentrated in the large particles relative to the smaller particles.

If the largest particle size of the field sample exceeds the allowable size for maintaining the fundamental error specified by the DQO *and* the analyte of interest is volatile, it may be necessary to analyze the sample as is and accept a large fundamental error. Guidance on handling VOCs in samples can be found in Section 6.3.4 and in ASTM Standard D 4547-98.

The *Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities* (ASTM D 6323-98) lists a variety of equipment for performing particle-size reduction (e.g., cutting mills, jar mills, disc mills, dish and puck mills, mortar grinders and jaw crushers) and tabulates their uses and limitations.

The techniques discussed below are most relevant to subsampling of solid particulate matter for analysis of nonvolatile constituents. Mason (1992, page 5-7) provides a field procedure that can be used to reduce the volume of a field soil sample for submission to the laboratory.

The issues regarding the subsampling of particulate-containing materials are identical to those considered when collecting the original field samples and are as follows:

- The tool used to collect the analytical sample must be correct and not discriminate against any portion of the sample (in other words, the tool should not introduce increment delimitation and increment extraction errors).
- The mass of the subsample must be enough to accommodate the largest of the particles contained within the parent sample (to reduce fundamental error).
- The sample mass and the manner in which it is collected must accommodate the short-term heterogeneity within the field sample (to reduce grouping and segregation error).

The sampling tool must be constructed such that its smallest dimension is at least three times greater than the largest particle size contained within the material being subsampled. The construction of the sampling tool must be such that it does not discriminate against certain areas of the material being sampled. For example, Pitard (1993) argues that all scoops for subsampling should be rectangular or square in design with flat bottoms as opposed to having curved surfaces (Figure 33).

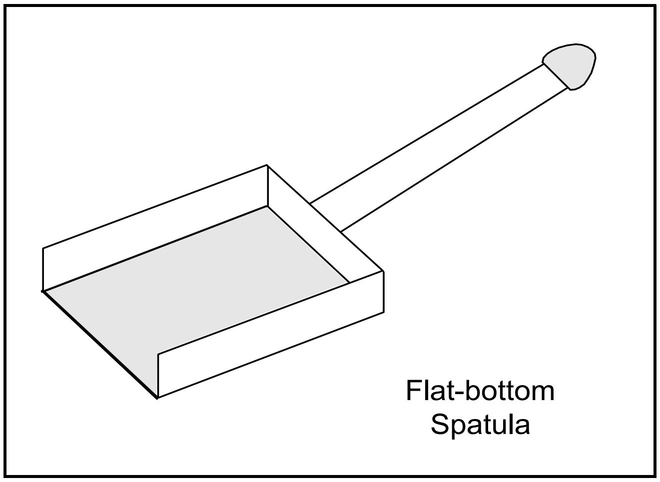

Figure 33. Example of correctly designed device for subsampling. Flat bottom and vertical side walls minimize increment delimitation error.

Pitard (1993) and ASTM D 6323-98 suggest subsampling from relatively flat elongated piles using a transversal subsampling technique that employs a sampling scoop or spatula and a flat working surface (Figure 34(a)). The objective is to convert the sampling problem to a one-dimensional approach. Specifically, Pitard (1993) recommends the following procedure:

- Empty the sample from the sample container onto a smooth and clean surface or appropriate material.
- Do not try to homogenize the sample, as this may promote segregation of particles.
- Reduce the sample by using the fractional shoveling technique (Figure 32) until a sample 5 to 10 times larger than the analytical sample is obtained.
- Shape the remaining material into an elongated pile with uniform width and thickness (Figure 34(a)).
- Take increments all across the pile through the entire thickness.
- Reshape the pile perpendicular to its long axis, and continue to take increments across the pile until the appropriate sample weight is reached.

Fractional shoveling and alternate scoop techniques alone (Figure 32) also can be used to generate subsamples.

When using these techniques, several stages or iterations of subsampling followed by particle size reduction may be needed to minimize fundamental error (also see Appendix D). At each stage, the number of increments should be at least 10 and preferably 25 to control grouping and segregation (short-term heterogeneity) within the sample. In the final stage, however, where very small analytical samples are required, the number of increments required will be much less.

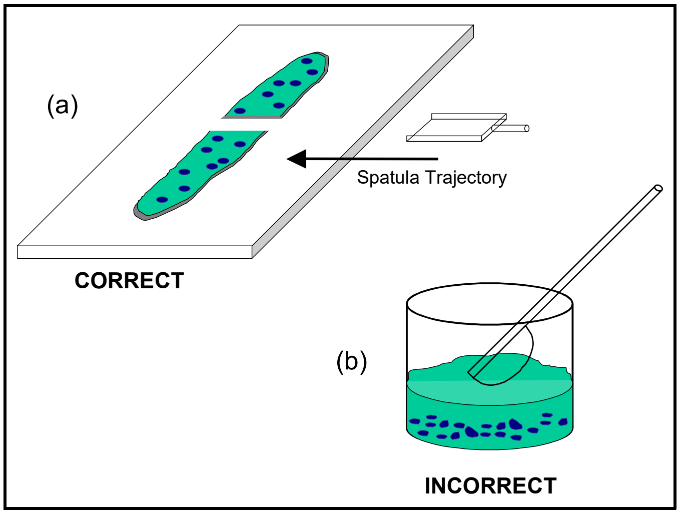

Figure 34. Correct (a) and incorrect (b) laboratory techniques for obtaining subsamples of granular solid media ((a) modified after Pitard 1993).

The subsampling procedures described above offer a more correct and defensible alternative to an approach to subsampling in which the analyst simply opens the sample jar or vial and removes a small increment from the top for preparation and analysis (Figure 34(b)).

8 ASSESSMENT: ANALYZING AND INTERPRETING DATA

This section presents guidance for the assessment of sampling and analytical results. In performing data assessment, evaluate the data set to determine whether the data are sufficient to make the decisions identified in the DQO Process. The data assessment process includes (1) sampling assessment and analytical assessment, and (2) data quality assessment (DQA) (Figure 35) and follows a series of logical steps to determine if the data were collected as planned and to reach conclusions about a waste relative to RCRA requirements.

At the end of the process, EPA recommends reconciliation with the DQOs to ensure that they were achieved and to decide whether additional data collection activities are needed.

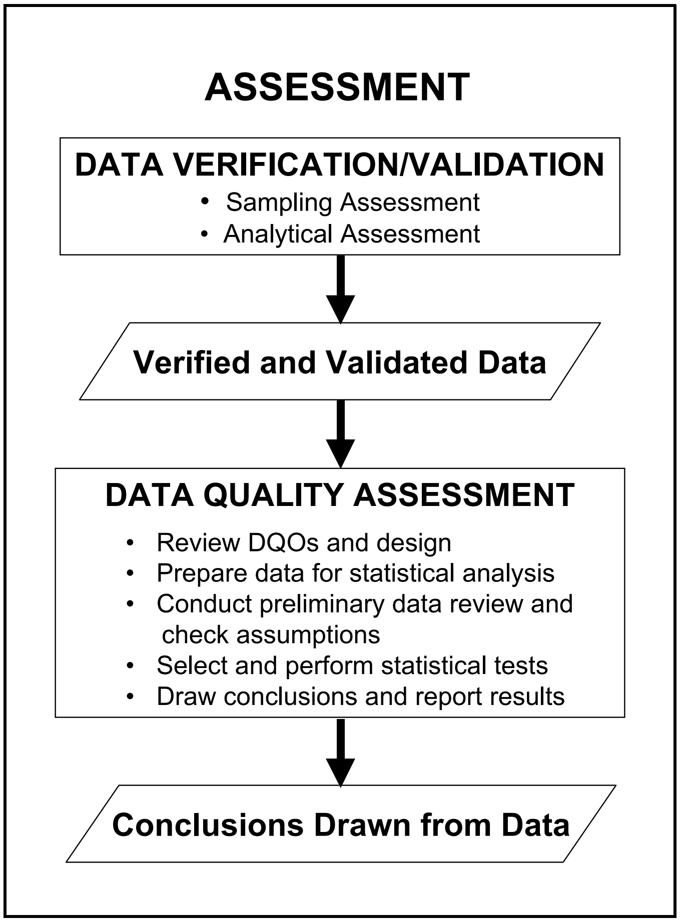

Figure 35. Elements of the quality assurance assessment process (modified after USEPA 1998a)

8.1 Data Verification and Validation

Data verification and validation are performed to ensure that the sampling and analysis protocols specified in the QAPP or WAP were followed and that the measurement systems performed in accordance with the criteria specified in the QAPP or WAP. The process is divided into two parts:

- sampling assessment (Section 8.1.1), and
- analytical assessment (Section 8.1.2).

Guidance on analytical assessment is provided in Chapter One of SW-846 and in the individual analytical methods. Additional guidance can be found in *Guidance on Environmental Data Verification and Data Validation EPA QA/G-8,* published by EPA's Office of Environmental Information (USEPA 2001c). For projects generating data for input into risk assessments, see EPA's *Guidance for Data Usability in Risk Assessment, Final* (USEPA 1992g).

8.1.1 Sampling Assessment

Sampling assessment is the process of reviewing field sampling and sample handling methods to check conformance with the requirements specified in the QAPP. Sampling assessment activities include a review of the sampling design, sampling methods, documentation, sampling handling and custody procedures, and preparation and use of quality control samples.

The following types of information are useful in assessing the sampling activity:

- Copies of the sampling plan, QAPP, and SOPs.

- Copies of logbooks, chain-of-custody records, bench sheets, well logs, sampling sequence logs, field instrument calibration records and performance records, and/or other records (including electronic records such as calculations) that describe and/or record all sampling operations, observations, and results associated with samples (including all QC samples) while in the custody of the sampling team. Records/results from the original sampling and any resampling, regardless of reason, should be retained. Also, retain copies of the shipping manifest and excess sample disposition (disposal) records describing the ultimate fate of any sample material remaining after submission to the laboratory.

- Copies of all records/comments associated with the sample team review of the original data, senior staff review, and QA/QC review of the sampling activity. Copies of any communication (telephone logs, faxes, E-mail, other records) between the sampling team and the customer dealing with the samples and any required resampling or reporting should be provided.

The following subsections outline the types of sampling information that should be assessed.

8.1.1.1 Sampling Design

Review the documentation of field activities to check if the number and type of samples called for in the sampling plan were, in fact, obtained and collected from the correct locations. Perform activities such as those described below:

- Sampling Design: Document any deviations from the sampling plan made during the field sampling effort and state what impact those modifications might have on the sampling results.

- Sample Locations/Times: Confirm that the locations of the samples in time or space match those specified in the plan.

- Number of Samples: Check for completeness in the sampling in terms of the number of samples obtained compared to the number targeted. Note the cause of the deficiencies such as structures covering planned locations, limited access due to unanticipated events, samples lost in shipment or in the laboratory, etc.

- Discrete versus Composite Samples: If composite sampling was employed, confirm that each component sample was of equal mass or volume. If not, determine if sufficient information is presented to allow adjustments to any calculations made on the data. Both field and laboratory records should be reviewed because compositing can occur at either location.

8.1.1.2 Sampling Methods

Details of how a sample was obtained from its original time/space location are important for properly interpreting the measurement results. Review the selection of sampling and ancillary equipment and procedures (including equipment decontamination) for compliance with the QAPP and sampling theory. Acceptable departures (for example, alternate equipment) from the QAPP and the action to be taken if the requirements cannot be satisfied should be specified for each critical aspect. Note potentially unacceptable departures from the QAPP and assess their potential impact on the quality and usefulness of the data. Comments from field surveillance on deviations from written sampling plans also should be noted.

Sampling records should be reviewed to determine if the sample collection and field processing were appropriate for the analytes being measured. For example, sampling for volatiles analysis poses special problems due to the likely loss of volatiles during sample collection. Also, determination of the appropriate “sample support” should be reviewed, whether it was obtained correctly in the field, whether any large particles or fragments were excluded from the sample, and whether any potential biases were introduced.

Laboratory subsampling and sample preparation protocols should be examined for the same types of potential bias as the field procedures. When found, they should be discussed in the assessment report.

8.1.1.3 Sample Handling and Custody Procedures

Details of how a sample is physically treated and handled between its original site or location and the actual measurement site are extremely important. Sample handling activities should be reviewed to confirm compliance with the QAPP or WAP for the following areas:

- Sample containers
- Preservation (physical and chemical)
- Chain-of-custody procedures and documentation
- Sample shipping and transport
- Conditions for storage (before analysis)
- Holding times.

8.1.1.4 Documentation

Field records generally consist of bound field notebooks with prenumbered pages, sample collection forms, sample labels or tags, sample location maps, equipment maintenance and calibration forms, chain-of-custody forms, sample analysis request forms, and field change request forms. Documentation also may include maps used to document the location of sample collection points or photographs or video to record sampling activities.

Review field records to verify they include the appropriate information to support technical interpretations, judgments, and discussions concerning project activities. Records should be legible, identifiable, and retrievable and protected against damage, deterioration, or loss. Especially note any documentation of deviations from SOPs and the QAPP.

8.1.1.5 Control Samples

Assess whether the control samples were collected or prepared as specified in the QAPP or WAP. Control samples include blanks (e.g., trip, equipment, and laboratory), duplicates, spikes, analytical standards, and reference materials that are used in different phases of the data collection process from sampling through transportation, storage, and analysis. There are many types of control samples, and the appropriate type and number of control samples to be used will depend on the data quality specifications.

See Section 7.2.4 for guidance on the type of control samples for RCRA waste-testing programs. Additional guidance on the preparation and use of QC samples can be found in the following publications:

- *Test Methods for Evaluating Solid Waste, SW-846* (USEPA 1986a), Chapter One
- *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5* (USEPA 1998a), Appendix D
- *Contract Laboratory Program (CLP) Guidance for Field Samplers - Draft Final* (USEPA 2001g), Section 3.1.1.

8.1.2 Analytical Assessment

Analytical assessment includes an evaluation of analytical and method performance and supporting documentation relative to the DQOs. Proper data review is necessary to minimize decision errors caused by out-of-control laboratory processes or calculation or transcription errors. The level and depth of analytical assessment is determined during the planning process and is dependent on the types of analyses performed and the intended use of the data.

Analytical records needed to perform the assessment of laboratory activities may include the following:

- Contract Statement of Work requirements
- SOPs
- QAPP or WAP
- Equipment maintenance documentation
- Quality assurance information on precision, bias, method quantitation limits, spike recovery, surrogate and internal standard recovery, laboratory control standard recovery, checks on reagent purity, and checks on glassware cleanliness

- Calibration records
- Traceability of standards/reagents (which provide checks on equipment cleanliness and laboratory handling procedures)
- Sample management records
- Raw data
- Correspondence
- Logbooks and documentation of deviation from procedures.

If data gaps are identified, then the assessor should prepare a list of missing information for correspondence and discussion with the appropriate laboratory representative. At that time, the laboratory should be requested to supply the information or to attest that it does not exist in any form.

8.1.2.1 Analytical Data Verification

The term **data verification** is confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. Data verification is the process of evaluating the completeness, correctness, and conformance/compliance of a specific data set against the method, procedural, or contractual requirements. The goal of data verification is to ensure that the data are what they purport to be, that is, that the reported results reflect what was actually done, and to document that the data fulfill specific requirements. When deficiencies in the data are identified, then those deficiencies should be documented for the data user's review and, where possible, resolved by corrective action (USEPA 2001c).

Data verification may be performed by personnel involved with the collection of samples or data, generation of analytical data, and/or by an external data verifier. The verification process normally starts with a list of requirements that apply to an analytical data package. It compares the laboratory data package to the requirements and produces a report that identifies those requirements that were met and not met. Requirements that were not met can be referred to as exceptions and may result in flagged data. Examples of the types of exceptions that are found and reported are listed below:

- Failure to analyze samples within the required holding times
- Required steps not carried out by the laboratory (i.e., failure to maintain sample custody, lack of proper signatures, etc.)
- Procedures not conducted at the required frequency (i.e., too few blanks, duplicates, etc.)
- Contamination found in storage, extraction, or analysis of blanks
- Procedures that did not meet pre-set acceptance criteria (poor laboratory control, poor sample matrix spike recovery, unacceptable duplicate precision, etc).

The verification report should detail all exceptions found with the data packages. If the laboratory was able to provide the missing information or a suitable narrative explanation of the exceptions, they should be made part of the report and included in the data package for use by the people who determine the technical defensibility of the data.

8.1.2.2 Analytical Data Validation (Evaluation)

The term **data validation** (also known as "evaluation") is the confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. Data validation is an analyte- and sample-specific process that extends the evaluation of data beyond method, procedural, or contractual compliance (i.e., data verification) to determine the analytical quality of a specific data set. Data validation criteria are based upon the measurement quality objectives developed in the QAPP or similar planning document, or presented in the sampling or analytical method. Data validation includes a determination, where possible, of the reasons for any failure to meet method, procedural, or contractual requirements, and an evaluation of the impact of such failure on the overall data set (USEPA 2001c)

Data validation includes inspection of the verified data and both field and analytical laboratory data verification documentation; a review of the verified data to determine the analytical quality of the data set; and the production of a data validation report and, where applicable, qualified data. A focused data validation may also be required as a later step. The goals of data validation are to evaluate the quality of the data, to ensure that all project requirements are met, to determine the impact on data quality of those requirements that were not met, and to document the results of the data validation and, if performed, the focused data validation. The main focus of data validation is determining data quality in terms of accomplishment of measurement quality objectives.

As in the data verification process, all planning documents and procedures not only must exist, but they should also be readily available to the data validators. A data validator's job cannot be completed properly without the knowledge of the specific project requirements. In many cases, the field and analytical laboratory documents and records are validated by different personnel. Because the data validation process requires knowledge of the type of information to be validated, a person familiar with field activities usually is assigned to the validation of the field documents and records. Similarly, a person with knowledge of analytical laboratory analysis, such as a chemist (depending on the nature of the project), usually is assigned to the validation of the analytical laboratory documents and records. The project requirements should assist in defining the appropriate personnel to perform the data validation (USEPA 2001c).

The personnel performing data validation should also be familiar with the project-specific data quality indicators (DQIs) and associated measurement quality objectives. One of the goals of the data validation process is to evaluate the quality of the data. In order to do so, certain data quality attributes are defined and measured. DQIs (such as precision, bias, comparability, sensitivity, representativeness, and completeness) are typically used as expressions of the quality of the data (USEPA 2001c).

The outputs that may result from data validation include validated data, a data validation report, and a focused validation report. For detailed guidance on data validation, see Chapter One of SW-846 and *Guidance on Environmental Data Verification and Data Validation EPA QA/G-8*

(USEPA 2001c).

8.2 Data Quality Assessment

Data quality assessment (DQA) is the scientific and statistical evaluation of data to determine if the data are of the right type, quality, and quantity to support their intended purpose (USEPA 2000d). The focus of the DQA process is on the use of statistical methods for environmental decision making – though not every environmental decisions necessarily must be made based on the outcome of a statistical test (see also Section 3). If the sampling design established in the planning process requires estimation of a parameter or testing of a hypothesis, then the DQA process can be used to evaluate the sample analysis results.

The DQA process described in this section includes five steps: (1) reviewing the DQOs and study design, (2) preparing the data for statistical analysis, (3) conducting a preliminary review of the data and checking statistical assumptions, (4) selecting and performing statistical test, and (5) drawing conclusions from the data (Figure 36).

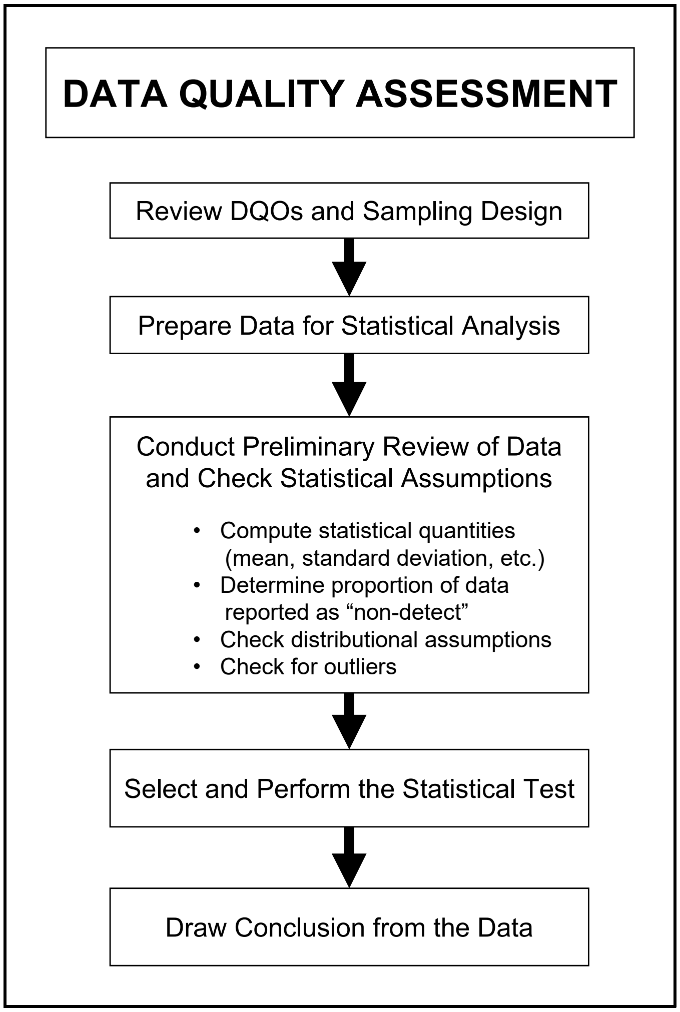

Figure 36. The DQA Process (modified from USEPA 2000d)

Detailed guidance on the statistical analysis of data can be found in Appendix F. Additional guidance can be found in *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d). A list of software tools to help you implement the DQA is provided in Appendix H.

8.2.1 Review the DQOs and the Sampling Design

Review the DQO outputs to ensure that they are still applicable. Refer back to Sections 4 and 5 of this document for more information on the DQO Process or see USEPA 2000a or 2000b. A clear understanding of the original project objectives, as determined during the systematic planning process, is critical to selecting the appropriate statistical tests (if needed) and interpreting the results relative to the applicable RCRA regulatory requirements.

8.2.2 Prepare Data for Statistical Analysis

After data validation and verification and before the data are available in a form for further analysis, several intermediate steps usually are required. For most situations, EPA recommends you prepare the data in computer-readable format. Steps in preparing data for statistical analysis are outlined below (modified from Ott 1988):

Steps in Preparing Data for Statistical Analysis

1. Receive the verified and validated data source.
2. Create a data base from the verified and validated data source.
3. Check and edit the data base.
4. Create data files from the data base.

1. *Receive the verified and validated source from the QA reports.* Data are supplied to the user in a variety of formats and readiness for use, depending on the size and complexity of the study and the types of analyses requested. Most laboratories supply a QA evaluation package that includes the verification/validation review, a narrative, tabulated summary forms (including the results of analyses of field samples, laboratory standards, and QC samples), copies of logbook pages, and copies of chain-of-custody records. From this information, you can create a data base for statistical analysis.

2. *Create a data base from the verified and validated data source.* For most studies in which statistical analyses are scheduled, a computer-readable data base is the most efficient method for managing the data. The steps required to create the data base and the format used will depend on the software systems used to perform the analysis. For example, the data base may be as simple as a string of concentration values for a single constituent input into a spreadsheet or word processor (such as required for use of EPA's DataQUEST software (USEPA 1997b)), or it may be more complex, requiring multiple and related data inputs, such as sample number, location coordinates, depth, date and time of collection, constituent name and concentration, units of measurements, test method, quantitation limit achieved, QC information, etc.

   If the data base is created via manual data entry, the verified and validated data should be checked for legibility. Any questions pertaining to illegible information should be resolved before the data are entered. Any special coding considerations, such as indicating values reported as "nondetect" should be specified in a coding guide or in the QAPP. For very large projects, it may be appropriate to prepare a separate detailed data management plan in advance.

3. *Check and edit the data base.* After creation of the data set, the data base should be checked against the data source to verify accurate data entry and to correct any errors discovered. Even if the data base is received from the laboratory in electronic format, it should be checked for obvious errors, such as unit errors, decimal errors, missing values, and quantitation limits.

4. *Create data files from the data base.* From the original data files, work files are created for use within the statistical software package. This step could entail separating data by constituent and by DQO decision unit and separating any QA/QC data from the record data. When creating the final data files for use in the statistical software, be sure to use a file naming and storage convention that facilitates easy retrieval for future use, reference, or reporting.

8.2.3 Conduct Preliminary Review of the Data and Check Statistical Assumptions

Many statistical tests and procedures require that certain assumptions be met for their use. Failure to satisfy these assumptions can result in biased estimates of the parameter of interest; therefore, it is important to conduct preliminary analyses of the data to learn about the characteristics. EPA recommends that you compute statistical quantities, determine the proportion of the data reported as “nondetect” for each constituent of concern, check whether the data exhibit a normal distribution, then determine if there are any “outliers” that deserve a closer look. The outputs of these activities are used to help select and perform the appropriate statistical tests.

8.2.3.1 Statistical Quantities

To help “visualize” and summarize the data, calculate basic statistical quantities such as the:

- Mean
- Maximum
- Percentiles
- Variance
- Standard deviation
- Coefficient of variation.

Calculate the quantities for each constituent of concern. Example calculations of the mean, variance, standard deviation, and standard error of the mean are given in Section 3. Detailed guidance on the calculation of statistical quantities is provided in Chapter Two of EPA’s *QA/G-9* guidance document (USEPA 2000d). The useful quantities easily can be computed using EPA’s DataQUEST software (USEPA 1997b, see also Appendix H) or any similar statistical software package.

When calculating statistical quantities, determine which data points were reported as below a limit of detection or quantitation - known as “nondetects” (NDs). See also Section 8.2.4.2 (“Treatment of Nondetects”).

8.2.3.2 Checking Data for Normality

Check the data sets for normality by using graphical methods, such as histograms, box and whisker plots, and normal probability plots (see also Section 3.1.3), or by using numerical tests, such as the Shapiro-Wilk test for normality (see Appendix F). Table 11 provides a summary of recommended methods. Detailed guidance on the use of graphical and statistical methods can be found in USEPA 1989b, 1992b, 1997b, and 2000d.

Table 11. Recommended Graphical and Statistical Methods for Checking Distributional Assumptions

Test	***Use***	***Reference***
Graphical Methods		
Histograms and frequency plots	Provides visual display of probability or frequency distribution	See USEPA 2000d. Construct via EPA's DataQUEST software (USEPA 1997b) or use a commercial software package.
Normal probability plot	Provides visual display of deviation from expected normality	See USEPA 2000d. Construct via EPA's DataQUEST software (USEPA 1997b) or use a commercial software package.
Box and Whisker Plot	Provides visual display of potential "outliers" or extreme values	See USEPA 2000d. Construct via EPA's DataQUEST software (USEPA 1997b) or use a commercial software package.
Numerical Tests for Normality		
Shapiro-Wilk Test	Use for sample sizes of ≤ 50	See procedure in Appendix F, Section F.1.2. This test also can be performed using EPA's DataQUEST software (USEPA 1997b).
Filliben's Statistic	Use for sample sizes of > 50	See USEPA 2000d. This test can be performed using EPA's DataQUEST software (USEPA 1997b).

Graphical methods allow you to visualize the central tendency of the data, the variability in the data, the location of extreme data values, and any obvious trends in the data. For example, a symmetrical "mound" shape of a histogram is an indicator of an approximately normal distribution. If a normal probability plot is constructed on the data (see Figure 5 in Section 3.1.3), a straight line plot usually is an indicator of normality. (Note that interpretation of a probability plot depends on the method used to construct it. For example, in EPA's DataQUEST software, normally distributed data will form an "S"-shaped curve rather than a straight line on a normal probability plot.)

The Shapiro-Wilk test is recommended as a superior method for testing normality of the data. The specific method for implementing the Shapiro-Wilk Test is provided in Appendix F. The method also is described in Gilbert (1987), EPA's guidance on the statistical analysis of ground-water monitoring data (USEPA 1992b), and can be performed with EPA's DataQUEST software or other commercially available statistical software.

8.2.3.3 How To Assess "Outliers"

A measurement that is very different from other values in the data set is sometimes referred to as an "outlier." EPA cautions that the term "outlier" be used advisedly, since a common reaction to the presence of "outlying" values has been to "cleanse the data," thereby removing any "outliers" prior to further analysis. In fact, such discrepant values can occur for many reasons, including (1) a catastrophic event such as a spill or process upset that impacts measurements at the sampling point, (2) inconsistent sampling or analytical chemistry methodology that may result in laboratory contamination or other anomalies, (3) errors in the transcription of data values or decimal points, and (4) *true* but extreme hazardous constituent measurements.

While any one of these events can cause an apparent “outlier,” it should be clear that the appropriate response to an outlier will be very different depending on the origin. Because high values due to contaminated media or waste are precisely what one may be trying to identify, it would not be appropriate to eliminate such data in the guise of “screening for outliers.” Furthermore, depending on the form of the underlying population, unusually high concentrations may be real but infrequent such as might be found in lognormally distributed data. Again, it would not be appropriate to remove such data without adequate justification.

A *statistical outlier* is defined as a value originating from a different underlying population than the rest of the data set. If the value is not consistent with the distributional behavior of the remaining data and is “too far out in one of the tails” of the assumed underlying population, it may test out as a statistical outlier. Defined as it is strictly in statistical terms, however, an outlier test may identify values as discrepant when no physical reason can be given for the aberrant behavior. One should be especially cautious about indiscriminate testing for statistical outliers for this reason.

If an outlier is suspected, an initial and helpful step is to construct a probability plot of the data set (see also Section 3.1.3 and USEPA 2000d). A probability plot is designed to judge whether the sample data are consistent with an underlying normal population model. If the rest of the data follow normality, but the outlier comes from a distinctly different population with higher (or lower) concentrations, this behavior will tend to show up on a probability plot as a lone value “out of line” with the remaining observations. If the data are lognormal instead, but the outlier is again from a distinct population, a probability plot on the logged observations should be constructed. Neither of these plots is a formal test; still, they provide invaluable visual evidence as to whether the suspected outlier should really be considered as such.

Methods for conducting outlier tests are described in Chapter 4 of EPA’s QA/G-9 guidance document (USEPA 2000d), and statistical tests are available in the DataQUEST software (for example, Rosner’s Test and Walsh’s Test) (USEPA 1997b).

8.2.4 Select and Perform Statistical Tests

This section provides guidance on how you can select the appropriate statistical test to make a decision about the waste or media that is the subject of the study. It is important to select the appropriate statistical test because decisions and conclusions derived from incorrectly used statistics can be expensive (Singh, et al. 1997).

Prior to selecting the statistical test, consider the following factors:

- The objectives of the study (identified in DQO Step 2)
- Whether assumptions of the test are fulfilled
- The nature of the underlying distribution

- The decision rule and null hypothesis (identified in DQO Step 5)
- The relative performance of the candidate tests (for example, parametric tests generally are more efficient than their nonparametric counterparts)
- The proportion of the data that are reported as nondetects (NDs).

The decision-tree presented in Figure 37 provides a starting point for selecting the appropriate statistical test. The statistical methods are offered as guidance and should not be used as a "cook book" approach to data analysis. The methods presented here usually will be adequate for the tests conducted under the specified conditions (see also Appendix F). An experienced statistician should be consulted whenever there are questions.

Based on the study objective (DQO Step 2), determine which category of statistical tests to use. Note the statistical methods recommended in the flow charts in Figure 38 and Figure 39 are for use when the objective is to compare the parameter of interest to a fixed standard. Other methods will be required if the objective is different (e.g., when comparing two populations, detecting trends, and evaluating spatial patterns or relationships of sampling points).

8.2.4.1 Data Transformations in Statistical Tests

Users of this guidance may encounter data sets that show significant evidence of non-normality. Due to the assumption of underlying normality in most parametric tests, a common statistical strategy when encountering this predicament is to search for a mathematical transformation that will lead to normally-distributed data on the transformed scale. Unfortunately, because of the complexities associated with interpreting statistical results from data that have been transformed to another scale and the common occurrence of lognormal patterns in environmental data, EPA generally recommends that the choice of scale be limited to either the original measurements (for normal data) or a log-transformed scale (for lognormal data). If neither of these scales results in approximate normality, it is typically easiest and wisest to switch to a nonparametric (or “distribution-free”) version of the same test.

If a transformation to the log scale is needed, and a confidence limit on the mean is desired, special techniques are required. If a data set exhibits a normal distribution on the log-transformed scale, it is a common mistake to assume that a standard normal-based confidence interval formula can be applied to the transformed data with the confidence interval endpoints retransformed to the original scale to obtain the confidence interval on the mean. Invariably, such an interval will be biased to the low side. In fact, the procedure just described actually produces a confidence interval around the *median* of a lognormal population, rather than the higher *mean*. To correctly account for this “transformation bias”, special procedures are required (Land 1971 and 1975, Gilbert 1987). See Section F.2.3 in Appendix F for detailed guidance on calculating confidence limits for the mean of a lognormal population.

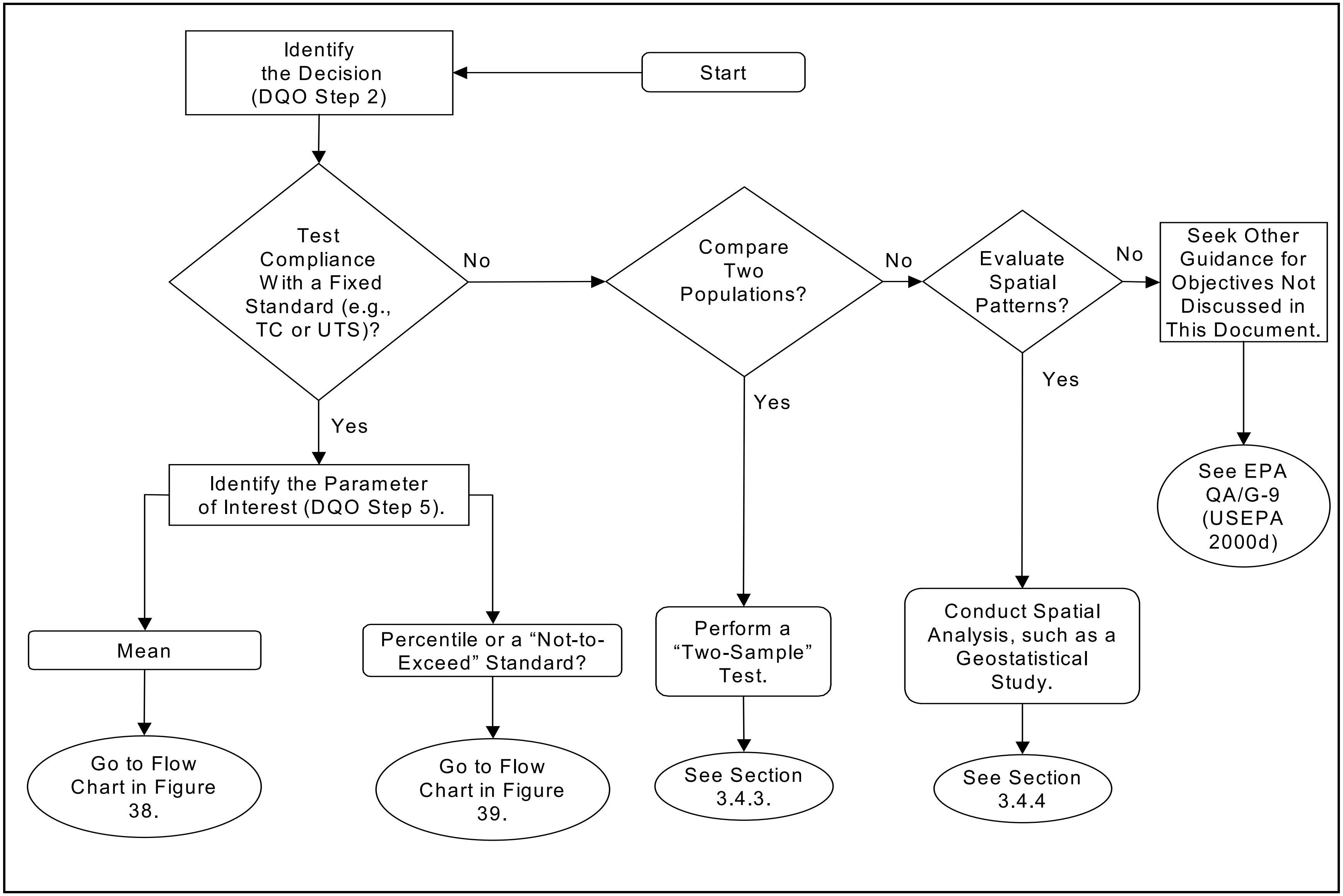

Figure 37. Flow chart for selecting a statistical method

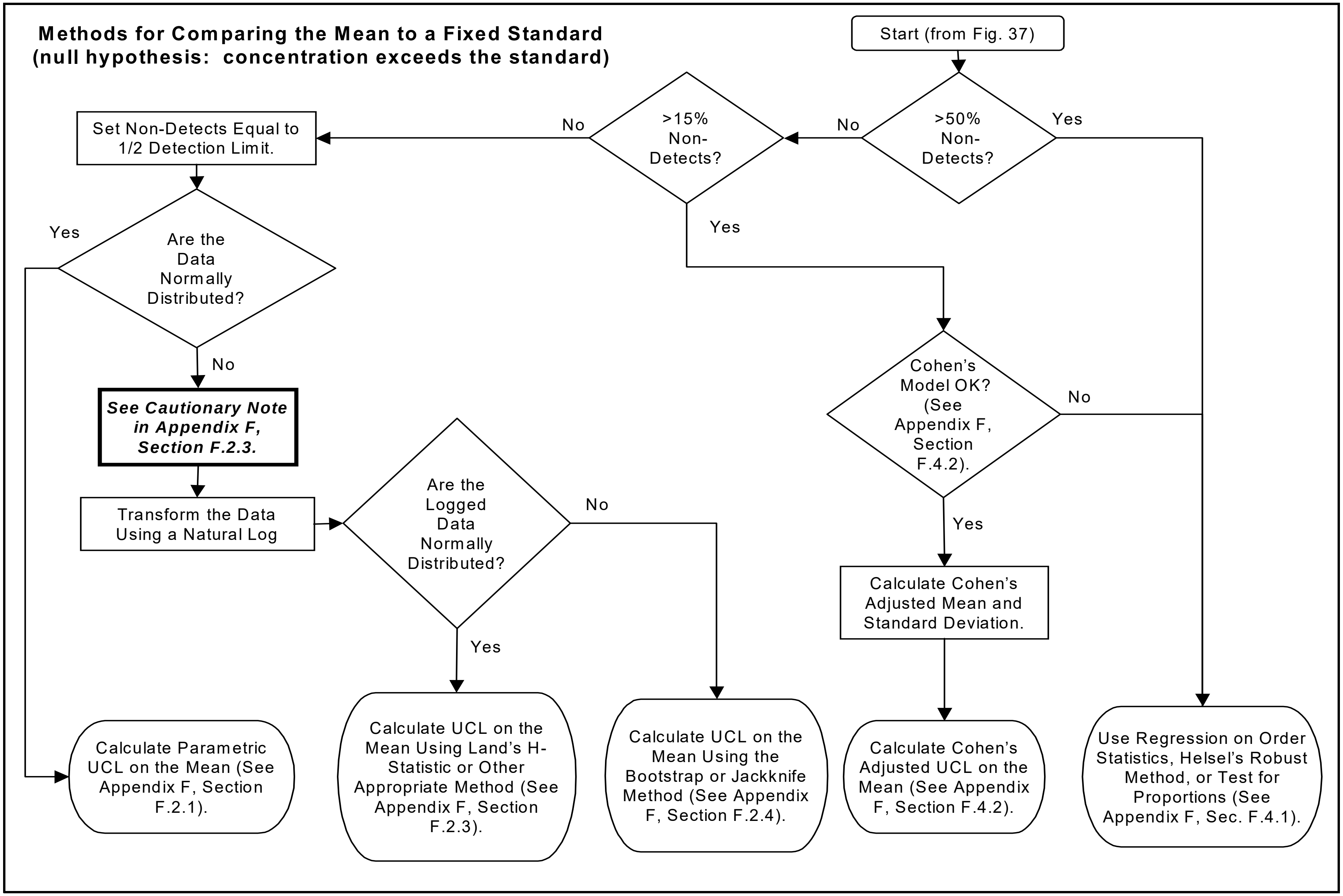

Figure 38. Flowchart of statistical methods for comparing the mean to a fixed standard (null hypothesis is "concentration exceeds the standard")

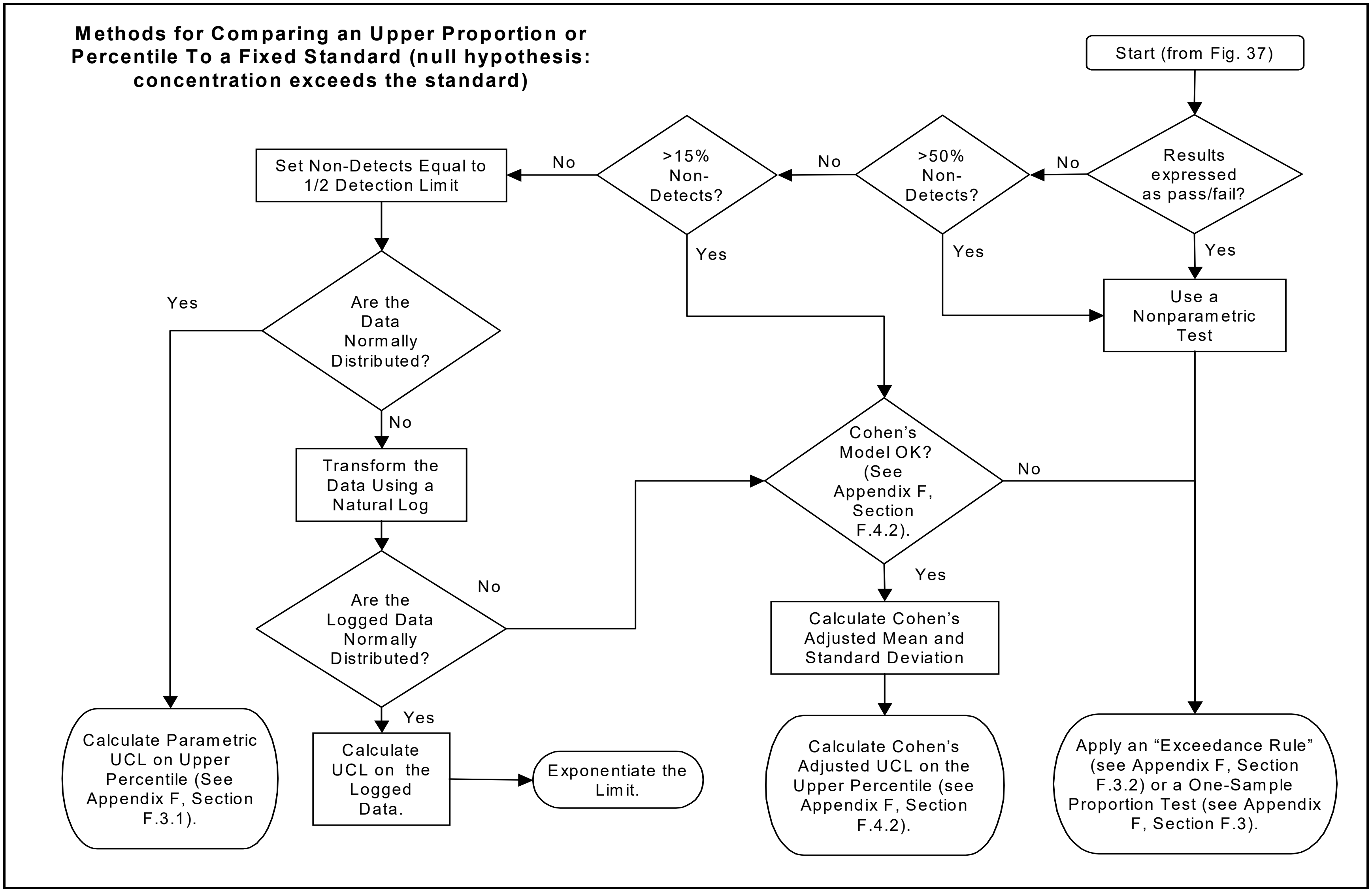

Figure 39. Flowchart of statistical methods for comparing an upper proportion or percentile to a fixed standard (null hypothesis is "concentration exceeds the standard")

If the number of samples is small, it may not be possible to tell whether the distribution is normal, lognormal, or any other specific function. You are urged not to read too much into small data sets and not to attempt overly sophisticated evaluations of data distributions based on limited information. If the distribution of data appears to be highly skewed, it is best to take operational measures (such as more samples or samples of a larger physical size) to better characterize the waste.

8.2.4.2 Treatment of Nondetects

If no more than approximately 15 percent of the samples for a given constituent are nondetect (i.e., reported as below a detection or quantitation limit), the results of parametric statistical tests will not be substantially affected if nondetects are replaced by half their detection limits (known as a substitution method) (USEPA 1992b). When a larger percentage of the sample analysis results are nondetect, however, the treatment of nondetects is more crucial to the outcome of statistical procedures. Indeed, simple substitution methods (such as replacing the detection limit with one-half the detection limit) tend to perform poorly in statistical tests when the nondetect percentage is substantial (Gilliom and Helsel 1986, Helsel 1990).

Guidance on selecting an approach for handling nondetects in statistical intervals is given in Appendix F, Section F.4. Guidance also is given in Section 4.7 of EPA's *Guidance for Data Quality Assessment Practical Methods for Data Analysis EPA QA/G-9* (USEPA 2000d).

8.2.5 Draw Conclusions and Report Results

The final step in the DQA Process is to draw conclusions from the data, determine if further sampling is required, and report the results. This step brings the planning, implementation, and assessment process "full circle" in that you attempt to resolve the problem and make the decision identified in Steps 1 and 2 of the DQO Process.

In the DQO Process, you establish a "null hypothesis" and attempt to gather evidence via sampling that will allow you to reject that hypothesis; otherwise, the null hypothesis must be accepted. If the decision making process involves use of a statistical method (such as the calculation of a statistical confidence limit or use of a statistical hypothesis test), then the outcome of the statistical test should be reported along with the uncertainty associated with the result. If other decision making criteria are used (such as use of a simple exceedance rule or a "weight of evidence" approach), then the outcome of that decision making process should be reported.

Detailed guidance on the use and interpretation of statistical methods for decision making can be found in **Appendix F**. Additional guidance can found in EPA's *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d).

Most of the statistical methods suggested in this document involve the construction of one-sided confidence limits (or bounds). The upper confidence limit, whether calculated on a mean, median, or percentile, provides a value below which one can claim with specified confidence that the true value of the parameter lies. Figure 40 demonstrates how you can use a confidence limit to test a hypothesis: In the situation depicted at "A," the upper confidence limit calculated from the sample data is less than the applicable standard and provides the evidence needed to reject the null hypothesis. The decision can be made that the waste concentration is below the standard with sufficient confidence and without further analysis.

In situation "B," we cannot reject the null hypothesis; however, because the interval "straddles" the standard, it is *possible* that the true mean lies below the standard and a Type II (false acceptance) error has been made (i.e., to conclude the concentration is above the standard, when in fact it is not). One possible remedy to this situation is to obtain more data to "tighten" the confidence interval.

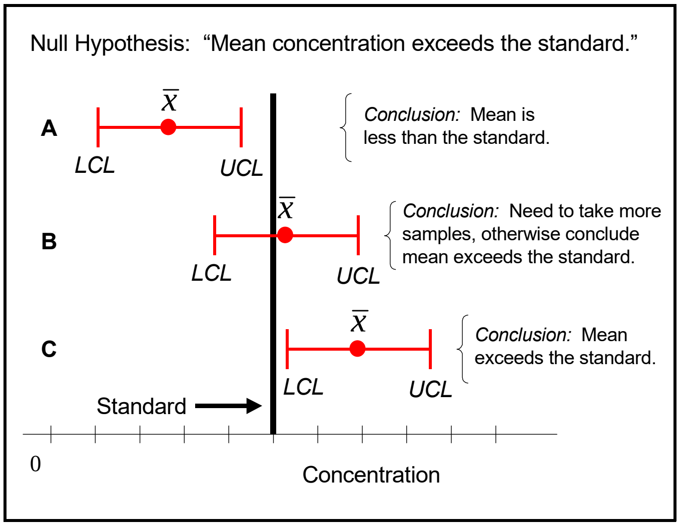

Figure 40. Using confidence limits on the mean to compare waste concentrations to a fixed standard.

In situation "C," the Type II (false acceptance) decision error rate is satisfied and we must conclude that the mean concentration exceeds the standard.

One simple method for checking the performance of the statistical test is use the information obtained from the samples to retrospectively estimate the number of samples required. For example, the sample variance can be input into the sample size equation used (see Section 5.4 and 5.5, DQO Process Step 7). (An example of this approach is presented in Appendix I.) If this theoretical sample size is less than or equal to the number of samples actually taken, then the test is sufficiently powerful. If the required number of samples is greater than the number actually collected, then additional samples would be required to satisfy the data user's performance criteria for the statistical test. See EPA's *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d) for additional guidance on this topic.

Finally, if a simple exceedance rule is used to measure compliance with a standard, then interpretation of the results is more straightforward. For example, if zero exceedances are allowed, and one or more samples exceeds the standard, then there is evidence of noncompliance with that standard (see Appendix F, Section F.3.2).

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APPENDIX A

GLOSSARY OF TERMS*

Accuracy - A measure of the closeness of an individual measurement or the average of a number of measurements to the true value. Accuracy includes a combination of random error (*precision*) and systematic error (bias) components that are due to sampling and analytical operations. EPA recommends using the terms "precision" and "bias," rather than the term "accuracy," to convey the information usually associated with accuracy. Pitard (1993) indicates that a sample is accurate when the absolute value of the bias is smaller than an acceptable standard of accuracy.

Action Level - The numerical value that causes the decision maker to choose one of the alternative actions (for example, compliance or noncompliance). It may be a regulatory threshold standard, such as the maximum contaminant level for drinking water, a risk-based concentration level, a technological limitation, or a reference-based standard (ASTM D 5792-95).

Alternative Hypothesis - See *Hypothesis*.

Assessment - The evaluation process used to measure the performance or effectiveness of a system and its elements. As used here, assessment is an all-inclusive term used to denote any of the following: *audit*, performance evaluation (PE), management systems review (MSR), peer review, inspection, or surveillance.

Audit (quality) - A systematic and independent examination to determine whether quality activities and related results comply with planned arrangements and whether these arrangements are implemented effectively and are suitable to achieve objectives.

Audit of Data Quality - A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

Baseline Condition - A tentative assumption to be proven either true or false. When *hypothesis* testing is applied to a site *assessment* decision, the data are used to choose between a presumed baseline condition of the environment and an alternative condition. The baseline condition is retained until overwhelming evidence indicates that the baseline condition is false. This is often called the *null hypothesis* in statistical tests.

Bias - The systematic or persistent distortion of a measured value from its true value (this can occur during sampling design, the sampling process, or laboratory analysis).

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* The definitions in this appendix are from USEPA 1998a, 2000b, 2000e, and 2001b, unless otherwise noted. Some definitions were modified based on comments received from technical reviewers during development of this document. These definitions do not constitute the Agency's official use of the terms for regulatory purposes and should not be construed to alter or supplant other terms in use.

Note: Terms in *italics* also are defined in this glossary.

Blank - A sample that is intended to contain none of the analytes of interest and is subjected to the usual analytical or measurement process to establish a zero baseline or background value. Sometimes used to adjust or correct routine analytical results. A blank is used to detect contamination during sample handling preparation and/or analysis (see also *Rinsate*, *Method Blank*, *Trip Blank*, and *Field Blank*).

Boundaries - The spatial and temporal limits and practical constraints under which environmental data are collected. Boundaries specify the area or volume (spatial boundary) and the time period (temporal boundary) to which the decision will apply. Samples are then collected within these boundaries.

Calibration - Comparison of a measurement standard, instrument, or item with a standard or instrument of higher *accuracy* to detect and quantify inaccuracies and to report or eliminate those inaccuracies by adjustments. Calibration also is used to quantify instrument measurements of a given concentration in a given sample.

Calibration Drift - The deviation in instrument response from a reference value over a period of time before recalibration.

Chain of Custody - An unbroken trail of accountability that ensures the physical security of samples, data, and records.

Characteristic - Any property or attribute of a datum, item, process, or service that is distinct, describable, and/or measurable.

Coefficient of Variation (CV) - A dimensionless quantity used to measure the spread of data relative to the size of the numbers. For a normal distribution, the coefficient of variation is given by $s / \overline{x}$. Also known as the *relative standard deviation (RSD)*.

Colocated Samples - Two or more portions collected as close as possible at the same point in time and space so as to be considered identical. If obtained in the field, these samples also are known as “field replicates.”

Comparability - A measure of the confidence with which one data set or method can be compared to another.

Completeness - A measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct, normal conditions.

Component - An easily identified item such as a large crystal, an agglomerate, rod, container, block, glove, piece of wood, or concrete (ASTM D 5956-96). An elementary part or a constituent that can be separated and quantified by analysis (Pitard 1993).

Composite Sample - A physical combination of two or more samples (ASTM D 6233-98). A sample collected across a temporal or spatial range that typically consists of a set of discrete samples (or "individual" samples) that are combined or "composited." Area-wide or long-term compositing should not be confused with localized compositing in which a sample of the desired support is created from many small increments taken at a single location. Four types of composite samples are listed below:

1. Time Composite - a sample comprising a varying number of discrete samples collected at equal time intervals during the compositing period. The time composite sample is typically used to sample waste water or streams.

2. Flow Proportioned Composite (FPC) - a sample collected proportional to the flow during the compositing period by either a time-varying/constant volume (TVCV) or a time-constant/varying volume method (TCVV). The TVCV method typically is used with automatic samplers that are paced by a flow meter. The TCVV method is a manual method that individually proportions a series of discretely collected samples. The FPC is typically used when sampling waste water.

3. Areal Composite - sample composited from individual equal-size samles collected on an areal or horizontal cross-sectional basis. Each discrete sample is collected in an identical manner. Examples include sediment composites from quarter-point sampling of streams and soil samples from within grids.

4. Vertical Composite - a sample composited from individual equal samples collected from a vertical cross section. Each discrete sample is collected in an identical manner. Examples include vertical profiles of soil/sediment columns, lakes, and estuaries (USEPA 1996c).

Confidence Level - The probability, usually expressed as a percent, that a confidence interval will contain the *parameter* of interest (ASTM D 5792-95). Also known as the confidence coefficient.

Confidence Limits - Upper and/or lower limit(s) within which the true value of a parameter is likely to be contained with a stated probability or confidence (ASTM D 6233-98).

Conformance - An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation. Also the state of meeting the requirements.

Consensus Standard - A standard established by a group representing a cross section of a particular industry or trade, or a part thereof.

Control Sample - A quality control sample introduced into a process to monitor the performance of the system (from Chapter One, SW-846).

Data Collection Design - A design that specifies the configuration of the environmental monitoring effort to satisfy the *data quality objectives*. It includes: the types of samples or monitoring information to be collected; where, when, and under what conditions they should be collected; what variables are to be measured; and the quality assurance/quality control (QA/QC) components that ensure acceptable sampling design error and measurement error to meet the *decision error* rates specified in the DQOs. The data collection design is the principal part of the quality assurance project plan (QAPP).

Data of Known Quality - Data that have the qualitative and quantitative components associated with their derivation documented appropriately for their intended use, and when such documentation is verifiable and defensible.

Data Quality Assessment (DQA) Process - A statistical and scientific evaluation of the data set to assess the validity and performance of the data collection design and statistical test and to establish whether a data set is adequate for its intended use.

Data Quality Indicators (DQIs) - The quantitative statistics and qualitative descriptors that are used to interpret the degree of acceptability or utility of data to the user. The principal data quality indicators are *bias*, *precision*, *accuracy* (precision and bias are preferred terms), *comparability*, *completeness*, and *representativeness*.

Data Quality Objectives (DQOs) - Qualitative and quantitative statements derived from the DQO Process that clarify study technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential *decision error*s that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Data Quality Objectives (DQO) Process - A systematic strategic planning tool based on the scientific method that identifies and defines the type, quality, and quantity of data needed to satisfy a specified use. The key elements of the process include:

- concisely defining the problem
- identifying the decision to be made
- identifying the key inputs to that decision
- defining the *boundaries* of the study
- developing the decision rule
- specifying tolerable limits on potential *decision errors*
- selecting the most resource efficient data collection design.

Data Reduction - The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collating them into a more useful and understandable form. Data reduction generally results in a reduced data set and an associated loss of detail.

Data Usability - The process of ensuring or determining whether the quality of the data produced meets the intended use of the data.

Data Validation - See *Validation*.

Debris - Under 40 CFR 268.2(g) (Land Disposal Restrictions regulations) debris includes “solid material exceeding a 60 mm particle size that is intended for disposal and that is a manufactured object; or plant or animal matter; or natural geologic material.” 268.2(g) also identifies materials that are not debris. In general, debris includes materials of either a large particle size or variation in the items present. When the constituent items are more than 2 or 3 inches in size or are of different compositions, *representative* sampling becomes more difficult.

Decision Error - An error made when drawing an inference from data in the context of *hypothesis* testing such that variability or *bias* in the data mislead the decision maker to draw a conclusion that is inconsistent with the true or actual state of the population under study. See also *False Negative Decision Error*, and *False Positive Decision Error*.

Decision Performance Curve - A graphical representation of the quality of a decision process. In statistical terms it is known as a power curve or function (or a reverse power curve depending on the hypotheses being tested).

Decision Performance Goal Diagram (DPGD) - A graphical representation of the tolerable risks of *decision errors*. It is used in conjunction with the decision performance curve.

Decision Unit - A volume or mass of material (such as waste or soil) about which a decision will be made.

Defensible - The ability to withstand any reasonable challenge related to the veracity, integrity, or quality of the logical, technical, or scientific approach taken in a decision-making process.

Design - Specifications, drawings, design criteria, and performance requirements. Also, the result of deliberate planning, analysis, mathematical manipulations, and design processes (such as experimental design and sampling design).

Detection Limit - A measure of the capability of an analytical method to distinguish samples that do not contain a specific analyte from samples that contain low concentrations of the analyte. The lowest concentration or amount of the target analyte that can be determined to be different from zero by a single measurement at a stated level of probability. Detection limits are analyte- and matrix-specific and may be laboratory-dependent.

Discrete Sample - A sample that represents a single location or short time interval. A discrete sample can be composed of more than one increment. The term has the same meaning as "individual sample."

Distribution - A probability function (density function, mass function, or distribution function) used to describe a set of observations (*statistical sample*) or a population from which the observations are generated.

Duplicate Samples - Two samples taken from and *representative* of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess the *variance* of the total method, including sampling and analysis. See also *Colocated Sample* and *Field Duplicate Samples*.

Dynamic Work Plan - A work plan that allows the project team to make decisions in the field about how subsequent site activities will progress (for example, by use field analytical methods that provide near real-time sample analysis results). Dynamic work plans provide the strategy for how dynamic field activities will take place. As such, they document a flexible, adaptive sampling and analytical strategy. (Adopted from EPA Superfund web site at http://www.epa.gov/superfund/programs/dfa/dynwork.htm).

Environmental Conditions - The description of a physical medium (e.g., air, water, soil, sediment) or a biological system expressed in terms of its physical, chemical, radiological, or biological characteristics.

Environmental Data - Any measurements or information that describe environmental processes, location, or conditions; ecological or health effects and consequences; or the performance of environmental technology. For EPA, environmental data include information collected directly from measurements, produced from models, and compiled from other sources, such as data bases or the scientific literature.

Environmental Monitoring - The process of measuring or collecting environmental data for evaluating a change in the environment (e.g., ground-water monitoring).

Environmental Processes - Manufactured or natural processes that produce discharges to or that impact the ambient environment.

Equipment Blank - See *Rinsate*.

Estimate - A characteristic from the sample from which inferences about population *parameters* can be made.

Evaluation - See *validation*.

Evidentiary Records - Records identified as part of litigation and subject to restricted access, custody, use, and disposal.

False Negative (False Acceptance) Decision Error (β) - A false negative decision error occurs when the decision maker does not reject the null *hypothesis* when the null hypothesis actually is false. In statistical terminology, a false negative decision error also is called a Type II error. The measure of the size of the error is expressed as a probability, usually referred to as "beta" (β). This probability also is called the complement of power (where "power" is expressed as $(1-\beta)$).

False Positive (False Rejection) Decision Error (α) - A false positive decision error occurs when a decision maker rejects the null *hypothesis* when the null hypothesis is true. In statistical terminology, a false positive decision error also is called a Type I error. The measure of the size of the error is expressed as a probability, usually referred to as "alpha" (α), the "level of significance," or "size of the critical region."

Field Blank - A *blank* used to provide information about contaminants that may be introduced during sample collection, storage, and transport. The clean sample is carried to the sampling site, exposed to sampling conditions, returned to the laboratory, and treated as an environmental sample.

Field Duplicates - Independent samples that are collected as close as possible to the same point in space and time. Two separate samples are taken from the same source, stored in separate containers, and analyzed independently. These duplicates are useful in documenting the *precision* of the sampling process (from Chapter One, SW-846, July 1992).

Field (matrix) Spike - A sample prepared at the sampling point (i.e., in the field) by adding a known mass of the target analyte to a specified amount of the sample. Field matrix spikes are used, for example, to determine the effect of the sample preservation, shipment, storage, matrix, and preparation on analyte recovery efficiency (the analytical *bias*).

Field Split Samples - Two or more *representative* portions taken from the same sample and usually submitted for analysis to different laboratories to estimate interlaboratory *precision*.

Fundamental Error - The fundamental error results when discrete units of the material to be sampled have different compositions with respect to the property of interest. The error is referred to as “fundamental” because it is an incompressible minimum sampling error that depends on the mass, composition, shape, fragment size distribution, and liberation factor of the material and is not affected by homogenization or mixing. The fundamental error is the only error that remains when the sampling operation is “perfect,” i.e., when all parts of the sample are obtained in a probabilistic manner and each part is independent. The fundamental error is never zero (unless the population is completely homogeneous or the entire population is submitted for exhaustive analysis) and it never “cancels out.” It can be reduced by taking larger physical samples and by using particle-size reduction steps in preparing the analytical sample.

Geostatistics - A branch of statistics, originating in the mining industry and greatly developed in the 1950s, that assesses the spatial correlation among samples and incorporates this information into the estimates of population *parameters*.

Goodness-of-Fit Test - In general, the level of agreement between an observed set of values and a set wholly or partly derived from a model of the data.

Grab Sample - A one-time sample taken from any part of the waste (62 FR 91, page 26047, May 12, 1997).

Graded Approach - The process of basing the level of application of managerial controls applied to an item or work according to the intended use of the results and the degree of confidence needed in the quality of the results. (See also *Data Quality Objectives Process*.)

Gray Region - A range of values of the population *parameter* of interest (such as mean contaminant concentration) within which the consequences of making a *decision error* are relatively minor. The gray region is bounded on one side by the *action level*. The width of the gray region is denoted by Δ in this guidance.

Guidance - A suggested practice that is not mandatory, but rather intended as an aid or example in complying with a standard or requirement.

Guideline - A suggested practice that is nonmandatory in programs intended to comply with a standard.

Hazardous Waste - Any waste material that satisfies the definition of "hazardous waste" as given in 40 CFR Part 261, "Identification and Listing of Hazardous Waste."

Heterogeneity - The condition of the population under which items of the population are not identical with respect to the *parameter* of interest (ASTM D 6233-98). (See Section 6.2.1).

Holding Time - The period of time a sample may be stored prior to its required analysis. While exceeding the holding time does not necessarily negate the veracity of analytical results, it causes the qualifying or “flagging” of any data not meeting all of the specified acceptance criteria.

Homogeneity - The condition of the population under which all items of the population are identical with respect to the *parameter* of interest (ASTM D 6233-98). The condition of a population or lot in which the elements of that population or lot are identical; it is an inaccessible limit and depends on the “scale” of the elements.

Hot Spots - Strata that contain high concentrations of the *characteristic* of interest and are relatively small in size when compared with the total size of the materials being sampled (ASTM D 6009-96).

Hypothesis - A tentative assumption made to draw out and test its logical or empirical consequences. In hypothesis testing, the hypothesis is labeled "null" (for the baseline condition) or "alternative," depending on the decision maker's concerns for making a *decision error*. The baseline condition is retained until overwhelming evidence indicates that the baseline condition is false. See also *baseline condition*.

Identification Error - The misidentification of an analyte. In this error type, the contaminant of concern is unidentified and the measured concentration is incorrectly assigned to another contaminant.

Increment - A group of particles extracted from a batch of material in a single operation of the sampling device. It is important to make a distinction between an increment and a sample that is obtained by the reunion of several increments (from Pitard 1989).

Individual Sample - See *Discrete Sample*.

Inspection - The examination or measurement of an item or activity to verify *conformance* to specific requirements.

Internal Standard - A standard added to a test portion of a sample in a known amount and carried through the entire determination procedure as a reference for calibrating and assessing the *precision* and *bias* of the applied analytical method.

Item - An all-inclusive term used in place of the following: appurtenance, facility, sample, assembly, *component*, equipment, material, module, part, product, structure, subassembly, subsystem, system, unit, documented concepts, or data.

Laboratory Split Samples - Two or more *representative* portions taken from the same sample for laboratory analysis. Often analyzed by different laboratories to estimate the interlaboratory *precision* or variability and the data *comparability*.

Limit of Quantitation - The minimum concentration of an analyte or category of analytes in a specific matrix that can be identified and quantified above the method detection limit and within specified limits of *precision* and *bias* during routine analytical operating conditions.

Limits on Decision Errors - The tolerable maximum decision error probabilities established by the decision maker. Potential economic, health, ecological, political, and social consequences of decision errors should be considered when setting the limits.

Matrix Spike - A sample prepared by adding a known mass of a target analyte to a specified amount of sample matrix for which an independent estimate of the target analyte concentration is available. Spiked samples are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Mean (arithmetic) ($\bar{x}$) - The sum of all the values of a set of measurements divided by the number of values in the set; a measure of central tendency.

Mean Square Error (MSE) - A statistical term equivalent to the *variance* added to the square of the *bias*. An overall measure of the representativeness of a sample.

Measurement Error - The difference between the true or actual state and that which is reported from measurements.

Median - The middle value for an ordered set of n values. Represented by the central value when n is odd or by the average of the two most central values when n is even. The median is the 50th percentile.

Medium - A substance (e.g., air, water, soil) that serves as a carrier of the analytes of interest.

Method - A body of procedures and techniques for performing an activity (e.g., sampling, chemical analysis, quantification) systematically presented in the order in which they are to be executed.

Method Blank - A *blank* prepared to represent the sample matrix as closely as possible and analyzed exactly like the *calibration* standards, samples, and QC samples. Results of method blanks provide an estimate of the within-batch variability of the blank response and an indication of *bias* introduced by the analytical procedure.

Natural Variability - The variability that is inherent or natural to the media, objects, or subjects being studied.

Nonparametric - A term describing statistical methods that do not assume a particular population probability distribution, and are therefore valid for data from any population with any probability distribution, which can remain unknown (Conover 1999).

Null Hypothesis - See *Hypothesis*.

Observation - (1) An *assessment* conclusion that identifies a condition (either positive or negative) that does not represent a significant impact on an item or activity. An observation may identify a condition that has not yet caused a degradation of quality. (2) A datum.

Outlier - An observation that is shown to have a low probability of belonging to a specified data population.

Parameter - A quantity, usually unknown, such as a mean or a standard deviation characterizing a population. Commonly misused for "variable," "*characteristic*," or "property."

Participant - When used in the context of environmental programs, an organization, group, or individual that takes part in the planning and design process and provides special knowledge or skills to enable the planning and design process to meet its objective.

Percent Relative Standard Deviation (%RSD) - The quantity, 100(RSD)%.

Percentile - The specific value of a distribution that divides the distribution such that *p* percent of the distribution is equal to or below that value. For example, if we say "the 95th percentile is X," then it means that 95 percent of the values in the *statistical sample* are less than or equal to X.

Planning Team - The group of people that will carry out the DQO Process. Members include the decision maker (senior manager), representatives of other data users, senior program and technical staff, someone with statistical expertise, and a QA/QC advisor (such as a QA Manager).

Population -The total collection of objects, media, or people to be studied and from which a sample is to be drawn. The totality of items or units under consideration (ASTM D 5956-96).

Precision - A measure of mutual agreement among individual measurements of the same property, usually under prescribed similar conditions, expressed generally in terms of the sample standard deviation. See also the definition for *precision* in Chapter One, SW-846.

Probabilistic Sample - See *statistical sample*.

Process - A set of interrelated resources and activities that transforms inputs into outputs. Examples of processes include analysis, design, data collection, operation, fabrication, and calculation.

Qualified Data - Any data that have been modified or adjusted as part of statistical or mathematical evaluation, data *validation*, or data verification operations.

Quality - The totality of features and characteristics of a product (including data) or service that bears on its ability to meet the stated or implied needs and expectations of the user (i.e., fitness for use).

Quality Assurance (QA) - An integrated system of management activities involving planning, implementation, *assessment*, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Assurance Manager - The individual designated as the principal manager within the organization having management oversight and responsibilities for planning, coordinating, and assessing the effectiveness of the quality system for the organization.

Quality Assurance Project Plan (QAPP) - A formal document describing, in comprehensive detail, the necessary QA, QC, and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria.

Quality Control (QC) - The overall system of technical activities that measures the attributes and performance (quality characteristics) of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer. Operational techniques and activities that are used to fulfill requirements for quality. The system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against "out-of-control" conditions and ensuring the results are of acceptable quality.

Quality Control (QC) Sample - An uncontaminated sample matrix spiked with known amounts of analytes from a source independent of the *calibration* standards. Generally used to establish intralaboratory or analyst-specific *precision* and *bias* or to assess the performance of all or a portion of the measurement system.

Quality Management - That aspect of the overall management system of the organization that determines and implements the quality policy. Quality management includes strategic planning, allocation of resources, and other systematic activities (e.g., planning, implementation, and *assessment*) pertaining to the quality system.

Quality Management Plan - A formal document that describes the quality system in terms of the organization's structure, the functional responsibilities of management and staff, the lines of authority, and the required interfaces for those planning, implementing, and assessing all activities conducted.

Quality System - A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

Random Error - The chance variation encountered in all measurement work, characterized by the random occurrence of deviations from the mean value.

Range - The numerical difference between the minimum and maximum of a set of values.

Relative Standard Deviation - See *Coefficient of Variation*.

Remediation - The process of reducing the concentration of a contaminant (or contaminants) in air, water, or soil media to a level that poses an acceptable risk to human health.

Repeatability - The degree of agreement between independent test results produced by the same analyst using the same test method and equipment on random aliquots of the same sample within a short time period; that is, within-rum precision of a method or set of measurements.

Reporting Limit - The lowest concentration or amount of the target analyte required to be reported from a data collection project. Reporting limits are generally greater than detection limits and usually are not associated with a probability level.

Representative Sample - RCRA regulations define a representative sample as “a sample of a universe or whole (e.g., waste pile, lagoon, ground water) which can be expected to exhibit the average properties of the universe or whole" (40 CFR § 260.10).

Representativeness - A measure of the degree to which data accurately and precisely represent a *characteristic* of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

Reproducible - The condition under which there is no statistically significant difference in the results of measurements of the same sample made at different laboratories.

Reproducibility - The degree of agreement between independent test results produced by the same method or set of measurements for very similar, but not identical, conditions (e.g., at different times, by different technicians, using different glassware, laboratories, or samples); that is, the between-run precision of a method or set of measurements.

Requirement - A formal statement of a need and the expected manner in which it is to be met.

Rinsate (Equipment Rinsate) - A sample of analyte-free medium (such as HPLC-grade water for organics or reagent-grade deionized or distilled water for inorganics) which has been used to rinse the sampling equipment. It is collected after completion of decontamination and prior to sampling. This *blank* is useful in documenting the adequate decontamination of sampling equipment (modified from Chapter One, SW-846).

Sample - A portion of material that is taken from a larger quantity for the purpose of estimating the properties or the composition of the larger quantity (ASTM D 6233-98).

Sample Support - See *Support*.

Sampling - The process of obtaining *representative* samples and/or measurements of a population or subset of a population.

Sampling Design Error - The error due to observing only a limited number of the total possible values that make up the population being studied. It should be distinguished from: errors due to imperfect selection; *bias* in response; and errors of observation, measurement, or recording, etc.

Scientific Method - The principles and processes regarded as necessary for scientific investigation, including rules for concept or *hypothesis* formulation, conduct of experiments, and validation of hypotheses by analysis of observations.

Sensitivity - The capability of a method or instrument to discriminate between measurement responses representing different levels of a variable of interest (i.e., the slope of the calibration).

Set of Samples - More than one individual sample.

Split Samples - Two or more *representative* portions taken from one sample and often analyzed by different analysts or laboratories as a type of QC sample used to assess analytical variability and *comparability*.

Standard Deviation - A measure of the dispersion or imprecision of a sample or population distribution expressed as the positive square root of the *variance* and that has the same unit of measurement as the mean. See *variance*.

Standard Operating Procedure (SOP) - A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps and that is officially approved (usually by the quality assurance officer) as the method for performing certain routine or repetitive tasks.

Statistic - A function of the sample measurements; e.g., the sample mean or standard deviation. A statistic usually, but not necessarily, serves as an estimate of a population *parameter*. A summary value calculated from a sample of observations.

Statistical Sample - A set of samples or measurements selected by probabilistic means (i.e., by using some form of randomness). Also known as a *probabilistic sample*.

Statistical Test - Any statistical method that is used to determine the acceptance or rejection of a hyothesis.

Stratum - A subgroup of a population separated in space or time, or both, from the remainder of the population and being internally consistent with respect to a target constituent or property of interest and different from adjacent portions of the population (ASTM D 5956-96).

Subsample - A portion of material taken from a larger quantity for the purpose of estimating properties or the composition of the whole sample (ASTM D 4547-98).

Support - The physical volume or mass, orientation, and shape of a sample, subsample, or decision unit.

Surrogate Spike or Analyte - A pure substance with properties that mimic the analyte of interest. It is unlikely to be found in environmental samples and is added to them to establish that the analytical method has been performed properly.

Technical Review - A documented critical review of work that has been performed within the state of the art. The review is accomplished by one or more qualified reviewers who are independent of those who performed the work, but are collectively equivalent in technical expertise to those who performed the original work. The review is an indepth analysis and evaluation of documents, activities, material, data, or items that require technical verification or *validation* for applicability, correctness, adequacy, *completeness*, and assurance that established requirements are satisfied.

Total Study Error - The combination of sampling design error and measurement error.

Traceability - The ability to trace the history, application, or location of an entity by means of recorded identifications. In a *calibration* sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the project's quality.

Trip Blank - A clean sample of a matrix that is taken to the sampling site and transported to the laboratory for analysis without having been exposed to sampling procedures. A trip blank is used to document contamination attributable to shipping and field handling procedures. This type of *blank* is useful in documenting contamination of volatile organics samples.

True - Being in accord with the actual state of affairs.

Type I Error (α) - A Type I error occurs when a decision maker rejects the null *hypothesis* when it is actually true. See also *False Positive Decision Error*.

Type II Error (β) - A Type II error occurs when the decision maker fails to reject the null *hypothesis* when it is actually false. See also *False Negative Decision Error*.

User - When used in the context of environmental programs, an organization, group, or individual that utilizes the results or products from environmental programs. A user also may be the client for whom the results or products were collected or created.

Vadose Zone - In soil, the unsaturated zone, limited above by the ground surface and below by the saturated zone.

Validation - Confirmation by examination and provision of objective evidence that the particular requirements for a specific intended use are fulfilled. In design and development, *validation* concerns the process of examining a product or result to determine *conformance* to user needs.

Variable - The attribute of the environment that is indeterminant. A quantity which may take any one of a specified set of values.

Variance - A measure of the variability or dispersion in (1) a population (population variance, σ^2), or (2) a sample or set of subsamples (sample variance, s^2). The variance is the second moment of a frequency distribution taken about the arithmetic mean as the origin. For a normal distribution, it is the sum of the squared deviations of the (population or sample) member observation about the (population or sample) mean divided by the degrees of freedom (N for σ^2 , or $n-1$ for s^2).

Verification - Confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. In design and development, verification concerns the process of examining a result of a given activity to determine *conformance* to the stated requirements for that activity.

APPENDIX B

SUMMARY OF RCRA REGULATORY DRIVERS FOR CONDUCTING WASTE SAMPLING AND ANALYSIS

Through RCRA, Congress provided EPA with the framework to develop regulatory programs for the management of solid and hazardous waste. The provisions of RCRA Subtitle C establish the criteria for identifying hazardous waste and managing it from its point of generation to ultimate disposal. EPA's regulations set out in 40 CFR Parts 260 to 279 are the primary reference for information on the hazardous waste program. These regulations include provisions for waste sampling and testing and environmental monitoring. Some of these RCRA regulations require sampling and analysis, while others do not specify requirements and allow sampling and analysis to be performed at the discretion of the waste handler or as specified in individual facility permits.

Table B-1 provides a comprehensive listing of the regulatory citations, the applicable RCRA standards, requirements for demonstrating attainment or compliance with the standards, and relevant USEPA guidance documents. The table is divided into three major sections addressing regulations for (1) hazardous waste identification, (2) land disposal restrictions, and (3) other programs. The table is meant to be used as a general reference guide. Consult the latest 40 CFR, related *Federal Register* notices, and EPA's World Wide Web site (www.epa.gov) for new or revised regulations and further clarification and definitive articulation of requirements. In addition, because some states have requirements that differ from EPA regulations and guidance, we recommend that you consult with a representative from your State if your State is authorized to implement the regulation.

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers for the Hazardous Waste Identification Program			
§261.3(a)(2)(v) - Used oil rebuttable presumption (see also Part 279, Subpart B and the Part 279 standards for generators, transporters, processors, re-refiners, and burners.)	Used oil that contains more than 1,000 parts per million (ppm) of total halogens is presumed to have been mixed with a regulated halogenated hazardous waste (e.g., spent halogenated solvents), and is therefore subject to applicable hazardous waste regulations. The rebuttable presumption does not apply to metalworking oils and oils from refrigeration units, under some circumstances.	A person may rebut this presumption by demonstrating, through analysis or other documentation, that the used oil has not been mixed with halogenated hazardous waste. One way of doing this is to show that the used oil does not contain significant concentrations of halogenated hazardous constituents (50 FR 49176; November 29, 1985). If the presumption is successfully rebutted, then the used oil will be subject to the used oil management standards instead of the hazardous waste regulations.	*Hazardous Waste Management System; Identification and Listing of Hazardous Waste; Recycled Used Oil Management Standards*, 57 FR 41566; September 10, 1992 *Part 279 Requirements: Used Oil Management Standards*, EPA530-H-98-001
§261.3(c)(2)(ii)(C) - Generic exclusion levels for K061, K062, and F006 nonwastewater HTMR residues	To be excluded from the definition of hazardous waste, residues must meet the generic exclusion levels specified at §261.3(c)(2)(ii)(C)(*1*) and exhibit no characteristics of hazardous waste.	Testing requirements must be incorporated in a facility's waste analysis plan or a generator's self-implementing waste analysis plan. At a minimum, composite samples of residues must be collected and analyzed quarterly and/or when the process or operation generating the waste changes. Claimant has the burden of proving by clear and convincing evidence that the material meets all of the exclusion requirements.	*Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers for the Hazardous Waste Identification Program (continued)			
§261.21- Characteristic of Ignitability	A solid waste exhibits the characteristic of ignitability if a representative sample of the waste is: (1) A liquid having a flashpoint of less than 140 degrees Fahrenheit (60 degrees Centigrade); (2) A non-liquid which causes fire through friction, absorption of moisture, or spontaneous chemical changes and, when ignited, burns so vigorously and persistently it creates a hazard; (3) An ignitable compressed gas; or (4) An oxidizer. (Aqueous solutions with alcohol content less than 24% are not regulated.)	If a representative sample of the waste exhibits the characteristic, then the waste exhibits the characteristic. Appendix I of 40 CFR Part 261 contains references to representative sampling methods; however a person may employ an alternative method without formally demonstrating equivalency. Also, for those methods specifically prescribed by regulation, the generator can petition the Agency for the use of an alternative method (see 40 CFR 260.21).	See Chapters Seven and Eight in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a)
§261.22 - Characteristic of Corrosivity	A solid waste exhibits the characteristic of corrosivity if a representative sample of the waste is: (1) Aqueous, with a pH less than or equal to 2, or greater than or equal to 12.5; or (2) Liquid and corrodes steel at a rate greater than 6.35 mm per year when applying a National Association of Corrosion Engineers Standard Test Method.	If a representative sample of the waste exhibits the characteristic, then the waste exhibits the characteristic. Appendix I of 40 CFR Part 261 contains references to representative sampling methods; however a person may employ an alternative method without formally demonstrating equivalency. Also, for those methods specifically prescribed by regulation, the generator can petition the Agency for the use of an alternative method (see 40 CFR 260.21).	See Chapters Seven and Eight in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers for the Hazardous Waste Identification Program (continued)			
§261.23 - Characteristic of Reactivity	A solid waste exhibits the characteristic of reactivity if a representative sample of the waste: (1) Is normally unstable and readily undergoes violent change; (2) Reacts violently with water; (3) Forms potentially explosive mixtures with water; (4) Generates toxic gases, vapors, or fumes when mixed with water; (5) Is a cyanide or sulfide-bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors, or fumes; (6) Is capable of detonation or explosion if subjected to a strong initiating source or if heated under confinement; (7) Is readily capable of detonation or explosive decomposition or reaction at standard temperature and pressure; or (8) Is a forbidden explosive as defined by DOT.	EPA relies on these narrative criterion to define reactive wastes. Waste handlers should use their knowledge to determine if a waste is sufficiently reactive to be regulated. Also, for those methods specifically prescribed by regulation, the generator can petition the Agency for the use of an alternative method (see 40 CFR 260.21).	EPA currently relies on narrative standards to define reactive wastes, and withdrew interim guidance related to sulfide and cyanide levels (see a Memorandum entitled, Withdrawal of Cyanide and Sulfide Reactivity Guidance" from David Bussard and Barnes Johnson to Diana Love, dated April 21, 1998).
§ 261.24 - Toxicity Characteristic	A solid waste exhibits the characteristic of toxicity if the extract of a representative sample of the waste contains any of the contaminants listed in Table 1 in 261.24, at or above the specified regulatory levels. The extract should be obtained through use of the Toxicity Characteristic Leaching Procedure (TCLP). If the waste contains less than .5 percent filterable solids, the waste itself, after filtering, is considered to be the extract.	Appendix I of 40 CFR Part 261 contains references to representative sampling methods; however, a person may employ an alternative method without formally demonstrating equivalency.	See Chapters Seven and Eight in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers for the Hazardous Waste Identification Program (continued)			
§261.38(c)(8)(iii)(A) - Exclusion of Comparable Fuels from the Definition of Solid and Hazardous Waste	For each waste for which an exclusion is claimed, the generator of the hazardous waste must test for all of the constituents on Appendix VIII to part 261, except those that the generator determines, based on testing or knowledge, should not be present in the waste. The generator is required to document the basis for each determination that a constituent should not be present.	For waste to be eligible for exclusion, a generator must demonstrate that "each constituent of concern is not present in the waste above the specification level at the 95% upper confidence limit around the mean."	See the final rule from June 19,1998 (63 *FR* 33781) For further information on the comparable fuels exclusion, see the following web site: http://www.epa.gov/combustion/fastrack/
Part 261- Appendix I - Representative Sampling Methods	Provides sampling protocols for obtaining a representative sample.	For the purposes of Subpart C, a sample obtained using Appendix I sampling methods will be considered representative. The Appendix I methods, however, are not formally adopted (see comment at §261.20(c)).	*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a) ASTM Standards

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers for the Land Disposal Restriction Program			
§268.6(b)(1) - Petitions to Allow Land Disposal of a Waste Prohibited Under Subpart C of Part 268 (No-Migration Petition)	The demonstration must meet the following criteria: (1) All waste and environmental sampling, test, and analysis data must be accurate and reproducible to the extent that state-of-the-art techniques allow; (2) All sampling, testing, and estimation techniques for chemical and physical properties of the waste and all environmental parameters must have been approved by the EPA Administrator.	• Waste analysis requirements will be specific to the petition. • Sampling methods are specified in the facility's Waste Analysis Plan.	*Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a) *Land Disposal Restrictions No Migration Variances; Proposed Rule.* Federal Register, August 11, 1992 (USEPA 1992)
§268.40 - Land Disposal Restriction (LDR) concentration-level standards	For total waste standards, all hazardous constituents in the waste or in the treatment residue must be at or below the values in the table at 268.40. For waste extract standards, the hazardous constituents in the extract of the waste or in the extract of the treatment residue must be at or below the values in the table at 268.40.	• Sampling methods are specified in the facility's Waste Analysis Plan. • Compliance with the standards for nonwastewater is measured by an analysis of grab samples. Compliance with wastewater standards is based on composite samples. No single sample may exceed the applicable standard.	*Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers for the Land Disposal Restriction Program (continued)			
§268.44 - Land Disposal Restriction Treatability Variance	If you are a generator or treatment facility whose wastes cannot be treated to achieve the established treatment standards, or for which treatment standards are not appropriate, you may petition EPA for a variance from the treatment standard. A treatment variance does not exempt your wastes from treatment, but rather establishes an alternative LDR treatment standard.	The application must demonstrate that the treatment standard for the waste in question is either “unachievable” or “inappropriate.”	Memorandum entitled “*Use of Site-Specific Land Disposal Restriction Treatability Variances Under 40 CFR 268.44(h) During Cleanups*” (Available from the RCRA Call Center or on EPA’s web site at http://www.epa.gov/epaoswer/hazwaste/ldr/tv-rule/guidmem.txt Variance Assistance Document: Land Disposal Restrictions Treatability Variances & Determinations of Equivalent Treatment (available from the RCRA Call Center or on EPA’s web site at http://www.epa.gov/epaoswer/hazwaste/ldr/guidance2.pdf
§268.49(c)(1) - Alternative LDR Treatment Standards for Contaminated Soil	All constituents subject to treatment must be treated as follows: (A) For non-metals, treatment must achieve 90 percent reduction in total constituent concentrations except where treatment results in concentrations less that 10 times the Universal Treatment Standard (UTS) at 268.48. (B) For metals, treatment must achieve 90 percent reduction in constituent concentrations as measured in TCLP leachate from the treated media or 90 percent reduction in total concentrations when a metal removal technology is used, except where treatment results in concentrations less that 10 times the UTS at 268.48.	Sampling methods are specified in the facility’s Waste Analysis Plan.	*Guidance on Demonstrating Compliance With the Land Disposal Restrictions (LDR) Alternative Soil Treatment Standards* (USEPA 2002) *Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers in Other RCRA Regulations			
§260.10 - Definitions	"Representative sample" means a sample of a universe or whole (e.g. waste pile, lagoon, ground water) which can be expected to exhibit the average properties of the universe or whole.	Representative samples may be required to measure compliance with various provisions within the RCRA regulations. See requirements specified in the applicable regulation or implementation guidance.	*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a)
Part 260 - Subpart C - Rulemaking Petitions	In the section for petitions to amend Part 261 to "delist" a hazardous waste, the petitioner must demonstrate that the waste does not meet any of the criteria under which the waste was listed as a hazardous waste (§260.22).	Demonstration samples must consist of enough representative samples, but in no case less than four samples, taken over a period of time sufficient to represent the variability or the uniformity of the waste.	*Petitions to Delist Hazardous Waste–A Guidance Manual*. 2nd ed. (USEPA 1993d) *Region 6 RCRA Delisting Program Guidance Manual for the Petitioner* (USEPA 1996d)
Part 262 - Subpart A - Purpose, Scope, and Applicability (including §262.11 - Hazardous Waste Determination)	Generators must make the following determinations if a secondary material is a solid waste: 1) whether the solid waste is excluded from regulation; 2) whether the waste is a listed waste; and 3) whether the waste is characteristic waste (§262.11)	Generators must document their waste determination and land disposal restriction determination.	*Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)
Part 262 - Subpart C - Pre-Transport Requirements	Under §262.34(a)(4), if generators are performing treatment within their accumulation units, they must comply with the waste analysis plan requirements of §268.7(a)(5).	Generators must develop a waste analysis plan (kept on-site for three years) which details the treatment they are performing to meet LDR treatment standards and the type of analysis they are performing to show completion of treatment.	*Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	***Applicable Standards***	***Requirements for Demonstrating Attainment of or Compliance With the Standards***	***Relevant USEPA Guidance***
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Part 264 - Subpart A - Purpose, Scope, and Applicability	§264.1(j)(2) - In an exemption established by the HWIR-media rulemaking, remediation waste can be exempt under circumstances that require chemical and physical analysis of a representative sample of the hazardous remediation waste to be managed at the site.	The analysis, at a minimum, must contain all the information needed to treat, store, or dispose of the waste according to Part 264 and Part 268. The waste analysis must be accurate and up-to-date.	See the final *Federal Register* notice from November 30, 1998 (63 *FR* 65873) For further documentation, see the following web site: http://www.epa.gov/epaoswer/hazwaste/id/hwirmdia.htm
Parts 264/265 - Subpart B - General Facility Standards	§264/265.13 - General waste analysis requirements specify: (a) Detailed chemical and physical analysis of a representative sample is required before an owner treats, stores, or disposes of any hazardous waste. Sampling method may be those under Part 261; and (b) Owner/operator must develop and follow a written waste-analysis plan.	All requirements are case-by-case and are determined in the facility permit.	*Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	***Applicable Standards***	***Requirements for Demonstrating Attainment of or Compliance With the Standards***	***Relevant USEPA Guidance***
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Part 264 - Subpart F - Groundwater Monitoring	Groundwater monitoring wells must be properly installed so that samples will yield representative results. All monitoring wells must be lined, or cased, in a manner that maintains the integrity of the monitoring well bore hole (§264.97(c)). Poorly installed wells may give false results. There are specific monitoring standards for all three sub-programs: • Detection Monitoring (§264.98); • Compliance Monitoring (§264.99); and • Corrective Action Program (§264.100). The Corrective Action Program is specific to the Groundwater Monitoring Program.	At a minimum, there must be procedures and techniques for sample collection, sample preservation and shipment, analytical procedures, and chain-of-custody control (§264.97(d)). Sampling and analytical methods must be appropriate for groundwater sampling and accurately measure the hazardous constituents being analyzed. The owner and operator must develop an appropriate sampling procedure and interval for each hazardous constituent identified in the facility's permit. The owner and operator may use an alternate procedure if approved by the RA. Requirements and procedures for obtaining and analyzing samples are detailed in the facility permit, usually in a Sampling and Analysis Plan.	*Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities (Interim Final Guidance)*. Office of Solid Waste (USEPA 1989b) *RCRA Ground-Water Monitoring: Draft Technical Guidance*. (USEPA 1992c) *Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities Addendum to Interim Final Guidance* (USEPA 1992b) *Methods for Evaluating the Attainment of Cleanup Standards. Volume 2: Ground Water* (USEPA. 1992i)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Part 265 - Subpart F - Ground-water Monitoring	To comply with Part 265, Subpart F, the owner/operator must install, operate, and maintain a ground-water monitoring system capable of representing the background groundwater quality and detecting any hazardous constituents that have migrated from the waste management area to the uppermost aquifer. Under Part 265, Subpart F, there are two types of groundwater monitoring programs: an indicator evaluation program designed to detect the presence of a release, and a ground-water quality assessment program that evaluates the nature and extent of contamination.	To determine existing ground-water conditions at an interim status facility, the owner and operator must install at least one well hydraulically upgradient from the waste management area. The well(s) must be able to accurately represent the background quality of ground water in the uppermost aquifer. The owner and operator must install at least three wells hydraulically downgradient at the limit of the waste management area, which are able to immediately detect any statistically significant evidence of a release. A separate monitoring system for each management unit is not required as long as the criteria in §265.91(a) are met and the system is able to detect any release at the edge of the waste management area.	*Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities (Interim Final Guidance)*. Office of Solid Waste (USEPA 1989b) *RCRA Ground-Water Monitoring: Draft Technical Guidance*. (USEPA 1992c) *Statistical Analysis of Ground-Water Monitoring Data at RCRA Facilities Addendum to Interim Final Guidance* (USEPA 1992b)
Part 264/265 - Subpart G - Closure and Post-Closure	The closure plan must include a detailed description of the steps for sampling and testing surrounding soils and criteria for determining the extent of decontamination required to satisfy the closure performance standards. (§264/265.112(b)(4))	All requirements are facility-specific and are set forth in the facility permit.	Closure/Postclosure Interim Status Standards (40 CFR 265, Subpart G): Standards Applicable to Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities Under RCRA, Subtitle C, Section 3004 *RCRA Guidance Manual for Subpart G Closure and Postclosure Care Standards and Subpart H Cost Estimating Requirements* (USEPA 1987)

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	***Applicable Standards***	***Requirements for Demonstrating Attainment of or Compliance With the Standards***	***Relevant USEPA Guidance***
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Part 264 - Subpart I - Use and Management of Containers	Spilled or leaked waste and accumulated precipitation must be removed from the sump or collection area in as timely a manner as is necessary to prevent overflow of the collection system (§264.175).	If the collected material is a hazardous waste under part 261 of this Chapter, it must be managed as a hazardous waste in accordance with all applicable requirements of parts 262 through 266 of the chapter. If the collected material is discharged through a point source to waters of the United States, it is subject to the requirements of section 402 of the Clean Water Act, as amended. Testing scope and requirements are site-specific and are set forth in the facility waste analysis plan.	*Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a) *Guidance for Permit Writers: Facilities Storing Hazardous Waste in Containers*, 11/2/82, PB88-105 689 *Model RCRA Permit for Hazardous Waste Management Facilities*, 9/15/88, EPA530-SW-90-049
Parts 264/265 - Subpart J - Tank Systems	Demonstrate the absence or presence of free liquids in the stored/treated waste using EPA Method 9095 (Paint Filter Liquid Tests) of SW-846 (§§264/265.196).	The Paint Filter Liquid Test is a positive or negative test.	*Method 9095* of *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a)

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40 CFR Citation and Description	***Applicable Standards***	***Requirements for Demonstrating Attainment of or Compliance With the Standards***	***Relevant USEPA Guidance***
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Part 264/265 - Subpart M - Land Treatment	To demonstrate adequate treatment (treatment demonstration), the permittee must perform testing, analytical, design, and operating requirements. (§264.272) Demonstration that food-chain crops can be grown on a treatment unit can include sample collection with criteria for sample selection, sample size, analytical methods, and statistical procedures. (§264/265.276) Owner/operator must collect pore-water samples and determine if there has been a statistically significant change over background using procedures specified in the permit. (§264/265.278) During post-closure period, owner may conduct pore-water and soil sampling to determine if there has been a statistically significant change in the concentration of hazardous constituents. (§264/265.280)	All requirements are facility-specific and are set forth in the facility permit.	See Chapters Twelve in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a) *Guidance Manual on Hazardous Waste Land Treatment Closure/Postclosure (40 CFR Part 265)*, 4/14/87, PB87-183 695 *Hazardous Waste Land Treatment*, 4/15/83, SW-874 *Permit Applicants' Guidance Manual for Hazardous Waste Land Treatment, Storage, and Disposal Facilities; Final Draft*, 5/15/84, EPA530-SW-84-004 *Permit Guidance Manual on Hazardous Waste Land Treatment Demonstrations*, 7/15/86, EPA530-SW-86-032 *Permit Guidance Manual on Unsaturated Zone Monitoring for Hazardous Waste Land Treatment Units*, 10/15/86, EPA530-SW-86-040

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40 CFR Citation and Description	***Applicable Standards***	***Requirements for Demonstrating Attainment of or Compliance With the Standards***	***Relevant USEPA Guidance***
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Part 264 - Subpart O - Incinerators	There are waste analysis requirements to verify that waste fed to the incinerator is within physical and chemical composition limits specified in the permit. (§§264/265.341) The owner/operator must conduct sampling and analysis of the waste and exhaust emissions to verify that the operating requirements established in the permit achieve the performance standards of §264.343 (§§264/265.347)	All requirements are facility-specific and are set forth in the facility permit.	See Chapter Thirteen in *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a)

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40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Corrective Action for Solid Waste Management Units	EPA includes corrective action in permits through the following statutory citations: Section 3008(h) - provides authority to require corrective action at interim status facilities Section 3004(u) - requires corrective action be addressed as a condition of a facility's Part B permit Section 3004(v) - provides authority to require corrective action for releases migrating beyond the facility boundary Section 3005(c)(3) - provides authority to include additional requirements in a facility's permit, including corrective action requirements Section 7003 - gives EPA authority to take action when contamination presents an imminent hazard to human health or the environment	Often the first activity in the corrective action process is the RCRA facility Assessment (RFA), which identifies potential and actual releases from solid waste management units (SWMUs) and make preliminary determinations about releases, the need for corrective action, and interim measures. Another activity in the corrective action process is the RCRA Facility Investigation (RFI), which takes place when a release has been identified and further investigation is necessary. The purpose of the RFI is to gather enough data to fully characterize the nature, extent, and rate of migration of contaminants to determine the appropriate response action. Once the implementing agency has selected a remedy, the facility enters the Corrective Measures Implementation (CMI) phase, in which the owner and operator of the facility implements the chosen remedy. Corrective action may include various sampling and monitoring requirements.	There is a substantial body of guidance and publications related to RCRA corrective action. See the following link for further information: http://www.epa.gov/epaoswer/hazwaste/ca/resource.htm
§264.552 - Corrective Action Management Units	There are ground-water monitoring, closure, and post-closure requirements for CAMUs.	All requirements are case-by-case and are determined in the facility permit.	There are numerous guidance documents available. See the following link for further information: http://www.epa.gov/epaoswer/hazwaste/ca/resource.htm

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40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Parts 264/265 - Subpart AA - Air Emission Standards	The following types of units are subject to the Subpart AA process vent standards: • Units subject to the permitting standards of Part 270 (i.e., permitted or interim status) • Recycling units located at hazardous waste management facilities otherwise subject to the permitting standards of Part 270 (i.e., independent of the recycling unit, the facility has a RCRA permit or is in interim status) • Less than 90-day large quantity generator units.	Testing and statistical methods are specified in the regulations at §264.1034(b).	The primary source of guidance is the regulations. See also the final rulemakings that promulgated the regulations: June 21, 1990 (55 *FR* 25494) November 25, 1996 (62 *FR* 52641) June 13, 1997 (62 *FR* 32462)
Parts 264/265 - Subpart BB - Air Emission Standards	The following types of units are subject to the Subpart BB equipment leak standards: • Units subject to the permitting standards of Part 270 (i.e., permitted or interim status) • Recycling units located at hazardous waste management facilities otherwise subject to the permitting standards of Part 270 (i.e., independent of the recycling unit, the facility already has a RCRA permit or is in interim status) • Less than 90-day large quantity generator units	The standards specify the type and frequency of all inspection and monitoring activities required. These requirements vary depending on the piece of equipment at the facility. Testing and statistical methods are specified in the regulations at §264.1063(c).	The primary source of guidance is the regulations. See also the final rulemakings that promulgated the regulations: June 21, 1990 (55 *FR* 25494) June 13, 1997 (62 *FR* 32462)

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40 CFR Citation and Description	***Applicable Standards***	***Requirements for Demonstrating Attainment of or Compliance With the Standards***	***Relevant USEPA Guidance***
Waste Analysis Drivers in Other RCRA Regulations (continued)			
§266.112 - Regulation of Residues	A residue from the burning or processing of hazardous waste may be exempt from hazardous waste determination if the waste derived residue is either: substantially similar to normal residue or below specific health based levels for both metal and nonmetal constituents.	Concentrations must be determined based on analysis of one or more samples obtained over a 24-hour period. Multiple samples may be analyzed and composite samples may be used provided the sampling period does not exceed 24 hours. If more than one sample is analyzed to represent the 24-hour period, the concentration shall be the arithmetic mean of the concentrations in the samples.	The regulations under §266.112 have specific sampling and analysis requirements Part 266, Appendix IX
Part 270 - Subpart B - Permit Application, Hazardous Waste Permitting	Provides the corresponding permit requirement to the general requirements (including the requirement for a waste analysis plan) under §270.14. There are also unit-specific waste analysis, monitoring, and sampling requirements incinerators (§270.19) and boilers and industrial furnaces (§270.22). There are also specific requirements for dioxin listings handled in waste piles (§270.18) and landfills (§270.21).	The permittee must conduct appropriate sampling procedures, and retain results of all monitoring. All requirements are facility specific and are set forth in the permit and waste analysis plan.	*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a) *Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)
Part 270 - Subpart C - Conditions Applicable to All Permits	Under §270.30, there are specific requirements for monitoring and recordkeeping. Section270.31 requires monitoring to be detailed in the permit.	The permittee must conduct appropriate sampling procedures, and retain results of all monitoring. All requirements are facility specific and are set forth in the permit and waste analysis plan.	*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a) *Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)

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40 CFR Citation and Description	***Applicable Standards***	***Requirements for Demonstrating Attainment of or Compliance With the Standards***	***Relevant USEPA Guidance***
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Part 270 - Subpart F - Special Forms of Permits	Specifies sampling and monitoring requirements based on trial burns for incinerators (§270.62) and Boiler and Industrial Furnaces (§270.66).	Waste analysis and sampling requirements are site specific and set forth in each facility's waste analysis plan required under 264.13.	*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a) *Waste Analysis at Facilities That Generate, Treat, Store, and Dispose of Hazardous Wastes, a Guidance Manual*, EPA530-R-94-024 (USEPA 1994a)
Part 273 - Universal Wastes	Handlers and transporters of universal wastes must determine if any material resulting from a release is a hazardous waste. (§273.17(b) for small quantity handlers, §273.37(b) for large quantity handlers, and §273.54 for transporters of universal wastes) Also, if certain universal wastes are dismantled, such as batteries or thermostats, in certain cases the resulting materials must be characterized for hazardous waste purposes. (§§273.13(a)(3) and (c)(3)(i))	Sampling and analysis requirements are identical to hazardous waste identification requirements.	*Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Updates I, II, IIA, IIB, III, and IIIA. SW-846*. (USEPA 1986a) *Universal Waste Final Rule, 60 FR 25492; May 11, 1995* Final rule adding Flourescent Lamps, *64 FR 36465; July 6, 1999*

Table B-1. Summary of Waste Analysis Drivers for Major RCRA Regulatory Program Areas

40 CFR Citation and Description	*Applicable Standards*	*Requirements for Demonstrating Attainment of or Compliance With the Standards*	*Relevant USEPA Guidance*
Waste Analysis Drivers in Other RCRA Regulations (continued)			
Part 279 - Standards for the Management of Used Oil	Specifies sampling and analysis procedures for owners or operators of used-oil processing and re-refining facilities.	Under §279.55, owners or operators of used oil processing and re-refining facilities must develop and follow a written analysis plan describing the procedures that will be used to comply with the analysis requirements of §279.53 and/or §279.72. The plan must be kept at the facility.	Sampling: Part 261, Appendix I *Hazardous Waste Management System; Identification and Listing of Hazardous Waste; Recycled Used Oil Management Standards*, 57 FR 41566, September 10, 1992 *Part 279 Requirements: Used Oil Management Standards*, EPA530-H-98-001

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APPENDIX C

STRATEGIES FOR SAMPLING HETEROGENEOUS WASTES

C.1 Introduction

“Heterogeneous wastes” include structures, demolition debris, waste-construction materials, containers (e.g., drums, tanks, and paint cans), solid waste from laboratories and manufacturing processes, and post-consumer wastes (e.g., electronics components, battery casings, and shredded automobiles) (USEPA and USDOE 1992). Heterogeneous wastes can pose challenges in the development and implementation of a sampling program due to the physical variety in size, shape, and composition of the material and the lack of tools and approaches for sampling heterogeneous waste. The application of conventional sampling approaches to heterogeneous waste is difficult and may not provide a representative sample.

To develop a sampling strategy for heterogeneous waste, it is first important to understand the scale, type, and magnitude of the heterogeneity. This appendix provides an overview of *large-scale heterogeneity* and provides some strategies that can be used to obtain samples of heterogeneous wastes. See also Section 6.2.1 for a description of other types of heterogeneity including short range (small-scale) heterogeneity (which includes distribution and constitution heterogeneity).

Additional guidance on sampling heterogeneous waste can be found in the following documents:

- *Characterizing Heterogeneous Wastes: Methods and Recommendations* (USEPA and USDOE 1992)
- *Standard Guide for Sampling Strategies for Heterogeneous Waste* (ASTM D 5956-96)
- *Pierre Gy's Sampling Theory and Sampling Practice: Heterogeneity, Sampling Correctness, and Statistical Process Control*. 2nd ed. (Chapter 21) (Pitard 1993), and
- *Geostatistical Error Management: Quantifying Uncertainty for Environmental Sampling and Mapping* (Myers 1997).

C.2 Types of Large-Scale Heterogeneity

The notion of heterogeneity is related to the scale of observation. An example given by Pitard (1993) and Myers (1997) is that of a pile of sand. From a distance of a few feet, a pile of sand appears to be uniform and homogeneous; however, at close range under magnification a pile of sand is heterogeneous. Substantial differences are found between the individual grains in their sizes, shapes, colors, densities, hardness, mineral composition, etc. For some materials, the differences between individual grains or items are not measurable or are not significant relative to the project objectives. In such a case, the degree of heterogeneity is so minor that for practical purposes the material can be considered homogeneous. The *Standard Guide for Sampling Strategies for Heterogeneous Waste* (ASTM D 5956-96) refers to this condition as "practical homogeneity," but recognizes that true homogeneity does not exist.

At a larger scale, such as an entire waste site, long-range (or large-scale) nonrandom heterogeneity is of interest. Large-scale heterogeneity reflects local trends and plays an important role in deciding whether to use a geostatistical appraisal to identify spatial patterns at the site, to use stratified sampling design to estimate a parameter (such as the overall mean), or to define the boundaries of the sampling problem so that it comprises two or more decision units that are each internally relatively homogeneous.

Items, particles, or phases within a waste or site can be distributed in various ways to create distinctly different types of heterogeneity. These types of heterogeneity include:

- **Random heterogeneity** – occurs when dissimilar items are randomly distributed throughout the population.
- **Non-random heterogeneity** – occurs when dissimilar items are nonrandomly distributed, resulting in the generation of strata. The term *strata* refers to subgroups of a population separated in space, in time, or by component from the remainder of the population. Strata are internally consistent with respect to a target constituent or a property of interest and are different from adjacent portions of the population.

The differences between items or particles that result in heterogeneity are due to differences in their composition or properties. One of these properties – particle size – deserves special consideration because significant differences in particle size are common and can complicate sampling due to the fundamental error. Fundamental error can be reduced only through particle-size reduction or the collection of sufficiently large samples. (Section 6 describes the impacts that fundamental error and particle size can have on sampling error.)

Figure C-1 depicts populations exhibiting the three types of heterogeneity described in ASTM D 5956-96 *Standard Guide for Sampling Strategies for Heterogeneous Waste*: (1) homogeneous, (2) randomly heterogeneous, (3) and nonrandomly heterogeneous populations. The drum-like populations portray different types of *spatial* distributions while the populations being discharged through the pipes represent different types of *temporal* distributions.

In the first scenario, very little spatial or temporal variation is found between the identical particles of the "homogeneous" population; however, in the second scenario, spatial and temporal variations are present due to the difference between the composition of the particles or items that make up the waste. ASTM D 5956-96 refers to this as a "randomly heterogeneous" population. In the third scenario, the overall composition of the particles or items remain the same as in the second scenario, but the two different components have segregated into distinct strata (e.g., due to gravity), with each strata being internally homogeneous. ASTM D 5956-96 refers to waste with this characteristic as "non-randomly heterogeneous."

C.3 Magnitude of Heterogeneity

The *magnitude* of heterogeneity is the degree to which there are differences in the characteristic of interest between fragments, particles, or volumes within the population. The magnitude of heterogeneity can range from that of a population whose items are so similar that it is practically

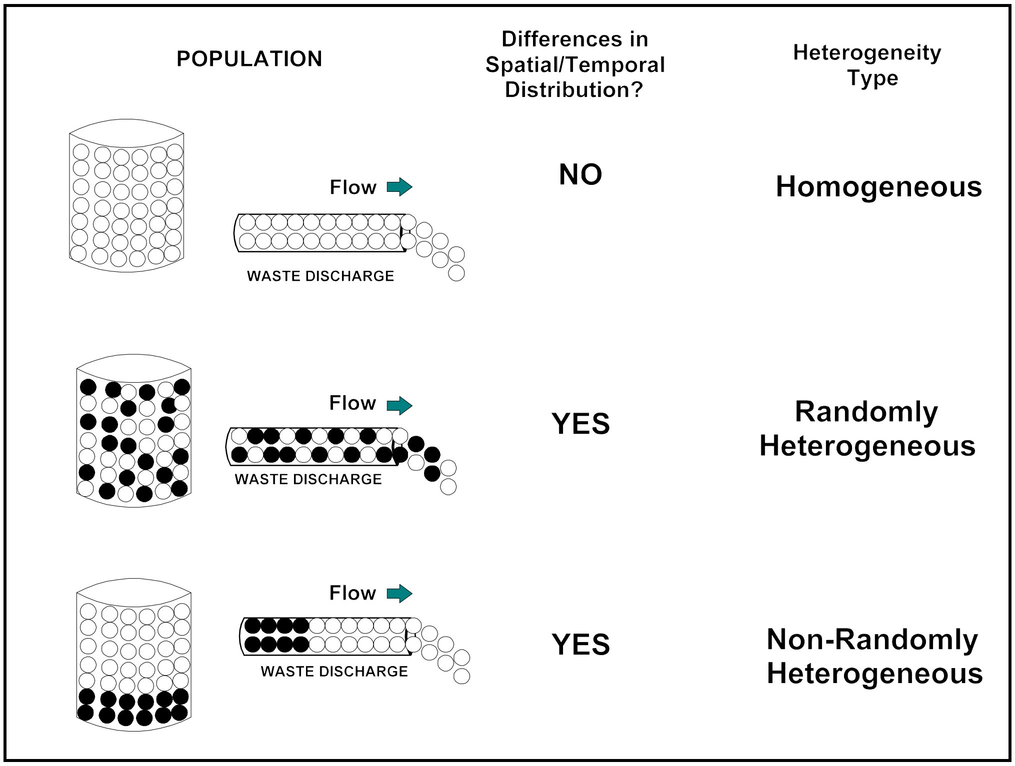

Figure C-1. Different types of spatial and temporal heterogeneity.

homogeneous to a population whose items are all dissimilar. Statistical measures of dispersion, the variance and standard deviation, are useful indicators of the degree of heterogeneity within a waste or waste site (assuming sampling error is not a significant contributor to the variance -- an optimistic assumption).

If the waste exhibits *nonrandom* heterogeneity and a *high magnitude* of heterogeneity, then consider segregating (e.g., at the point of generation) and managing the waste as two or more separate decision units (if physically possible and allowed by regulations). This approach will require prior knowledge (for example, from a pilot study) of the portions of the waste that fall into each specified category (such as hazardous debris and nonhazardous debris).

C.4 Sampling Designs for Heterogeneous Wastes

The choice of a sampling design to characterize heterogeneous waste will depend upon the regulatory objective of the study (e.g., waste identification or classification, site characterization, etc.), the data quality objectives, the type and magnitude of the heterogeneity, and practical considerations such as access to all portions of the waste, safety, and the availability of equipment suitable for obtaining and preparing samples.

As described in Section 5 of this document, there are two general categories of sampling designs: *probability* sampling design and *authoritative* (nonprobability) sampling designs. Probability sampling refers to sampling designs in which all parts of the waste or media under study have a known probability of being included in the sample. This assumption may be difficult to support when sampling highly heterogeneous materials such as construction debris.

All parts of a highly heterogeneous waste may not be accessible by conventional sampling tools, limiting the ability to introduce some form of randomness into the sampling design.

> **Random Heterogeneous Waste:** For random heterogeneous waste, a probability sampling design such as simple random or systematic sampling can be used. At least one of two sample collection strategies, however, also should be used to improve the precision (reproducibility) of the sampling design: (1) take very large individual samples (to increase the sample support), or (2) take many increments to form each individual sample (i.e., use composite sampling). The concept of sample support is described in Section 6.2.3. Composite sampling is discussed in Section 5.3.
>
> **Non-Random Heterogeneous Waste:** For non-random heterogeneous wastes, one of two strategies can be used to improve sampling: (1) If the objective is to estimate an *overall* population parameter (such as the mean), then stratified random sampling could be used. Stratified random sampling is discussed in detail in Section 5.2.2. (2) If the objective is to characterize each stratum separately (e.g., to classify the stratum as either a hazardous waste or a nonhazardous waste), then an appropriate approach is to separate or divert each stratum at its point of generation into discrete, nonoverlapping decision units and characterize and manage each decision unit separately (i.e., to avoid mixing or managing hazardous waste with nonhazardous waste).

If some form of stratified sampling is used, then one of three types of stratification must be considered. There are three types of stratification that can be used in sampling:

- stratification by space
- stratification by time
- stratification by component.

The choice of the type of stratification will depend on the type and magnitude of heterogeneity present in the population under consideration.

Figure C-2 depicts these different types of strata which are often generated by different processes or a significant variant of the same process. The different origins of the strata usually result in a different concentration or property distribution and different mean concentrations or average properties. While stratification over time or space is widely understood, stratification by component is less commonly employed. Some populations lack obvious spatial or temporal stratification yet display high levels of heterogeneity. If these populations contain easily identifiable components, such as bricks, gloves, pieces of wood or concrete, then it may be advantageous to consider the population as consisting of a number of component strata. An advantage of component stratification is that it can simplify the sampling and analytical process and allow a mechanism for making inferences to a highly stratified population. Component stratification shares many similarities with the gender or age stratification applied to demographic data by pollsters (i.e., the members of a given age bracket belonging to the same stratum regardless of where they reside). Component stratification is used by the mining industry to assay gold ore and other commodities where the analyte of interest is found in

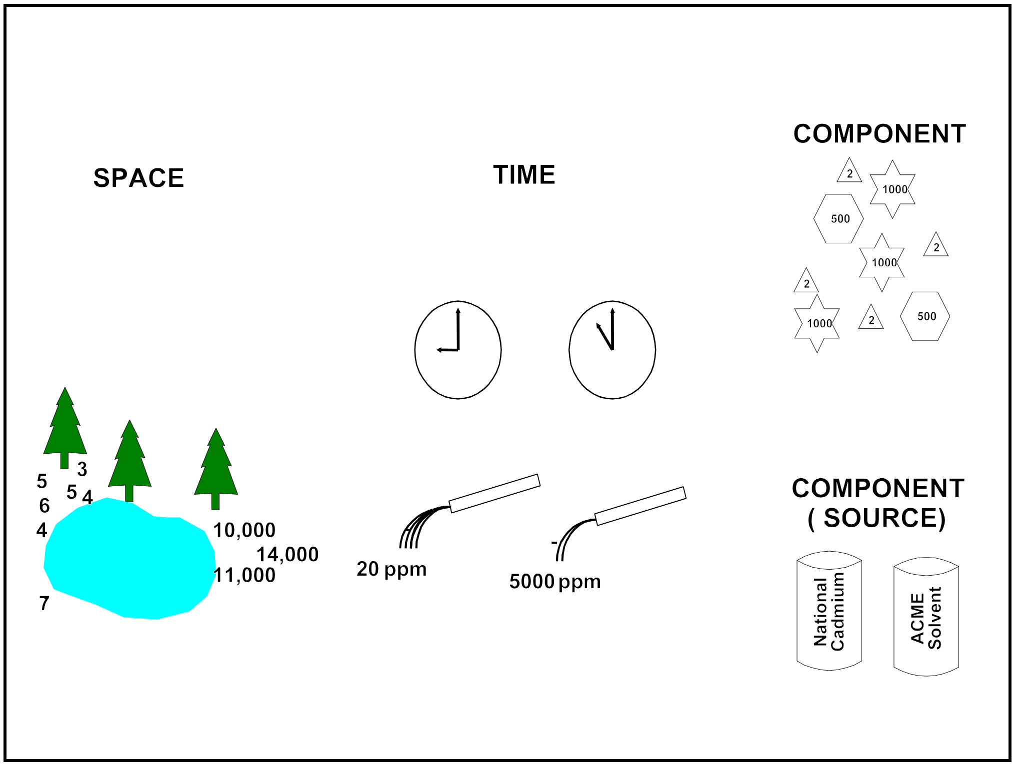

Figure C-2. Three different types of strata (from ASTM 5956-96)

discrete particles relative to a much greater mass of other materials.

Component stratification, although not commonly employed, is applicable to many waste streams, including the more difficult-to-characterize waste streams such as building debris. Additional guidance on stratification by component can be found in ASTM D 5956-96.

Table C-1 offers practical examples when wastes considered “non-randomly heterogeneous” might be good candidates for stratification across space, time, or by component.

The stratification approach can result in a more precise estimate of the mean compared to simple random sampling. However, keep in mind that greater precision is likely to be realized only if a waste exhibits substantial nonrandom chemical heterogeneity and stratification efficiently "divides" the waste into strata that exhibit maximum between-strata variability and minimum within-strata variability. If that does not occur, stratified random sampling can produce results that are less precise than in the case of simple random sampling; therefore, it is reasonable to employ stratified sampling only if the distribution of chemical contaminants in a waste is sufficiently known to allow an intelligent identification of the strata and at least two or three samples can be collected in each stratum.

Note that failure to recognize separate strata might lead one to conclude incorrectly, via a statistical test, that the underlying population is lognormal or some other right-skewed distribution.

Table C-1. Examples of Three Types of Stratification

Type of Stratification	*Example Scenario*
Stratification Across Space	A risk-based cleanup action requires a site owner to remove the top two feet of soil from a site. Prior to excavation, the waste hauler wants to know the average concentration of the constituent of concern in the soil to be removed. The top six inches of soil are known to be more highly contaminated than the remaining 18-inches of soil. Sampling of the soil might be carried out more efficiently by stratifying the soil into two subpopulations - the upper six-inch portion and the lower 18-inch portion.
Stratification Across Time	A waste discharge from a pipe varies across time. If the objective is to estimate the overall mean, then an appropriate sampling design might include stratification across time.
Stratification by Component	Construction debris covered with lead-based paint in the same structure with materials such as glass and unpainted wood could be sampled by components (lead-based paint debris, glass, and unpainted wood). This strategy is known as “stratification by component” (from ASTM D 5956-96).

C.5 Sampling Techniques for Heterogeneous Waste

Due to practical constraints, conventional sampling approaches may not be suitable for use in sampling of heterogeneous wastes. For example, sampling of contaminated debris can pose significant challenges due to the high degree of heterogeneity encountered. Methods used to sample contaminated structures and debris have included the following:

- Coring and cutting pieces of debris followed by crushing and grinding of the matrix (either in the field or within the laboratory) so the laboratory can handle the sample in a manner similar to a soil sample (Koski, et al 1991)

- Drilling of the matrix (e.g., with a hand held drill) followed by collection of the cuttings for analysis. This technique may require 20 to 50 drill sites in a local area to obtain a sufficient volume for an individual field sample (Koski, et al 1991)

- Grinding an entire structure via a tub grinder followed by conventional sampling approaches (AFCEE 1995).

ASTM has published a guide for sampling debris for lead-based paint (LBP) in ASTM E1908-97 *Standard Guide for Sample Selection of Debris Waste from a Building Renovation or Lead Abatement Project for Toxicity Characteristic Leaching Procedure (TCLP) testing for Leachable Lead (Pb)* .

Additional methods are described in Chapter Five, “Sample Acquisition,” of *Characterizing Heterogeneous Wastes: Methods and Recommendations* (USEPA and USDOE 1992) and in Rupp (1990).

APPENDIX D

A QUANTITATIVE APPROACH FOR CONTROLLING FUNDAMENTAL ERROR

This appendix provides a basic approach for determining the particle-size sample-weight relationship sufficient to achieve the fundamental error level specified in the DQOs. The procedure is based on that described by Pitard (1989, 1993), Gy (1998), and others; however, a number of simplifying assumptions have been made for ease of use. *The procedure described in this appendix is applicable to sampling of granular solid media (including soil) to be analyzed for nonvolatile constituents. It is not applicable to liquids, oily wastes, or debris.*

The mathematical derivation of the equation for the fundamental error is complex and beyond the scope of this guidance. Readers interested in the full documentation of the theory and underlying mathematics are encouraged to review Gy (1982) and Pitard (1993). Several authors have developed example calculations for the variance of the fundamental sampling error for a "typical" contaminated soil to demonstrate its practical application.[1] Examples found in Mason (1992), and Myers (1997) may be particularly useful.

The equation for the variance of the fundamental error is extremely practical for optimization of sampling protocols (Pitard 1993). In a relatively simple "rule of thumb" form, the equation for the variance of the fundamental error (s_{FE}^2) is estimated by

$$S_{FE}^2 = \frac{\mathrm{f}\lambda}{\mathrm{M_s}}\left(\frac{1}{\mathrm{a_{LC}}} - 2\right)d^3 \qquad \textbf{Equation D.1}$$

where

f = a dimensionless "shape" factor for the shape of particles in the material to be sampled where cubic = 1.0, sphere = 0.523, flakes = 0.1, and needles = 1 to 10
λ = average density (gm/cm^3) of the material
$\mathrm{M_s}$ = the sample weight (or mass of sample) in grams
$\mathrm{a_{LC}}$ = proportion of the sample with a particle size less than or equal to the particle size of interest
d = diameter of the largest fragment (or particle) in the waste, in centimeters.

Pitard's methodology suggests the particle size of interest should be set at 95 percent of the largest particle in the population (or "lot"), such that $\mathrm{a_{LC}}$ = 0.05. Equation D.1 then reduces to

$$s_{FE}^2 = \frac{\mathrm{f}\lambda}{\mathrm{M_a}} 18 d^3 \qquad \textbf{Equation D.2}$$

---

[1] It is important to note that discussion of the "variance of the fundamental error" refers to the relative variance, which is the ratio of the sample variance over square of the sample mean ($s^2 / \bar{x}^2$). The relative variance is useful for comparing results from different experiments.

The equation demonstrates that the variance of the fundamental error is directly proportional to the size of the largest particle and inversely proportional to the mass of the sample. To calculate the appropriate mass of the sample, Equation D.2 easily can be rearranged as

$$\mathrm{M_a} = \frac{\mathrm{f}\lambda}{(s_{FE})^2} 18d^3 \qquad \textbf{Equation D.3}$$

Pitard (1989, 1993) proposed a "Quick Safety Rule" for use in environmental sampling using the following input assumptions for Equation D.3:

f = 0.5 (dimensionless shape factor for a sphere)
λ = 2.7 (density of a waste in gm/cm^3)
s_{FE} = $\pm 5\%$ (standard deviation of the fundamental error).

By putting these assumed factors into Equation D.3, we get:

$$\mathrm{M_s} = \frac{0.5 \times 2.7}{(0.05)^2} 18d^3 \qquad \textbf{Equation D.4}$$

Pitard (1993) rounds up, to yield the relationship

$$\mathrm{M_s} \geq 10000d^3 \qquad \textbf{Equation D.5}$$

Alternatively, if we are willing to accept $s_{FE} = \pm 16\%$, Equation D.4 yields

$$\mathrm{M_s} \geq 1000d^3 \qquad \textbf{Equation D.6}$$

Equation D.4 was used to develop Table D-1 showing the maximum particle size that is allowed for a given sample mass with the standard deviation of the fundamental error (s_{FE}) prespecified at various levels (e.g., 5%, 10%, 16%, 20%, and 50%). A table such as this one can be used to estimate the optimal weight of field samples and the optimal weight of subsamples prepared within the laboratory. An alternative graphical method is presented by Pitard (1993) and Myers (1997).

An important feature of the fundamental error is that it does not "cancel out." On the contrary, the variance of the fundamental error adds together at each stage of subsampling. As pointed out by Myers (1997), the fundamental error can quickly accumulate and exceed 50%, 100%, 200%, or greater unless it is controlled through particle-size reduction. The variance of the fundamental error, s_{FE}^2 , calculated at each stage of subsampling and particle-size reduction must be added together at the end to derive the total s_{FE}^2 .

Table D-1. Maximum Allowable Particle Size (cm) for a Given Sample Mass for Selected Standard Deviations of the Fundamental Error

Sample Mass (g)	***Maximum Allowable Particle Size d (cm)***				
	S_{FE} = 5%	***S_{FE} = 10%***	***S_{FE} = 16%****	***S_{FE} = 20%***	***S_{FE} = 50%***
0.1	0.02	0.03	0.05	0.05	0.10
1	0.05	0.07	0.10	0.12	0.22
2	0.06	0.09	0.13	0.15	0.27
3	0.07	0.11	0.15	0.17	0.31
4	0.07	0.12	0.16	0.19	0.35
5	0.08	0.13	0.17	0.20	0.37
10	0.10	0.16	0.22	0.25	0.47
20	0.13	0.20	0.28	0.32	0.59
30	0.15	0.23	0.32	0.37	0.68
40	0.16	0.25	0.35	0.40	0.74
50	0.17	0.27	0.37	0.43	0.80
75	0.20	0.31	0.43	0.50	0.92
100	0.22	0.35	0.47	0.55	1.01
500	0.37	0.59	0.81	0.94	1.73
1000	0.47	0.74	1.02	1.18	2.17
5000	0.80	1.27	1.74	2.02	3.72

*A maximum standard deviation of the fundamental error of 16% has been recommended by Pitard (1993) and is included in this table as a point of reference only. Project-specific fundamental error rates should be set in the DQO Process.

Two important assumptions underlie the use of Table D-1:

1. The table is valid only if each and all steps of the sampling and subsampling minimize other sampling error through use of careful and correct sampling procedures
2. The table is valid only for wastes or soils with a shape factor (f) and density (λ) similar to that used to derive the table; otherwise, waste-specific tables or graphical methods (see Pitard 1993, Mason 1992, or Myers 1997) should be used.

Hypothetical Example

Suppose we have a waste that is a particulate solid to be analyzed for total metals. The laboratory requires an analytical sample of only 1 gram. The DQO planning team wants to maintain the total standard deviation of the fundamental error (s_{FE}) within $\pm 16\%$. The sample masses are determined at each stage of sampling and subsampling as follows:

Primary Stage: Based on prior inspection of the waste, it is known that 95 percent of the particles are 0.47 cm in diameter or less. Using Table D-1, we determine that a field sample of 1,000 grams (or 1 Kg) will generate a fundamental error s_{FE} not greater than $\pm 5\%$.

Secondary Stage: After shipment of the 1,000-gram sample to the laboratory, particle-size reduction to about 0.23 cm (2.36 mm or a No. 8 sieve) is performed, and a 30-gram subsample is taken. This step generates a fundamental error s_{FE} of $\pm 10\%$.

Final Stage: A 1-gram subsample is required for the analysis. Particle-size reduction to 0.07 cm or less (e.g., a No. 30 sieve) is performed, and a 1-g subsample is taken. This step generates a fundamental error s_{FE} of $\pm 10\%$.

The variance (s_{FE}^2) from each stage is then summed to determine the *total* variance of the fundamental error. As shown in Table D-2, the total standard deviation of the fundamental error is less than ±16 percent and the DQO is achieved.

Table D-2. Example Calculation of the Total Variance of the Fundamental Error

Sampling and Subsampling Stage	***Mass (grams)***	***d (cm)***	s_{FE}	s_{FE}^2
Primary Stage	1000	0.47	.05	.0025
Secondary Stage	30	0.23	.10	.01
Final Stage	1	0.07	.10	.01
Sum of the variances of the fundamental errors (s_{FE}^2)				s_{FE}^2 = 0.0225
Total standard deviation of the fundamental error (s_{FE}) (DQO = 16%)				s_{FE} = 0.15 or 15%

One final word of caution is provided regarding the use of the particle-size reduction and subsampling routine outlined above. The approach can reduce bias and improve precision of analyses for *total* constituent analyses, but the particle-size reduction steps may actually *introduce bias* when used in conjunction with some leaching tests. For example, the TCLP specifies a minimum sample mass of 100 grams (for nonvolatile extractions) and maximum particle size of 9.5 mm. While this combination would generate a s_{FE} of almost ±50 percent, excessive particle-size reduction below 9.5 mm to lower s_{FE} would *increase* the leachability of the material during the test due to the increased surface area-to-volume ratio of smaller particles. Therefore, it is important to remember that particle-size reduction to control fundamental error is beneficial when total constituent analyses are performed, but may introduce bias for some leaching tests. Furthermore, particle-size reduction below 9.5 mm is *not required* by Method 1311 (TCLP) (except during Step 7.1.4, “Determination of Appropriate Extraction Fluid”).

APPENDIX E

SAMPLING DEVICES

The key features of recommended sampling devices are summarized in this appendix. For each sampling device, information is provided in a uniform format that includes a brief description of the device and its use, advantages and limitations of the device, and a figure to indicate the general design of the device. Each summary also identifies sources of other guidance on each device, particularly any relevant ASTM standards.

Much of the information in this appendix was drawn from ASTM standards (see also Appendix J for summaries of ASTM standards). In particular, much of the information came from ASTM D 6232, *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*.

Devices not listed in this appendix or described elsewhere in this chapter also may be appropriate for use in RCRA-related sampling. For example, other more innovative or less common technologies may allow you to meet your performance goals and may be appropriate for your sampling effort. Therefore, we encourage and recommend the selection and use of sampling equipment based on a performance-based approach. In future revisions to this chapter, we will include new technologies, as appropriate.

Internet Resource

Information on sampling devices can be found on the Internet at the Federal Remediation Technologies Roundtable site at http://www.frtr.gov/. The Field Sampling and Analysis Technologies Matrix and accompanying Reference Guide are intended as an initial screening tool to provide users with an introduction to innovative site characterization technologies and to promote the use of potentially cost-effective methods for onsite monitoring and measurement.

This appendix is divided into subsections based on various categories of sampling technologies. The categories are based on those listed in ASTM D 6232. The equipment categories covered within this appendix are as follows:

E.1 Pumps and Siphons
E.2 Dredges
E.3 Discrete Depth Samplers
E.4 Push Coring Devices
E.5 Rotating Coring Devices
E.6 Liquid Profile Devices
E.7 Surface Sampling Devices

E.1 Pumps and Siphons

Pumps and siphons can be used to obtain samples of liquid wastes and ground water. For detailed guidance on the selection and use of pumps for sampling of ground water, see *RCRA Ground-Water Monitoring: Draft Technical Guidance* (USEPA 1992c).

In this section, you will find summaries for the following pumps or siphons:

E.1.1 Automatic Sampler

An automatic sampler (see Figure E-1) is a type of pumping device used to periodically collect samples. It is recommended for sampling surface water and point discharges. It can be used in waste-water collection systems and treatment plants and in stream sampling investigations. An automatic sampler designed for collection of samples for volatile organic analyses is available.

An automatic sampler typically uses peristaltic pumps as the sampling mechanism. It can be programmed to obtain samples at specified intervals or to obtain a continuous sample. It also can be programmed to collect time composite or flow proportional samples.

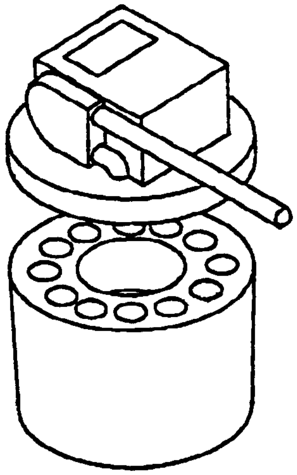

Figure E-1. Automatic sampler

Advantages

- Can provide either grab sample or composite samples over time.
- Operates unattended, and it can be programmed to sample variable volumes at variable times.

Limitations

- Requires power to operate (either AC or battery power).
- May be difficult to decontaminate.
- May not operate correctly when sampling liquid streams containing a high percentage of solids.
- Highly contaminated water or waste can degrade sampler components.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232.

E.1.2 Bladder Pump

The bladder pump is recommended for the sampling of surface water, ground water, and point discharges. It also can be used to sample other liquids in surface impoundments.

A bladder pump consists of a flexible membrane (bladder) enclosed by a rigid sample container and can be constructed of a variety of materials, such as neoprene, rubber, stainless steel, nitrile, etc. There are two types of bladder pumps - the squeeze type and the expanding type (see Figure E-2). The squeeze type has the bladder connected to the sample discharge line. The chamber between the bladder and the sampler body is connected to the gas line. The expanding type has the bladder connected to the gas line. In this type of bladder pump, the chamber between the bladder and the sampler body is connected to the sample discharge line.

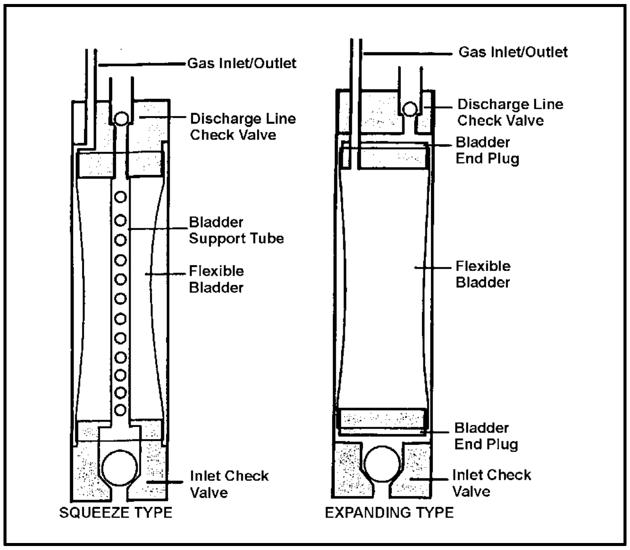

Figure E-2. Bladder pump

During sampling, water enters the sampler through a check valve at the bottom of the device. Compressed air or gas is then injected into the sampler. This causes the bladder to expand or compress depending on the type of bladder pump. The inlet valve closes and the contents of the sampler are forced through the top check valve into the discharge line. The top check valve prevents water from re-entering the sampler. By removing the pressure, the process is repeated to collect more sample. Automated sampling systems have been developed to control the time between pressurization cycles.

Advantages

- Is suitable for sampling liquids containing volatile compounds.
- Can collect samples up to a depth of 60 m (200 ft.) (ASTM D 6232).

Limitations

- Operation requires large volumes of compressed air or gas and a controller.
- Requires a power source.
- Can be heavy and difficult to operate.
- Decontamination can be difficult.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232
- *Standard Guide for Sampling Groundwater Monitoring Wells*, ASTM D 4448

E.1.3 Peristaltic Pump

A peristaltic pump (Figure E-3) is a suction lift pump used at the surface to collect liquid from ground-water monitoring wells or surface impoundments. It can be used for sampling surface water, ground water, point discharges, impounded liquids, and multi-layer liquid wastes.

A peristaltic pump consists of a rotor with ball bearing rollers and it has a piece of flexible tubing threaded around the pump rotor and connected to two pieces of polytetrafluroethylene (PTFE) or other suitable tubing. One end of the tubing is placed in the sample. The other end is connected to a sample container. Silicone tubing is commonly used within the pumphead; however, for organic sampling purposes, medical grade silicone is recommended to avoid contamination of the sample (ASTM D 4448). Fluorocarbon resin tubing is also sometimes used for high hazard materials and for samples to be analyzed for organics (ASTM D 6063). The device can be modified to avoid contact of the sample with the flexible tubing. In such a case, the pump is connected to a clean glass container using a PTFE insert. A second PTFE tubing is used to connect the glass container to the sample source.

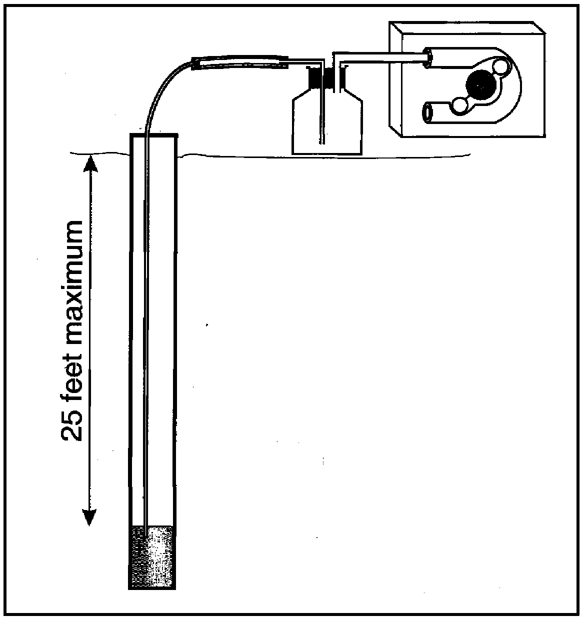

Figure E-3. Peristaltic pump

During operation, the rotor squeezes the flexible tubing, causing a vacuum to be applied to the inlet tubing. The sample material is drawn up the inlet tubing and discharged through the outlet end of the flexible tubing. In the modified peristaltic pump, the sample is emptied into the glass container without coming in contact with the flexible tubing. To sample liquids from drums, the peristaltic pump is first used to mix the sample. Both ends of the tubing are placed in the sample and the pump is turned on. Once the drum contents are mixed, the sample is collected as described above. To collect samples for organic volatile analyses, the PTFE tubing attached to the intake end of the pump is filled with the sample and then disconnected from the pump. The tube is then drained into the sample vials.

Advantages

- Can collect samples from multiple depths and small diameter wells.
- Easy to use and readily available.

- The pump itself does not need to be decontaminated. The tubing can be either decontaminated or replaced.

Limitations

- The drawing of a vacuum to lift the sample may cause the loss of volatile contaminants.
- Sampling depth cannot exceed about 7.6 m (25 ft.) (ASTM D 6232).
- Requires a power source.
- Flexible tubing may be incompatible with certain matrices.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Guide for Sampling of Drums and Similar Containers by Field Personnel*, ASTM D 6063
- *Standard Guide for Sampling Groundwater Monitoring Wells,* ASTM D 4448

E.1.4 Centrifugal Submersible Pump

The centrifugal submersible pump (Figure E-4) is a type of pump used for purging and sampling monitoring wells, sampling of waste water from impoundments, and sampling point discharges.

A centrifugal submersible pump uses a set of impellers, powered by an electric motor, to draw water up and through a discharge hose. Parts in contact with liquid may be made of PTFE and stainless steel. The pump discharge hose can be made of PTFE or other suitable material. The motor cavity is filled with either air or deionized or distilled water that may be replaced when necessary. Flow rates for centrifugal submersible pumps range from 100 mL per minute to 9 gallons per minute (ASTM D 6232).

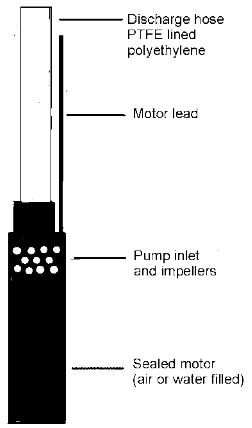

Figure E-4. Centrifugal submersible pump

During operation, water is drawn into the pump by a slight suction created by the rotation of the impellers. The impellers work against fixed stator plates and pressurize the water which is driven to the surface through the discharge hose. The speed at which the impellers are driven controls the pressure and, thus, the flow rate.

Advantages

- Can be constructed of materials (PTFE and stainless steel) that are chemically resistant.
- Can be used to pump liquids up to a 76 m (250 ft) head (ASTM D 6232).
- Flow rate is adjustable.

Limitations

- May be incompatible with liquids containing a high percentage of solids.
- May not be appropriate for collection of samples for volatile organics analysis. Loss of volatiles can occur as a result of motor heating and sample pressurization.
- Requires an electric power source; e.g., either a 12 v (DC) or a 110/220 v (AC) converter (ASTM D 6232).
- May require a winch or reel system for portable use.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232

E.1.5 Displacement Pumps

The displacement pump (Figure E-5) is a type of pump used for the sampling of surface water, ground water, point discharges and other liquids (e.g., in impoundments).

A displacement pump forces a discrete column of water to the surface via a mechanical lift. During sampling, water enters the sampler through the check valve at the bottom of the device. It is commonly constructed of PVC, stainless steel, or both. It also can be made of PTFE to reduce the risk of contamination when collecting samples with trace levels of organic compounds. Two common types of displacement pumps include the air/gas and piston displacement pumps.

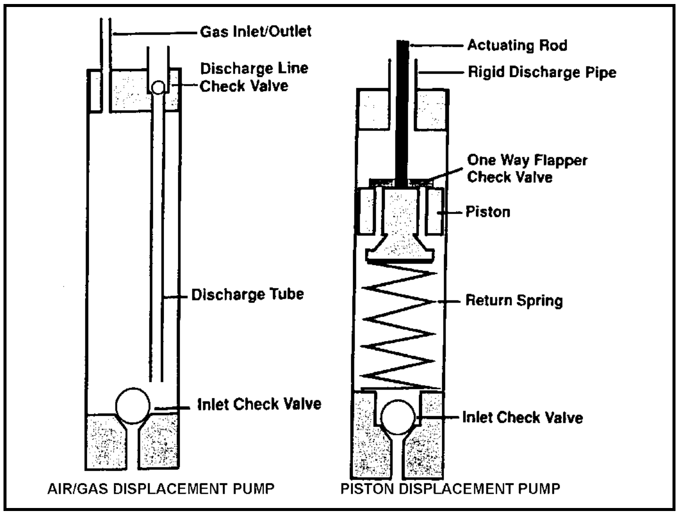

Figure E-5. Displacement pump

The air/gas displacement pump uses compressed gas and it operates by applying positive pressure to the gas line. This causes the inlet check valve to close and the discharge line check valve to open, forcing water up the discharge line to the surface. Removal of the gas pressure causes the top valve to close and the bottom valve to open. Water enters the sampler and the process is repeated.

The piston displacement pump uses an actuating rod powered from the surface or from an air or electric actuator. The mechanically operated plunger delivers the sample to the surface at the same time the chamber fills. It has a flap valve on the piston and an inlet check valve at the bottom of the sampler.

Advantages

- Can be constructed of PTFE to reduce the risk of contamination caused by materials of construction when collecting samples for trace levels of organics.

Limitations

- May be difficult to decontaminate.
- Displacement pumps require large volumes of air or gas and a power source.
- Loss of dissolved gases or sample contamination from the driving gas may occur during sampling.
- Displacement pumps may be heavy.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Guide for Sampling Groundwater Monitoring Wells*, ASTM D 4448

E.2 Dredges

Dredges include equipment that is often used to collect bottom material (e.g., sediments) from beneath a layer of stationary or moving liquid. A variety of dredges are available including the **Ekman** bottom grab sampler and the **Ponar** dredge. The Ponar dredge is described below.

E.2.1 Ponar Dredge

The ponar dredge is recommended for sampling sediment. It has paired jaws that penetrate the substrate and close to retain the sample. The sample volume range is 0.5 to 3.0 liters (ASTM D 6232).

The Ponar dredge is lowered slowly with controlled speed so that the dredge will properly land and avoid blowout of the surface layer to be sampled. The weight of the dredge causes it to penetrate the substrate surface. The slack in tension unlocks the open jaws and allows the dredge to close as it is raised. The dredge is raised slowly to minimize disturbance and sample washout as the dredge is retrieved through the liquid column. The collected sample is emptied into a suitable container. Auxiliary weight may be added to the dredge to increase penetration.

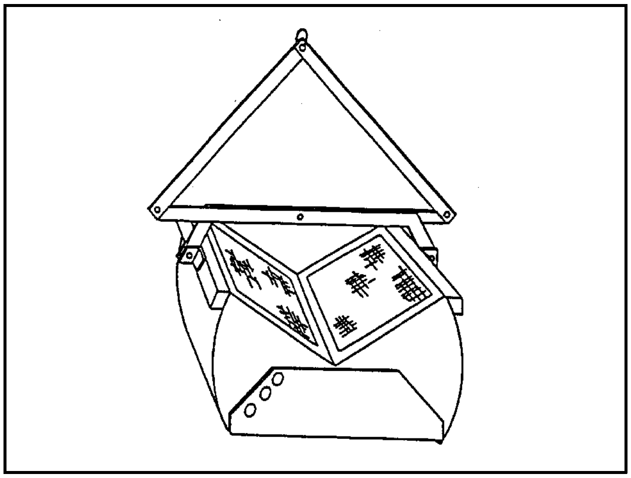

Figure E-6. Ponar dredge

Advantages

- Reusable
- Can obtain samples of most types of stationary sediments ranging from silt to granular material
- Available in a range of sizes and weights
- Some models may be available in either stainless steel or brass.

Limitations

- Not capable of collecting undisturbed samples
- May be difficult to decontaminate (depending upon the dredge's design and characteristics of the sampled material)
- Cannot collect a representative lift or repeatedly sample to the same depth and position
- Can be heavy and require a winch or portable crane to lift; however, a smaller version, the petit Ponar, is available and can be operated by a hand-line (ASTM D 4342).

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Practice for Collecting of Benthic Macroinvertebrates with Ponar Grab Sampler*, ASTM D 4342
- *Standard Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates*, ASTM D 4387

- “Sediment Sampling” (USEPA 1994e)

E.3 Discrete Depth Samplers

Discrete depth samplers include equipment that can collect samples at a specific depth. Such samplers are sometimes used to collect samples from layered liquids in tanks or surface impoundments. You will find summaries for the following discrete depth samplers in this section:

E.3.1 Bacon Bomb
E.3.2 Kemmerer Sampler
E.3.3 Syringe Sampler
E.3.4 Lidded Sludge/Water Sampler
E.3.5 Discrete Level Sampler

Besides the samplers listed below, a self-purging, discrete depth sampler is available for sampling ground-water monitoring wells. It fills when stopped at the desired depth and eliminates the need for well purging. It samples directly into a 40-mL glass VOA sample vial contained within the sampler; therefore, the loss of volatile organic compounds is minimized.

E.3.1 Bacon Bomb

A bacon bomb (Figure E-7) is a type of discrete level sampler that provides a sample from a specific depth in a stationary body of water or waste. A bacon bomb is recommended for sampling surface water and is usually used to collect samples from a lake or pond. It can also be used to collect liquid waste samples from large tanks or lagoons. It originally was designed to collect oil samples. The sample volume range is from 0.1 to 0.5 liters (100 to 500 mL) (ASTM D 6232).

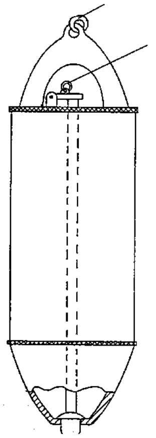
Figure E-7. Bacon bomb

A bacon bomb has a cylindrical body sometimes constructed of stainless steel, but it is sometimes made of chrome-plated brass and bronze. It is lowered into material by a primary support line and has an internal tapered plunger that acts as a valve to admit the sample. A secondary line attached to the top of the plunger opens and closes the plunger valve. The top cover has a locking mechanism to keep the plunger closed after sampling. The bacon bomb remains closed until triggered to collect the sample. Sample collection is triggered by raising the plunger line and allowing the sampler to fill. The device is then closed by releasing the plunger line. It is returned to the surface by raising the primary support line, and the sample is transferred directly to a container.

Advantages

- Collects a discrete depth sample; it is not opened until the desired depth.
- Easy to use, without physical requirement limitations.

Limitations

- May be difficult to decontaminate due to design or construction materials.
- Maximum sample capacity is only 500 mL.
- Materials of construction may not be compatible with parameters of concern.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- “Tank Sampling” (USEPA 1994c)

E.3.2 Kemmerer Sampler

A kemmerer sampler (Figure E-8) is a type of discrete level sampler that provides a sample from a specific depth. Recommended for sampling surface water, it is usually used to collect samples from a lake or pond. It can also be used to collect liquid waste samples from large tanks or lagoons. The sample volume range is from 1 to 2 liters (ASTM D 6232).

The sampler comprises a stainless steel or brass cylinder with rubber stoppers for the ends, but all PFTE construction also is available. The ends are left open while being lowered in a vertical position, allowing free passage of water or liquid through the cylinder. When the device is at the designated depth, a messenger is sent down a rope to close the stoppers at each end. The cylinder is then raised and the sample is removed through a valve to fill sample containers.

Advantages

- Can collect a discrete depth sample.

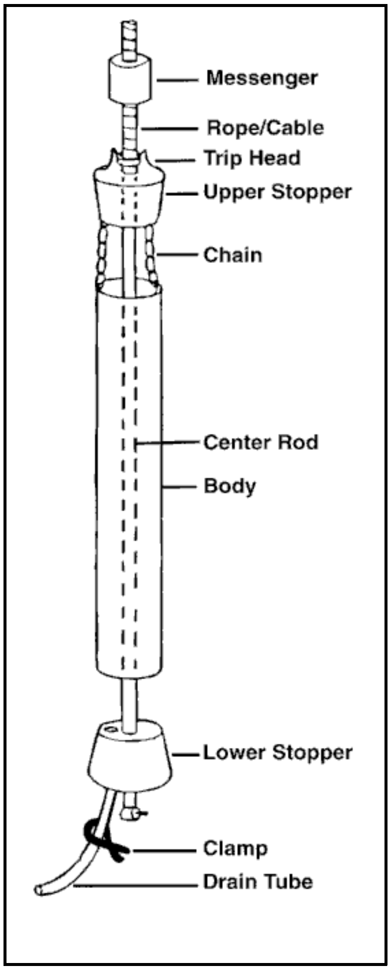

Figure E-8. Kemmerer sampler

- Provides correct delimitation and extraction of sample (Pitard 1989)
- Easy to use
- All PTFE construction is available.

Limitations

- May be difficult to decontaminate due to construction or materials.
- The sampler is exposed to the medium at other depths while being lowered to a sampling point at the desired depth.
- Materials of construction may not be compatible with parameters of concern.

Other Guidance:

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232

E.3.3 Syringe Sampler

A syringe sampler (Figure E-9) is a discrete depth sampler used to sample liquids. With the optional coring tip, it can be used as a coring device to sample highly viscous liquids, sludges, and tar-like substances. It is used to collect samples from drums, tanks, and surface impoundments, and it can also draw samples when only a small amount remains at the bottom of a tank or drum. The sample volume range is 0.2 to 0.5 liters (ASTM D 6232).

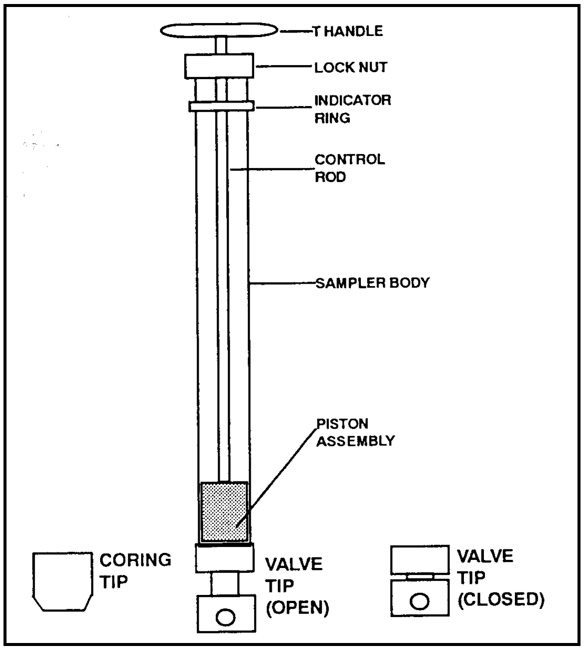

Figure E-9. Syringe sampler

A syringe sampler generally is constructed of a piston assembly that comprises a T-handle, safety locking nut, control rod, piston body assembly, sampling tube assembly, and two tips for the lower end (a closeable valve and a coring tip). When used as a syringe, the sampler is slowly lowered to the sampling point and the T-handle is gradually raised to collect the sample. Once the desired sample is obtained, the lock nut is tightened to secure the piston rod and the bottom valve is closed by pressing down on the sampler against the side or bottom of the container. When used as a coring device, the sampler is slowly pushed down into the material. Once the desired sample is obtained, the lock nut is tightened to secure the piston rod and the sampler is removed from the media. The sample material is extruded into the sample container by opening the bottom valve (if fitted), loosening the lock nut, and pushing the piston down.

Advantages

- The syringe sampler is easy to use and decontaminate.
- The syringe sampler can sample at discrete depths, including the bottom of a container.

Limitations

- The syringe sampler can be used to depths of about 1.8 meters only (ASTM D 6232).
- Material to be sampled must be viscous enough to remain in the device when the coring tip is used; the valve tip is not recommended for viscous materials (ASTM D 6063).

Other Guidance

- *Standard Guide for Sampling Single or Multilayered Liquids,* ASTM D 5743
- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Guide for Sampling of Drums and Similar Containers by Field Personnel,* ASTM D 6063

E.3.4 Lidded Sludge/Water Sampler

A lidded sludge/water sampler (Figure E-10) is a type of discrete depth device that provides a sample from a specific depth. It is used to collect sludges or waste fluids from tanks, tank trucks, and ponds. It can sample liquids, multi-layer liquid wastes, and mixed-phase solid/liquid wastes. The typical sample volume is 1.0-liter (ASTM D 6232).

A lidded sludge/water sampler comprises a removable glass jar, sometimes fitted with a cutter for sampling materials containing more than 40-percent solids (ASTM D 6232), that is mounted on a stainless steel device.

The sampler is lowered into the material to be sampled and opened at the desired depth. The top handle is rotated to upright the jar and open and close the lid. Then, the device is carefully retrieved from the material. The jar is removed from the sampler by lifting it from the holder, and

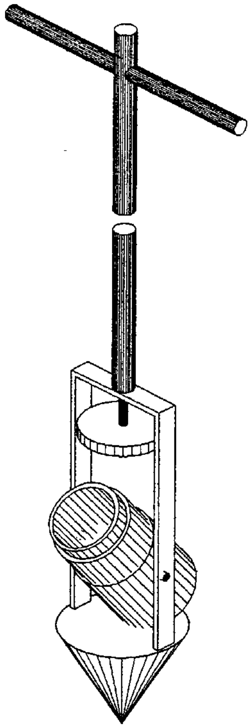

Figure E-10. Lidded sludge/water sampler the jar serves as a sample container so there is no need to transfer the sample.

Advantages

- The jar in the sampling device also serves as a sample container reducing the risk of cross-contamination.
- Bottles and lids are unique to each sample, therefore, decontamination of an intermediate transfer container is not required.

Limitations

- Heavy and limited to one bottle size
- Thick sludge is difficult to sample (ASTM D 6232).

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232

E.3.5 Discrete Level Sampler

A discrete level sampler (Figure E-11) is a dismountable cylindrical sampler fitted with a manually-operated valve(s). It is recommended for sampling surface water, ground water, point discharges, liquids, and multi-layer liquids and is used for sampling drums, tanks, containers, wells, and surface impoundments. The typical sample volume range is 0.2 to 0.5 liters (ASTM D 6232).

A discrete level sampler is made from PTFE and stainless steel and is designed to be reusable. It comprises a tube fitted with manually-operated valve or valves, which are operated by a control assembly attached to the upper end of the sampler. This assembly consists of a rigid tube and rod or a flexible tube and inner cable. The standard level sampler has a manually operated upper valve and a lower spring-retained bottom dump valve. The dual valve model may be emptied by opening the valves manually or with a metering device attached to the lower end of the sampler (not shown).

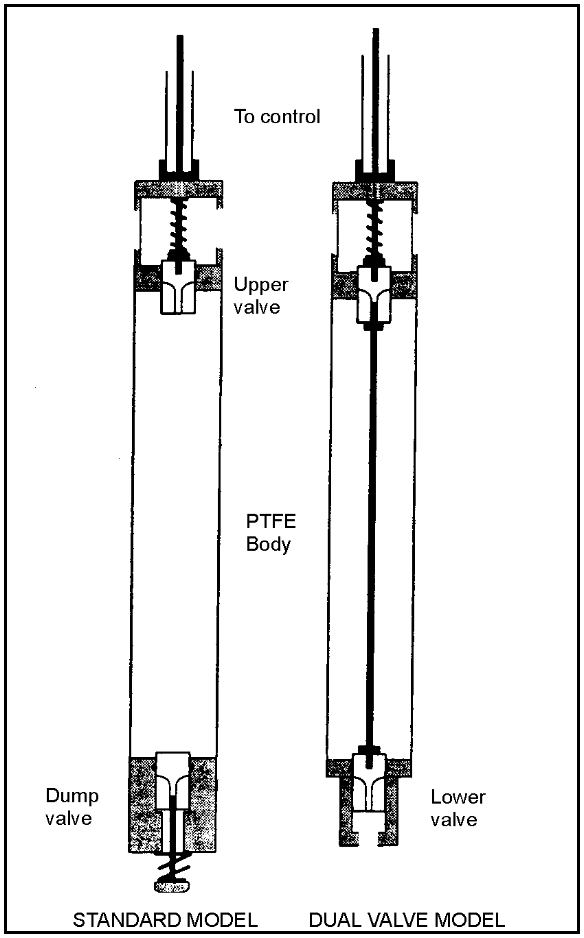

Figure E-11. Discrete level sampler

To collect a sample, the discrete level sampler is lowered into the sample material to the desired sampling depth. The valve or valves are opened manually to collect the sample and closed before retrieving the sampler. The standard model is emptied by pressing the dump valve against the side of the sample container. The dual valve sampler is emptied by opening the valves manually. Alternatively, the collected sample may be taken to the laboratory in the sampler body by replacing the valves with solid PTFE end caps.

Advantages

- Relatively easy to decontaminate and reuse
- May be used to sample liquids in most environmental situations.
- Can be remotely operated in hazardous environments.
- Sample representativeness is not affected by liquids above the sampling point.
- The sampling body can be used for sample storage and transport.

Limitations

- Limited to sample chamber capacities of 240-475 mL (ASTM D 6232).
- May be incompatible with liquids containing a high percentage of solids.

Other Guidance

- Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities, ASTM D 6232

E.4 Push Coring Devices

Push coring devices include equipment that use a pushing action to collect a vertical column of a solid sample. You will find summaries for the following push coring devices in this section:

E.4.1 Penetrating Probe Sampler
E.4.2 Split Barrel Sampler
E.4.3 Concentric Tube Thief
E.4.4 Trier
E.4.5 Thin-Walled Tube
E.4.6 Coring Type Sampler (with Valve)
E.4.7 Miniature Core Sampler
E.4.8 Modified Syringe Sampler

E.4.1 Penetrating Probe Sampler

The penetrating probe sampler (Figure E-12) is a push coring device and, therefore, provides a core sample. The probe sampler is recommended for sampling soil and other solids. The sample volume range is 0.2 to 2.0 liters (ASTM D 6232).

The probe sampler typically consists of single or multiple threaded steel tubes, a threaded top cap, and a detachable steel tip. The steel tubes are approximately 1 inch or less in diameter. Specialized attachments may be used for various matrices. Some probes are equipped with adjustable screens or retractable inner rods to sample soil vapor or ground water.

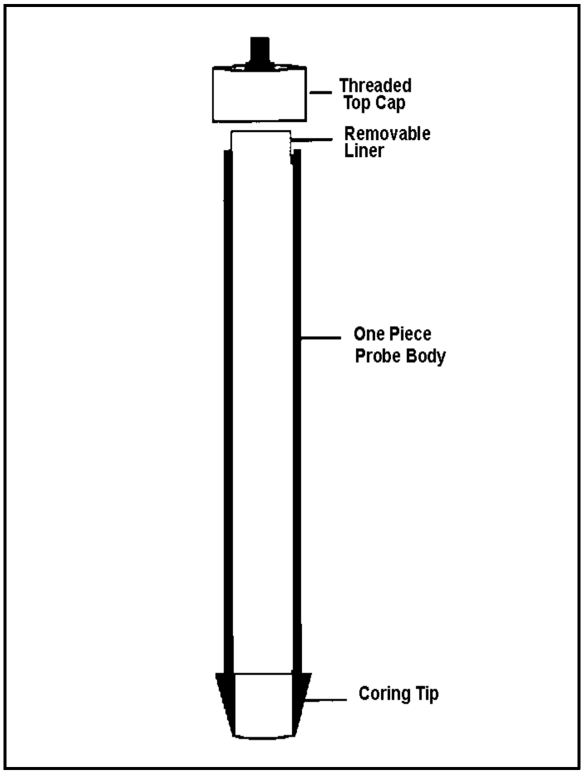

Figure E-12. Probe sampler

Advantages

- Easy to decontaminate and is reusable.
- Can provide samples for onsite analysis (ASTM D 6232).
- Versatile and may sample 15 to 20 locations a day for any combination of matrices (ASTM D 6232).
- Can reduce quantity of investigative derived wastes.

Limitations

- May be heavy and bulky depending on the size used.
- Limited by composition of subsurface materials and accessibility to deeper depth materials.
- May be inappropriate for sampling materials that require mechanical strength to penetrate.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232

E.4.2 Split Barrel Sampler

A split barrel sampler (Figure E-13) is a push coring device often used with a drill rig to collect deep subsurface samples. The device is recommended for soil sampling, but can be used to sample other solids. The materials to be sampled should be moist enough to remain in the sampler. The sample volume range is 0.5 to 30.0 liters (ASTM D 6232).

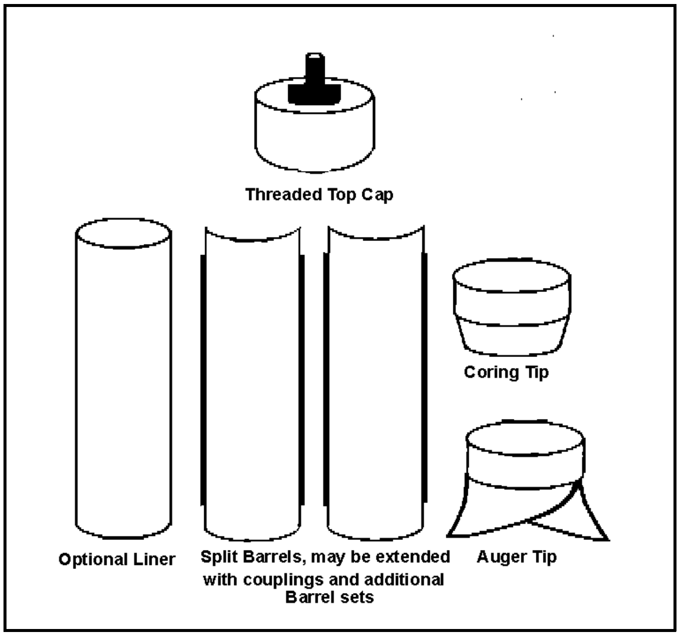

Figure E-13. Split barrel sampler

The sampler consists of a length of steel tubing split longitudinally and equipped with a drive shoe, made of steel, and a drive head. The drive shoe is detachable and should be replaced when dented or distorted. The samplers are available in a variety of diameters and lengths. The split barrel is typically 18 to 30 inches in length with an inside diameter of 1.5 to 2.5 inches (ASTM D 4700, ASTM D 1586). The split barrel sampler can be used to collect relatively undisturbed soil samples at considerable depths.

The split barrel sampler may be driven manually, but is usually driven with a drill rig drive weight assembly or hydraulically pushed using rig hydraulics. The sampler is placed on the surface of the material to be sampled, then pushed downward while being twisted slightly. Because pushing by hand may be difficult, a drop hammer typically is attached to a drill rig used to finish inserting the sampler. When the desired depth is reached the sampler is twisted again to break the core; then, the sampler is pulled straight up and out of the material. The sample may be removed from the barrel or the liner may be capped off for analysis. Barrels may be extended to 5 inches in diameter (ASTM D 6232). Liners often are used when sampling for volatile organic compounds or other trace constituents of interest. With a liner, the sample can be removed with a minimum amount of disturbance. Liners must be compatible with the matrix and compounds of interest; plastic liners may be inappropriate if analyzing for organics.

Advantages

- Reusable, easily decontaminated, and easy to use.
- Provides a relatively undisturbed sample, therefore, can minimize the loss of volatile organic compounds.

Limitations

- Requires a drill or direct push rig for deep samples.
- Made of steel and may penetrate underground objects such as a pipe or drum.

- Only accommodates samples that contain particles smaller than the opening of the drive shoe (ASTM D 4700).

Other Guidance:

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232
- *Standard Guide for Soil Sampling from the Vadose* Zone, ASTM D 4700
- *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils,* ASTM D 1586

E.4.3 Concentric Tube Thief

The concentric tube thief (also known as a grain sampler) (Figure E-14) is a push coring device that the user directly pushes into the material to be sampled. It can be used to sample powdered or granular solids and wastes in piles or in bags, drums, or similar containers. The concentric tube thieves are generally 61 to 100 cm (24 to 40 inches) long by 1.27 to 2.54 cm (½ to 1 inch) in diameter (USEPA 1994i). The sample volume range is 0.5 to 1.0 liters (ASTM D 6232).

The concentric tube thief consists of two slotted telescoping tubes, which are constructed of stainless steel, brass, or other material. The outer tube has a conical pointed tip on one end which allows the thief to penetrate the material being sampled. The thief is opened and closed by rotating the inner tube, and it is inserted into the material while in the closed position. Once inserted, the inner tube is rotated into the open position and the device is wiggled to allow the material to enter the open slots. The thief then is closed and withdrawn.

Advantages

- Is a good direct push sampler for dry unconsolidated materials.
- Easy to use.

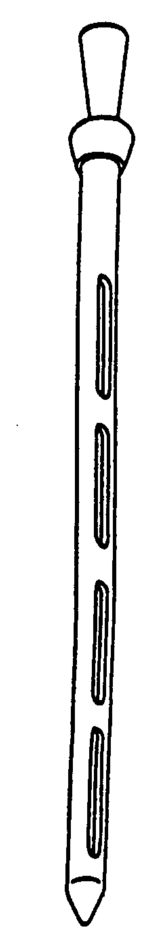

Figure E-14. Concentric tube thief

Limitations

- May be difficult to decontaminate, depending on the matrix
- Not recommended for sampling of moist or sticky materials.
- Does not collect samples containing all particle sizes if the diameter of the largest solid particle is greater than one-third of the slot width (ASTM D 6232). Most useful when the solids are no greater than 0.6 cm (1/4-inch) in diameter (USEPA 1994i).
- Depth of sample is limited by the length of the thief.
- Not recommended for use when volatiles are of interest. Collects a somewhat disturbed sample, which may cause loss of some volatiles.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- “Waste Pile Sampling” (USEPA 1994d)

E.4.4 Trier

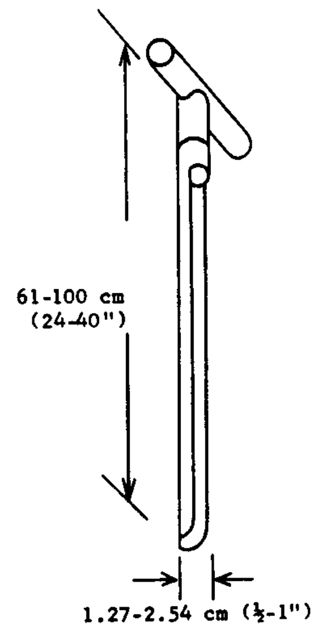

Figure E-15. Trier

A trier (Figure E-15) is a push coring device that resembles an elongated scoop and is used to sample moist or sticky solids with a particle diameter less than one-half the diameter of the tube portion. The trier can be used to sample soils and similar fine-grained cohesive materials. The typical sample volume range is 0.1 to 0.5 liters (ASTM D 6232).

A trier comprises a handle connected to a tube cut in half lengthwise, with a sharpened tip that allows it to cut into the material. Triers are made of stainless steel, PTFE-coated metal, or plastic. One should be selected who materials of construction are compatible with the sampled material.

A trier, typically 61 to 100 cm long and 1.27 to 2.54 cm in diameter, is used as a vertical coring device when a relatively complete and cylindrical sample can be extracted.

The trier is pushed into the material to be sampled and turned one or two times to cut a core. The rotation is stopped with the open face pointing upward. The core is then carefully removed from the hole, preventing overburden material from becoming a part of the sample. The sample is inspected for irregularities (e.g., pebbles) or breakage. If breakage occurred and if the core does not satisfy minimum length requirements, discard it and extract another from an immediately adjacent location (ASTM D 5451). The sample is emptied into the appropriate container for analysis.

Advantages

- A good direct push sampler for moist or sticky materials.
- Lightweight, easy to use, and easy to decontaminate for reuse.

Limitations

- Limited to sample particle sizes within the diameter of the inserted tube and will not collect particles greater than the slot width.
- Not recommended for sampling of dry unconsolidated materials. (A concentric tube thief is good for such materials.)
- Only for surface sampling, and the depth of sample is limited by the length of the trier.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232
- *Standard Practice for Sampling Using a Trier Sampler,* ASTM D 5451
- *Sampling of Drums and Similar Containers by Field Personnel,* ASTM D 6063
- *Standard Practice for Sampling Unconsolidated Solids in Drums or Similar Containers,* ASTM D 5680

E.4.5 Thin-Walled Tube

A thin-walled tube (Figure E-16) is a type of push coring device recommended for sampling cohesive, unconsolidated solids – particularly soil. It is not recommended for gravel or rocky soil. The sample volume range is 0.5 to 5.0 liters (ASTM D 6232).

The tube generally is constructed of carbon stainless steel, but can be manufactured from other metals (ASTM D 4700). It is commonly 30-inches long and is readily available in 2-, 3-, and 5-inch outside diameters (ASTM D 4700). The tube is attached with set screws to a length of a solid or tubular rod, and the upper end of the rod, or sampler head, is threaded to accept a handle or extension rod. Typically, the length of the tube depends on the desired sampling depth. Its advancing end is beveled and has a cutting edge with a smaller diameter than the tube inside diameter. The tube can be used in conjunction with drills – from hand-held to full-sized rigs.

The end of the sampler is pushed directly into the media using a downward force on the handle. It can be pushed downward by hand, with a jack-like system, or with a hydraulic piston. Once the desired depth is reached, the tube is twisted to break the continuity of the tip and is pulled from the media. The sample material is extruded into the sample container by forcing a rod through the tube. A paring device has been developed to remove the outer layer during extrusion (ASTM D 4700). Plastic and PFTE sealing caps for use after sampling are available for the 2-, 3-, and 5-inch tubes.

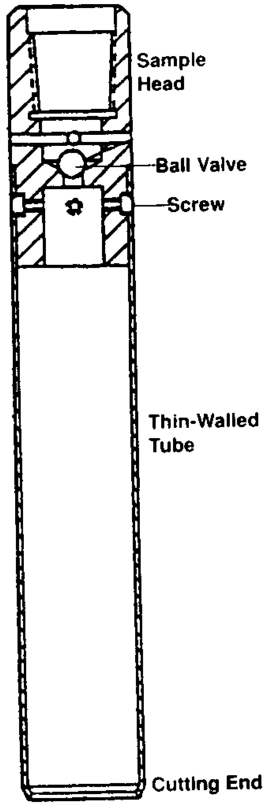

Figure E-16. Thin-walled tube

Advantages

- Readily available, inexpensive, and easy to use.
- Reusable and can be decontaminated.
- Obtains a relatively undisturbed sample.

Limitations

- Some thin-walled tubes are large and heavy.
- The material to be sampled must be of a physical consistency (cohesive sold material) to be cored and retrieved within the tube. It cannot be used to sample gravel or rocky soils.
- Some volatile loss is possible when the sample is removed from the tube.
- The most disturbed portion in contact with the tube may be considered unrepresentative. Shorter tubes provide less-disturbed samples than longer tubes.
- Materials with particles larger than one-third of the inner diameter of the tube should not be sampled with a thin-walled tube.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Guide for Core Sampling of Submerged, Unconsolidated Sediments,* ASTM D 4823
- *Standard Practice for Thin-Walled Type Geotechnical Sampling of Soils,* ASTM D 1587
- *Standard Guide for Soil Sampling from the Vadose Zone,* ASTM D 4700

E.4.6 Coring Type Sampler (with Valve)

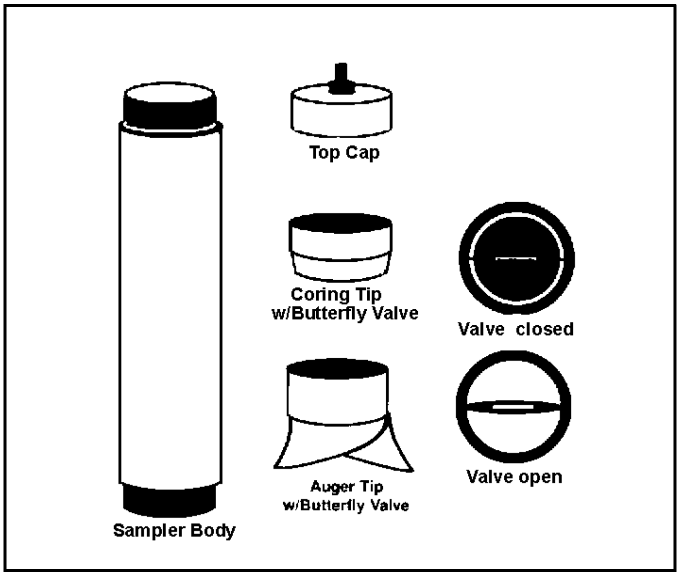

Figure E-17. Coring type sampler (with valve)

A coring type sampler with valve (Figure E-17) is a type of push coring device recommended for wet soil, and can also be used to sample unconsolidated solid waste, mixed-phase solid/liquid waste, and free-flowing powders. The coring device may be used in drums and small containers as well as tanks, lagoons, and waste impoundments. The sample volume range is 0.2 to 1.5 liters (ASTM D 6232).

The coring type sampler with valve is a stainless steel cylindrical sampler with a coring tip, top cap, an extension with a cross handle, and a non-return valve at the lower end behind a coring or augering tip. The valve is a retaining device to hold the sample in place as the coring device is removed. Samples are normally collected in an optional liner. It is operated by attaching a handle or an extension with a handle to the top of the coring device. The corer is lowered to the surface, pushed into the material being sampled and removed. The top cap is removed and the contents emptied into a sample container. Alternatively, the liner can be removed (with the sampled material retained inside) and capped on both ends for shipment to a laboratory.

Advantages

- Reusable and is easily decontaminated.
- Provides a relatively undisturbed sample if not extruded.
- Can be hand operated and does not require significant physical strength.

Limitations

- Can not be used in gravel, large particle sediments, or sludges.
- When sampling for volatile organic compounds, it must be used with a liner and capped to minimize the loss of volatiles.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232
- *Guide for Core Sampling Submerged, Unconsolidated Sediments*, ASTM D 4823

E.4.7 Miniature Core Sampler

The miniature core sampler (Figure E-18) can be used to collect soil and waste samples for volatile organics analysis. These include devices such as the Purge-and-Trap Soil Sampler™, the EnCore™ sampler, or a cut plastic syringe (see Section 6.0 of SW-846 Method 5035). A miniature core sampler is a single-use push coring sampling device that also can be used as an air-tight sample storage and shipping container. It collects a small contained subsample and is particularly useful for the sampling and analysis of volatile organic compounds.

It is recommended for sampling soil, from the ground or the side of a trench, and may be used for sampling sediment and unconsolidated solid wastes. It cannot be used for sampling cemented material, consolidated material, or material having fragments coarse enough to interfere with proper coring. The EnCore™ sampler can be used to collect subsamples from soil cores and has a sample volume range of 0.01 to 0.05 liters (ASTM D 6232).

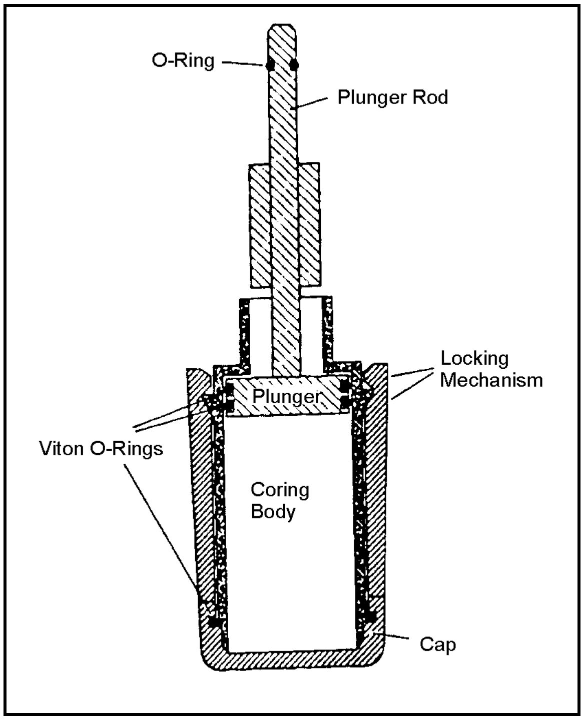

Figure E-18. Miniature core sample (Encore™ sampler)

The device is available from the manufacturer in two sizes for collection of 5- and 25-gram samples (assuming a soil density of 1.7 g/cm^3). The size is chosen based on the sample size required by the analytical procedure.

SW-846 Method 5035, “Closed-System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples,” recommends that samples not be stored in the device longer than 48 hours prior to sample preparation for analysis. The manufacturer's instructions for sample extrusion should be followed carefully.

Advantages

- Maintains sample structure in a device that also can be used to store and transport the sample directly to the laboratory.
- Recommended for collecting samples for the analysis of volatile compounds. It collects a relatively undisturbed sample that is contained prior to analysis to minimize the loss of volatile compounds.
- Usually is compatible with the chemicals and physical characteristics of the sampled media.
- No significant physical limitations for its use.
- Cross-contamination should not be a concern if the miniature core sampler is certified clean by the manufacturer and employed as a single-use device.

Limitations

- Cannot be used to sample gravel or rocky soils.
- Instructions must be followed carefully for proper use to avoid trapping air with the sample and to ensure that the sample does not compromise the seals.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Practice for Using the Disposable EnCore™ Sampler for Sampling and Storing Soil for Volatile Organic Analysis,* ASTM D 6418
- *Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds,* ASTM D 4547

E.4.8 Modified Syringe Sampler

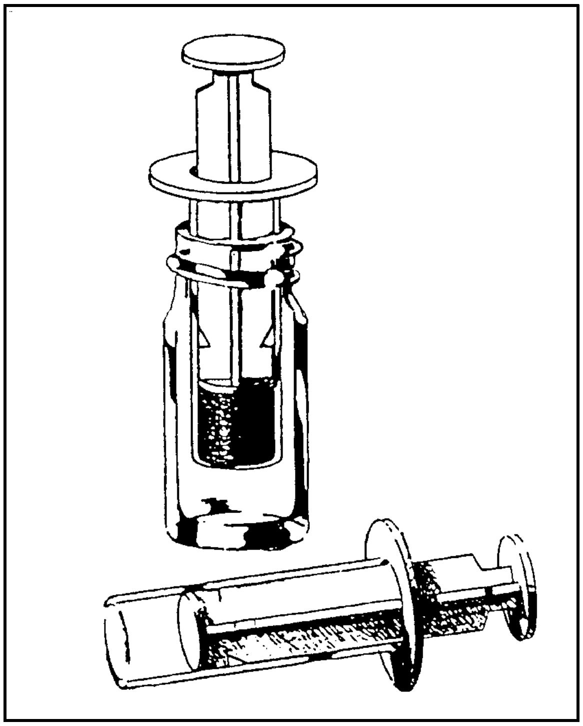

Figure E-19. Modified syringe sampler

A modified syringe sampler (Figure E-19) is a push coring sampling device constructed by the user by modifying a plastic, single-use, medical syringe. It can be used to provide a small, sub-sample of soil, sediments, and unconsolidated solid wastes. It is sometimes used to sub-sample a larger core of soil. It is not recommended for sampling cemented material, consolidated material, or material having fragments coarse enough to interfere with proper coring. Unlike the EnCore™ sampler, it should not be used to store and ship a sample to the laboratory. Instead, the sample should be extruded into another container. Although the modified syringe sampler does not provide as contained a sample as the EnCore™ sampler, it can be used for sampling volatile compounds, as long as sample extrusion into another container is quickly and carefully executed. The modified syringe sample has a volume range of 0.01 to 0.05 liters (ASTM D 6232).

A modified syringe sampler is constructed by cutting off the lower end of the syringe attachment for the needle. The rubber cap is removed from the plunger, and the plunger is pushed in until it is flush with the cut end. For greater ease in pushing into the solid matrix, the front edge sometimes can be sharpened (ASTM D 4547). The syringe sampler is then pushed into the media to collect the sample, which then may be placed in a glass VOA vial for storage and transport to the laboratory. The sample is immediately extruded into the vial by gently pushing the plunger. The volume of material collected should not cause excessive stress on the device during intrusion into the material, or be so large that the sample falls apart easily during extrusion.

Advantages

- Obtains a relatively undisturbed profile sample.
- Can be used for the collection of samples for the analysis of volatile compounds as long as sample extrusion is quickly and carefully executed.
- No significant physical limitations for its use.
- Low-cost, single-use device.

Limitations

- Cannot be used to sample gravel or rocky soils.
- Material of construction may be incompatible with highly contaminated media.
- Care is required to ensure that the device is clean before use.
- The device cannot be used to store and transport a sample.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232
- *Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds,* ASTM D 4547

E.5 Rotating Coring Devices

Rotating coring devices include equipment that obtains vertical columns of a solid sample through a rotating action. Some of these devices (such as augers) also can be used for just boring a hole for sample collection at a certain depth using another piece of equipment. You will find summaries for the following rotating coring devices in this section:

E.5.1 Bucket Auger
E.5.2 Rotating Coring Device

E.5.1 Bucket Auger

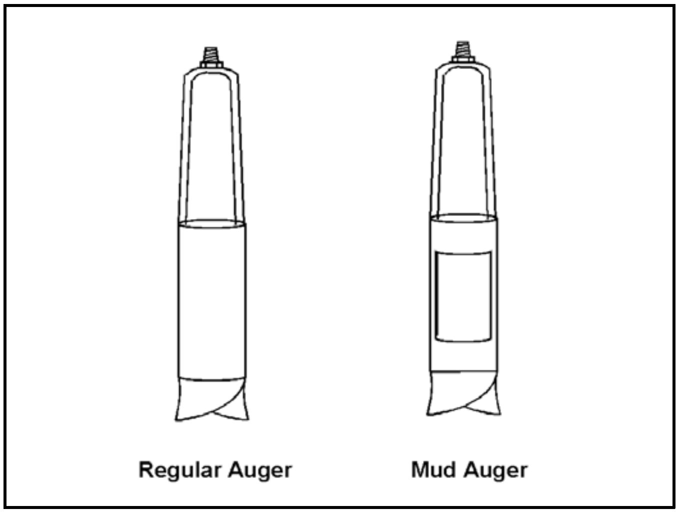

Figure E-20. Bucket auger

The bucket auger (Figure E-20) is a hand-operated rotating coring device generally used to sample soil, sediment, or unconsolidated solid waste. It can be used to obtain samples from drums, storage containers, and waste piles. The sample volume range is 0.2 to 1.0 liters (ASTM D 6232).

The cutting head of the auger bucket is pushed and twisted by hand with a downward force into the ground and removed as the bucket is filled. The empty auger is returned to the hole and the procedure is repeated. The sequence is continued until the required depth is reached. The same bucket may be used to advance the hole if the vertical sample is a composite of all intervals; however, discrete grab samples should be collected in separate clean auger buckets. The top several inches of material should be removed from the bucket to minimize chances of cross-contamination of the sample from fall-in material from the upper portions of the hole.

Note that hand augering may be difficult in tight clays or cemented sands. At depths approaching 20 feet (6 m), the tension of hand auger extension rods may make operation of the auger too difficult. Powered methods are recommended if deeper samples are required (ASTM D 6232).

Advantages

- Reusable and easy to decontaminate.
- Easy to use and relatively quick for shallow subsurface samples.
- Allows the use of various auger heads to sample a wide variety of soil conditions (USEPA 1993c).
- Provides a large volume of sample in a short time.

Limitations

- Depth of sampling is limited to about 20 feet (6 m) below the surface.
- Not suitable for obtaining undisturbed samples.
- Requires considerable strength to operate and is labor intensive.
- Not ideal for sampling soils for volatile organic compounds.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Practice for Soil Investigation and Sampling by Auger Borings,* ASTM D 1452
- *Standard Guide for Soil Sampling from the Vadose Zone,* ASTM D 4700
- *Standard Practice for Sampling Unconsolidated Waste From Trucks*, ASTM D 5658
- *Standard Guide for Sampling of Drums and Similar Containers by Field Personnel,* ASTM D 6063
- “Waste Pile Sampling” (USEPA 1994d)

- “Sediment Sampling” (USEPA 1994e)

E.5.2 Rotating Coring Device

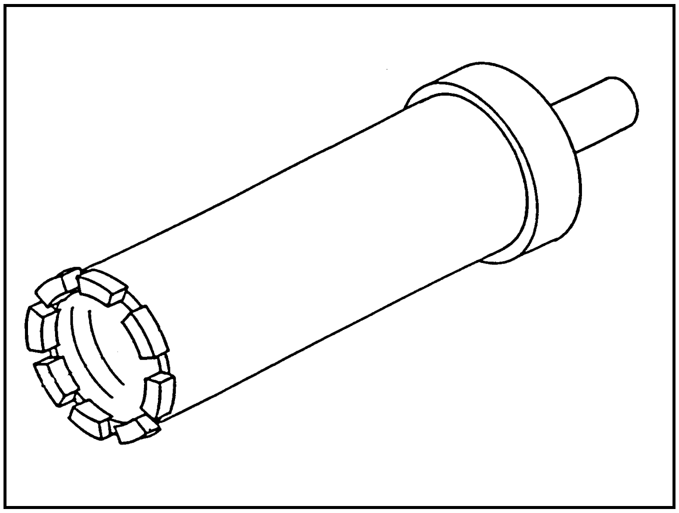

Figure E-21. Rotating coring device

The rotating coring device (Figure E-21) collects vertical columns of a solid sample through a rotating action and can be used in sampling consolidated solid waste, soil, and sediment. The sample volume range is 0.5 to 1.0 liters (ASTM D 6232).

The rotating coring device consists of a diamond- or carbide-tipped open steel cylinder attached to an electric drill. The coring device may be operated with the drill hand-held or with the drill mounted on a stand. When on a portable stand, full-depth core samples can be obtained. The barrel length is usually 1- to 1.5-feet long and the barrel diameter ranges from 2 to 6 inches (ASTM D 6232 and ASTM D 5679). The rotating coring device may be used for surface or depth samples.

The rotating coring device is placed vertical to the surface of the media to be sampled, then turned on before contact with the surface. Uniform and continuous pressure is supplied to the device until the specified depth is reached. The coring device is then withdrawn and the sample is placed into a container for analysis, or the tube itself may be capped and sent to the laboratory. Capping the tube is preferred when sampling for volatile organic compounds. The rotating tube must be cooled and lubricated with water between samples.

Advantages

- Easy to decontaminate.
- Reusable.
- Can obtain a solid core sample.

Limitations

- Requires a battery or other source of power.
- Requires a supply of water, used for cooling the rotating tube.
- Not easy to operate.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Practice for Sampling Consolidated Solids in Drums or Similar Containers*, ASTM D 5679
- "Drum Sampling" (USEPA 1994b)
- "Sediment Sampling" (USEPA 1994e)

E.6 Liquid Profile Devices

Liquid profile devices include equipment that can collect a vertical column of a liquid, sludge, or slurry sample. You will find summaries for the following liquid profile devices in this section:

E.6.1 Composite Liquid Waste Sampler (COLIWASA)
E.6.2 Drum Thief
E.6.3 Valved Drum Sampler
E.6.4 Plunger Type Sampler
E.6.5 Settleable Solids Profiler (Sludge Judge)

E.6.1 COLIWASA (Composite Liquid Waste Sampler)

The COLIWASA (Figure E-22) is a type of liquid profile sampling device used to obtain a vertical column of sampled material. A COLIWASA is recommended for sampling liquids, multi-layer liquid wastes, and mixed-phase solid/liquid wastes and is commonly used to sample containerized liquids, such as tanks and drums. It also may be used for sampling open bodies of stagnant liquids. The sample volume range is 0.5 to 3 liters (ASTM D 6232).

A COLIWASA can be constructed of polyvinyl chloride (PVC), glass, metal, PTFE or any other material compatible with the sample being collected. In general, a COLIWASA comprises a tube with a tapered end and an inner rod that has some type of stopper on the end. The design can be modified or adapted to meet the needs of the sampler. One configuration comprises a piston valve attached by an inner rod to a locking

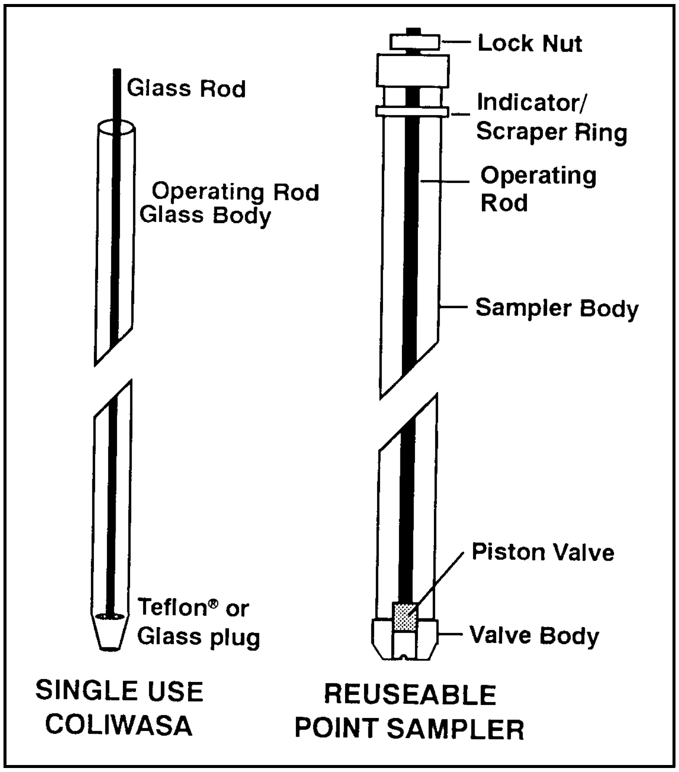

Figure E-22. COLIWASA

mechanism at the other end. Designs are available for specific sampling situations (i.e., drums, tanks). COLIWASAs specifically designed for sampling liquids, viscous materials, and heavy sludges are also available. COLIWASAs come in a variety of diameters (0.5 to 2 inches) and lengths (4 to 20 feet) (ASTM D 6232).

COLIWASAs are available commercially with different types of stoppers and locking mechanisms, but all have the same operating principle. To draw a sample, the COLIWASA is slowly lowered into the sample at a right angle with the surface of the material. (If the COLIWASA sampler is lowered too fast, the level of material inside and outside the sampler may not be the same, causing incorrect proportions in the sample. In addition, the layers of multi-layered materials may be disturbed.) The sampler is opened at both ends as it is lowered to allow the material to flow through it. When the device reaches the desired sampling depth, the sampler is closed by the stopper mechanism and both tubes are removed from the material. The sampled material is then transferred to a sample container by opening the COLIWASA. A COLIWASA can be reused following proper decontamination (reusable point sampler) or disposed after use (single-use COLIWASA). The reusable point sampler is used in the same way as the single use COLIWASA; however, it can also sample at a specific point in the liquid column.

Advantages

- Provides correct delimitation and extraction of waste (Pitard 1989).
- Easy to use.
- Inexpensive.
- Reusable.
- Single-use models are available.

Limitations

- May break if made of glass and used in consolidated matrices.
- Decontamination may be difficult.
- The stopper may not allow collection of material in the bottom of a drum.
- High viscosity fluids are difficult to sample.

Other Guidance

- *Standard Practice for Sampling with a Composite Liquid Waste Sampler* (COLIWASA), ASTM D 5495
- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232

- *Standard Guide for Sampling Drums and Similar Containers by Field Personnel*, ASTM D 6063
- *Standard Practice for Sampling Single or Multilayered Liquids, With or Without Solids, in Drums or Similar Containers*, ASTM D 5743
- "Drum Sampling" (USEPA 1994b)
- "Tank Sampling" (USEPA 1994c)

E.6.2 Drum Thief

A drum thief (Figure E-23) is an open-ended tube and liquid profile sampling device that provides a vertical column of the sampled material. It is recommended for sampling liquids, multi-layer liquid wastes, and mixed-phase solid/liquid wastes and can be used to sample liquids in drums or similar containers. The typical sample volume range is 0.1 to 0.5 liters (ASTM D 6232).

Drum thieves can be made of glass, stainless steel, or any other suitable material. Drum thieves are typically 6 mm to 16 mm inside diameter and 48-inches long (USEPA 1994c). To sample liquids with low surface tension, a narrow bailer works best. In most cases, tubes with a 1-centimeter inside diameter work best. Wider tubes can be used to sample sludges.

The drum thief is lowered vertically into the material to be sampled, inserted slowly to allow the level of material inside and outside the tube to be approximately the same. This avoids incorrect proportions in the sample. The upper end is then sealed with the thumb or a rubber stopper to hold the sample in the tube as it is removed from the container. The thief is emptied by removing the thumb or stopper.

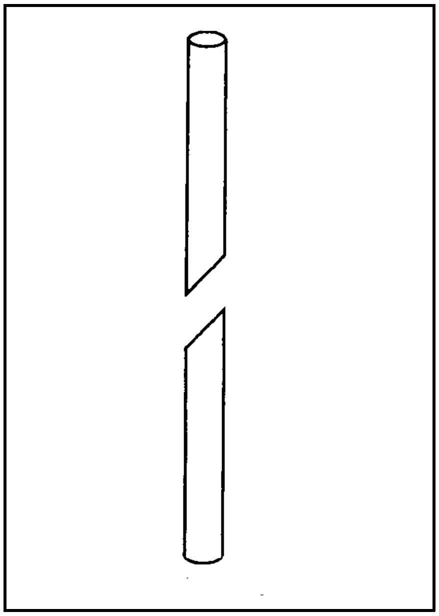

Figure E-23. Drum thief

Advantages

- Easy to use and inexpensive.
- Available in reusable and single-use models.

Limitations

- Sampling depth is limited to the length of the sampler.
- May not collect material in the bottom of a drum. The depth of unsampled material depends on the density, surface tension, and viscosity of the material being sampled.

- Highly viscous materials are difficult to sample.
- May be difficult to retain sample in the tube when sampling liquids of high specific gravity.
- If made of glass, may break if used in consolidated matrices. In addition, chips and cracks in a glass drum thief may cause an imperfect seal.
- Decontamination is difficult.
- When sampling a drum, repeated use of the drum thief to obtain an adequate volume of sample may disturb the drum contents.
- Drum-size tubes have a small volume and sometimes require repeated use to obtain a sample. Two or more people may be required to use larger sizes.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232
- *Standard Guide for Sampling of Drums and Similar Containers by Field Personnel*, ASTM D 6063
- *Standard Practice for Sampling Single or Multilayered Liquids, With or Without Solids, in Drums or Similar Containers*, ASTM D 5743
- "Drum Sampling" (USEPA 1994b)
- "Tank Sampling" (USEPA 1994c)

E.6.3 Valved Drum Sampler

A valved drum sampler (Figure E-24) is a liquid profile device often used to sample liquids in drums or tanks and provides a vertical column of the sampled material. A valved drum sampler is recommended for sampling liquids, multi-layered liquid wastes, and mixed-phase solid/liquid wastes. The typical sample volume range is 0.3 to 1.6 liters (ASTM D 6232).

The sampler can be constructed from PTFE for reuse or polypropylene for single use and comprises a tube fitted with a top plug and a bottom valve. A sliding indicator ring allows specific levels or fluids interfaces to be identified.

The valved drum sampler is open at both ends during

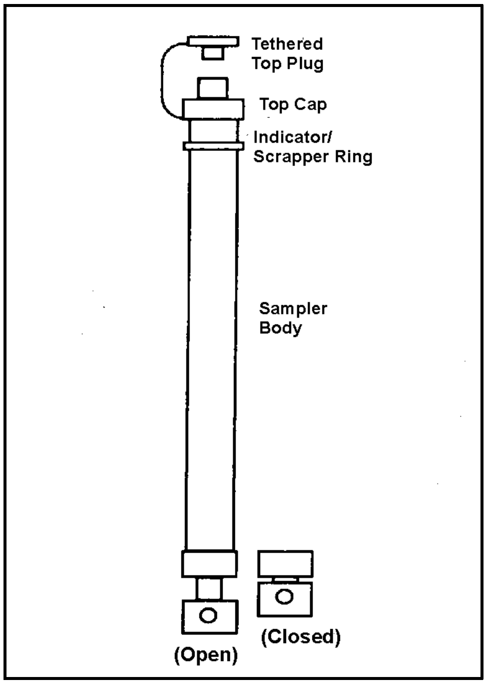

Figure E-24. Valved drum sampler

sample collection and lowered vertically into the material to be sampled. The sampler is inserted slowly to allow the level of material inside and outside the tube to equalize. Once the desired amount of sample is collected, the top plug and the bottom valve are closed. The top plug is closed manually and the bottom valve is closed by pressing against the side or bottom of the container. The sample is poured from the top of the sampler into a suitable container.

Advantages

- Easy to use, inexpensive, and unbreakable.
- Obtains samples to depths of about 8 feet (2.4 m) (ASTM D 6232).
- Reusable if made from PTFE (single-use if made from polypropylene) (ASTM D 6232).

Limitations

- Somewhat difficult to decontaminate
- The bottom valve may prevent collection of the bottom 1.25 cm of material (ASTM D 6232).
- High viscosity fluids are difficult to sample.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232

E.6.4 Plunger Type Sampler

The plunger type sampler (Figure E-25) is a liquid profile sampling device used to collect a vertical column of liquid and is recommended for the sampling of single and multi-layered liquids or mixtures of liquids and solids. The plunger type sampler can be used to collect samples from drums, surface impoundments, and tanks. Sample volume is at least 0.2 liters and ultimately depends on the size of the sample container (ASTM D 6232).

A plunger type sampler comprises a sample tube, sample line or rod, head section, and plunger and is made of HDPE, PTFE, or glass. A sample jar is connected to the head section. The sample tube is lowered into the liquid to the desired depth. The plunger is engaged into the tube to secure the sample within the tube and the cord or rod is raised to transfer the sample directly into the

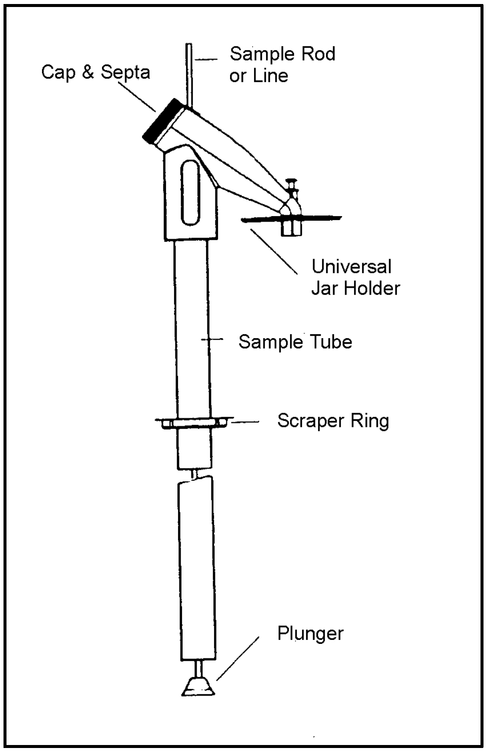

Figure E-25. Plunger type sampler

sampling bottle or jar. The plunger can be pushed back down the sampling tube to reset the sampler.

Advantages

- Easy to use.
- Provides a sealed collection system.
- Relatively inexpensive and available in various lengths.

Limitations

- Care is needed when using a glass sampling tube.
- Decontamination may be difficult, particularly when a glass sampling tube is used.

Other Guidance:

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Practice for Sampling Single or Multilayered Liquids, With or Without Solids, in Drums or Similar Containers*, ASTM D 5743

E.6.5 Settleable Solids Profiler (Sludge Judge)

The settleable solids profiler (Figure E-26), also known as the sludge judge, primarily is used to measure or sample settleable (suspended) solids found in sewage treatment plants, waste settling ponds and impoundments containing waste. It also can be used to sample drums and tanks. It has a sample volume range of 1.3 to 4.0 liters (ASTM D 6232).

The sludge judge is made from clear PVC and has 1-foot-depth markings on its 5-foot-long body sections. It has a check valve on the lower section and a cord on the upper section and is assembled using the threaded connections of the sections to the length needed for the sampling event. The sampler is lowered into the media to allow it to fill. A tug on the cord sets the check valve and it is removed from the sampled material. The level of settleable solids can be measured using the markings. It is emptied by pressing in the protruding pin on the lower end.

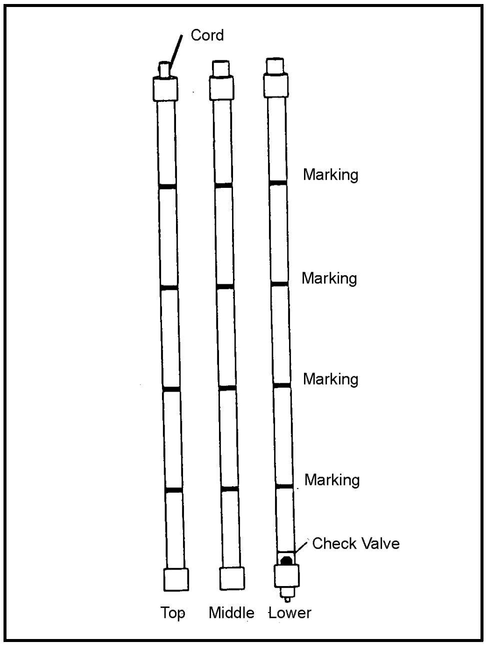

Figure E-26. Settleable solids profiler

Advantages

- Allows measurement of the liquid/settleable solids columns of any length.
- Easy to assemble and use.
- Unbreakable in normal use and reusable.

Limitations

- Suitable for sampling noncaustic liquids only.
- May be difficult to sample high viscosity materials.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232

E.7 Surface Sampling Devices

Surface sampling devices include equipment that by design are limited to sample collection at the surface of material or can sample material of limited depth or width only. You will find summaries for the following surface sampling devices in this section:

E.7.1 Bailer
E.7.2 Dipper
E.7.3 Liquid Grab Sampler
E.7.4 Swing Jar Sampler
E.7.5 Spoons, Scoops, Trowels, and Shovels

E.7.1 Bailer

Bailers (Figure E-27) are designed for obtaining samples of ground water; however, they also can be used to obtain samples of liquids and multi-layered liquid wastes from tanks and surface impoundments. Bailers are not suitable for sampling sludges. The sample volume range is 0.5 to 2 liters (ASTM D 6232).

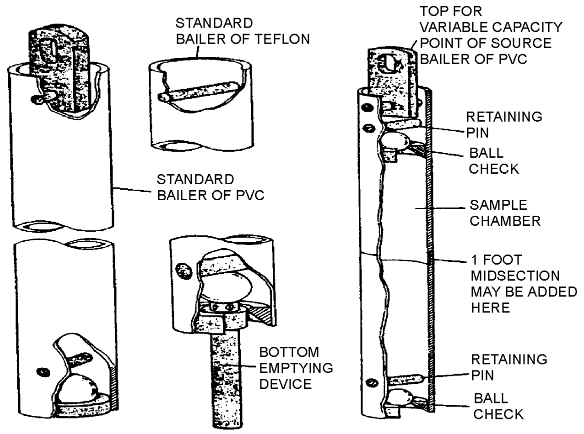

Figure E-27. Bailer

A bailer is a hollow tube with a check valve at the base (open bailer) or valves at both ends (point-source bailer). A bailer can be threaded in the middle so that extension tubes can be added to increase the sampling volume. It can be constructed of stainless steel, PVC, PTFE, or any other suitable material and is available in numerous sizes for use in a variety of well sizes. The bailer is attached to a line and gradually lowered into the sample. As the bailer is lowered, the bottom check valve allows water to flow through the tube. The bailer is then slowly raised to the surface. The weight of the water closes the bottom check valve. A point-source bailer allows sampling at a specific depth. The check valve at the top of the tube limits water or particles from entering the bailer as it is retrieved.

The bailer is emptied either by pouring from the top or by a bottom emptying device. When using a top-emptying bailer, the bailer should be tipped slightly to allow a slow discharge into the sample container to minimize aeration. A bottom-emptying model has controlled flow valves, which is good for collecting samples for volatile organic analysis since agitation of the sample is minimal.

Advantages

- Easy to use, inexpensive, and does not require an external power source.
- Can be constructed of almost any material that is compatible with the parameters of interest.
- Relatively easy to decontaminate between samples. Single-use models are available.
- Bottom-emptying bailers with control valves can be used to obtain samples for volatile compound analysis.

Limitations

- Not designed to obtain samples from specific depths below liquid surface (unless it is a point-source bailer).
- If using a top-emptying bailer, the sample may become aerated if care is not taken during transfer to the sample container.
- May disturb the sample in a water column if it is lowered too rapidly.
- High suspended solids' content or freezing temperatures can impact operation of check valves.
- One of the least preferred devices for obtaining samples of ground water for low concentration analyses due to their imprecision and agitation of the sample (see USEPA 1992a and Puls and Barcelona 1996).

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Guide for Sampling Groundwater Monitoring Wells*, ASTM D 4448

- “Tank Sampling” (USEPA 1994c)

E.7.2 Dipper

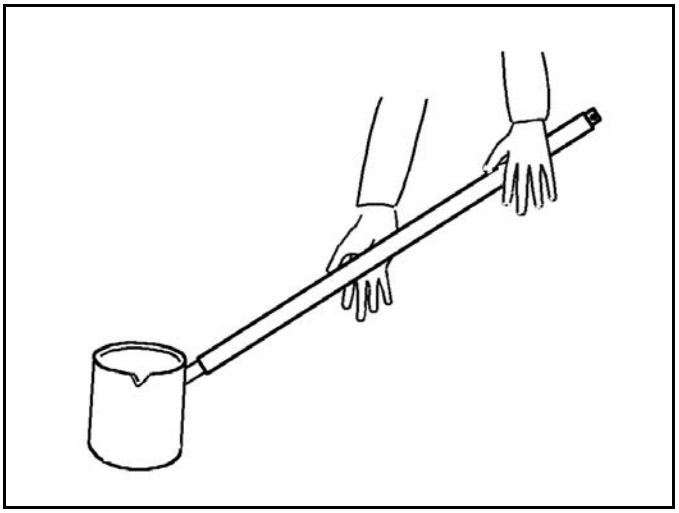

Figure E-28. Dipper

A dipper (Figure E-28) is a type of surface sampling device used to sample surface samples from drums, surface impoundments, tanks, pipes, and point source discharges. Sampling points are shallow (10 inches) and taken at, or just below, the surface. The typical sample volume range is 0.5 to 1.0 liters (ASTM D 6232).

A dipper comprises a glass, metal, or plastic beaker clamped to the end of a two- or three-piece telescoping aluminum or fiberglass pole, which serves as a handle. A dipper may vary in the number of assembled pieces. Some dippers have an adjustable clamp attached to the end of a piece of metal tubing. The tubing forms the handle; the clamp secures the beaker. Another type of dipper is a stainless steel scoop clamped to a movable bracket that is attached to a piece of rigid tube. The scoop may face either toward or away from the person collecting the sample, and the angle of the scoop to the pipe is adjustable. The dipper, when attached to a rigid tube, can reach easily 10 to 13 feet (3 to 4 m) away from the person collecting the samples (ASTM D 6232).

The dipper is used by submerging the beaker end into the material slowly (to minimize surface disturbance). It should be on its side so that the liquid runs into the container without swirling or bubbling. The beaker is filled and rotated up, then brought slowly to the surface. Dippers and their beakers should be compatible with the sampled material.

Advantages

- Inexpensive.
- Easy to construct and adapt to the sampling scenario by modifying the length of the tubing or the type of container.

Limitations

- Not appropriate for sampling subsurface layers or to characterize discrete layers of stratified liquids.
- Can only be used to collect surface samples.

Other Guidance

- *Standard Practice for Sampling with a Dipper or Pond Sampler*, ASTM D 5358
- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities,* ASTM D 6232
- *Standard Practice for Sampling Wastes from Pipes and Other Point Discharges,* ASTM D 5013

E.7.3 Liquid Grab Sampler

A liquid grab sampler (Figure E-29) is a surface sampling device designed to collect samplers at a specific shallow depth beneath the liquid surface. It can be used to collect samples of liquids or slurries from surface impoundments, tanks, and drums. Its sample volume range is from 0.5 to 1.0 liters (ASTM D 6232).

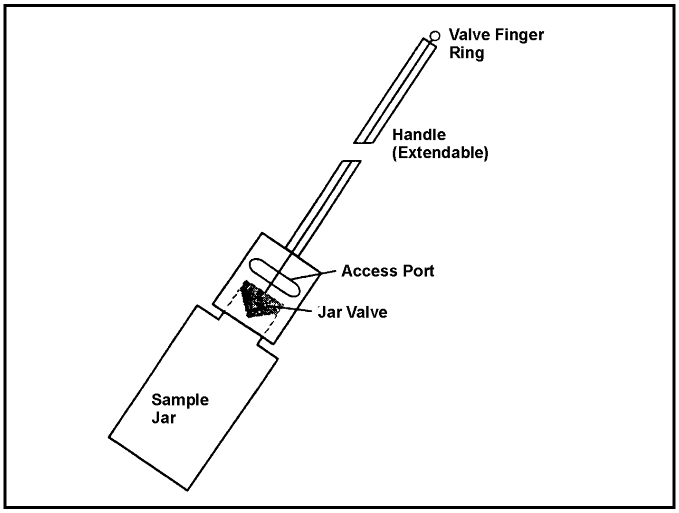

Figure E-29. Liquid grab sampler

The liquid grab sampler is usually made from polypropylene or PTFE with an aluminum or stainless steel handle and stainless steel fittings. The sampling jar is usually made of glass, although plastic jars are available. The jar is threaded into the sampler head assembly, then lowered by the sampler to the desired sampling position beneath the liquid surface. The valve is then opened by pulling up on a finger ring to fill the jar. The valve is closed before retrieving the sample.

Advantages

- Easy to use.
- The sample jar can be capped and used for transport to the laboratory, thus minimizing the loss of volatile organic compounds.
- The closed sampler prevents contaminants in upper layers from compromising the sample.

Limitations

- Care is required to prevent breakage of glass sample jar.
- Materials of construction need to be compatible with the sampled media.

- Cannot be used to collect deep samples.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232

E.7.4 Swing Sampler (Swing Jar Sampler)

The swing jar sampler (Figure E-30) is a surface sampler that may be used to sample liquids, powders, or small solids at distances of up to 12 feet (3.5 m). It can be used to sample many different types of units, including drums, surface impoundments, tanks, pipe/point source discharges, sampling ports, and storage bins. It has a sample volume range of 0.5 to 1.0 liters.

The swing jar sampler is normally used with high density polyethylene sample jars and has an extendable aluminum handle with a pivot at the juncture of the handle and the jar holder. The jar is held in the holder with an adjustable clamp. The pivot allows samples to be collected at different angles.

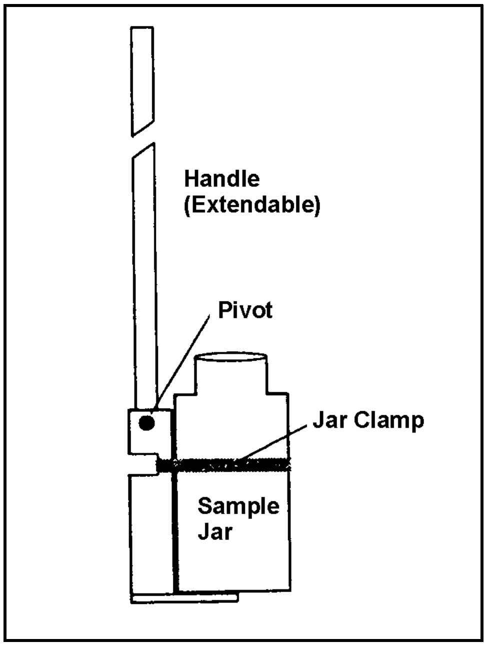

Figure E-30. Swing jar sampler

Advantages

- Easy to use.
- Easily adaptable to samples with jars of different sizes and materials, which can be used to facilitate compatibility with the material to be sampled.
- Can be pivoted to collect samples at different angles.
- Can sample from a wide variety of locations and units.

Limitations

- Cannot collect discrete depth samples.
- Care is required to prevent breakage when using a glass sample jar.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232

E.7.5 Spoons, Scoops, Trowels, and Shovels

Spoons, scoops, trowels, or shovels are types of surface sampling devices used to sample sludge, soil, powder, or solid wastes. The typical sample volume range is 0.1 to 0.6 liters for scoops or trowels and 1.0 to 5.0 Liters for shovels (ASTM D 6232). The typical sample volume for a spoon is 10 to 100 grams (USEPA 1993c).

Spoons, available in stainless steel or PTFE (reusable) or in plastic (disposable), easily sample small volumes of liquid or other waste from the ground or a container.

Scoop samplers provide best results when the material is uniform and may be the only sampler possible for materials containing fragments or chunks. The scoop size should be suitable for the size and quantity of the collected material. Scoops and trowels come in a variety of sizes and materials, although unpainted stainless steel is preferred (ASTM D 6232). Scoops may be attached to an extension, similar to the dipper, in order to reach a particular area. Scoops and trowels are used by digging and rotating the sampler. The scoop is used to remove a sample and transfer it into a sample container.

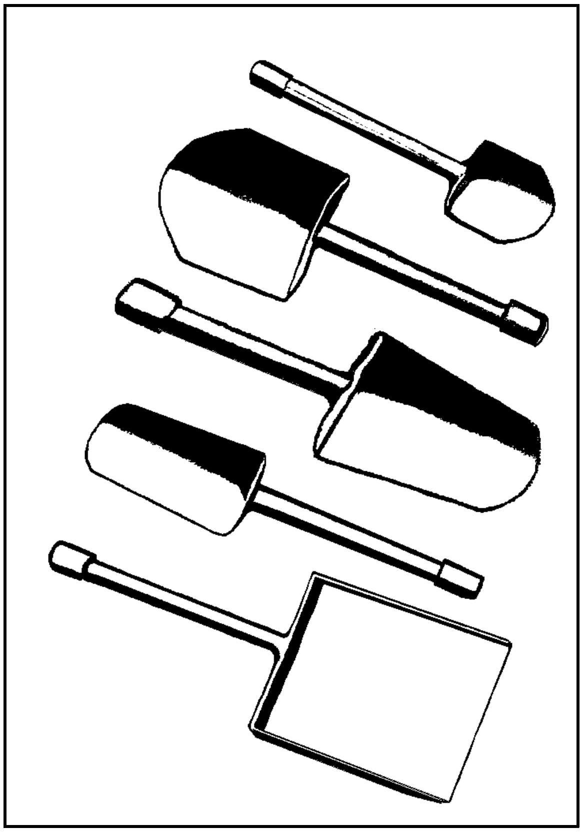

Figure E-31. Scoops

Shovels, usually made from stainless steel or suitable plastic materials, are typically used to collect surface samples or to remove overburden material so that a scoop may remove a sample.

Advantages

- A correctly designed scoop or spatula (i.e., with a flat bottom and vertical sides) is one of the preferred devices for sampling a one-dimensional mass of granular solids (see also Sections 6.3.2.1 and 7.3.3.3).
- Spoons, scoops, trowels, and shovels are reusable, easy to decontaminate, and do not require significant physical strength to use.
- Spoons and scoops are inexpensive and readily available.
- Spoons and scoops are easily transportable and often disposable -- hence, their use can reduce sampling time.
- Shovels are rugged and can be used to sample hard materials.

Limitations

- Spoons, scoops, trowels, and shovels are limited to shallow and surface sampling.
- Shovels may be awkward to handle and cannot be used to easily fill small sample containers.
- Sampling with a spoon, scoop, trowel, or shovel may cause loss of volatile organic compounds through disturbance of the media.
- Spoons, scoops, trowels, and shovels of incorrect design (e.g., with rounded bottoms) can introduce bias by preferentially selecting certain particle sizes.

Other Guidance

- *Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities*, ASTM D 6232
- *Standard Practice for Sampling with a Scoop*, ASTM D 5633
- "Waste Pile Sampling" (USEPA 1994d)
- "Sediment Sampling" (USEPA 1994e).

APPENDIX F

STATISTICAL METHODS

This appendix provides guidance on the statistical analysis of waste testing and environmental monitoring data. You should select the statistical test during the Data Quality Assessment (DQA) phase after you review the data quality objectives, the sampling design, and the characteristics of the data set. See guidance provided in Section 8.

The statistical methods in this appendix are appropriate for use in evaluating sample analysis results when comparing constituent concentrations in a waste or environmental medium to a *fixed standard*. Users of this guidance may have other objectives such as comparing two populations, detecting trends, or characterizing the spatial pattern of contamination. If so, review other guidance or seek assistance from a professional environmental statistician.

Additional Guidance on the Statistical Analysis of Waste Testing and Environmental Monitoring Data

USEPA. 2000d. *Guidance For Data Quality Assessment, EPA QA/G-9*, (QA00 version). EPA/600/R-96/084. Office of Research and Development, Washington, D.C.

Note that not all RCRA standards require the waste handler to use sampling, analysis, and statistical tests to measure compliance. However, if sampling and analysis is used by the waste handler to measure compliance with a RCRA standard, then statistical methods may be used to help quantify uncertainty associated with the decisions made using the data – even where there is no regulatory obligation to do so (see also Sections 2 and 3).

This appendix is divided into subsections that describe the following statistical methods:

F.1 Testing Distributional Assumptions
  F.1.1 Overview and Recommendations
  F.1.2 Shapiro-Wilk Test for Normality ($n \le 50$)

F.2 Confidence Limits for the Mean
  F.2.1 Confidence Limits for the Mean of a Normal Distribution
  F.2.2 Confidence Limits for a Normal Mean When Composite Sampling Is Used
  F.2.3 Confidence Limits for a Lognormal Mean
  F.2.4 Confidence Limits for the Mean of a Non-normal or Unknown Distribution

F.3 Tests for a Proportion or a Percentile
  F.3.1 Parametric Upper Confidence Limits for an Upper Percentile
  F.3.2 Using a Simple Exceedance Rule Method for Determining Compliance With A Fixed Standard

F.4 Treatment of Nondetects
  F.4.1 Recommendations
  F.4.2 Cohen's Adjustment

Table F-1 provides a summary of frequently used statistical equations. See Appendix G for statistical tables used with these methods.

Table F-1. Summary of Basic Statistical Terminology Applicable to Sampling Plans for Solid Waste

Terminology	***Symbol***	***Mathematical Equation***	***Equation No.***
Variable (e.g., barium or endrin)	x	--	--
Individual measurement of variable	x_i	--	--
Simple Random Sampling and Systematic Random Sampling			
Mean of measurements generated from the samples (sample mean)	$\bar{x}$	$\bar{x} = \frac{1}{n}\sum_{i=1}^{n} x_i$ where n = number of sample measurements.	1
Variance of sample	s^2	$s^2 = \frac{1}{n-1}\sum_{i=1}^{n}(x_i - \bar{x})^2$	2
Standard deviation of sample	s	$s = \sqrt{s^2}$	3
Standard error (also standard deviation of the mean)	$s_{\bar{x}}$	$s_{\bar{x}} = \frac{s}{\sqrt{n}}$	4
Approximate number of samples to estimate the mean (financial constraints not considered) (See Section 5.4.1)	n	$n = \frac{(z_{1-\alpha} + z_{1-\beta})^2 s^2}{\Delta^2} + \frac{z_{1-\alpha}^2}{2}$ where the "z" values are obtained from the last row of Table G-1 in Appendix G.	8
Approximate number of samples to test a proportion against a fixed standard (See Section 5.5.1).	n	$n = \left[\frac{z_{1-\beta}\sqrt{GR(1-GR)} + z_{1-\alpha}\sqrt{AL(1-AL)}}{\Delta^2}\right]^2$	15
Number of samples to test a proportion when the decision rule specifies zero nonconforming samples (See Section 5.5.2).	n	$n = \log(\alpha)/\log(p)$ where p equals the proportion of the waste or media exceeded by the largest sample	16

Table F-1. (Continued)

Terminology	*Symbol*	*Mathematical Equation*	*Equation No.*
Stratified Random Sampling (Proportional Allocation)			
Arithmetic mean of the measurements generated from the samples obtained from each hth stratum	$\bar{x}_h$	$\bar{x}_h = \frac{1}{n_h}\sum_{i=1}^{n_h} x_{hi}$ where n_h = number of sample measurements obtained from each hth stratum.	--
Variance of measurements generated from the samples obtained from each hth stratum	s_h^2	$s_h^2 = \frac{1}{n_h - 1}\sum_{i=1}^{n_h}(x_{hi} - x_h)^2$	--
The weighting factor assigned to each hth stratum when stratified random sampling is used	W_h	--	--
Overall sample mean using stratified random sampling	$\bar{x}_{st}$	$\bar{x}_{st} = \sum_{h=1}^{L} W_h \bar{x}_h$	9
Standard error of the mean for a stratified random sample	$s_{\bar{x}_{st}}$	$s_{\bar{x}_{st}} = \sqrt{\sum_{h=1}^{L} W_h^2 \frac{s_h^2}{n_h}}$	10
Total number of samples to collect from a solid waste to estimate the mean using stratified random sampling (proportional allocation)	n	$n = \frac{\left[t_{1-\alpha,df} + t_{1-\beta,df}\right]^2}{\Delta^2}\sum_{h=1}^{L} W_h s_h^2$	11
Degrees of freedom associated with the *t*-quantile in Table G-1, Appendix G, when stratified random sampling is used	df	$df = \left(\sum_{h=1}^{L} W_h s_h^2\right)^2 \Big/ \sum_{h=1}^{L} \frac{W_h^2 s_h^4}{nW_h - 1}$	12

F.1 Testing Distributional Assumptions

F.1.1 Overview and Recommendations

The assumption of normality is very important as it is the basis for many statistical tests. A normal distribution is a reasonable model of the behavior of certain random phenomena and often can be used to approximate other probability distributions. In addition, the Central Limit Theorem and other limit theorems state that as the sample size gets large, some of the sample summary statistics (such as the sample mean) behave as if they are normally distributed variables. As a result, a common assumption associated with parametric tests or statistical models is that the errors associated with data or a model follow a normal distribution.

While assumption of a normal distribution is convenient for statistical testing purposes, it is not always appropriate. Sometimes data are highly skewed. In environmental applications, it is not unusual to encounter data that exhibit a lognormal distribution in which the *natural* logarithms of the data exhibit a normal distribution. Statistical tests can be used to verify the assumption of normality or lognormality, but the conclusion of lognormality should not be based on the outcome of a statistical test alone. There are several physical phenomena that can cause the underlying distribution to appear lognormal when in fact it is not. For example, Singh, et al. (1997) note that the presence of a relatively small highly contaminated area in an otherwise uncontaminated area can cause sampling results to indicate a lognormal distribution. In such a situation, it may be more appropriate to treat the areas as two separate decision units or use a stratified sampling design. In other cases, sampling bias may cause a population to appear lognormal. For example, analytical results could be skewed if highly concentrated portions of the waste are over- or under-represented by the sampling procedure.

There are many methods available for verifying the assumption of normality ranging from simple to complex. This guidance recommends use of the Shapiro-Wilk test for normality. Use of the test is appropriate when the number of samples (n) is 50 or less. For n greater than 50, an alternative test for normality should be used. One alternative presented in EPA's QA/G-9 guidance (USEPA 2000d) and the DataQUEST software (USEPA 1997b) is Filliben's Statistic (Filliben 1975). Refer to EPA's QA/G-9 (USEPA 2000d) guidance or EPA's statistical guidance for ground-water monitoring data (USEPA 1989b and 1992b) for other graphical and statistical goodness-of-fit tests.

F.1.2 Shapiro-Wilk Test for Normality ($n \leq 50$)

Purpose and Background

This section provides the method for performing the Shapiro-Wilk test for normality. The test is easily performed using statistical software such as EPA's DataQUEST freeware (USEPA 1997b); however, the test also can be performed manually, as described here.

The Shapiro-Wilk test is recommended as a superior method for testing normality of the data. It is based on the premise that if the data are normally distributed, the ordered values should be highly correlated with corresponding quantiles (z-scores) taken from a normal distribution (Shapiro and Wilk 1965). In particular, the Shapiro-Wilk test gives substantial weight to evidence of non-normality in the tails of a distribution, where the robustness of statistical tests based on the normality assumption is most severely affected.

The Shapiro-Wilk test statistic (W) will tend to be large when a probability plot of the data indicates a nearly straight line. Only when the plotted data show significant bends or curves will the test statistic be small. The Shapiro-Wilk test is considered to be one of the very best tests of normality available (Miller 1986, Madansky 1988).

Procedure

Step 1. Order the data from least to greatest, labeling the observations as x_i for $i = 1 \ldots n$. Using the notation $x_{(j)}$, let the $j\text{th}$ order statistic from any data set represent the $j\text{th}$ smallest value.

Step 2. Compute the differences $\left[x_{(n-i+1)} - x_{(i)}\right]$ for each $i = 1 \ldots n$. Then determine k as the greatest integer less than or equal to $(n/2)$.

Step 3. Use Table G-4 in Appendix G to determine the Shapiro-Wilk coefficients, a_{n-i+1}, for $i = 1 \ldots n$. Note that while these coefficients depend only on the sample size (n), the order of the coefficients must be preserved when used in step 4 below. The coefficients can be determined for any sample size from n = 3 up to n = 50.

Step 4. Compute the quantity b given by the following formula:

$$b = \sum_{i=1}^{k} b_i = \sum_{i=1}^{k} a_{n-i+1}(x_{(n-i+1)} - x_{(i)}) \qquad \textbf{Equation F.1}$$

Note that the values b_i are simply intermediate quantities represented by the terms in the sum of the right-hand expression in the above equation.

Step 5. Calculate the standard deviation (s) of the data set. Then compute the Shapiro-Wilk test statistic using the following formula:

$$W = \left[\frac{b}{s\sqrt{n-1}}\right]^2 \qquad \textbf{Equation F.2}$$

Step 6. Given the significance level (α) of the test (for example, 0.01 or 0.05), determine the critical point of the Shapiro-Wilk test with n observations using Table G-5 in Appendix G. Compare the Shapiro-Wilk statistic (W) against the critical point (w_c). If the test statistic exceeds the critical point, accept normality as a reasonable model for the underlying population; however, if $W < w_c$, reject the null hypothesis of normality at the α-level and decide that another distributional model would provide a better fit.

An example calculation of the Shapiro-Wilk test for normality is presented in Box F.1.

Box F.1. Example Calculation of the Shapiro-Wilk Test for Normality

Use the Shapiro-Wilk test for normality to determine whether the following data set, representing the total concentration of nickel in a solid waste, follows a normal distribution: 58.8, 19, 39, 3.1, 1, 81.5, 151, 942, 262, 331, 27, 85.6, 56, 14, 21.4, 10, 8.7, 64.4, 578, and 637.

Solution

Step 1. Order the data from smallest to largest and list, as in Table F-2. Also list the data in reverse order alongside the first column.

Step 2. Compute the differences $\left[x_{(n-i+1)} - x_{(i)}\right]$ in column 4 of the table by subtracting column 2 from column 3. Because the total number of samples is $n = 20$, the largest integer less than or equal to $(n/2)$ is $k = 10$.

Step 3. Look up the coefficients a_{n-i+1} from Table G-4 in Appendix G and list in column 4.

Step 4. Multiply the differences in column 4 by the coefficients in column 5 and add the first k products (b_i) to get quantity b_i, using Equation F.1.

$$b = \left[.4734(941.0)+.3211(633.9) + \cdots .0140(2.8)\right] = 932.88$$

Step 5. Compute the standard deviation of the sample, S = 259.72, then use Equation F.2 to calculate the Shapiro-Wilk test statistic:

$$W = \left[\frac{932.88}{259.72\sqrt{19}}\right]^2 = 0.679$$

Step 6. Use Table G-5 in Appendix G to determine the .01-level critical point for the Shapiro-Wilk test when n = 20. This gives w_c = 0.868. Then, compare the observed value of W = 0.679 to the 1-percent critical point. Since W < 0.868, the sample shows significant evidence of non-normality by the Shapiro-Wilk test. The data should be transformed using natural logs and rechecked using the Shapiro-Wilk test before proceeding with further statistical analysis.

Table F-2. Example Calculation of the Shapiro-Wilk Test (see example in Box F.1)

i	$x_{(i)}$	$x_{(n-i+1)}$	$x_{(n-i+1)} - x_{(i)}$	a_{n-i+1}	b_i
1	1	942	941	0.4734	445.47
2	3.1	637	634	0.3211	203.55
3	8.7	578	569	0.2565	146.03
4	10	331	321	0.2085	66.93
5	14	262	248	0.1686	41.81
6	19	151	132	0.1334	17.61
7	21.4	85.6	64.2	0.1013	6.5
8	27	81.5	54.5	0.0711	3.87
9	39	64.4	25.4	0.0422	1.07
10	56	58.8	2.8	0.0140	0.04
11	58.8	56	–2.8		*b* = 932.88
12	64.4	39	–25.4		
13	81.5	27	–54.5		
14	85.6	21.4	–64.2		
15	151	19	–132.0		
16	262	14	–248.0		
17	331	10	–321.0		
18	578	8.7	–569.3		
19	637	3.1	–633.9		
20	942	1	–941.0		

F.2 Confidence Limits for the Mean

When a fixed standard or limit is meant to represent an average or mean concentration level, attainment of the standard can be measured using a confidence limit on the mean. A confidence limit is then compared with the fixed compliance limit. Under the null hypothesis that the mean concentration in the waste exceeds the standard unless proven otherwise, statistically significant evidence of compliance with the standard is shown if and only if the entire confidence interval lies below the standard. By implication, the key test then involves comparing the upper confidence limit (UCL) to the standard. In other words, the entire confidence interval must lie below the standard for the waste to be compliant with the standard. If the UCL exceeds the regulatory limit, on the other hand, we cannot conclude the mean concentration is below the standard.

F.2.1 Confidence Limits for the Mean of a Normal Distribution

Requirements and Assumptions

Confidence intervals for the mean of a normal distribution should be constructed only if the data pass a test of approximate normality or at least are reasonably symmetric. It is strongly recommended that a confidence interval not be constructed with less than four measurements, though the actual number of samples should be determined as part of the planning process. The reason for this is two-fold: (1) the formula for a normal-based confidence interval on the mean involves calculation of the sample standard deviation (s), which is used as an estimate of the underlying population standard deviation (this estimate may not be particularly accurate when the sample size is smaller than four), and (2) the confidence interval formula also involves a Student's *t*-quantile based on $n - 1$ degrees of freedom, where n equals the number of samples used in the calculation (see Table G-1 in Appendix G). When n is quite small, the *t*-quantile will be relatively large, leading to a much wider confidence interval than would be expected with a larger n. For example, at a 90-percent confidence level, the appropriate *t*-quantile would be $t = 3.078$ for $n = 2$, $t = 1.638$ for $n = 4$, and $t = 1.415$ for $n = 8$.

Procedure

Step 1. Check the n sample concentrations for normality. If the normal model is acceptable, calculate the mean ($\bar{x}$) and standard deviation (s) of the data set. If the lognormal model provides a better fit, see Section F.2.3.

Step 2. Given the desired level of confidence, ($1-\alpha$), calculate the upper confidence limit as follows:

$$UCL = \bar{x} + t_{1-\alpha,df}\,\frac{s}{\sqrt{n}} \qquad \textbf{Equation F.3}$$

where $t_{1-\alpha,df}$ is obtained from a Student's *t*-table (Table G-1) with the appropriate degrees of freedom. If simple random or systematic sampling is used, then $df = n-1$.

If stratified random sampling is used, calculate the UCL as follows:

$$UCL_{st} = \bar{x}_{st} + t_{1-\alpha,df}\, s_{\bar{x}_{st}} \qquad \textbf{Equation F.4}$$

where $\bar{x}_{st}$ is the overall mean from Equation 8, the df is obtained from Equation 11, and the standard error ($s_{\bar{x}_{st}}$) is obtained from Equation 9 (see also Table F-1 for these equations).

Step 3. Compare the UCL calculated in Step 2 to the fixed standard. If the UCL is less than the standard, then you can conclude, with 100($1-\alpha$)% confidence, that the mean concentration of the constituent of concern is less than the standard. If, however, the upper confidence bound is greater than the standard, then there is not sufficient evidence that the mean is less than the standard.

An example calculation of the UCL on the mean is provided in Box F.2.

Box F.2. Example Calculation of the UCL for a Normal Mean

A generator obtains ten samples of waste to demonstrate that the waste qualifies for the comparable fuels exclusion under 40 CFR 261.38. The samples are obtained using a simple random sampling design. Analysis of the samples for lead generated the following results: 16, 17.5, 21, 22, 23, 24, 24.5, 27, 31, and 38 ppm. The regulation requires comparison of a 95% UCL on the mean to the specification level. The specification level is 31 ppm.

Solution

Step 1. Using the Shapiro-Wilk test, we confirmed that the normal model is acceptable. The mean is calculated as 24.4 ppm and the standard deviation as 6.44 ppm.

Step 2. The RCRA regulations at 40 CFR 261.38(c)(8)(iii)(A) require that the determination be made with a level of confidence, 100($1-\alpha$)%, of 95 percent. We turn to Table G-1 (Appendix G) and find the Student's *t* value is 1.833 for $n-1=9$ degrees of freedom. The UCL is calculated as follows:

$$UCL = 24.4 + 1.833\frac{6.44}{\sqrt{10}} = 28.1 \approx 28$$

Step 3. We compare the limit calculated in step 2 to the fixed standard. Because the UCL (28 ppm) is less than the regulatory level (31 ppm), we can conclude with at least 95-percent confidence that the mean concentration of the constituent in the waste is less than 31 ppm.

F.2.2 Confidence Limits for a Normal Mean When Composite Sampling Is Used

If a composite sampling strategy has been employed to obtain a more precise estimate of the mean, confidence limits can be calculated from the analytical results using the same procedure outlined above in Section F.2.1, except that n represents the number of composite samples and s represents the standard deviation of the n composite samples.

F.2.3 Confidence Limits for a Lognormal Mean

If the results of a test for normality indicate the data set may have a lognormal distribution, and a confidence limit on the mean is desired, then a special approach is required. It is *not* correct to simply transform the data to the log scale, calculate a normal-based mean and confidence interval on the logged data, and transform the results back to the original scale. It is a common mistake to do so. Invariably, a transformation bias will be introduced and the approach will underestimate the mean and UCL. In fact, the procedure just described actually produces a confidence interval around the *median* of a lognormal population rather than the higher-valued *mean*.

To calculate a UCL on the mean for data that exhibit a lognormal distribution, this guidance recommends use of a procedure developed by Land (1971, 1975); however, as noted below, Land's procedure should be used with caution because it relies heavily on the lognormal assumption, and if that assumption is not true, the results may be substantially biased.

Requirements and Assumptions

Confidence intervals for the mean of a lognormal distribution should be constructed only if the data pass a test of approximate normality *on the log-scale*. While many environmental populations tend to follow the lognormal distribution, it is usually wisest to first test the data for normality on the original scale. If such a test fails, the data can then be transformed to the log-scale and retested.

Cautionary Note: Even if a data set passes a test for normality on the log scale, do not proceed with calculation of the confidence limits using Land's procedure until you have considered the following:

- The skewness of the data set may be due to biased sampling, mixed distributions of multiple populations, or outliers, and not necessarily due to lognormally distributed data (see Singh, et al. 1997). Review the sampling approach, the physical characteristics of the waste or media, and recheck any unusually high values before computing the confidence limits. Where there is spatial clustering of sample data, declustering and distribution weighting techniques (Myers 1997) may also be appropriate.

- If the number of samples (n) is small, the confidence interval obtained by Land's procedure could be remarkably wide. Singh, et al. (1997) have recommended that Land's procedure not be used for cases in which the number of samples is less than 30. They argue that in many cases the resulting UCL will be an order of magnitude larger than the maximum observed data value. Even higher values for the UCL could be generated if the coefficient of variation (CV or the standard deviation divided by the mean) is greater than 1.

If the lognormal distribution is the best fit, and the number of samples (n) is small, then Land's method (provided below) can still be used, though a "penalty" will be paid for the small sample size. If the number of samples is small and the distribution is skewed to the right, one of the following alternative approaches should be considered: (1) Simply treat the data set as if the parent distribution were normal and use the parametric Student-*t* method to calculate confidence limits using the *untransformed* (original scale) data (as described in Section F.2.1). If, however, this normal theory approach is used with highly skewed data, the actual confidence level achieved by the test will be less than that desired (Porter, et al. 1997); (2) UCLs on the mean could be constructed using procedures such as the "bootstrap" or the "jackknife," as recommended by Singh, et al. (1997) (see Section F.2.4).

The approach for Land's "H-statistic" method is given below:

Procedure

Step 1. Test the data for normality on the log-scale. After determining that the lognormal distribution is a good fit, transform the data via logarithms (the natural log is used) and denote the transformed measurements by y_i.

Step 2. Compute the sample mean and the standard deviation (s_y) from the log-scale measurements.

Step 3. Obtain Land's bias-correction factor(s) ($H_{1-\alpha}$) from Table G-6 in Appendix G, where the correct factor depends on the sample size (n), the log-scale sample standard deviation (s_y), and the desired confidence level ($1-\alpha$).[1]

Step 4. Plug all these factors into the equations given below for the UCL.

$$UCL_{1-\alpha} = \exp\left(\bar{y} + .5 s_y^2 + \frac{s_y H_{1-\alpha}}{\sqrt{n-1}} \right)$$

Equation F.5

Step 5. Compare the UCL against the fixed standard. If the UCL is less than the standard, then you can conclude with 100($1-\alpha$)% confidence that the mean concentration of the constituent of concern is less than the standard. If, however, the upper confidence bound is greater than the standard, then there is not sufficient evidence that the mean is less than the standard.

An example calculation of the UCL on a lognormal mean is given in Box F.3.

Box F.3: Example Calculation of the UCL on a Lognormal Mean

This example is modified after an example provided in *Supplemental Guidance to RAGS: Calculating the Concentration Term* (USEPA 1992a).

The concentration of lead (total in mg/Kg) in 31 soil samples obtained using a simple random sampling design are: 1, 3, 13, 14, 18, 20, 21, 36, 37, 41, 42, 45, 48, 59, 60, 110, 110, 111, 111, 136, 137, 140, 141, 160, 161, 200, 201, 230, 400, 1300, and 1400. Using these data, calculate a 90% UCL on the mean.

Solution

Step 1. Using the Shapiro-Wilk test, the natural logarithms of the data set are shown to exhibit a normal distribution. The data are then transformed to natural logs.

Step 2. The mean of logged data is $\bar{y} = 4.397$. The standard deviation is $s_y = 1.509$.

Step 3. The bias-correction factor ($H_{1-\alpha} = 2.282$) is obtained from Table G-6 for $n = 31$ and a confidence level of 90 percent .

Step 4. Plug the factors into the equation for the upper (UCL) confidence limit.

$$\begin{aligned} UCL_{1-\alpha} &= \exp\left(4.222 + 0.5(1.509)^2 + \frac{1.509(2.282)}{\sqrt{31-1}} \right) \\ &= \exp(5.989) = 399 \,\mathrm{mg/kg} \end{aligned}$$

Step 5. The 90-percent UCL on the mean is 399 mg/kg.

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[1] For a more extensive tabulation of Land's factors, see Land (1975) or Tables A10 through A13 in Gilbert (1987).

F.2.4 Confidence Limits for the Mean of a Non-normal or Unknown Distribution

If the assumption of a normal or lognormal distribution cannot be justified, then you may construct a UCL on the mean using one of several alternative methods described in this section.

Bootstrap or Jackknife Methods: Bootstrap and jackknife procedures, as discussed by Efron (1981) and Miller (1974), typically are nonparametric statistical techniques which can be used to reduce the bias of point estimates and construct approximate confidence intervals for parameters such as the population mean. These procedures require no assumptions regarding the statistical distribution (e.g., normal or lognormal) for the underlying population.

Using a computer, the bootstrap method randomly samples n values with replacement from the original set of n random observations. For each bootstrap sample, the mean (or some other statistic) is calculated. This process of “resampling” is repeated hundreds or perhaps thousands of times and the multiple estimates of the mean are used to define the confidence limits on the mean. The jackknife approximates the bootstrap. Rather than resampling randomly from the entire sample like the bootstrap does, the jackknife takes the entire sample except for one value, and then calculates the statistic of interest. It repeats the process, each time leaving out a different value, and each time recalculating the test statistic.

Both the bootstrap and the jackknife methods require a great deal of computer power, and, historically have not been widely adopted by environmental statisticians (Singh, et al. 1997). However, with advances in computer power and availability of software, computationally intensive statistical procedures have become more practical and accessible. Users of this guidance interested in applying a “resampling” method such as the bootstrap or jackknife should check the capabilities of available software packages and consult with a professional statistician on the correct use and application of the procedures.

Nonparametric Confidence Limits: If the data are not assumed to follow a particular distribution, then it may not be possible to calculate a UCL on the mean using normal theory techniques. If, however, the data are non-normal but approximately *symmetric*, a nonparametric UCL on the *median* (or the 50th percentile) may serve as a reasonable alternative to calculation of a parametric UCL on the mean. *One severe limitation of this approach is that it involves changing the parameter of interest (as determined in the DQO Process) from the mean to the median, potentially biasing the result if the distribution of the data is not symmetric.* Accordingly, the procedure should be used with caution.

Lookup tables can be used to determine the confidence limits on the median (50th percentile). For example, see Conover (1999, Table A3) or Gilbert (1987, Table A14). In general, when the sample size is very small (e.g., less than about nine or ten samples) and the required level of confidence is high (e.g., 95 to 99 percent), the tables will designate the maximum value in the data set as the upper confidence limit. Conover (1999, page 143) gives a large sample approximation for a confidence interval on a proportion (quantile). Methods also are given in Gilbert (1987, page 173), Hahn and Meeker (1991, page 83), and USEPA (1992i, page 5-30).

F.3 Tests for a Proportion or Percentile

Some RCRA standards represent concentrations that should rarely or never be exceeded for the waste or media to comply with the standard. To measure compliance with such a standard, a waste handler may want to know with some specified level of confidence that a high proportion of the waste complies with the standard (or conversely, that at most only a small proportion of all possible samples could exceed the standard). Two approaches are given for measuring compliance with such a standard:

1. Under the assumption of a normal distribution, use a parametric UCL on a percentile to demonstrate that the true pth percentile (x_p) concentration in the set of all possible samples is less than the concentration standard. The method is given below in **Section F.3.1**.
2. By far, the simplest method for testing proportions is to use an "exceedance rule" in which the proportion of the population with concentrations less than the standard can be estimated based on the total number of sample values and the number of those (if any) that exceed the standard. The exceedance rule method is given below in **Section F.3.2**.

If the number of samples is relatively large, then a "one-sample proportion test" also can be used to test a proportion against a fixed standard. The one-sample proportion test is described in Section 3.2.2.1 in *Guidance for Data Quality Assessment, EPA QA/G-9 (QA00 Update)* (USEPA 2000d).

F.3.1 Parametric Upper Confidence Limits for an Upper Percentile

If the study objective is to demonstrate that the true pth percentile (x_p) concentration in the set of all possible samples (of a given sample support) is less than the applicable standard or Action Level, then a UCL on the upper percentile can be used to determine attainment of the standard.

Requirements and Assumptions

The formulas for constructing parametric UCL on an upper percentile assume that the data are at least approximately normally distributed. Therefore, such a limit should be constructed only if the data pass a test of normality. If the data are best fit by a lognormal distribution instead, the observations should first be transformed to the log-scale. Unlike confidence limits for a lognormal mean, no special equations are required to construct similar limits on an upper percentile. The same formula used when the data are normally distributed can be applied to the log-scale data. The only additional step is that the confidence interval limits must be re-exponentiated before comparing them against the regulatory standard.

It is strongly recommended that a confidence limit not be constructed with less than four measurements, and preferably more (the actual number, however, should be determined during Step Seven of the DQO Process). There are three reasons for this: (1) the formula for a normal-based confidence interval on an upper percentile involves calculation of the sample standard deviation, s, which is used as an estimate of the underlying population standard deviation. This estimate may not be accurate when fewer than four samples are used. (2) The confidence interval formula also involves a special factor κ ("kappa"), which depends on both the desired confidence level ($1-\alpha$) and the number of samples, n, used in the calculation. When n is quite small, the κ factor is more extreme, leading to a much wider confidence interval than would be expected with a larger n. For example, at a confidence level of 90 percent, the appropriate κ factor for an upper one-sided limit on the 99th percentile is κ = 18.50 when n = 2, κ = 5.438 when n = 4, and κ = 3.783 when n = 8. (3) The third reason is that the power of the test for normality or lognormality is very low with a small number of samples.

Procedure

Step 1. First test the data for normality on the original scale. If a test of normality is passed, calculate the limit on the raw measurements. If the data violate the assumption of normality, but pass a test of lognormality, calculate the limit using the log-scale data.

Step 2. If the data are normal, compute the mean and standard deviation of the raw data. If the data are consistent with lognormality instead, compute the mean and standard deviation after first transforming the data to the log-scale.

Step 3. Given the percentile (p) being estimated, the sample size (n), and the desired confidence level ($1-\alpha$), use Table G-2 (in Appendix G) to determine the κ factor(s) needed to construct the appropriate UCL. A one-sided upper confidence bound is then computed with the formula

$$UL_{1-\alpha}(x_p) = \bar{x} + s \cdot \kappa_{1-\alpha,p} \quad \textbf{Equation F.6}$$

where $\kappa_{1-\alpha,p}$ is the upper $1-\alpha$ factor for the pth percentile with n sample measurements.

Again, if the data are lognormal instead of normal, the same formula would be used but with the log-scale mean and standard deviation substituted for the raw-scale values. Then the limit must be exponentiated to get the final upper confidence bound, as in the following formula for an upper bound with $(1-\alpha)100\%$ confidence:

$$UL_{1-\alpha}(x_p) = \exp\left[\bar{y} + s_y \cdot \kappa_{1-\alpha,p}\right] \quad \textbf{Equation F.7}$$

Step 4. Compare the upper $(1-\alpha)100\%$ confidence bound against the fixed standard. If the upper limit exceeds the standard, then the standard is not met.

An example calculation of the UCL on a percentile is given in Box F.4.

Box F.4. Example Calculation of a UCL on an Upper Percentile To Classify a Solid Waste

A secondary lead smelter produces a slag that under some operating conditions exhibits the Toxicity Characteristic (TC) for lead. The facility owner needs to classify a batch of waste as either hazardous or nonhazardous at the point of waste generation. During the planning process, the owner determined based on previous sampling studies that the constituent of interest is lead, TCLP results for lead tend to exhibit a normal distribution, and a sample size of ten 200-gram samples (not including QC samples) should satisfy the study objectives. The TC regulatory level for lead is 5 mg/L. The owner wants to determine, with 90-percent confidence, whether a large proportion (e.g., at least 95 percent) of all possible samples of the waste will be below the regulatory limit.

At the point of waste generation, the facility representative takes a series of systematic samples of the waste. The following sample analysis results were generated for ten samples analyzed for lead via the TCLP and SW-846 Method 6010B: <0.5, 0.55, 0.60, 0.80, 0.90, 1.00, 1.50, 1.80, 2.00, and 3.00 mg/L.

Calculate a 90-percent upper confidence limit on the 95th percentile.

Solution

Step 1. Based on the shape of the histogram and normal probability plot, the data were judged to exhibit a normal distribution. Therefore, we proceed with the calculation on the original (untransformed) scale.

Step 2. One value (10% of the measurements) is reported below the quantitation limit of 0.5 mg/L so we replace that value with half the quantitation limit (0.25 mg/L) (see also Section F.4). The mean and standard deviation of the data set are then calculated as $\bar{x} = 1.24$ mg/L and $s = 0.836$.

Step 3. Use Table G-2 (in Appendix G) to determine the κ factor for $n = 10$ needed to construct a 90-percent UCL on the 95th percentile. The table indicates $\kappa = 2.568$. Plug $\bar{x}$, s, and κ into Equation F.6, as follows:

$$UL_{0.90}(x_{0.95}) = 1.24 + (0.836)(2.568) = 3.39 \approx 3.4 \text{mg / L}$$

Step 4. All of the sample analysis results are less than the TC regulatory limit of 5 mg/L TCLP for lead, and the owner concludes that the waste is a nonhazardous waste under RCRA. The owner also can conclude with at least 90-percent confidence that at least 95 percent of all possible sample analysis results representing the batch of waste in the roll-off bin are nonhazardous.

F.3.2 Using a Simple Exceedance Rule Method for Determining Compliance With A Fixed Standard

Some RCRA standards represent concentration limits that should never or rarely be exceeded or waste properties that should never or rarely be exhibited for the waste to comply with the standard. One of the simplest nonparametric methods for determining compliance with such a standard is to use an “exceedance rule” (USEPA 1989a). To apply this method, simply require that a number of samples be acquired and that zero or a small number (e.g., one) of the concentration measurements be allowed to exceed the standard. This kind of rule is easy to implement and evaluate once the data are collected. It only requires specification of a number of samples and the number of exceedances allowed (usually zero, for example, for compliance with the LDR concentration level treatment standards). Alternately, one can specify the statistical performance criteria in advance and then determine the number of samples required.

Requirements and Assumptions for Use of an Exceedance Rule

The method given here is a simple nonparametric method and requires only the ability to identify the number of samples in the data set and whether each sample analysis result complies with the applicable standard or does not comply with the standard. Unfortunately, this ease of use comes with a price. Compared to parametric methods that assume underlying normality or lognormality of the data, the nonparametric method given here requires significantly more samples to achieve the same level of confidence.

Procedure

Step 1: Specify the degree of confidence desired, $100(1-\alpha)\%$, and the proportion (p) of the population that must comply with the standard.

Step 2: If the decision rule permits no exceedance of the standard for any single sample in a set of samples, then obtain and analyze the number of samples (n) indicated in Table G-3a in Appendix G.

If the decision rule permits a single exceedance of the standard in a set of samples, then obtain and analyze the number of samples (n) indicated in Table G-3b in Appendix G.

Step 3: Based on the number of samples obtained and the statistical performance required, determine whether the applicable standard has been attained.

An example application of the exceedance rule is Box F.5.

Box F.5: Example Application of a Simple Exceedance Rule

A facility has treated nonwastewater F003 solvent waste containing carbon disulfide to attain the LDR UTS. Samples of the treatment residue are obtained systematically as the waste treatment is completed. The treater wants to have at least 90% confidence that at least 90% of the batch of treated waste attains the standard. To comply with the LDR regulations, no samples can exceed the UTS. TCLP analyses for carbon disulfide in the treated waste are required to measure compliance with the treatment standard of 4.8 mg/L TCLP.

From **Table G-3a** we find that for a confidence level ($1-\alpha$) of .90 (or 90%) and a proportion of .90, at least 22 samples are required. All sample analysis results must be less than or equal to the UTS of 4.8 mg/L TCLP for the statistical performance criteria to be achieved.

If only 9 samples are obtained (with all sample analysis results less than or equal to the standard), what level of confidence can the treater have that at least 90-percent (or p = 0.90) of all possible samples drawn from the waste meet the treatment standard?

From **Table G-3a** we find for p = 0.90 and n = 9, $1-\alpha$ = 0.60. Therefore, the $100(1-\alpha)\%$ confidence level equals only 60 percent.

F.4 Treatment of Nondetects in Statistical Tests

Data generated from chemical analysis may fall below a limit of detection of the analytical procedure. These measurement data generally are described as “nondetects”, (rather than as zero or not present) and the appropriate limit of detection - such as a quantitation limit - usually is reported. Data sets that include both detected and nondetected results are called “censored” data in the statistical literature.

If a relatively small proportion of the data are reported below detection limit values, replacing the nondetects with a small number (between zero and the detection limit) and proceeding with the usual analysis may be satisfactory. For moderate amounts of data below the detection limit, a more detailed adjustment is appropriate. In situations in which relatively large amounts of data below the detection limit exist, one may need only to consider whether the chemical was detected as above some level or not.

F.4.1 Recommendations

If no more than approximately 15 percent of the sample analysis results are nondetect for a given constituent, then the results of parametric statistical tests will not be substantially affected if nondetects are replaced by half their detection limits (USEPA 1992b).[2] When more than approximately 15 percent of the samples are nondetect, however, the handling of nondetects is more crucial to the outcome of statistical procedures. Indeed, simple substitution methods tend to perform poorly in statistical tests when the nondetect percentage is substantial (Gilliom and Helsel 1986). If the percentage of nondetects is between approximately 15 percent and 50 percent, we recommend use of Cohen’s Adjustment (see method below).

The conditions for use of Cohen's method, however, are limited (see method given below) and numerous alternative techniques for imputing left-censored data should be considered if the conditions for use of Cohen’s method do not apply. Other methods available include iterative techniques, regression on order statistics (ROS) methods, bias-corrected maximum likelihood estimator (MLE), restricted MLE, modified probability plotting, Winsorization, and lognormalized statistics (EPA Delta log). A modified probability plotting method called Helsel's Robust Method (Helsel 1990) is a popular method that should be considered. Most of the above methods can be performed using publicly available software entitled UnCensor© v. 4.0 (Newman et al. 1995). Although EPA’s Office of Solid Waste has not reviewed or tested this software, users of this guidance may be interested in investigating its use.

If the percentage of nondetects is greater than 50 percent, then the regression on order statistics method or Helsel’s Robust Method should be considered. As an alternative, EPA’s *Guidance for Data Quality Assessment EPA QA/G-9* (USEPA 2000d) suggests the use of a test for proportions when the percentage of nondetects is in the range of greater than 50 percent to 90 percent.

This guidance does not advocate a specific method for imputing or replacing values that lie

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[2] Additional experience and research for EPA supporting development of guidance on the statistical analysis of ground-water monitoring data indicates that if the percentage of nondetects is as high as 20 to 25 percent, the results of parametric statistical tests may not be substantially affected if the nondetects are replaced with half their detection limits (Cameron 1999).

below the limit of detection, however, whichever method is selected should be adequately supported. Table F-3 provides a summary of approaches for handling nondetects in statistical intervals.

Table F-3. Guidance for Handling Nondetects In Statistical Intervals

Percentage of Data Reported as "Nondetect"	*Recommended Treatment of Data Set*
< 15%	Replace nondetects with DL/2
15% to 50%	Cohen's adjustment, regression order statistics, or Helsel's Robust Method
> 50%	Regression on order statistics, Helsel's Robust Method, or a test for proportions

Even with a small proportion of nondetects, care should be taken when choosing which value should be used as the "detection limit". There are important differences between the method detection limit and the quantitation limit (QL) in characterizing "nondetect" concentrations. Many nondetects are characterized by analytical laboratories with one of three data qualifier flags: "U," "J," or "E." Samples with a "U" data qualifier represent "undetected" measurements, meaning that the signal characteristic of that analyte could not be observed or distinguished from "background noise" during lab analysis. Inorganic samples with an "E" flag and organic samples with a "J" flag may or may not be reported with an estimated concentration. If no concentration estimate is reported, these samples represent "detected but not quantified" measurements. In this case, the actual concentration is assumed to be positive, falling somewhere between zero and the QL. Because the actual concentration is unknown, the suggested substitution for parametric statistical procedures is to replace each nondetect qualified with an "E" or "J" with one-half the QL. Note, however, that "E" and "J" samples reported *with* estimated concentrations should be treated, for statistical purposes, as valid measurements. In other words, substitution of one-half the QL is *not recommended* for samples for which an estimated concentration is provided.

As a general rule, nondetect concentrations should not be assumed to be bounded above by the MDL. The MDL is usually estimated on the basis of ideal laboratory conditions with analyte samples that may or may not account for matrix or other interferences encountered when analyzing specific, actual field samples. For this reason, the QL typically should be taken as the most reasonable upper bound for nondetects when imputing specific concentration values to these measurements.

If a constituent is reported only as "not detected" and a detection limit is not provided, then review the raw data package to determine if a detection limit was provided. If not, identify the analytical method used and consult a qualified chemist for guidance on an appropriate QL.

F.4.2 Cohen's Adjustment

If a confidence limit is used to compare waste concentrations to a fixed standard, and a significant fraction of the observed measurements in the data set are reported as nondetects, simple substitution techniques (such as putting in half the detection limit for each nondetect) can lead to biased estimates of the mean or standard deviation and inaccurate confidence limits.

By using the detection limit and the pattern seen in the detected values, Cohen's method (Cohen 1959) attempts to reconstruct the key features of the original population, providing explicit estimates of the population mean and standard deviation. These, in turn, can be used to calculate confidence intervals, where Cohen's adjusted estimates are used as replacements for the sample mean and sample standard deviation.

Requirements and Assumptions

Cohen's Adjustment assumes that the common underlying population is normal. As such, the technique should only be used when the observed sample data approximately fit a normal model. Because the presence of a large fraction of nondetects will make explicit normality testing difficult, if not impossible, the most helpful diagnostic aid may be to construct a censored probability plot on the detected measurements. If the censored probability plot is clearly linear on the original measurement scale but not on the log-scale, assume normality for purposes of computing Cohen's Adjustment. If, however, the censored probability plot is clearly linear on the log-scale, but not on the original scale, assume the common underlying population is lognormal instead; then compute Cohen's Adjustment to the estimated mean and standard deviation on the log-scale measurements and construct the desired statistical interval using the algorithm for lognormally-distributed observations (see also Gilbert 1987, page 182).

When more than 50 percent of the observations are nondetect, the accuracy of Cohen's method breaks down substantially, getting worse as the percentage of nondetects increases. Because of this drawback, EPA does not recommend the use of Cohen's adjustment when more than half the data are nondetect. In such circumstances, one should consider an alternate statistical method (see Section F.4.1).

One other requirement of Cohen's method is that there be just a single censoring point. As discussed previously, data sets with multiple detection or quantitation limits may require a more sophisticated treatment.

Procedure

Step 1. Divide the data set into two groups: detects and nondetects. If the total sample size equals n, let m represent the number of detects and (n - m) represent the number of nondetects. Denote the ith detected measurement by x_i, then compute the mean and sample variance of the group of detects (i.e., above the quantitation limit data) using the following formulas:

$$\bar{x}_d = \frac{1}{m}\sum_{i=1}^{m} x_i \qquad \textbf{Equation F.8}$$

and

$$s_d^2 = \frac{1}{m-1}\left[\sum_{i=1}^{m} x_i^2 - m\bar{x}_d^2\right] \qquad \textbf{Equation F.9}$$

Step 2. Denote the single censoring point (e.g., the quantitation limit) by QL. Then compute the two intermediate quantities, h and γ, necessary to derive Cohen's adjustment via the following equations:

$$h = (n - m)/n$$ **Equation F.10**

and

$$\gamma = s_d^2 / (\overline{x}_d - QL)^2$$ **Equation F.11**

Step 3. Use the intermediate quantities, h and γ to determine Cohen's adjustment parameter $\hat{\lambda}$ from Table G-7 in Appendix G. For example, if h = 0.4 and γ = 0.30, then $\hat{\lambda}$ = 0.6713.

Step 4. Using the adjustment parameter $\hat{\lambda}$ found in step 3, compute adjusted estimates of the mean and standard deviation with the following formulas:

$$\overline{x} = \overline{x}_d - \hat{\lambda}(\overline{x}_d - QL)$$ **Equation F.12**

and

$$s = \sqrt{s_d^2 + \hat{\lambda}(\overline{x}_d - QL)^2}$$ **Equation F.13**

Step 5. Once the adjusted estimates for the population mean and standard deviation are derived, these values can be substituted for the sample mean and standard deviation in formulas for the desired confidence limit.

An example calculation using Cohen's method is given in Box F.6.

Box F.6. An Example of Cohen's Method

To determine attainment of a cleanup standard at SWMU, 24 random soil samples were obtained and analyzed for pentachlorophenol. Eight of the 24 values (33%) were below the matrix/laboratory-specific quantitation limit of 1 mg/L. The 24 values are <1.0, <1.0, <1.0, <1.0, <1.0, <1.0, <1.0, <1.0, 1.1, 1.5, 1.9, 2.0, 2.5, 2.6, 3.1, 3.3, 3.2, 3.2, 3.3, 3.4, 3.5, 3.8, 4.5, 5.8 mg/L. Cohen's Method will be used to adjust the sample mean and standard deviation for use in constructing a UCL on the mean to determine if the cleanup has attained the site-specific risk-based cleanup standard of 5.0 mg/kg.

Solution

Step 1: The sample mean of the m = 16 values greater than the quantitation limit is $\overline{x}_d$ = 3.044

Step 2: The sample variance of the 16 quantified values is s_d^2 = 1.325.

Step 3: $h = (24 - 16) / 24 = 0.333$ and $\gamma = 1.325 / (3.044 - 1.0)^2 = 0.317$

Step 4: Table G-7 of Appendix G was used for h = 0.333 and γ = 0.317 to find the value of $\hat{\lambda}$. Since the table does not contain these entries exactly, double linear interpolation was used to estimate $\hat{\lambda}$ = 0.5223.

Step 5: The adjusted sample mean and standard deviation are then estimated as follows:

$\overline{x} = 3.044 - 0.5223\,(3.044 - 1.0) = 1.976 \approx 2.0$ and

$$s = \sqrt{1.325 + 0.5223(3.044 - 1.0)^2} = 1.873 \approx 1.9$$

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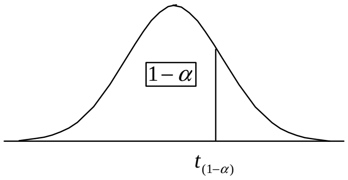
APPENDIX G

STATISTICAL TABLES

Table G-1. Critical Values of Student's t Distribution (One-Tailed)



Degrees of Freedom (see note)	*t values for ($1-\alpha$) or ($1-\beta$)*								
	0.70	***0.75***	***0.80***	***0.85***	***0.90***	***0.95***	***0.975***	***0.99***	***0.995***
1	0.727	1.000	1.376	1.963	3.078	6.314	12.706	31.821	63.657
2	0.617	0.816	1.061	1.386	1.886	2.920	4.303	6.965	9.925
3	0.584	0.765	0.978	1.250	1.638	2.353	3.182	4.541	5.841
4	0.569	0.741	0.941	1.190	1.533	2.132	2.776	3.747	4.604
5	0.559	0.727	0.920	1.156	1.476	2.015	2.571	3.365	4.032
6	0.553	0.718	0.906	1.134	1.440	1.943	2.447	3.143	3.707
7	0.549	0.711	0.896	1.119	1.415	1.895	2.365	2.998	3.499
8	0.546	0.706	0.889	1.108	1.397	1.860	2.306	2.896	3.355
9	0.543	0.703	0.883	1.100	1.383	1.833	2.262	2.821	3.250
10	0.542	0.700	0.879	1.093	1.372	1.812	2.228	2.764	3.169
11	0.540	0.697	0.876	1.088	1.363	1.796	2.201	2.718	3.106
12	0.539	0.695	0.873	1.083	1.356	1.782	2.179	2.681	3.055
13	0.538	0.694	0.870	1.079	1.350	1.771	2.160	2.650	3.012
14	0.537	0.692	0.868	1.076	1.345	1.761	2.145	2.624	2.977
15	0.536	0.691	0.866	1.074	1.340	1.753	2.131	2.602	2.947
16	0.535	0.690	0.865	1.071	1.337	1.746	2.120	2.583	2.921
17	0.534	0.689	0.863	1.069	1.333	1.740	2.110	2.567	2.898
18	0.534	0.688	0.862	1.067	1.330	1.734	2.101	2.552	2.878
19	0.533	0.688	0.861	1.066	1.328	1.729	2.093	2.539	2.861
20	0.533	0.687	0.860	1.064	1.325	1.725	2.086	2.528	2.845
21	0.532	0.686	0.859	1.063	1.323	1.721	2.080	2.518	2.831
22	0.532	0.686	0.858	1.061	1.321	1.717	2.074	2.508	2.819
23	0.532	0.685	0.858	1.060	1.319	1.714	2.069	2.500	2.807
24	0.531	0.685	0.857	1.059	1.318	1.711	2.064	2.492	2.797
25	0.531	0.684	0.856	1.058	1.316	1.708	2.060	2.485	2.787
26	0.531	0.684	0.856	1.058	1.315	1.706	2.056	2.479	2.779
27	0.531	0.684	0.855	1.057	1.314	1.703	2.052	2.473	2.771
28	0.530	0.683	0.855	1.056	1.313	1.701	2.048	2.467	2.763
29	0.530	0.683	0.854	1.055	1.311	1.699	2.045	2.462	2.756
30	0.530	0.683	0.854	1.055	1.310	1.697	2.042	2.457	2.750
40	0.529	0.681	0.851	1.050	1.303	1.684	2.021	2.423	2.704
60	0.527	0.679	0.848	1.046	1.296	1.671	2.000	2.390	2.660
120	0.526	0.677	0.845	1.041	1.289	1.658	1.980	2.358	2.617
∞	0.524	0.674	0.842	1.036	1.282	1.645	1.960	2.326	2.576

Note: For simple random or systematic sampling, degrees of freedom (df) are equal to the number of samples (n) collected from a solid waste and analyzed, less one (in other words, $df = n-1$). If stratified random sampling is used, calculate df using Equation 12 or 14 in Section 5.4.2.2.

The last row of the table (∞ degrees of freedom) gives the critical values for a standard normal distribution (z). For example, the z value for $1-\alpha$ where $\alpha = 0.10$ is found in the last row as 1.282.

Table G-2. Factors (κ) for Parametric Upper Confidence Bounds on Upper Percentiles (p)

n	p = 0.80					p = 0.90				
$1-\alpha$	**0.800**	**0.900**	**0.950**	**0.975**	**0.990**	**0.800**	**0.900**	**0.950**	**0.975**	**0.990**
2	3.417	6.987	14.051	28.140	70.376	5.049	10.253	20.581	41.201	103.029
3	2.016	3.039	4.424	6.343	10.111	2.871	4.258	6.155	8.797	13.995
4	1.675	2.295	3.026	3.915	5.417	2.372	3.188	4.162	5.354	7.380
5	1.514	1.976	2.483	3.058	3.958	2.145	2.742	3.407	4.166	5.362
6	1.417	1.795	2.191	2.621	3.262	2.012	2.494	3.006	3.568	4.411
7	1.352	1.676	2.005	2.353	2.854	1.923	2.333	2.755	3.206	3.859
8	1.304	1.590	1.875	2.170	2.584	1.859	2.219	2.582	2.960	3.497
9	1.266	1.525	1.779	2.036	2.391	1.809	2.133	2.454	2.783	3.240
10	1.237	1.474	1.703	1.933	2.246	1.770	2.066	2.355	2.647	3.048
11	1.212	1.433	1.643	1.851	2.131	1.738	2.011	2.275	2.540	2.898
12	1.192	1.398	1.593	1.784	2.039	1.711	1.966	2.210	2.452	2.777
13	1.174	1.368	1.551	1.728	1.963	1.689	1.928	2.155	2.379	2.677
14	1.159	1.343	1.514	1.681	1.898	1.669	1.895	2.109	2.317	2.593
15	1.145	1.321	1.483	1.639	1.843	1.652	1.867	2.068	2.264	2.521
16	1.133	1.301	1.455	1.603	1.795	1.637	1.842	2.033	2.218	2.459
17	1.123	1.284	1.431	1.572	1.753	1.623	1.819	2.002	2.177	2.405
18	1.113	1.268	1.409	1.543	1.716	1.611	1.800	1.974	2.141	2.357
19	1.104	1.254	1.389	1.518	1.682	1.600	1.782	1.949	2.108	2.314
20	1.096	1.241	1.371	1.495	1.652	1.590	1.765	1.926	2.079	2.276
21	1.089	1.229	1.355	1.474	1.625	1.581	1.750	1.905	2.053	2.241
22	1.082	1.218	1.340	1.455	1.600	1.572	1.737	1.886	2.028	2.209
23	1.076	1.208	1.326	1.437	1.577	1.564	1.724	1.869	2.006	2.180
24	1.070	1.199	1.313	1.421	1.556	1.557	1.712	1.853	1.985	2.154
25	1.065	1.190	1.302	1.406	1.537	1.550	1.702	1.838	1.966	2.129
26	1.060	1.182	1.291	1.392	1.519	1.544	1.691	1.824	1.949	2.106
27	1.055	1.174	1.280	1.379	1.502	1.538	1.682	1.811	1.932	2.085
28	1.051	1.167	1.271	1.367	1.486	1.533	1.673	1.799	1.917	2.065
29	1.047	1.160	1.262	1.355	1.472	1.528	1.665	1.788	1.903	2.047
30	1.043	1.154	1.253	1.344	1.458	1.523	1.657	1.777	1.889	2.030
31	1.039	1.148	1.245	1.334	1.445	1.518	1.650	1.767	1.877	2.014
32	1.035	1.143	1.237	1.325	1.433	1.514	1.643	1.758	1.865	1.998
33	1.032	1.137	1.230	1.316	1.422	1.510	1.636	1.749	1.853	1.984
34	1.029	1.132	1.223	1.307	1.411	1.506	1.630	1.740	1.843	1.970
35	1.026	1.127	1.217	1.299	1.400	1.502	1.624	1.732	1.833	1.957
36	1.023	1.123	1.211	1.291	1.391	1.498	1.618	1.725	1.823	1.945
37	1.020	1.118	1.205	1.284	1.381	1.495	1.613	1.717	1.814	1.934
38	1.017	1.114	1.199	1.277	1.372	1.492	1.608	1.710	1.805	1.922
39	1.015	1.110	1.194	1.270	1.364	1.489	1.603	1.704	1.797	1.912
40	1.013	1.106	1.188	1.263	1.356	1.486	1.598	1.697	1.789	1.902
41	1.010	1.103	1.183	1.257	1.348	1.483	1.593	1.691	1.781	1.892
42	1.008	1.099	1.179	1.251	1.341	1.480	1.589	1.685	1.774	1.883
43	1.006	1.096	1.174	1.246	1.333	1.477	1.585	1.680	1.767	1.874
44	1.004	1.092	1.170	1.240	1.327	1.475	1.581	1.674	1.760	1.865
45	1.002	1.089	1.165	1.235	1.320	1.472	1.577	1.669	1.753	1.857
46	1.000	1.086	1.161	1.230	1.314	1.470	1.573	1.664	1.747	1.849
47	0.998	1.083	1.157	1.225	1.308	1.468	1.570	1.659	1.741	1.842
48	0.996	1.080	1.154	1.220	1.302	1.465	1.566	1.654	1.735	1.835
49	0.994	1.078	1.150	1.216	1.296	1.463	1.563	1.650	1.730	1.828
50	0.993	1.075	1.146	1.211	1.291	1.461	1.559	1.646	1.724	1.821
55	0.985	1.063	1.130	1.191	1.266	1.452	1.545	1.626	1.700	1.790
60	0.978	1.052	1.116	1.174	1.245	1.444	1.532	1.609	1.679	1.764
65	0.972	1.043	1.104	1.159	1.226	1.437	1.521	1.594	1.661	1.741
70	0.967	1.035	1.094	1.146	1.210	1.430	1.511	1.581	1.645	1.722
75	0.963	1.028	1.084	1.135	1.196	1.425	1.503	1.570	1.630	1.704
80	0.959	1.022	1.076	1.124	1.183	1.420	1.495	1.559	1.618	1.688
85	0.955	1.016	1.068	1.115	1.171	1.415	1.488	1.550	1.606	1.674
90	0.951	1.011	1.061	1.106	1.161	1.411	1.481	1.542	1.596	1.661
95	0.948	1.006	1.055	1.098	1.151	1.408	1.475	1.534	1.586	1.650
100	0.945	1.001	1.049	1.091	1.142	1.404	1.470	1.527	1.578	1.639

Table G-2. Factors (K) for Parametric Upper Confidence Bounds on Upper Percentiles (p) (continued)

n	p = 0.95					p = 0.99				
$1-\alpha$	**0.800**	**0.900**	**0.950**	**0.975**	**0.990**	**0.800**	**0.900**	**0.950**	**0.975**	**0.990**
2	6.464	13.090	26.260	52.559	131.426	9.156	18.500	37.094	74.234	185.617
3	3.604	5.311	7.656	10.927	17.370	5.010	7.340	10.553	15.043	23.896
4	2.968	3.957	5.144	6.602	9.083	4.110	5.438	7.042	9.018	12.387
5	2.683	3.400	4.203	5.124	6.578	3.711	4.666	5.741	6.980	8.939
6	2.517	3.092	3.708	4.385	5.406	3.482	4.243	5.062	5.967	7.335
7	2.407	2.894	3.399	3.940	4.728	3.331	3.972	4.642	5.361	6.412
8	2.328	2.754	3.187	3.640	4.285	3.224	3.783	4.354	4.954	5.812
9	2.268	2.650	3.031	3.424	3.972	3.142	3.641	4.143	4.662	5.389
10	2.220	2.568	2.911	3.259	3.738	3.078	3.532	3.981	4.440	5.074
11	2.182	2.503	2.815	3.129	3.556	3.026	3.443	3.852	4.265	4.829
12	2.149	2.448	2.736	3.023	3.410	2.982	3.371	3.747	4.124	4.633
13	2.122	2.402	2.671	2.936	3.290	2.946	3.309	3.659	4.006	4.472
14	2.098	2.363	2.614	2.861	3.189	2.914	3.257	3.585	3.907	4.337
15	2.078	2.329	2.566	2.797	3.102	2.887	3.212	3.520	3.822	4.222
16	2.059	2.299	2.524	2.742	3.028	2.863	3.172	3.464	3.749	4.123
17	2.043	2.272	2.486	2.693	2.963	2.841	3.137	3.414	3.684	4.037
18	2.029	2.249	2.453	2.650	2.905	2.822	3.105	3.370	3.627	3.960
19	2.016	2.227	2.423	2.611	2.854	2.804	3.077	3.331	3.575	3.892
20	2.004	2.208	2.396	2.576	2.808	2.789	3.052	3.295	3.529	3.832
21	1.993	2.190	2.371	2.544	2.766	2.774	3.028	3.263	3.487	3.777
22	1.983	2.174	2.349	2.515	2.729	2.761	3.007	3.233	3.449	3.727
23	1.973	2.159	2.328	2.489	2.694	2.749	2.987	3.206	3.414	3.681
24	1.965	2.145	2.309	2.465	2.662	2.738	2.969	3.181	3.382	3.640
25	1.957	2.132	2.292	2.442	2.633	2.727	2.952	3.158	3.353	3.601
26	1.949	2.120	2.275	2.421	2.606	2.718	2.937	3.136	3.325	3.566
27	1.943	2.109	2.260	2.402	2.581	2.708	2.922	3.116	3.300	3.533
28	1.936	2.099	2.246	2.384	2.558	2.700	2.909	3.098	3.276	3.502
29	1.930	2.089	2.232	2.367	2.536	2.692	2.896	3.080	3.254	3.473
30	1.924	2.080	2.220	2.351	2.515	2.684	2.884	3.064	3.233	3.447
31	1.919	2.071	2.208	2.336	2.496	2.677	2.872	3.048	3.213	3.421
32	1.914	2.063	2.197	2.322	2.478	2.671	2.862	3.034	3.195	3.398
33	1.909	2.055	2.186	2.308	2.461	2.664	2.852	3.020	3.178	3.375
34	1.904	2.048	2.176	2.296	2.445	2.658	2.842	3.007	3.161	3.354
35	1.900	2.041	2.167	2.284	2.430	2.652	2.833	2.995	3.145	3.334
36	1.895	2.034	2.158	2.272	2.415	2.647	2.824	2.983	3.131	3.315
37	1.891	2.028	2.149	2.262	2.402	2.642	2.816	2.972	3.116	3.297
38	1.888	2.022	2.141	2.251	2.389	2.637	2.808	2.961	3.103	3.280
39	1.884	2.016	2.133	2.241	2.376	2.632	2.800	2.951	3.090	3.264
40	1.880	2.010	2.125	2.232	2.364	2.627	2.793	2.941	3.078	3.249
41	1.877	2.005	2.118	2.223	2.353	2.623	2.786	2.932	3.066	3.234
42	1.874	2.000	2.111	2.214	2.342	2.619	2.780	2.923	3.055	3.220
43	1.871	1.995	2.105	2.206	2.331	2.615	2.773	2.914	3.044	3.206
44	1.868	1.990	2.098	2.198	2.321	2.611	2.767	2.906	3.034	3.193
45	1.865	1.986	2.092	2.190	2.312	2.607	2.761	2.898	3.024	3.180
46	1.862	1.981	2.086	2.183	2.303	2.604	2.756	2.890	3.014	3.168
47	1.859	1.977	2.081	2.176	2.294	2.600	2.750	2.883	3.005	3.157
48	1.857	1.973	2.075	2.169	2.285	2.597	2.745	2.876	2.996	3.146
49	1.854	1.969	2.070	2.163	2.277	2.594	2.740	2.869	2.988	3.135
50	1.852	1.965	2.065	2.156	2.269	2.590	2.735	2.862	2.980	3.125
55	1.841	1.948	2.042	2.128	2.233	2.576	2.713	2.833	2.943	3.078
60	1.832	1.933	2.022	2.103	2.202	2.564	2.694	2.807	2.911	3.038
65	1.823	1.920	2.005	2.082	2.176	2.554	2.677	2.785	2.883	3.004
70	1.816	1.909	1.990	2.063	2.153	2.544	2.662	2.765	2.859	2.974
75	1.810	1.899	1.976	2.047	2.132	2.536	2.649	2.748	2.838	2.947
80	1.804	1.890	1.964	2.032	2.114	2.528	2.638	2.733	2.819	2.924
85	1.799	1.882	1.954	2.019	2.097	2.522	2.627	2.719	2.802	2.902
90	1.794	1.874	1.944	2.006	2.082	2.516	2.618	2.706	2.786	2.883
95	1.790	1.867	1.935	1.995	2.069	2.510	2.609	2.695	2.772	2.866
100	1.786	1.861	1.927	1.985	2.056	2.505	2.601	2.684	2.759	2.850

Table G-3a. Sample Size Required to Demonstrate With At Least $100(1-\alpha)\%$ Confidence That At Least $100p\%$ of a Lot or Batch of Waste Complies With the Applicable Standard (No Samples Exceeding the Standard)

p	$1-\alpha$										
	0.50	**0.55**	**0.60**	**0.65**	**0.70**	**0.75**	**0.80**	**0.85**	**0.90**	**0.95**	**0.99**
0.50	1	2	2	2	2	2	3	3	4	5	7
0.55	2	2	2	2	3	3	3	4	4	6	8
0.60	2	2	2	3	3	3	4	4	5	6	10
0.65	2	2	3	3	3	4	4	5	6	7	11
0.70	2	3	3	3	4	4	5	6	7	9	13
0.75	3	3	4	4	5	5	6	7	9	11	17
0.80	4	4	5	5	6	7	8	9	11	14	21
0.85	5	5	6	7	8	9	10	12	15	19	29
0.90	7	8	9	10	12	14	16	19	22	29	44
0.95	14	16	18	21	24	28	32	37	45	59	90
0.99	69	80	92	105	120	138	161	189	230	299	459

Table G-3b. Sample Size Required to Demonstrate With At Least $100(1-\alpha)\%$ Confidence That At Least $100p\%$ of a Lot or Batch of Waste Complies With the Applicable Standard (One Sample Exceeding the Standard)

p	$1-\alpha$										
	0.50	**0.55**	**0.60**	**0.65**	**0.70**	**0.75**	**0.80**	**0.85**	**0.90**	**0.95**	**0.99**
0.50	3	4	4	4	5	5	5	6	7	8	11
0.55	4	4	4	5	5	6	6	7	8	9	12
0.60	4	5	5	5	6	6	7	8	9	10	14
0.65	5	5	6	6	7	7	8	9	10	12	16
0.70	6	6	7	7	8	9	9	10	12	14	20
0.75	7	7	8	9	9	10	11	13	15	18	24
0.80	9	9	10	11	12	13	14	16	18	22	31
0.85	11	12	13	15	16	18	19	22	25	30	42
0.90	17	19	20	22	24	27	29	33	38	46	64
0.95	34	37	40	44	49	53	59	67	77	93	130
0.99	168	184	202	222	244	269	299	337	388	473	662

Table G-4. Coefficients $[a_{n-i+1}]$ for the Shapiro-Wilk Test for Normality

i \ n	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	
1	.7071	.7071	.6872	.6646	.6431	.6233	.6052	.5888	.5739	
2		.0000	.1677	.2413	.2806	.3031	.3164	.3244	.3291	
3				.0000	.0875	.1401	.1743	.1976	.2141	
4						.0000	.0561	.0947	.1224	
5								.0000	.0399	

i \ n	**11**	**12**	**13**	**14**	**15**	**16**	**17**	**18**	**19**	**20**
1	.5601	.5475	.5359	.5251	.5150	.5056	.4968	.4886	.4808	.4734
2	.3315	.3325	.3325	.3318	.3306	.3290	.3273	.3253	.3232	.3211
3	.2260	.2347	.2412	.2460	.2495	.2521	.2540	.2553	.2561	.2565
4	.1429	.1586	.1707	.1802	.1878	.1939	.1988	.2027	.2059	.2085
5	.0695	.0922	.1099	.1240	.1353	.1447	.1524	.1587	.1641	.1686
6	.0000	.0303	.0539	.0727	.0880	.1005	.1109	.1197	.1271	.1334
7			.0000	.0240	.0433	.0593	.0725	.0837	.0932	.1013
8					.0000	.0196	.0359	.0496	.0612	.0711
9							.0000	.0163	.0303	.0422
10									.0000	.0140

i \ n	**21**	**22**	**23**	**24**	**25**	**26**	**27**	**28**	**29**	**30**
1	.4643	.4590	.4542	.4493	.4450	.4407	.4366	.4328	.4291	.4254
2	.3185	.3156	.3126	.3098	.3069	.3043	.3018	.2992	.2968	.2944
3	.2578	.2571	.2563	.2554	.2543	.2533	.2522	.2510	.2499	.2487
4	.2119	.2131	.2139	.2145	.2148	.2151	.2152	.2151	.2150	.2148
5	.1736	.1764	.1787	.1807	.1822	.1836	.1848	.1857	.1864	.1870
6	.1399	.1443	.1480	.1512	.1539	.1563	.1584	.1601	.1616	.1630
7	.1092	.1150	.1201	.1245	.1283	.1316	.1346	.1372	.1395	.1415
8	.0804	.0878	.0941	.0997	.1046	.1089	.1128	.1162	.1192	.1219
9	.0530	.0618	.0696	.0764	.0823	.0876	.0923	.0965	.1002	.1036
10	.0263	.0368	.0459	.0539	.0610	.0672	.0728	.0778	.0822	.0862
11	.0000	.0122	.0228	.0321	.0403	.0476	.0540	.0598	.0650	.0697
12			.0000	.0107	.0200	.0284	.0358	.0424	.0483	.0537
13					.0000	.0094	.0178	.0253	.0320	.0381
14							.0000	.0084	.0159	.0227
15									.0000	.0076

Source: After Shapiro and Wilk (1965)

Table G-4. Coefficients $[a_{n-i+1}]$ for the Shapiro-Wilk Test for Normality (Continued)

i \ n	**31**	**32**	**33**	**34**	**35**	**36**	**37**	**38**	**39**	**40**
1	.4220	.4188	.4156	.4127	.4096	.4068	.4040	.4015	.3989	.3964
2	.2921	.2898	.2876	.2854	.2834	.2813	.2794	.2774	.2755	.2737
3	.2475	.2463	.2451	.2439	.2427	.2415	.2403	.2391	.2380	.2368
4	.2145	.2141	.2137	.2132	.2127	.2121	.2116	.2110	.2104	.2098
5	.1874	.1878	.1880	.1882	.1883	.1883	.1883	.1881	.1880	.1878
6	.1641	.1651	.1660	.1667	.1673	.1678	.1683	.1686	.1689	.1691
7	.1433	.1449	.1463	.1475	.1487	.1496	.1505	.1513	.1520	.1526
8	.1243	.1265	.1284	.1301	.1317	.1331	.1344	.1356	.1366	.1376
9	.1066	.1093	.1118	.1140	.1160	.1179	.1196	.1211	.1225	.1237
10	.0899	.0931	.0961	.0988	.1013	.1036	.1056	.1075	.1092	.1108
11	.0739	.0777	.0812	.0844	.0873	.0900	.0924	.0947	.0967	.0986
12	.0585	.0629	.0669	.0706	.0739	.0770	.0798	.0824	.0848	.0870
13	.0435	.0485	.0530	.0572	.0610	.0645	.0677	.0706	.0733	.0759
14	.0289	.0344	.0395	.0441	.0484	.0523	.0559	.0592	.0622	.0651
15	.0144	.0206	.0262	.0314	.0361	.0404	.0444	.0481	.0515	.0546
16	.0000	.0068	.0131	.0187	.0239	.0287	.0331	.0372	.0409	.0444
17			.0000	.0062	.0119	.0172	.0220	.0264	.0305	.0343
18					.0000	.0057	.0110	.0158	.0203	.0244
19							.0000	.0053	.0101	.0146
20									.0000	.0049

i \ n	**41**	**42**	**43**	**44**	**45**	**46**	**47**	**48**	**49**	**50**
1	.3940	.3917	.3894	.3872	.3850	.3830	.3808	.3789	.3770	.3751
2	.2719	.2701	.2628	.2667	.2651	.2635	.2620	.2604	.2589	.2574
3	.2357	.2345	.2334	.2323	.2313	.2302	.2291	.2281	.2271	.2260
4	.2091	.2085	.2078	.2072	.2065	.2058	.2052	.2045	.2038	.2032
5	.1876	.1874	.1871	.1868	.1865	.1862	.1859	.1855	.1851	.1847
6	.1693	.1694	.1695	.1695	.1695	.1695	.1695	.1693	.1692	.1691
7	.1531	.1535	.1539	.1542	.1545	.1548	.1550	.1551	.1553	.1554
8	.1384	.1392	.1398	.1405	.1410	.1415	.1420	.1423	.1427	.1430
9	.1249	.1259	.1269	.1278	.1286	.1293	.1300	.1306	.1312	.1317
10	.1123	.1136	.1149	.1160	.1170	.1180	.1189	.1197	.1205	.1212
11	.1004	.1020	.1035	.1049	.1062	.1073	.1085	.1095	.1105	.1113
12	.0891	.0909	.0927	.0943	.0959	.0972	.0986	.0998	.1010	.1020
13	.0782	.0804	.0824	.0842	.0860	.0876	.0892	.0906	.0919	.0932
14	.0677	.0701	.0724	.0745	.0775	.0785	.0801	.0817	.0832	.0846
15	.0575	.0602	.0628	.0651	.0673	.0694	.0713	.0731	.0748	.0764
16	.0476	.0506	.0534	.0560	.0584	.0607	.0628	.0648	.0667	.0685
17	.0379	.0411	.0442	.0471	.0497	.0522	.0546	.0568	.0588	.0608
18	.0283	.0318	.0352	.0383	.0412	.0439	.0465	.0489	.0511	.0532
19	.0188	.0227	.0263	.0296	.0328	.0357	.0385	.0411	.0436	.0459
20	.0094	.0136	.0175	.0211	.0245	.0277	.0307	.0335	.0361	.0386
21	.0000	.0045	.0087	.0126	.0163	.0197	.0229	.0259	.0288	.0314
22			.0000	.0042	.0081	.0118	.0153	.0185	.0215	.0244
23					.0000	.0039	.0076	.0111	.0143	.0174
24							.0000	.0037	.0071	.0104
25									.0000	.0035

Table G-5. α -Level Critical Points for the Shapiro-Wilk Test

n	α	
	0.01	**0.05**
3	0.753	0.767
4	0.687	0.748
5	0.686	0.762
6	0.713	0.788
7	0.730	0.803
8	0.749	0.818
9	0.764	0.829
10	0.781	0.842
11	0.792	0.850
12	0.805	0.859
13	0.814	0.866
14	0.825	0.874
15	0.835	0.881
16	0.844	0.887
17	0.851	0.892
18	0.858	0.897
19	0.863	0.901
20	0.868	0.905
21	0.873	0.908
22	0.878	0.911
23	0.881	0.914
24	0.884	0.916
25	0.888	0.918
26	0.891	0.920
27	0.894	0.923
28	0.896	0.924
29	0.898	0.926
30	0.900	0.927
31	0.902	0.929
32	0.904	0.930
33	0.906	0.931
34	0.908	0.933
35	0.910	0.934
36	0.912	0.935
37	0.914	0.936
38	0.916	0.938
39	0.917	0.939
40	0.919	0.940
41	0.920	0.941
42	0.922	0.942
43	0.923	0.943
44	0.924	0.944
45	0.926	0.945
46	0.927	0.945
47	0.928	0.946
48	0.929	0.947
49	0.929	0.947
50	0.930	0.947

Source: After Shapiro and Wilk (1965)

Table G-6. Values of $H_{1-\alpha} = H_{0.90}$ for Calculating a One-Sided 90-Percent UCL on a Lognormal Mean

s_y	n									
	3	5	7	10	12	15	21	31	51	101
0.10	1.686	1.438	1.381	1.349	1.338	1.328	1.317	1.308	1.301	1.295
0.20	1.885	1.522	1.442	1.396	1.380	1.365	1.348	1.335	1.324	1.314
0.30	2.156	1.627	1.517	1.453	1.432	1.411	1.388	1.370	1.354	1.339
0.40	2.521	1.755	1.607	1.523	1.494	1.467	1.437	1.412	1.390	1.371
0.50	2.990	1.907	1.712	1.604	1.567	1.532	1.494	1.462	1.434	1.409
0.60	3.542	2.084	1.834	1.696	1.650	1.606	1.558	1.519	1.485	1.454
0.70	4.136	2.284	1.970	1.800	1.743	1.690	1.631	1.583	1.541	1.504
0.80	4.742	2.503	2.119	1.914	1.845	1.781	1.710	1.654	1.604	1.560
0.90	5.349	2.736	2.280	2.036	1.955	1.880	1.797	1.731	1.672	1.621
1.00	5.955	2.980	2.450	2.167	2.073	1.985	1.889	1.812	1.745	1.686
1.25	7.466	3.617	2.904	2.518	2.391	2.271	2.141	2.036	1.946	1.866
1.50	8.973	4.276	3.383	2.896	2.733	2.581	2.415	2.282	2.166	2.066
1.75	10.48	4.944	3.877	3.289	3.092	2.907	2.705	2.543	2.402	2.279
2.00	11.98	5.619	4.380	3.693	3.461	3.244	3.005	2.814	2.648	2.503
2.50	14.99	6.979	5.401	4.518	4.220	3.938	3.629	3.380	3.163	2.974
3.00	18.00	8.346	6.434	5.359	4.994	4.650	4.270	3.964	3.697	3.463
3.50	21.00	9.717	7.473	6.208	5.778	5.370	4.921	4.559	4.242	3.965
4.00	24.00	11.09	8.516	7.062	6.566	6.097	5.580	5.161	4.796	4.474
4.50	27.01	12.47	9.562	7.919	7.360	6.829	6.243	5.763	5.354	4.989
5.00	30.01	13.84	10.61	8.779	8.155	7.563	6.909	6.379	5.916	5.508
6.00	36.02	16.60	12.71	10.50	9.751	9.037	8.248	7.607	7.048	6.555
7.00	42.02	19.35	14.81	12.23	11.35	10.52	9.592	8.842	8.186	7.607
8.00	48.03	22.11	16.91	13.96	12.96	12.00	10.94	10.08	9.329	8.665
9.00	54.03	24.87	19.02	15.70	14.56	13.48	12.29	11.32	10.48	9.725
10.0	60.04	27.63	21.12	17.43	16.17	14.97	13.64	12.56	11.62	10.79

Source: Land (1975)

Table G-7. Values of the Parameter $\hat{\lambda}$ for Cohen's Adjustment for Nondetected Values

γ	h											
	.01	.02	.03	.04	.05	.06	.07	.08	.09	.10	.15	.20
.00	.010100	.020400	.030902	.041583	.052507	.063625	.074953	.08649	.09824	.11020	.17342	.24268
.05	.010551	.021294	.032225	.043350	.054670	.066159	.077909	.08983	.10197	.11431	.17925	.25033
.10	.010950	.022082	.033398	.044902	.056596	.068483	.080563	.09285	.10534	.11804	.18479	.25741
.15	.011310	.022798	.034466	.046318	.058356	.070586	.083009	.09563	.10845	.12148	.18985	.26405
.20	.011642	.023459	.035453	.047829	.059990	.072539	.085280	.09822	.11135	.12469	.19460	.27031
.25	.011952	.024076	.036377	.048858	.061522	.074372	.087413	.10065	.11408	.12772	.19910	.27626
.30	.012243	.024658	.037249	.050018	.062969	.076106	.089433	.10295	.11667	.13059	.20338	.28193
.35	.012520	.025211	.038077	.051120	.064345	.077736	.091355	.10515	.11914	.13333	.20747	.28737
.40	.012784	.025738	.038866	.052173	.065660	.079332	.093193	.10725	.12150	.13595	.21129	.29250
.45	.013036	.026243	.039624	.053182	.066921	.080845	.094958	.10926	.12377	.13847	.21517	.29765
.50	.013279	.026728	.040352	.054153	.068135	.082301	.096657	.11121	.12595	.14090	.21882	.30253
.55	.013513	.027196	.041054	.055089	.069306	.083708	.098298	.11208	.12806	.14325	.22225	.30725
.60	.013739	.027849	.041733	.055995	.070439	.085068	.099887	.11490	.13011	.14552	.22578	.31184
.65	.013958	.028087	.042391	.056874	.071538	.086388	.10143	.11666	.13209	.14773	.22910	.31630
.70	.014171	.028513	.043030	.057726	.072505	.087670	.10292	.11837	.13402	.14987	.23234	.32065
.75	.014378	.029927	.043652	.058556	.073643	.088917	.10438	.12004	.13590	.15196	.23550	.32489
.80	.014579	.029330	.044258	.059364	.074655	.090133	.10580	.12167	.13775	.15400	.23858	.32903
.85	.014773	.029723	.044848	.060153	.075642	.091319	.10719	.12225	.13952	.15599	.24158	.33307
.90	.014967	.030107	.045425	.060923	.075606	.092477	.10854	.12480	.14126	.15793	.24452	.33703
.95	.015154	.030483	.045989	.061676	.077549	.093611	.10987	.12632	.14297	.15983	.24740	.34091
1.00	.015338	.030850	.046540	.062413	.078471	.094720	.11116	.12780	.14465	.16170	.25022	.34471

Table G-7. Values of the Parameter $\hat{\lambda}$ for Cohen's Adjustment for Nondetected Values (Continued)

γ	h											
	.25	.30	.35	.40	.45	.50	.55	.60	.65	.70	.80	.90
.05	.32793	.4130	.5066	.6101	.7252	.8540	.9994	1.166	1.358	1.585	2.203	3.314
.10	.33662	.4233	.5184	.6234	.7400	.8703	1.017	1.185	1.379	1.608	2.229	3.345
.15	.34480	.4330	.5296	.6361	.7542	.8860	1.035	1.204	1.400	1.630	2.255	3.376
.20	.35255	.4422	.5403	.6483	.7673	.9012	1.051	1.222	1.419	1.651	2.280	3.405
.25	.35993	.4510	.5506	.6600	.7810	.9158	1.067	1.240	1.439	1.672	2.305	3.435
.30	.36700	.4595	.5604	.6713	.7937	.9300	1.083	1.257	1.457	1.693	2.329	3.464
.35	.37379	.4676	.5699	.6821	.8060	.9437	1.098	1.274	1.475	1.713	2.353	3.492
.40	.38033	.4735	.5791	.6927	.8179	.9570	1.113	1.290	1.494	1.732	2.376	3.520
.45	.38665	.4831	.5880	.7029	.8295	.9700	1.127	1.306	1.511	1.751	2.399	3.547
.50	.39276	.4904	.5967	.7129	.8408	.9826	1.141	1.321	1.528	1.770	2.421	3.575
.55	.39679	.4976	.6061	.7225	.8517	.9950	1.155	1.337	1.545	1.788	2.443	3.601
.60	.40447	.5045	.6133	.7320	.8625	1.007	1.169	1.351	1.561	1.806	2.465	3.628
.65	.41008	.5114	.6213	.7412	.8729	1.019	1.182	1.368	1.577	1.824	2.486	3.654
.70	.41555	.5180	.6291	.7502	.8832	1.030	1.195	1.380	1.593	1.841	2.507	3.679
.75	.42090	.5245	.6367	.7590	.8932	1.042	1.207	1.394	1.608	1.851	2.528	3.705
.80	.42612	.5308	.6441	.7676	.9031	1.053	1.220	1.408	1.624	1.875	2.548	3.730
.85	.43122	.5370	.6515	.7781	.9127	1.064	1.232	1.422	1.639	1.892	2.568	3.754
.90	.43622	.5430	.6586	.7844	.9222	1.074	1.244	1.435	1.653	1.908	2.588	3.779
.95	.44112	.5490	.6656	.7925	.9314	1.085	1.255	1.448	1.668	1.924	2.607	3.803
1.00	.44592	.5548	.6724	.8005	.9406	1.095	1.287	1.461	1.882	1.940	2.626	3.827

APPENDIX H

STATISTICAL SOFTWARE

Since publication of Chapter Nine ("Sampling Plan") of SW-846 in 1986, great advances have been made in desktop computer hardware and software. In implementing the procedures recommended in this chapter, you should take advantage of the powerful statistical software now available for low cost or no cost. A number of useful "freeware" packages are available from EPA and other organizations, and many are downloadable from the Internet. Commercially available software also may be used.

This appendix provides a list of software that you might find useful. *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5* (USEPA 1998a) also provides an extensive list of software that can assist you in developing and preparing a quality assurance project plan.

Sampling Design Software

Title	**Description**
*Decision Error Feasibility Trials (DEFT)**	This software package allows quick generation of cost information about several simple sampling designs based on DQO constraints, which can be evaluated to determine their appropriateness and feasibility before the sampling and analysis design is finalized. This software supports the *Guidance for the Data Quality Objectives Process EPA QA/G-4* (USEPA 2000b), which provides general guidance to organizations developing data quality criteria and performance specifications for decision making. The *Data Quality Objectives Decision Error Feasibility Trials Software (DEFT) - User's Guide* (EPA/240/B-01/007) contains detailed instructions on how to use DEFT software and provides background information on the sampling designs that the software uses. Download from EPA's World Wide Web site at: http://www.epa.gov/quality/qa_docs.html.
*GeoEAS**	*Geostatistical Environmental Assessment Software (GeoEAS)* (USEPA 1991b) is a collection of interactive software tools for performing two-dimensional geostatistical analyses of spatially distributed data. Programs are provided for data file management, data transformations, univariate statistics, variogram analysis, cross-validation, kriging, contour mapping, post plots, and line/scatter plots. Users may alter parameters and re-calculate results or reproduce graphs, providing a "what-if" analysis capability. GeoEAS Version 1.2.1 (April 1989) software and documentation is available from EPA's Web site at http://www.epa.gov/ada/csmos/models/geoeas.html

* Also available on EPA's CD-ROM *Site Characterization Library Volume 1* (Release 2) (USEPA 1998c)

Sampling Design Software (Continued)	
Title	**Description**
ELIPGRID-PC	*ELIPGRID-PC* is a program for the design and analysis of sampling grids for locating elliptical targets (e.g., contamination "hot spots"). It computes the probability of success in locating targets based on the assumed size, shape, and orientation of the targets, as well as the specified grid spacing. It also can be used to compute a grid spacing from a specified success probability, compute cost information associated with specified sampling grids, determine the size of the smallest "hot spot" detected given a particular grid, and create graphs of the results. Information, software, and user's guide are available on the World Wide Web at: http://dqo.pnl.gov/software/elipgrid.htm The site is operated for the U.S. Department of Energy Office of Environmental Management by the Pacific Northwest National Laboratory.
DQO-PRO	This software comprises a series of programs with a user interface such as a common calculator and it is accessed using Microsoft Windows. *DQO-PRO* provides answers for three objectives: 1. Determining the rate at which an event occurs 2. Determining an estimate of an average within a tolerable error 3. Determining the sampling grid necessary to detect "hot spots." *DQO-PRO* facilitates understanding the significance of DQOs by showing the relationships between numbers of samples and DQO parameters, such as (1) confidence levels versus numbers of false positive or negative conclusions; (2) tolerable error versus analyte concentration, standard deviation, etc., and (3) confidence levels versus sampling area grid size. The user has only to type in his or her requirements and the calculator instantly provides the answers. Contact: Information and software are available on the Internet at the American Chemical Society, Division of Environmental Chemistry Web site at http://www.acs-envchem.duq.edu/dqopro.htm
Visual Sample Plan (VSP)	VSP provides statistical solutions for optimizing the sampling design. The software can answer two important questions in sample planning: (1) How many samples are needed? VSP can quickly calculate the number of samples needed for various scenarios at different costs. (2) Where should the samples be taken? Sample placement based on personal judgment is prone to bias. VSP provides random or grided sampling locations overlaid on the site map. Information and software available at http://dqo.pnl.gov/VSP/Index.htm VSP was developed in part by Department of Energy's (DOE's) National Analytical Management Program (NAMP) and through a joint effort between Pacific Northwest National Laboratory (PNNL) and Advanced Infrastructure Management Technologies (AIMTech).

Data Quality Assessment Software	
Title	**Description**
DataQUEST	This software tool is designed to provide a quick-and-easy way for managers and analysts to perform baseline Data Quality Assessment. The goal of the system is to allow those not familiar with standard statistical packages to review data and verify assumptions that are important in implementing the DQA Process. This software supports the *Guidance for Data Quality Assessment, EPA QA/G-9* (USEPA 2000d) which demonstrates the use of the DQA Process in evaluating environmental data sets. Download from EPA's World Wide Web site at http://www.epa.gov/quality/qa_docs.html
*ASSESS 1.01a**	This software tool was designed to calculate variances for quality assessment samples in a measurement process. The software performs the following functions: (1) transforming the entire data set, (2) producing scatter plots of the data, (3) displaying error bar graphs that demonstrate the variance, and (4) generating reports of the results and header information. Available on EPA's CD-ROM *Site Characterization Library Volume 1* (Release 2) (USEPA 1998c)
MTCA*Stat*	This software package is published by the Washington Department of Ecology and can be used to calculate sample sizes (for both normal and lognormal distributions), basic statistical quantities, and confidence intervals. Requires MS Excel 97. The USEPA Office of Solid Waste has not evaluated this software for use in connection with RCRA programs, however, users of this guidance may wish to review the software for possible application to some of the concepts described in this document. Available from Washington Department of Ecology's "Site Cleanup, Sediments, and Underground Storage Tanks" World Wide Web site at http://www.ecy.wa.gov/programs/tcp/tools/toolmain.html

* Also available on EPA's CD-ROM *Site Characterization Library Volume 1* (Release 2) (USEPA 1998c)

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APPENDIX I

EXAMPLES OF PLANNING, IMPLEMENTATION, AND ASSESSMENT FOR RCRA WASTE SAMPLING

This appendix presents the following two hypothetical examples of planning, implementation, and assessment for RCRA waste sampling:

Example 1:	Sampling soil in a RCRA Solid Waste Management Unit (SWMU) to confirm attainment of the cleanup standard (using the mean to measure compliance with a standard)
Example 2:	Sampling of a process waste to make a hazardous waste determination (using a maximum or upper percentile to measure compliance with a standard).

Example 1: Sampling Soil at a RCRA SWMU to Confirm Attainment of a Cleanup Standard

Introduction

In this example, the owner of a permitted TSDF completed removal of contaminated soil at a SWMU as required under the facility's RCRA permit under EPA's RCRA Corrective Action Program. The permit required the facility owner to conduct sampling and analysis to determine if the remaining soil attains the facility-specific risk-based standard specified in the permit. This hypothetical example describes how the planning, implementation, and assessment activities were conducted.

Planning Phase

The planning phase included implementation of EPA's systematic planning process known as the Data Quality Objectives (DQO) Process and preparation of a quality assurance project plan (QAPP). A DQO planning team was assembled, and the DQO Process was implemented following EPA's guidance in *Guidance for the Data Quality Objectives Process for Hazardous Waste Site Operations EPA QA/G-4HW* (USEPA 2000a), *Guidance for the Data Quality Objectives Process EPA QA/G-4* (USEPA 2000b), and Chapter Nine of SW-846.

The outputs of the seven steps of the DQO Process are outlined below.

DQO Step 1: Stating the Problem

- The DQO planning team included the facility owner, a technical project manager, a chemist, environmental technician (sampler), and a facility engineer familiar with statistical methods. As part of the DQO Process, the team consulted with their state regulator to determine if the State has any additional regulations or guidance that applies. A state guidance document provided recommendations for the parameter of interest and the acceptable Type I decision error rate.

- A concise description of the problem was developed as follows: The facility conducted a soil removal action at the SWMU. Soil with concentrations greater than the risk-based cleanup standard of 10 mg/kg of pentachlorophenol (PCP) was excavated for off-site disposal. Removal was guided by the results of grab samples analyzed for PCP using a semi-quantitative field analytical method.

- The conceptual site model (CSM) assumed that the PCP migrated downward into the soil, and that if a soil layer were found to be "clean," then the underlying soil layer also would be assumed "clean."

- The technical staff were given six weeks to complete the study and submit a draft report to the regulatory agency.

DQO Step 2: Identifying Possible Decisions

- Decision statement: The study objective was to determine if the soil remaining in the SWMU after removal of the contaminated soil attained the cleanup standard. If the standard is attained, then the area will be backfilled with clean fill and reserved for future industrial development. If the standard is not attained, then the next layer of soil within the SWMU will be removed.

DQO Step 3: Identifying Inputs to the Decision

- The sample analysis results for total PCP (in mg/kg) in soil were used to decide whether or not the soil attained the cleanup. PCP was designated as the only constituent of concern, and its distribution within the SWMU was assumed to be random. The risk-based cleanup level for PCP in soil was set at 10 mg/kg.

- The decision was based on the concentrations in the top six-inch layer of soil across the entire SWMU. The study was designed to determine whether the entire unit attains the standards, or does not.

- The chemist identified two candidate analytical methods for measuring PCP concentrations in soil: (1) SW-846 Method 4010A "Screening For Pentachlorophenol By Immunoassay" ($20/analysis), and (2) SW-846 Method 8270 (and prep method 3550) ($110/analysis). The project chemist confirmed that both methods were capable of achieving a quantitation limit well below the action level of 10 mg/kg. During Step 7 of the DQO Process, the chemist revisited this step to select a final method and prepare method performance criteria as part of the overall specification of decision performance criteria.

- The planning team identified the need to specify the size, shape, and orientation of each sample to satisfy the acceptable sampling error (specified in DQO Process Step 7) and to enable selection of the appropriate sampling device (during development of the QAPP). Because the soil exists in a relatively flat stationary three-dimensional unit, it was considered a series of overlapping two-dimensional surfaces for the purposes of sampling. The correct orientation, size, and shape of each sample was a vertical core capturing the full six-inch thickness of the soil unit. The minimum mass of each primary field sample was determined during DQO Process Step 7 using the particle size-weight relationship required to control fundamental error at an acceptable level.

DQO Step 4: Defining Boundaries

- The dimensions of the SWMU were approximately 125 feet by 80 feet (10,000 square feet). The SWMU was relatively flat. The depth of interest was limited to the top six inches of soil in the unit after removal of the contaminated soil. The spatial boundary of the SWMU was defined by the obvious excavation and by wooden stakes at the corners of the excavation.

- The soil within the study boundary was loamy sand with a maximum particle size of about 1.5 mm (0.15 cm).

- The project team planned to collect samples within a reasonable time frame, and degradation or transformation of the PCP over the investigation period was not a concern.

DQO Step 5: Developing Decision Rules

- The population parameter of interest was the *mean.* The mean was selected as the parameter of interest because the risk-based cleanup standard (Action Level) was derived based upon long-term average health effects predicted from exposures to the contaminated soil.

- The risk-based action level was 10 mg/kg total pentachlorophenol (PCP) in soil.

- The decision rule was then established as follows: “If the mean concentration for PCP in the soil is less than 10 mg/kg, then the cleanup standard is attained. Otherwise, the SWMU will be considered contaminated and additional remedial action will be required.”

DQO Step 6: Specifying Limits on Decision Errors

- The major sources of variability (measured as the relative variance) were identified as within-sample unit variability (s_w^2) (including analytical imprecision and Gy’s fundamental error) and between-sample unit variability (s_b^2) (or population variability). The total study variance (s_T^2) , expressed as the relative variance, was estimated using the following relationship:

$$\begin{aligned} s_T^2 &= s_b^2 + s_w^2 \\ &= s_b^2 + s_s^2 + s_a^2 \end{aligned}$$

where s_b^2 = between-unit variance (population variance), s_s^2 = sample collection imprecision (estimated by Gy's fundamental error, s_{FE}^2), and s_a^2 = analytical imprecision (determined from the measurement of laboratory control samples with concentrations near the Action Level).

- Sample analysis results for eight samples of soil excavated from the previous lift gave a standard deviation and mean of s = 7.1 and $\bar{x}$ = 10.9 respectively. The total study relative standard deviation (s_T) was then estimated as 0.65.

- The relative standard deviation (RSD) of the sampling error (s_s) was estimated as 0.10 (as estimated by Gy's fundamental error), based a maximum observed particle size of approximately 1.5 mm (0.15 cm) and a sample mass of 10 grams.

- The RSD for the analytical imprecision (s_a) associated with the field screening method (SW-846 Method 4010A - "Screening For Pentachlorophenol By Immunoassay") was estimated from replicate measurements as 0.40.

- The between-unit (population) relative standard deviation (s_b) was then estimated as:

$$\begin{aligned} s_b &= \sqrt{s_T^2 - (s_s^2 + s_a^2)} \\ &= \sqrt{(.65)^2 - (.10^2 + .40^2)} = 0.50 \end{aligned}$$

- Two potential decision errors could be made based on interpreting sampling and analytical data:

  Decision Error A: Concluding that the mean PCP concentration within the SWMU was less than 10 mg/kg when it was truly greater than 10 mg/kg, or

  Decision Error B: Concluding that the mean PCP concentration within the SWMU was greater than 10 mg/kg when it was truly less than 10 mg/kg.

The consequences of Decision Error A, incorrectly deciding the SWMU was "clean" (mean PCP concentration less than 10 mg/kg), would leave contaminated soil undetected and would likely increase health risks for onsite workers and pose potential future legal problems for the owner.

The consequences of Decision Error B, incorrectly deciding the SWMU was "not clean" (mean PCP concentration greater than or equal to 10 mg/kg), would cause the needless expenditure of resources (e.g., funding, time, backhoe and operator, soil disposal, sampling crew labor, and analytical capacity) for unnecessary further remedial action.

Error A, incorrectly deciding that the mean PCP concentration is less than the action level of 10 mg/kg, posed more severe consequences for human health plus liability and compliance concerns. Consequently, the baseline condition chosen for the SWMU was that the mean PCP concentration within the SWMU is truly greater than or equal to the action level of 10 mg/kg.

Table I-1. Null Hypothesis and Possible Decision Errors for Example 1

"Null Hypothesis" (baseline condition)	***Possible Decision Errors***	
	Type I Error (α), False Rejection	***Type II Error (β), False Acceptance***
The true mean concentration of PCP in the SWMU is greater than or equal to the risk-based cleanup standard (i.e., the SWMU is contaminated).	Concluding the site is "clean" when, in fact, it is contaminated.	Concluding the site is still contaminated when, in fact, it is "clean."

- Next, it was necessary to specify the boundaries of the gray regions. The gray region defines a range that is less than the action limit, but too close to the Action Level to be considered "clean," given uncertainty in the data. When the null hypothesis (baseline condition) assumes that the site is contaminated (as in this example), the upper limit of the gray region is bounded by the Action Level; the lower limit is determined by the decision maker. The project team sets the lower bound of the gray region at 7.5 mg/kg, with the understanding that this bound could be modified after review of the outputs of Step 7 of the DQO Process.

- The planning team set the acceptable probability of making a Type I (false rejection) error at 5 percent ($\alpha = 0.05$) based on guidance provided by the State regulatory agency. In other words, the team was willing to accept a 5 percent chance of concluding the SWMU was clean, if in fact it was not. While a Type II (false acceptance) error could prove to be costly to the company, environmental protection and permit compliance are judged to be most important. The planning team decides to set the Type II error rate at only 20 percent.

- The information collected in Step 6 of the DQO Process is summarized below.

Table I-2. Initial Outputs of Step 6 of the DQO Process

Needed Parameter	**Output**
Action Level (AL)	10 mg/kg
Gray Region	7.5 - 10 mg/kg (width of gray region, Δ = 2.5)
Relative Width of Gray Region	(10 - 7.5)/7.5 = 0.33
Null Hypothesis (H_o)	Mean (PCP) $\geq$ 10 mg/kg
False Rejection Decision Error Limit (probability of a Type I error)	$\alpha = 0.05$
False Acceptance Decision Error Limit (probability of a Type II error)	$\beta = 0.20$

DQO Step 7: Optimizing the Data Collection Design

1. ***Review outputs from the first six steps of the DQO Process.*** The project team reviewed the outputs of the first six steps of the DQO Process. They expected the PCP concentration to be near the cleanup standard (Action Level); thus, it was decided that a probabilistic sampling design would be used so that the results could be stated with a known probability of making a decision error.

2. ***Consider various data collection designs.*** The objective of this step was to find cost-effective design alternatives that balance the number of samples and the measurement performance, given the feasible choices for sampling designs and measurement methods. Based on characterization data from the excavated soil, the planning team assumed that the between-sample unit variability or population variability would remain relatively stable at approximately $s_b = 0.50$, independent of the sampling and analytical methods used. The planning team investigated various combinations of sampling and analytical methods (with varying associated levels of precision and cost) as a means find the optimal study design.

   The planning team considered three probabilistic sampling designs: simple random, stratified random, and systematic (grid-based) designs. A composite sampling strategy also was considered. All designs allowed for an estimate of the mean to be made. Because the existence of strata was not expected (although could be discovered during the investigation), the stratified design was eliminated from consideration. A simple random design is the simplest of the probabilistic sampling methods, but it may not provide very even coverage of the SWMU; thus, if spatial variability becomes a concern, then it may go undetected with a simple random design. The systematic design provides more even coverage of the SWMU and typically is easy to implement.

   The practical considerations were considered for each alternative design, including site access and conditions, equipment selection/use, experience needed, special analytical needs, health and safety requirements, and scheduling. There were no significant practical constraints that would limit the use of either the systematic or the simple random sampling designs; however, the systematic design was preferred because it provides sampling locations that are easier to survey and locate in the field, and it provides better spatial coverage. Ultimately, two sampling designs were evaluated: a systematic sampling design and a systematic sampling design that incorporates composite sampling.

The acceptable mass of each primary field sample was determined using the particle size-weight relationship required to control fundamental error. The soil in the SWMU is a granular solid, and the 95th percentile particle size (d) was estimated at 1.5 mm (0.15 cm). To maintain the relative standard deviation of the fundamental error at 0.10, a sample mass of at least 8.2 grams was required (using Equation D.4 in Appendix D). To maintain the relative standard deviation of the fundamental error at 0.05, a sample mass of at least 30 grams would be required. There were no practical constraints on obtaining samples of these sizes.

Next, it was necessary to estimate unit costs for sampling and analysis. Based on prior experience, the project team estimated the cost of collecting a grab sample at $40 – plus an additional $30 per sample for documentation, processing of field screening samples, and $60 per sample for documentation, processing, and shipment for samples sent for fixed laboratory analysis.

3. ***Select the optimal number of samples.*** Using the initial outputs of Step 6, the appropriate number of samples was calculated for each sampling design:

For the systematic sampling design (without compositing), the following formula was used (Equation 8 from Section 5.4.1):

$$n = \frac{(z_{1-\alpha} + z_{1-\beta})^2 s_T^2}{\Delta^2} + \frac{z_{1-\alpha}^2}{2}$$

where

$z_{1-\alpha}$ = the pth quantile of the standard normal distribution (from the last row of Table G-1, Appendix G), where α is the probability of making a Type I error (the significance level of the test) set in DQO Step 6.

$z_{1-\beta}$ = the pth quantile of the standard normal distribution (from the last row of Table G-1, Appendix G), where β is the probability of making a Type II error set in DQO Step 6.

s_T = an estimate of the total study relative standard deviation.

Δ = the width of the gray region from DQO Step 6 (expressed as the relative error in this example).

[EPA's DEFT software could be used to calculate the appropriate number of samples (see *Data Quality Objectives Decision Error Feasibility Trials Software (DEFT) - User's Guide,* USEPA 2001h). Note, however, that the DEFT program asks for the bounds of the gray region specified in absolute units. If the planning team uses the relative standard deviation (or coefficient of variation) in the sample size equation rather than the absolute standard deviation, then the bounds of the gray region also must be input into DEFT as relative values. Thus, the Action Level would be set equal to 1, and the other bound of the gray region would be set equal to 1 - (relative width of gray region) or 1 + (relative width of gray region) depending what baseline condition is selected.]

Note that if there were more than one constituent of concern, then the appropriate number of samples would need to be calculated for each constituent using preliminary estimates of their standard deviations. The number of samples would then be determined by the highest number of samples obtained for any single constituent of concern.

The sample size for <u>systematic composite sampling</u> also was evaluated. In comparison to non-composite sampling, composite sampling can have the effect of minimizing between-sample variation, thereby reducing somewhat the total number of composite samples that must be submitted for analysis. In addition, composite samples are expected to generate normally distributed data thereby allowing the team to apply normal theory statistical methods. To estimate the sample size, the planning team again required an estimate of the standard deviation. However, since the original estimate of the standard deviation was based on available individual or "grab" sample data rather than composite samples, it was necessary to adjust the variance term in the sample size equation for the appropriate number of composite samples. In the sample size equation, the between-unit (population) component of variance (s_b^2) was replaced with s_b^2/g , where g is the number of individual or "grab" samples used to form each composite. Sample sizes were then calculated assuming $g = 4$.

Table I-3 and Table I-4 summarize the inputs and outputs of Step 7 of the DQO Process and provides the estimated costs for the various sampling and analysis designs evaluated.

Table I-3. Summary of Inputs for Candidate Sampling Designs

Parameter	**Systematic Sampling - Fixed Lab Analyses**	**Systematic Sampling - Field Analyses**	**Systematic Composite Sampling - Fixed Lab Analyses**	**Systematic Composite Sampling - Field Analyses**
Inputs				
Sampling Costs				
Collection Cost (per "grab")	\$40 ea.	\$40 ea.	\$40 ea.	\$40 ea.
Documentation, processing, shipment	\$60 ea.	\$30 ea.	\$60 ea.	\$30 ea.
Analytical Costs				
SW-846 Method 3550/8270 (fixed lab)	\$110 ea.	\$110 ea.*	\$110 ea.	\$110 ea.*
SW-846 Method 4010A (field screening)	NA	\$20 ea.	NA	\$20 ea.
Relative Width of Gray Region (Δ)	0.33	0.33	0.33	0.33
Null Hypothesis (H_o)	Mean (PCP) $\geq$ 10 mg/kg	Mean (PCP) $\geq$ 10 mg/kg	Mean (PCP) $\geq$ 10 mg/kg	Mean (PCP) $\geq$ 10 mg/kg
False Rejection Decision Error Limit	$\alpha = 0.05$	$\alpha = 0.05$	$\alpha = 0.05$	$\alpha = 0.05$
False Acceptance Decision Error Limit	$\beta = 0.20$	$\beta = 0.20$	$\beta = 0.20$	$\beta = 0.20$
Relative Std. Dev.				
Sampling (s_s)	0.10	0.10	0.10	0.10
Analytical (s_a), SW-846 Method 8270	0.10	NA	0.10	NA
Analytical (s_a) SW-846 Method 4010A	NA	0.40	NA	0.40
"Population" (s_b)	0.50	0.50	0.50	0.50
Total Study $s_T = \sqrt{s_s^2 + s_a^2 + s_b^2}$	0.52	0.65	0.29**	0.48**

NA: Not applicable

* Assumes 20-percent of all field analyses must be confirmed via fix laboratory method.

** For composite sampling, the total study relative standard deviation (s_T) was estimated by replacing s_b^2 with s_b^2 / g , where g = the number of "grabs" per composite.

Table I-4. Summary of Outputs for Candidate Sampling Designs

Parameter	Systematic Sampling - Fixed Lab Analyses	Systematic Sampling - Field Analyses	Systematic Composite Sampling - Fixed Lab Analyses	Systematic Composite Sampling - Field Analyses
Outputs				
Number of Samples (n)	17	25	6	15
Cost Estimate				
“Grab” Sampling	$40 x 17	$40 x 25	$40 x 4 x 6 (see note 1)	$40 x 4 x 15 (see note 1)
Documentation, processing, and shipment	$60 x 17	($30 x 25) + ($60 x 5) (see note 2)	$60 x 6	($30 x 15) + ($60 x 3) (see note 2)
SW-846 Method 3550/8270 (fixed lab)	$110 x 17	$110 x 5 (see note 2)	$110 x 6	$110 x 3 (see note 2)
SW-846 Method 4010A (field screening)	NA	$20 x 25	NA	$20 x 15
Cost	**$3,570**	**$3,100**	**$1,980**	**$3,660**

1. The calculation assumes four grabs per composite sample.
2. The calculation includes costs for shipment and analysis of 20% of field screening samples for fixed laboratory analysis.
NA: Not applicable

4. ***Select a resource-effective design.*** It was determined that all of the systematic designs and systematic composite sampling designs would meet the statistical performance requirements for the study in estimating the mean PCP concentration in the SWMU. The project team selected the systematic composite sampling design - with fixed laboratory analysis - based on the cost savings projected over the other sampling designs.

   The planning team decided that one additional field quality control sample (an equipment rinsate blank), analyzed by SW-846 Method 8720, was required to demonstrate whether the sampling equipment was free of contamination.

   The outputs of the DQO Process were summarized in a memo report which was then used help prepare the QAPP.

5. ***Prepare a QAPP.*** The operational details of the sampling and analytical activities were documented in the QAPP using *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5* (USEPA 1998a) and Chapter One of SW-846 for guidance.

Implementation Phase

The QAPP was implemented in accordance with the schedule, sampling plan, and safety plan. The exact location of each field sample was established using a grid on a map of the SWMU. The start point for constructing the grid was selected at random.

The QAPP established the following DQOs and performance goals for the sampling equipment:

- The correct orientation and shape of each sample is a vertical core.
- Each sample must capture the full depth of interest (six inches).
- The minimum mass of each sample is 10 g.
- The device must be constructed of materials that will not alter analyte concentrations due to loss or gain of analytes via sorption, desorption, degradation, or corrosion.
- The device must be easy to use, safe, and low cost.

A sampling device was selecting using the four-steps described in Figure 28 in Section 7.1.

Step 1 - Identify the Medium to be Sampled

The material to be sampled is a soil. Using Table 8 in Section 7.1, we find the media descriptor that most closely matches the waste in the first column of the table: “Soil and other unconsolidated geologic material.”

Step 2 - Select the Sample Location

The second column of Table 8 in Section 7.1 provides a list of possible sampling sites (or units types) for soil (i.e., surface or subsurface). In this example, the sampling location is surface soil and “Surface” is found in the second column in the table.

Step 3 - Identify Candidate Sampling Devices

The third column of Table 8 in Section 7.1 provides a list of candidate sampling devices. For the waste stream in this example, the list includes bucket auger, concentric tube thief, coring type sampler, miniature core sampler, modified syringe, penetrating probe sampler, sampling scoop/trowel/shovel, thin-walled tube, and trier.

Step 4 - Select Devices

Sampling devices were selected from the list of candidate sampling devices after review of Table 9 in Section 7.1. Selection of the equipment was made after consideration of the DQOs for the sample support (i.e., required volume, depth, shape, and orientation), the performance goals established for the sampling device, ease of use and decontamination, worker safety issues, cost, and any practical considerations.

Table I-5 demonstrates how the DQOs and performance goals can be used together to narrow the candidate devices down to just one or two.

Table I-5. Using DQOs and Performance Goals to Select a Final Sampling Device

Candidate Devices	***Data Quality Objectives and Performance Goals***				
	Required Depth	***Orientation and Shape***	***Sample Volume***	***Operational Considerations***	***Desired Material of Construction***
	6 inches	Vertical undisturbed core	>10 g	Device is portable, safe, & low cost?	Stainless or carbon steel
Bucket auger	Y	N	Y	Y	Y
Concentric tube thief	Y	N	Y	Y	Y
Coring Type Sampler	Y	N	Y	Y	Y
Miniature core sampler	Y	Y	N	Y	N
Modified syringe sampling	N	N	N	Y	N
Penetrating Probe Sampler	Y	Y	Y	Y	Y
Scoop, trowel, or shovel	Y	N	Y	Y	Y
Thin-walled tube	Y	Y	Y	Y	Y
Trier	Y	N	Y	Y	Y

Key: Y = The device is capable of achieving the specified DQO or performance goal.
N = The device is not capable of achieving the DQO or performance goal.

The “penetrating probe sampler” and the “thin-walled tube” were identified as the preferred devices because they could satisfy all of the DQOs and performance goals for the sampling devices. The penetrating probe was selected because it was easy to use and was readily available to the field sampling crew.

A penetrating probe sampler was then used to take the field samples at each location on the systematic square grid (see Figure I-1). Each composite sample was formed by pooling and mixing individual samples collected from within each of four quadrants. The process was repeated until six composite samples were obtained. Because the total mass of each individual (grab) sample used to form composite samples exceeded that required by the laboratory for analysis, a field subsampling routine was used to reduce the volume of material submitted to the laboratory.

The field samples and associated field QC samples were submitted to the laboratory where a subsample was taken from each field sample for analysis. The samples were analyzed in accordance with the QAPP.

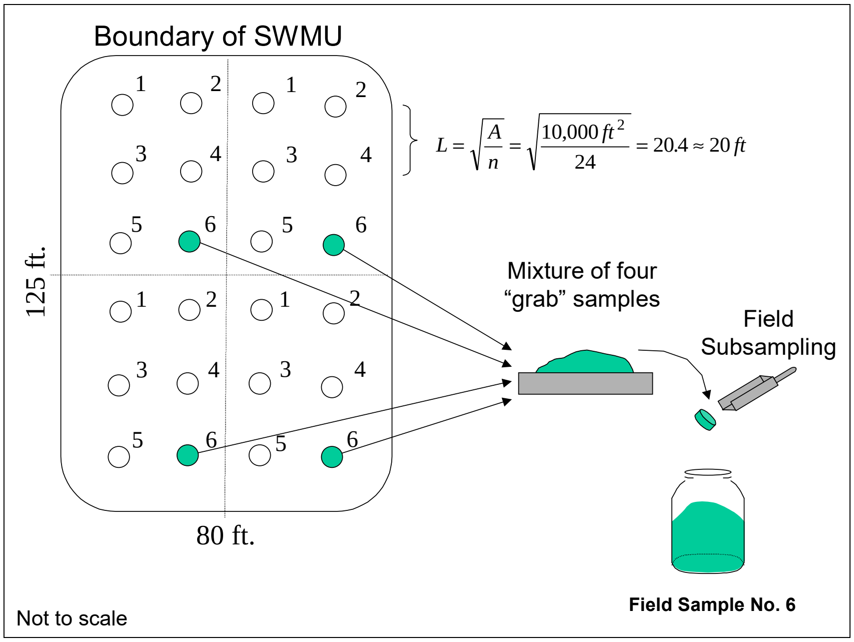


Figure I-1. Systematic sampling with compositing. The distance between sampling points (*L*) is determined using the approach described in Section 5.2.3 (Box 5). Samples with the same number are pooled and mixed to form each composite sample. A field sample is formed from each composite using one of the subsampling methods described in Section 7.3.2 (e.g., by fractional shoveling).

Assessment Phase

Data Verification and Validation

Sampling and analytical records were reviewed to check compliance with the QAPP. The data collected during the study met the measurement objectives. Sampling and analytical error were minimized through the use of a statistical sampling design, correct field sampling and subsampling procedures, and adherence to the requirements of the analytical methods. The soil that was sampled did not present any special problems concerning access to sampling locations, equipment usage, particle-size distribution, or matrix interferences. A quantitation limit of 0.5 mg/kg was achieved. The analytical package was verified and validated, and the data generated were judged acceptable for their intended purpose.

Data Quality Assessment (DQA)

DQA was performed using the approach outlined in Section 8.2:

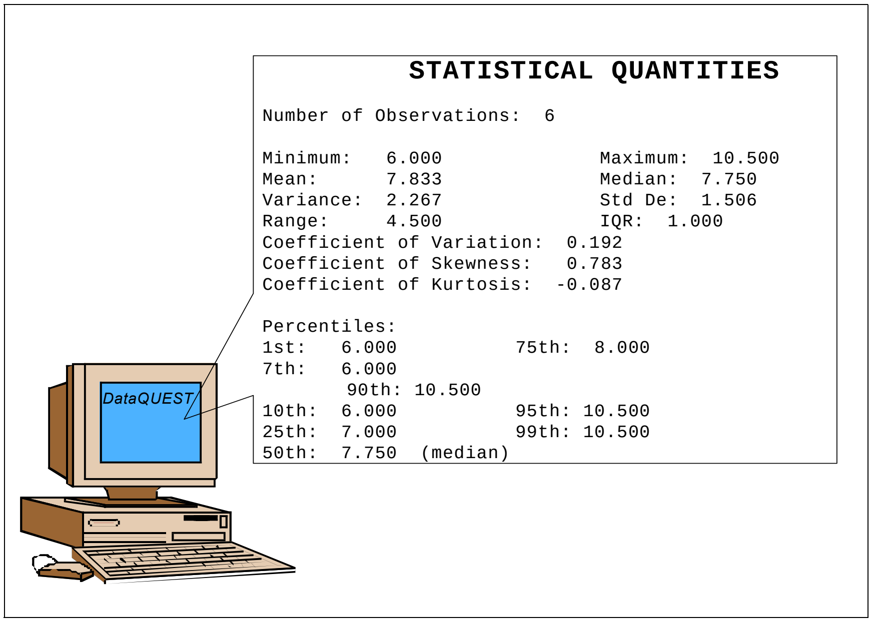
1. ***Review DQOs and sampling design.*** The DQO planning team reviewed the original objectives: "If the mean concentration for PCP in the soil is less than 10 mg/kg, then the cleanup standard is attained. Otherwise, the SWMU will be considered contaminated and additional remedial action will be required."

2. ***Prepare the data for statistical analysis.*** The summary of the verified and validated data were received in hard-copy format and an electronic data base was created by manual data entry into spreadsheet software. The data base was checked by a second person for accuracy. The results for the data collection effort are listed in Table I-6. A data file was created in a format suitable for import into EPA's *DataQUEST* software.

Table I-6. Soil Sample Analysis Results for PCP (mg/kg)

Sample Identification	*Result (PCP, mg/kg)*
1	8.0
2	8.0
3	7.0
4	6.0
5	10.5
6	7.5

3. ***Conduct preliminary analysis of data and check distributional assumptions***: Using EPA's *DataQUEST*, statistical quantities were computed as shown in Figure I-2.



```
             STATISTICAL QUANTITIES

Number of Observations:  6

Minimum:   6.000              Maximum:  10.500
Mean:      7.833              Median:  7.750
Variance:  2.267              Std De:  1.506
Range:     4.500              IQR:  1.000
Coefficient of Variation:  0.192
Coefficient of Skewness:   0.783
Coefficient of Kurtosis:  -0.087

Percentiles:
1st:   6.000          75th:  8.000
7th:   6.000
       90th: 10.500
10th:  6.000          95th: 10.500
25th:  7.000          99th: 10.500
50th:  7.750  (median)
```

Figure I-2. Statistical quantities using *DataQUEST* software

On a normal probability plot, the data plot as a straight line, indicating approximate normality (see Figure I-3).

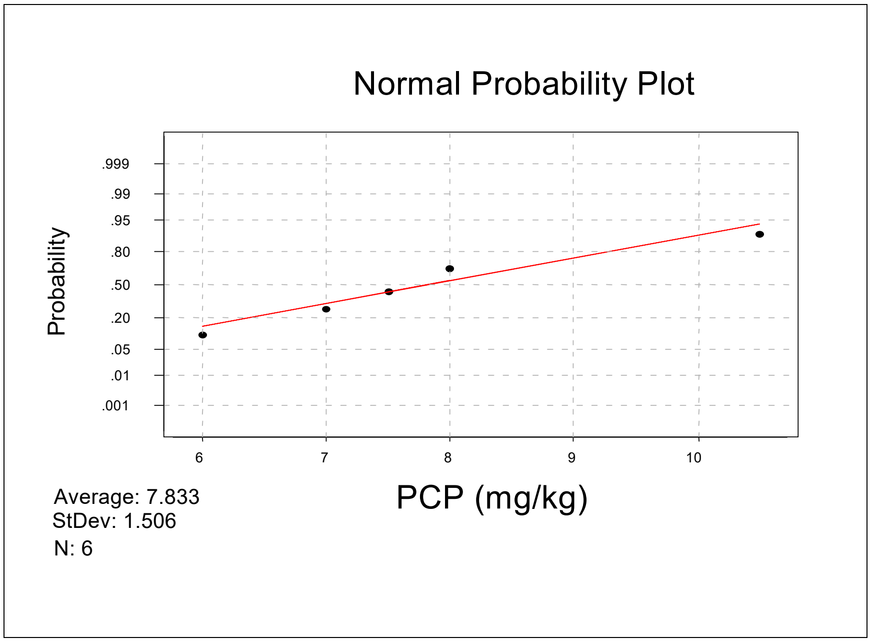
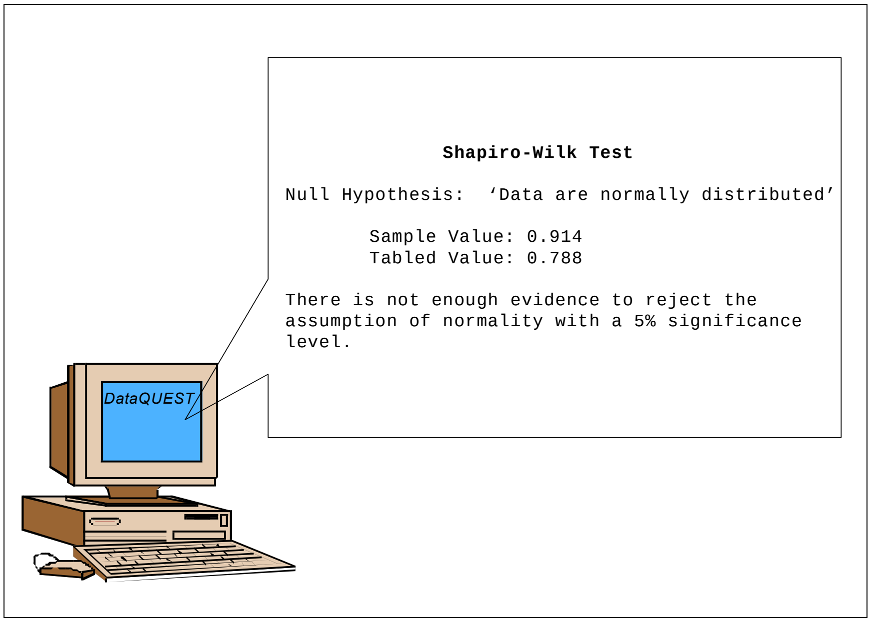


Figure I-3. Normal probability plot

The data also were checked for normality by the Shapiro-Wilk test. Using the *DataQUEST* software, the Shapiro-Wilk test was performed at the 0.05 percent significant level. The Shapiro-Wilk test did not reject the null hypothesis of normality (see Figure I-4).



```
                  Shapiro-Wilk Test

Null Hypothesis:  'Data are normally distributed'

       Sample Value: 0.914
       Tabled Value: 0.788

There is not enough evidence to reject the
assumption of normality with a 5% significance
level.
```

Figure I-4. Results of the Shapiro-Wilk test using EPA's *DataQUEST* software

4. ***Select and perform the statistical test***: The analysis of the data showed there were no "non-detects" and a normal distribution was an acceptable model. Using the guidance in Figure 38 (Section 8.2.4), a parametric upper confidence limit (UCL) on the mean was selected as the correct statistic to compare to the regulatory level. The 95% UCL on the mean was calculated as follows:

$$\begin{aligned} UCL_{0.95} &= \bar{x} + t_{0.95,n-1}\frac{s}{\sqrt{n}} \\ &= 7.833 + 2.015\left(\frac{1.506}{\sqrt{6}}\right) \\ &= 9.1 \ \text{mg/kg} \end{aligned}$$

   The tabulated "t value" (2.015) was obtained from Table G-1 in Appendix G and based on a 95-percent one-tailed confidence interval with $\alpha = 0.05$ and 5 degrees of freedom.

5. ***Draw conclusions and report results:*** The 95% UCL for the mean of the sample analysis results for PCP, 9.1 mg/kg, was less than the specified cleanup level of 10 mg/kg. Thus, the null hypothesis was rejected, and the owner made the determination that the soil remaining in the SWMU attains the cleanup standard for PCP based on the established decision rule.

   A summary report including a description of all planning, implementation, and assessment activities was submitted to the regulatory agency for review.

Example 2: Sampling of a Process Waste to Make a Hazardous Waste Determination

Introduction

An aircraft manufacturing and maintenance facility strips paint from parts before remanufacturing them. The facility recently switched its paint stripping process from a solvent-based system to use of an abrasive plastic blasting media (PBM). The waste solvent, contaminated with stripped paint, had to be managed as a hazardous waste. The facility owner changed the process to reduce - or possibly eliminate - the generation of hazardous waste from this operation and thereby reduce environmental risks and lower waste treatment and disposal costs.

The plant operators thought the spent PBM could include heavy metals such as chromium and cadmium from the paint, and therefore there was a need to make a hazardous waste determination in order to comply with the RCRA regulations at 40 CFR Part 262.11. The facility owner determined that the spent PBM is a solid waste under RCRA but not a listed hazardous waste. The facility owner then needed to determine if the solid waste exhibits any of the characteristics of hazardous waste: ignitability (§261.21), corrosivity (§261.22), reactivity (§261.23), or toxicity (§261.24). Using process and materials knowledge, the owner determined that the waste blasting media would not exhibit the characteristics of ignitability, corrosivity, or reactivity. The facility owner elected to conduct waste testing to determine if the waste blasting media exhibits the characteristic of toxicity.

This hypothetical example describes how the planning, implementation, and assessment activities were conducted.

Planning Phase

The planning phase comprises the Data Quality Objectives (DQO) Process and preparation of a quality assurance project plan (QAPP) including a sampling and analysis plan. A DQO planning team was assembled and the DQO Process was implemented following EPA's guidance in *Guidance for the Data Quality Objectives Process EPA QA/G-4* (USEPA 2000b) and SW-846.

The outputs of the seven steps of the DQO Process are outlined below.

DQO Step 1: Stating the Problem

- The DQO planning team included the plant manager, a technical project manager, a consulting chemist, and the paint stripping booth operator who also served as the sampler.

- The conceptual model of the waste generation process was developed as follows: The de-painting operation consists of a walk-in blast booth with a reclamation floor. After blasting, the plastic blast media, mixed with paint fines, is passed through a reclamation system; the reusable media is separated out for reloading to the blast unit, while the spent media and paint waste is discharged to a container.

- A concise description of the problem was developed as follows: The problem was described as determining whether the new waste stream (the spent plastic blasting media and waste paint) should be classified as a hazardous waste that requires treatment and subsequent disposal in a RCRA Subtitle C landfill (at $300 per ton), or whether it is a nonhazardous industrial waste that can be land-disposed in an industrial landfill (at $55 per ton).

- The plant manager gave the plant staff and consultant 60 days to complete the study. The turn-around time was established to minimize the amount of time that the waste was stored at the facility while the data were being generated, and to allow adequate time to have the waste shipped off site - if it were found to be a hazardous waste - within the 90-day accumulation time specified at 40 CFR Part 262.34(a).

DQO Step 2: Identifying Possible Decisions

- Decision statement: The decision statement was determining whether the spent PBM paint waste was hazardous under the RCRA regulations.

- Alternative actions: If the waste was hazardous, then treatment and subsequent disposal in a RCRA landfill would be required.

DQO Step 3: Identifying Inputs to the Decision

- The decision was to be based on the quantity of waste generated over approximately a one-month period, but not to exceed the quantity placed in a single 10-cubic yard roll off box.

- Based on process and materials knowledge, the team specified cadmium and chromium as the constituents of concern.

- To resolve the decision statement, the planning team needed to determine if, using the Toxicity Characteristic Leaching Procedure (TCLP) SW-846 Method 1311, the extract from a representative sample of the waste contained the constituents of concern at concentrations equal to or greater than their regulatory levels as required by the RCRA regulations at 40 CFR 261.24. The chemist noted, however, that the TCLP method allows the following: “If a total analysis of the waste demonstrates that individual analytes are not present in the waste, or that they are present but at such low concentrations that the appropriate regulatory levels could not possibly be exceeded, the TCLP need not be run.” With that flexibility in mind, the planning team identified a candidate method for total analysis (including SW-846 Method 3050B/6010), and noted that the TCLP would be required if the total analysis indicated TC levels could be exceeded.

- The project chemist found that SW-846 Methods 3010A (prep) and 6010B were suitable for analysis of the TCLP extracts at quantitation limits at or below the applicable regulatory levels.

- The minimum sample "support" was determined as follows: Method 1311 (TCLP) specifies a minimum sample mass of 100 grams for analysis of nonvolatile constituents and a maximum particle size of 9.5 mm. The waste stream, composed of dry fine to medium-grained plastic and paint chips, was well within the particle size requirements of the TCLP. During Step 7 of the DQO Process, the planning team revisited this step to determine whether a sample mass larger than 100-grams would be necessary to satisfy the overall decision performance criteria.

DQO Step 4: Defining Boundaries

- The paint stripping operation includes a blast booth, a PBM reclamation unit, and a waste collection roll-off box that complies with the applicable container requirements of Subparts I and CC of 40 CFR part 265. The spent blast media and paint waste is discharged to the roll-off box from the reclamation unit. Each discharge event was considered a "batch" for the purposes of the waste classification study.

- When testing a solid waste to determine if it exhibits a characteristic of hazardous waste, the determination must be made when management of the solid waste would potentially be subject to the RCRA hazardous waste regulations at 40 CFR Part 262 through 265. Accordingly, the planning team decided samples should be obtained at the point where the waste discharges from the reclamation unit into the roll-off container (i.e., the point of generation). Until such time that the generator determined that the waste is not a hazardous waste, the generator complied with the applicable pre-transport requirements at 40 CFR Part 262 - Subpart C (i.e., packaging, labeling, marking, and accumulation time).

- The boundary of the decision was set as the extent of time over which the decision applies. The boundary would change only if there were a process or materials change that would alter the composition of the waste. Such a process or materials change could include, for example, a change in the composition, particle size or particle shape of the blasting media, or a significant change in the application (pressure) rate of the blast media.

DQO Step 5: Developing Decision Rules

- The planning team reviewed the RCRA regulations at for the Toxicity Characteristic at 40 CFR 261.24 and found the regulation does not specify a parameter of interest (such as the mean or a percentile). They observed, however, that the Toxicity Characteristic (TC) regulatory levels specified in Table 1 of Part 261.24 represent "maximum" concentrations that cannot be equaled or exceeded; otherwise, the solid waste must be classified as hazardous. While the regulations for hazardous waste determination do not require the use of any statistical test to make a hazardous waste determination, the planning team decided to use a high percentile value as a reasonable approximation of the maximum TCLP sample analysis result that could be obtained from a sample of the waste. Their objective was to "prove the negative" - that is, to demonstrate with a desired level of confidence that the vast majority of the waste was nonhazardous. The upper 90th percentile was selected. The team specified an additional constraint that no single sample could exceed the standard. Otherwise, there may be evidence that the waste is hazardous at least part of the time.

- The Action Levels were set at the TC regulatory limits specified in Table 1 of 40 CFR Part 261.24:

Cadmium:	1.0 mg/L TCLP
Chromium:	5.0 mg/L TCLP

- The decision rule was then established as follows: "If the upper 90th percentile TCLP concentration for cadmium or chromium in the waste <u>and</u> all samples analysis results are less than their respective action levels of 1.0 and 5.0 mg/L TCLP, then the waste can be classified as nonhazardous waste under RCRA; otherwise, the waste will be considered a hazardous waste."

DQO Step 6: Specifying Limits on Decision Errors

- The null hypothesis was that the waste is hazardous, i.e., the true proportion (P) of samples with concentrations of cadmium or chromium less than their regulatory thresholds is less than 0.90, or Ho: $P < 0.90$.

- Two potential decision errors could be made based on interpreting sampling and analytical data:

  Decision Error A: Concluding that the true proportion (P) of the waste that is nonhazardous was greater than 0.90 when it was truly less than 0.90, or

  Decision Error B: Concluding that the true proportion (P) of the waste that is nonhazardous was less than 0.90 when it was truly greater than 0.90.

  The consequences of Decision Error A - incorrectly deciding the waste was nonhazardous - would lead the facility to ship untreated hazardous waste off site for disposal in solid waste landfill, likely increase health risks for onsite workers, and pose potential future legal problems for the owner.

  The consequences of Decision Error B - incorrectly deciding the waste was hazardous when in fact it is not hazardous - would cause the needless costs for treatment and disposal, but with no negative environmental consequences.

  Error A, incorrectly deciding that a hazardous waste is a nonhazardous waste, posed more severe consequences for the generator in terms of liability and compliance concerns. Consequently, the baseline condition (null hypothesis) chosen was that the true proportion of waste that is nonhazardous is less than 90 percent.

Table I-7. Null Hypothesis and Possible Decision Errors for Example 2

"Null Hypothesis" (baseline condition)	***Possible Decision Errors***	
	Type I Error (α), False Rejection	***Type II Error (β), False Acceptance***
The true proportion (P) of waste that is nonhazardous is less than 0.90.	Concluding the waste is nonhazardous when, in fact, it is hazardous.	Concluding the waste is hazardous when, in fact, it is nonhazardous.

- Next, it was necessary to specify the boundaries of the gray region. When the null hypothesis (baseline condition) assumes that the waste is hazardous (as in this example), one limit of the gray region is bounded by the Action Level and the other limit is set at a point where it is desirable to control the Type II (false acceptance) error. The project team set one bound of the gray region at 0.90 (the Action Level). Since a "no exceedance" criterion is included in the decision rule, the other bound of the gray region is effectively set at 1.

- The DQO planning team then sets the acceptable probability of making a Type I (false rejection) error at 10 percent ($\alpha = 0.10$). In other words, they are willing to accept a 10 percent chance of concluding the waste is nonhazardous when at least a portion of the waste is hazardous. The use of the exceedance rule method does not require specification of the Type II (false acceptance) error rate.

- The information collected in Step 6 of the DQO Process is summarized below.

Table I-8. Initial Outputs of Step 6 of the DQO Process - Example 2

Needed Parameter	Output
Action Level	0.90
Gray Region	0.90 to 1.0 (Δ = 0.10)
Null Hypothesis (H_o)	P < 0.90
False Rejection Decision Error Limit (probability of a Type I error)	$\alpha = 0.10$
False Acceptance Decision Error Limit (probability of a Type II error)	Not specified

DQO Step 7: Optimizing the Data Collection Design

- ***Review outputs from the first six steps of the DQO Process.*** The planning team reviewed the outputs of the first six steps of the DQO Process.

- ***Consider various data collection designs.*** The DQO planning team considered two probabilistic sampling designs: simple random and systematic (random within time intervals). Both the simple random and the systematic design would allow the facility owner to estimate whether a high percentage of the waste complies with the standard. The team also considered using an authoritative "biased" sampling design to estimate the high end or "worst case" waste characteristics.

  Two analytical plans were then considered: One in which the full TCLP would be performed on each sample, and one in which TCLP concentrations could be estimated from total concentration by comparing each total sample analysis result to 20 times the TC regulatory limit (to account for the 20:1 dilution used in the TCLP).

  The laboratory requested a sample mass of at least 300 grams (per sample) to allow the laboratory to perform the preliminary analyses required by the TCLP and to provide sufficient mass to perform the full TCLP (if required).

  The practical considerations were then evaluated for each alternative design, including access to sampling locations, worker safety, equipment selection/use, experience needed, special analytical needs, and scheduling.

- ***Select the optimal number of samples.*** Since the decision rule specified no exceedance of the standard in any sample, the number of samples was determined from Table G-3a in Appendix G. The table is based on the formula $n = \log(\alpha)/\log(p)$. For a desired $p = 0.90$ and $(1-\alpha) = 0.90$, the number of samples (n) for a simple random or systematic sampling design was 22.

  The team also considered how many samples might be required if a nonprobabilistic authoritative sampling design were used. Some members of the planning team thought that significantly fewer samples (e.g., four) could be used to make a hazardous waste determination, and they pointed out that the RCRA regulations do not require statistical sampling for waste classification. On the other hand, other members of the planning team argued against the authoritative design. They argued that there was insufficient knowledge of the waste to implement authoritative sampling and noted that a few samples taken in a non-probabilistic manner would limit their ability to quantify any possible decision errors.

- ***Select a resource-effective design.*** The planning team evaluated the sampling and analytical design options and costs. The following table summarizes the estimated costs for the four sampling designs evaluated.

Table I-9. Estimated Costs for Implementing Candidate Sampling Designs

	Simple Random or Systematic Sampling (total metals only)	**Simple Random or Systematic Sampling (TCLP metals)**	**Authoritative (Biased) Sampling (total metals only)**	**Authoritative (Biased) Sampling (TCLP metals)**
Sample collection cost (per sample)	$50	$50	$50	$50
Analysis cost				
• SW-846 Methods 3050B/ 6010B (total Cd and Cr) (per sample)	$40		$40	
• SW-846 TCLP Method 1311. Extract analyzed by SW-846 Methods 3010A/6010B (per sample)		$220		$220
Number of samples	22	22	4	4
Total Estimated Cost	**$1,980**	**$5,940**	**$360**	**$1,080**

While the authoritative design with total metals analysis offered the least cost compared to the probabilistic designs, the team decided that they did not have sufficient knowledge of the waste, its leaching characteristics, or the process yet to use an authoritative sampling approach with total metals analysis only. Furthermore, the team needed to quantify the probability of making a decision error. The planning team selected the systematic design with total metals analysis for Cd and Cr with the condition that if any total sample analysis result indicated the maximum theoretical TCLP result could exceed the TC limit, then the TCLP would be performed for that sample. This approach was selected for its ease of implementation, it would provide adequate waste knowledge for future waste management decisions (assuming no change in the waste generation process), and would satisfy other cost and performance objectives specified by the planning team.

- ***Prepare a QAPP/SAP.*** The operational details of the sampling and analytical activities are documented in a Quality Assurance Project Plan and Sampling and Analysis Plan (QAPP/SAP).

Implementation Phase

The QAPP/SAP was implemented in accordance with the schedule and the facility's safety program. Based on the rate of waste generation, it was estimated that the roll-off box would be filled in about 30 work days assuming one "batch" of waste was placed in the roll off box each day. It was decided to obtain one random sample from each batch as the waste was discharge from the reclamation unit to the roll-off container (i.e., at the *point of waste generation*). See Figure I-5.

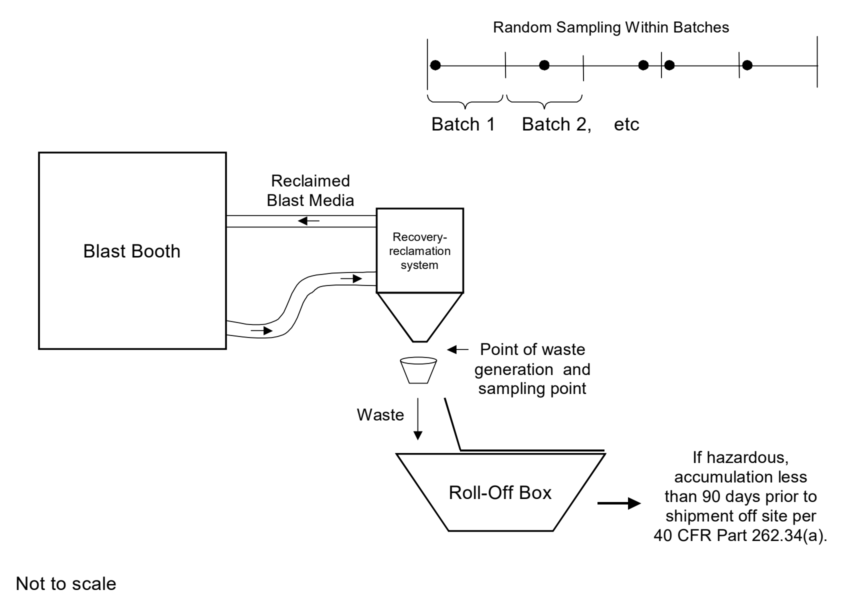


Figure I-5. Systematic sampling design with random sampling times selected within each batch

The QAPP/SAP established the following DQOs and performance goals for the equipment.

The sampling device must meet the following criteria:

- Be able to obtain a minimum mass of 300 grams for each sample
- Be constructed of materials that will not alter analyte concentrations due to loss or gain of analytes via sorption, desorption, degradation, or corrosion
- Be easy to use, safe, and low cost
- Be capable of obtaining increments of the waste at the discharge drop without introducing sampling bias.

The following four steps were taken to select the sampling device (from Section 7.1):

Step 1 - Identify the Medium To Be Sampled

Based on a prior inspection, it was known that the waste is a unconsolidated dry granular solid. Using Table 8 in Section 7.1, we find the media descriptor that most closely matches the waste in the first column of the table: “Other Solids - Unconsolidated.”

Step 2 - Select the Sample Location

The second column of Table 8 provides a list of common sampling locations for unconsolidated solids. The discharge drop opening is four inches wide, and the waste is released downward into the collection box. “Pipe or Conveyor” found in the table is the closest match to the configuration of the waste discharge point.

Step 3 - Identify Candidate Sampling Devices

The third column of Table 8 provides a list of candidate sampling devices for sampling solids from a pip or conveyor. For this waste stream, the list of devices for sampling a pipe or conveyor includes bucket, dipper, pan, sample container, miniature core sampler, scoop/trowel/shovel, and trier. The planning team immediately eliminated miniature core sampler, scoop/trowel/shovel, and trier because they are not suitable for obtaining samples from a falling stream or vertical discharge.

Step 4 - Select Devices

From the list of candidate sampling devices, one device was selected for use in the field from Table 9 in Section 7.1. Selection of the equipment was made after consideration of the DQOs for the sample support (i.e., required volume, width, shape, and orientation), the performance goals established for the sampling device, ease of use and decontamination, worker safety issues, cost, and any practical considerations. Table I-10 demonstrates how the DQOs and performance goals were used to narrow the candidate devices down to just one or two.

Table I-10. Using DQOs and Performance Goals To Select a Final Sampling Device

Candidate Devices	***Data Quality Objectives and Performance Goals***				
	Required Width	***Orientation and Shape***	***Sample Volume***	***Operational Considerations***	***Desired Material of Construction***
	4 inches	Cross-section of entire stream	>300 g	Device is portable, safe, and low cost?	Polyethylene or PTFE
Bucket	Y	Y	Y	Y	Y
Dipper	N	Y	Y	Y	Y
Pan	Y	Y	Y	Y	Y
Sample container	N	N	Y	Y	Y

Key: Y = The device is capable of achieving the specified DQO or performance goal.
N = The device is not capable of achieving the specified DQO or performance goal.

The sampling mode was “one-dimensional,” that is, the material is relatively linear in time and space. The ideal sampling device would obtain a sample of constant thickness and must be capable of obtaining the entire width of the stream for a fraction of the time (see discussion at Section 6.3.2.1). Either a bucket or pan wide enough (preferably 3 times the width of the stream) to obtain all of the flow for a fraction of the time are identified as suitable devices because they are capable of achieving all the performance goals.

A flat 12-inch wide polyethylene pan with vertical sides was used to collect each primary field sample. Each primary field sample was approximately 2 kilograms, therefore, the field team used the “fractional shoveling” technique (see Section 7.3.2) to reduce the sample mass to a subsample of approximately 300 grams. The field samples (each in a 32-oz jar) and associated field QC samples were submitted to the laboratory in accordance with the sample handling and shipping instructions specified in the QAPP/SAP.

A total of 30 samples were obtained by the time the roll-off box was filled, so it was necessary to randomly select 22 samples from the set of 30 for laboratory analysis.

All 22 samples were first analyzed for total cadmium and chromium to determine if the maximum theoretical TCLP concentration in any one sample could exceed the applicable TC limit. Samples whose maximum theoretical TCLP value exceeded the applicable TC limit were then analyzed using the full TCLP.

For the TCLP samples, no particle-size reduction was required for the sample extraction because the maximum particle size in the waste passed through a 9.5 mm sieve (the maximum particle size allowed for the TCLP). (On a small subsample of the waste, however, particle size reduction to 1 mm was required to determine the TCLP extract type (I or II)). A 100-gram subsample was taken from each field sample for TCLP analysis.

Assessment Phase

Data Verification and Validation

Sampling and analytical records were reviewed to check compliance with the QAPP/SAP. The data collected during the study met the DQOs. Sampling and analytical error were minimized through the use of a statistical sampling design, correct field sampling and subsampling procedures, and adherence to the requirements of the analytical methods. The material that was sampled did not present any special problems concerning access to sampling locations, equipment usage, particle-size distribution, or matrix interferences. Quantitation limits achieved for total cadmium and chromium were 5 mg/kg and 10 mg/kg respectively. Quantitation limits achieved for cadmium and chromium in the TCLP extract were 0.10 mg/L and 1.0 mg/L respectively. The analytical package was validated and the data generated were judged acceptable for their intended purpose.

Data Quality Assessment

DQA was performed using the approach outlined in Section 9.8.2 and EPA QA/G-9 (USEPA 2000d):

1. ***Review DQOs and sampling design.*** The DQO planning team reviewed the original objectives: “If the upper 90th percentile TCLP concentration for cadmium or chromium in the waste and all samples analysis results are less than their respective action levels of 1.0 and 5.0 mg/L TCLP, then the waste can be classified as nonhazardous waste under RCRA; otherwise, the waste will be considered a hazardous waste.”

2. ***Prepare the data for statistical analysis.*** The summary of the verified and validated data were received in hard copy format, and summarized in a table. The table was checked by a second person for accuracy. The results for the data collection effort are listed in Table I-11.

Table I-11. Total and TCLP Sample Analysis Results

Sample No.	***Cadmium***		***Chromium***	
	Total (mg/kg)	***Total / 20 (TC limit = 1 mg/L)***	***Total (mg/kg)***	***Total / 20 (TC limit = 5 mg/L)***
1	<5	<0.25	11	0.55
2	6	0.3	<10	<0.5
3	29	1.45 (full TCLP = 0.72)	<10	<0.5
4	<5	<0.25	<10	<0.5
5	<5	<0.25	42	2.1
6	7	0.35	<10	<0.5
7	7	0.35	<10	<0.5
8	13	0.65	26	1.3
9	<5	<0.25	19	0.95
10	<5	<0.25	<10	<0.5
11	36	1.8 (full TCLP = 0.8)	<10	<0.5
12	<5	<0.25	<10	<0.5
13	<5	<0.25	<10	<0.5
14	<5	<0.25	12	0.6
15	<5	<0.25	<10	<0.5
16	9	0.45	<10	<0.5
17	<5	<0.25	<10	<0.5
18	<5	<0.25	<10	<0.5
19	<5	<0.25	31	1.55
20	20	1 (full TCLP = <0.10)	<10	<0.5
21	<5	<0.25	<10	<0.5
22	<5	<0.25	<10	<0.5

3. ***Conduct preliminary analysis of data and check distributional assumptions.*** To use the nonparametric “exceedance rule” no distributional assumptions are required. The only requirements are a random sample, and that the quantitation limit is less than the applicable standard. These requirements were met.

4. ***Select and perform the statistical test***: The maximum TCLP sample analysis results for cadmium and chromium were compared to their respective TC regulatory limits. While several of the total results indicated the maximum theoretical TCLP result could exceed the regulatory limit, subsequent analysis of the TCLP extracts from these samples indicated the TCLP concentrations were below the regulatory limits.

5. ***Draw conclusions and report results.*** All 22 sample analysis results were less than the applicable TC limits, therefore the owner concluded with at least 90-percent confidence that at least 90-percent of all possible samples of the waste would be below the TC regulatory levels. Based on the decision rule established for the study, the owner decided to manage the waste as a nonhazardous waste.[1]

   A summary report including a description of all planning, implementation, and assessment activities was placed in the operating record.

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[1] Note that if fewer than 22 samples were analyzed - for example, due to a lost sample - and all sample analysis results indicated concentrations less than the applicable standard, then one still could conclude that 90-percent of all possible samples are less than the standard but with a lower level of confidence. See Section 5.5.2, Equation 17.

APPENDIX J

SUMMARIES OF ASTM STANDARDS

ASTM (the American Society for Testing and Materials) is one of the entities that can provide additional useful information on sampling. This appendix references many of the standards published by ASTM that are related to sampling.

ASTM is a not-for-profit organization that provides a forum for writing standards for materials, products, systems, and services. The Society develops and publishes standard test methods, specifications, practices, guides, classifications, and terminology.

Each ASTM standard is developed within the consensus principles of the Society and meets the approved requirements of its procedures. The voluntary, full-consensus approach brings together people with diverse backgrounds and knowledge. The standards undergo intense round-robin testing. Strict balloting and due process procedures guarantee accurate, up-to-date information.

Contact ASTM

For more information on ASTM or how to purchase their publications, including the standards referenced by this appendix, contact them at: ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428-2959; telephone: 610-832-9585; World Wide Web: http://www.astm.org.

To help you determine which ASTM standards may be most useful, this appendix includes text found in the scope of each standard. The standards, listed in alpha-numerical order, each deal in some way with sample collection. ASTM has future plans to publish these standards together in one volume on sampling.

D 140 Standard Practice for Sampling Bituminous Materials

This practice applies to the sampling of bituminous materials at points of manufacture, storage, or delivery.

D 346 Standard Practice for Collection and Preparation of Coke Samples for Laboratory Analysis

This practice covers procedures for the collection and reduction of samples of coke to be used for physical tests, chemical analyses, and the determination of total moisture.

D 420 Guide to Site Characterization for Engineering, Design, and Construction Purposes

This guide refers to ASTM methods by which soil, rock, and ground-water conditions may be determined. The objective of the investigation should be to identify and locate, both horizontally and vertically, significant soil and rock types and ground-water conditions present within a given site area and to establish the characteristics of the subsurface materials by sampling or *in situ* testing, or both.

D 1452 Standard Practice for Soil Investigation and Sampling by Auger Borings

This practice covers equipment and procedures for the use of earth augers in shallow geotechnical exploration. It does not apply to sectional continuous flight augers. This practice applies to any purpose for which disturbed samples can be used. Augers are valuable in connection with ground water level determinations, to help indicate changes in strata, and in the advancement of a hole for spoon and tube sampling.

D 1586 Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils

This test method describes the procedure, generally known as the Standard Penetration Test, for driving a split-barrel sampler. The procedure is used to obtain a representative soil sample and to measure the resistance of the soil to penetration of the sampler.

D 1587 Standard Practice for Thin-Walled Tube Geotechnical Sampling of Soils

This practice covers a procedure for using a thin-walled metal tube to recover relatively undisturbed soil samples suitable for laboratory tests of structural properties. Thin-walled tubes used in piston, plug, or rotary-type samplers, such as the Denison or Pitcher sampler, should comply with the portions of this practice that describe the thin-walled tubes. This practice is used when it is necessary to obtain a relatively undisturbed sample. It does not apply to liners used within the above samplers.

D 2113 Standard Practice for Diamond Core Drilling for Site Investigation

This practice describes equipment and procedures for diamond core drilling to secure core samples of rock and some soils that are too hard to sample by soil-sampling methods. This method is described in the context of obtaining data for foundation design and geotechnical engineering purposes rather than for mineral and mining exploration.

D 2234 Standard Practice for Collection of a Gross Sample of Coal

This practice covers procedures for the collection of a gross sample of coal under various conditions of sampling. The practice describes general and special purpose sampling procedures for coals by size and condition of preparation (e.g., mechanically cleaned coal or raw coal) and by sampling characteristics. The sample is to be crushed and further prepared for analysis in accordance with ASTM Method D 2013. This practice also gives procedures for dividing large samples before any crushing.

D 3213 Standard Practices for Handling, Storing, and Preparing Soft Undisturbed Marine Soil

These practices cover methods for project/cruise reporting; and for the handling, transporting and storing of soft cohesive undisturbed marine soil. The practices also cover procedures for preparing soil specimens for triaxial strength, and procedures for consolidation testing. These practices may include the handling and transporting of sediment specimens contaminated with hazardous materials and samples subject to quarantine regulations.

D 3326 Standard Practice for Preparation of Samples for Identification of Waterborne Oils

This practice covers the preparation for analysis of waterborne oils recovered from water. The identification is based on the comparison of physical and chemical characteristics of the waterborne oils with oils from suspect sources. These oils may be of petroleum or vegetable/animal origin, or both. The practice covers the following seven procedures (A through G): Procedure A, for samples of more than 50-mL volume containing significant quantities of hydrocarbons with boiling points above 280°C; Procedure B, for samples containing significant quantities of hydrocarbons with boiling points above 280°C; Procedure C, for waterborne oils containing significant amounts of components boiling below 280°C and to mixtures of these and higher boiling components; Procedure D, for samples containing both petroleum and vegetable/animal derived oils; Procedure E, for samples of light crudes and medium distillate fuels; Procedure F, for thin films of oil-on-water; and Procedure G, for oil-soaked samples.

D 3370 Standard Practices for Sampling Water from Closed Conduits

These practices cover the equipment and methods for sampling water from closed conduits (e.g., process streams) for chemical, physical, and microbiological analyses. It provides practices for grab sampling, composite sampling, and continual sampling of closed conduits.

D 3550 Standard Practice for Ring-Lined Barrel Sampling of Soils

This practice covers a procedure for using a ring-lined barrel sampler to obtain representative samples of soil for identification purposes and other laboratory tests. In cases in which it has been established that the quality of the sample is adequate, this practice provides shear and consolidation specimens that can be used directly in the test apparatus without prior trimming. Some types of soils may gain or lose significant shear strength or compressibility, or both, as a result of sampling. In cases like these, suitable comparison tests should be made to evaluate the effect of sample disturbance on shear strength and compressibility. This practice is not intended to be used as a penetration test; however, the force required to achieve penetration or a blow count, when driving is necessary, is recommended as supplemental information.

D 3665 Standard Practice for Random Sampling of Construction Materials

This practice covers the determination of random locations (or timing) at which samples of construction materials can be taken. For the exact physical procedures for securing the sample, such as a description of the sampling tool, the number of increments needed for a sample, or the size of the sample, reference should be made to the appropriate standard method.

D 3975 Standard Practice for Development and Use (Preparation) of Samples for Collaborative Testing of Methods for Analysis of Sediments

This practice establishes uniform general procedures for the development, preparation, and use of samples in the collaborative testing of methods for chemical analysis of sediments and similar materials. The principles of this practice are applicable to aqueous samples with suitable technical modifications.

D 3976 Standard Practice for Preparation of Sediment Samples for Chemical Analysis

This practice describes standard procedures for preparing test samples (including the removal of occluded water and moisture) of field samples collected from locations such as streams, rivers, ponds, lakes, and oceans. These procedures are applicable to the determination of volatile, semivolatile, and nonvolatile constituents of sediments.

D 3694 Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents

These practices cover the various means of (1) preparing sample containers used for collection of waters to be analyzed for organic constituents and (2) preservation of such samples from the time of sample collection until the time of analysis. The sample preservation practice depends on the specific analysis to be conducted. Preservation practices are listed with the corresponding applicable general and specific constituent test method. The preservation method for waterborne oils is given in Practice D 3325. Use of the information given will make it possible to choose the minimum number of sample preservation practices necessary to ensure the integrity of a sample designated for multiple analysis.

D 4136 Standard Practice for Sampling Phytoplankton with Water-Sampling Bottles

This practice covers the procedures for obtaining quantitative samples of a phytoplankton community by the use of water-sampling bottles.

D 4220 Standard Practices for Preserving and Transporting Soil Samples

These practices cover procedures for preserving soil samples immediately after they are obtained in the field and accompanying procedures for transporting and handling the samples. These practices are not intended to address requirements applicable to transporting of soil samples known or suspected to contain hazardous materials.

D 4342 Standard Practice for Collecting of Benthic Macroinvertebrates with Ponar Grab Sampler

This practice covers the procedures for obtaining qualitative or quantitative samples of macroinvertebrates inhabiting a wide range of bottom substrate types (e.g., coarse sand, fine gravel, clay, mud, marl, and similar substrates. The Ponar grab sampler is used in freshwater lakes, rivers, estuaries, reservoirs, oceans, and similar habitats.

D 4343 Standard Practice for Collecting Benthic Macroinvertebrates with Ekman Grab Sampler

This practice covers the procedures for obtaining qualitative or quantitative samples of macroinvertebrates inhabiting soft sediments. The Ekman grab sampler is used in freshwater lakes, reservoirs, and, usually, small bodies of water.

D 4387 Standard Guide for Selecting Grab Sampling Devices for Collecting Benthic Macroinvertebrates

This guide covers the selection of grab sampling devices for collecting benthic macroinvertebrates. Qualitative and quantitative samples of macroinvertebrates in sediments or substrates are usually taken by grab samplers. The guide discusses the advantages and limitations of the Ponar, Peterson, Ekman and other grab samplers.

D 4411 Standard Guide for Sampling Fluvial Sediment in Motion

This guide covers the equipment and basic procedures for sampling to determine discharge of sediment transported by moving liquids. Equipment and procedures were originally developed to sample mineral sediments transported by rivers but they also are applicable to sampling a variety of sediments transported in open channels or closed conduits. Procedures do not apply to sediments transported by flotation. This guide does not pertain directly to sampling to determine nondischarge-weighted concentrations, which in special instances are of interest. However, much of the descriptive information on sampler requirements and sediment transport phenomena is applicable in sampling for these concentrations and the guide briefly specifies suitable equipment.

D 4448 Standard Guide for Sampling Groundwater Monitoring Wells

This guide covers procedures for obtaining valid representative samples from ground-water monitoring wells. The scope is limited to sampling and "in the field" preservation and does not include well location, depth, well development, design and construction, screening, or analytical procedures. This guide provides a review of many of the most commonly used methods for sampling ground-water quality monitoring wells and is not intended to serve as a ground-water monitoring plan for any specific application. Because of the large and ever-increasing number of options available, no single guide can be viewed as comprehensive. The practitioner must make every effort to ensure that the methods used, whether or not they are addressed in this guide, are adequate to satisfy the monitoring objectives at each site.

D 4489 Standard Practices for Sampling of Waterborne Oils

These practices describe the procedures to be used in collecting samples of waterborne oils, oil found on adjoining shorelines, or oil-soaked debris, for comparison of oils by spectroscopic and chromatographic techniques, and for elemental analyses. Two practices are described. Practice A involves "grab sampling" macro oil samples. Practice B involves sampling most types of waterborne oils and is particularly applicable in sampling thin oil films or slicks. Practice selection will be dictated by the physical characteristics and the location of the spilled oil. Specifically, the two practices are (1) Practice A, for grab sampling thick layers of oil, viscous oils or oil soaked debris, oil globules, tar balls, or stranded oil, and (2) Practice B, for TFE-fluorocarbon polymer strip samplers. Each of the two practices collect oil samples with a minimum of water, thereby reducing the possibility of chemical, physical, or biological alteration by prolonged contact with water between the time of collection and analysis.

D 4547 Standard Guide for Sampling Waste and Soils for Volatile Organic Compounds

This guide describes recommended procedures for the collection, handling, and preparation of solid waste, soil, and sediment subsamples for subsequent determination of volatile organic compounds (VOCs). This class of compounds includes low molecular weight aromatics, hydrocarbons, halogenated hydrocarbons, ketones, acetates, nitriles, acrylates, ethers, and sulfides with boiling points below 200°C that are insoluble or slightly soluble in water. Methods of subsample collection, handling, and preparation for analysis are described. This guide does not cover the details of sampling design, laboratory preparation of containers, and the analysis of the subsamples.

D 4687 Standard Guide for General Planning of Waste Sampling

This guide provides information for formulating and planning the many aspects of waste sampling that are common to most waste-sampling situations. This guide addresses the following aspects of sampling: Sampling plans, safety plans, quality assurance considerations, general sampling considerations, preservation and containerization, cleaning equipment, labeling and shipping procedures, and chain-of-custody procedures. This guide does not provide comprehensive sampling procedures for these aspects, nor does it serve as a guide to any specific application.

D 4696 Standard Guide for Pore-Liquid Sampling from the Vadose Zone

This guide discusses equipment and procedures used for sampling pore-liquid from the vadose zone (unsaturated zone). The guide is limited to *in-situ* techniques and does not include soil core collection and extraction methods for obtaining samples. The term "pore-liquid" is applicable to any liquid from aqueous pore-liquid to oil, however, all of the samplers described in this guide are designed to sample aqueous pore-liquids only. The abilities of these samplers to collect other pore-liquids may be quite different than those described. Some of the samplers described in the guide currently are not commercially available. These samplers are presented because they may have been available in the past, and may be encountered at sites with established vadose zone monitoring programs. In addition, some of these designs are particularly suited to specific situations. If needed, these samplers could be fabricated.

D 4700 Standard Guide for Soil Sampling from the Vadose Zone

This guide addresses procedures that may be used for obtaining soil samples from the vadose zone (unsaturated zone). Samples can be collected for a variety of reasons, including the following:

- Stratigraphic description
- Hydraulic conductivity testing
- Moisture content measurement
- Moisture release curve construction
- Geotechnical testing
- Soil gas analyses
- Microorganism extraction
- Pore-liquid and soil chemical analyses.

This guide focuses on methods that provide soil samples for chemical analyses of the soil or contained liquids or contaminants. Comments on how methods may be modified for other objectives, however, also are included. This guide does not describe sampling methods for lithified deposits and rocks (e.g., sandstone, shale, tuff, granite).

D 4823 Standard Guide for Core Sampling Submerged, Unconsolidated Sediments

This guide covers core-sampling terminology, advantages and disadvantages of various core samplers, core distortions that may occur during sampling, techniques for detecting and minimizing core distortions, and methods for dissecting and preserving sediment cores. In this guide, sampling procedures and equipment are divided into the following categories (based on water depth): sampling in depths shallower than 0.5 m, sampling in depths between 0.5 m and 10 m, and sampling in depths exceeding 10 m. Each category is divided into two sections: (1) equipment for collecting short cores and (2) equipment for collecting long cores. This guide also emphasizes general principles. Only in a few instances are step-by-step instructions given. Because core sampling is a field-based operation, methods and equipment usually must be modified to suit local conditions. Drawings of samplers are included to show sizes and proportions. These samplers are offered primarily as examples (or generic representations) of equipment that can be purchased commercially or built from plans in technical journals. This guide is a brief summary of published scientific articles and engineering reports, and the references are listed. These documents provide operational details that are not given in the guide but are nevertheless essential to the successful planning and completion of core sampling projects.

D 4840 Standard Guide for Sampling Chain-of-Custody Procedures

This guide contains a comprehensive discussion of potential requirements for a sample chain-of-custody program and describes the procedures involved in sample chain-of-custody. The purpose of these procedures is to provide accountability for and documentation of sample integrity from the time of sample collection until sample disposal. These procedures are intended to document sample possession during each stage of a sample's life cycle, that is, during collection, shipment, storage, and the process of analysis. Sample chain of custody is just one aspect of the larger issue of data defensibility. A sufficient chain-of-custody process (i.e., one that provides sufficient evidence of sample integrity in a legal or regulatory setting) is situationally dependent. The procedures presented in this guide are generally considered sufficient to assure legal defensibility of sample integrity. In a given situation, less stringent measures may be adequate. It is the responsibility of the users of this guide to determine their exact needs. Legal counsel may be needed to make this determination.

D 4854 Standard Guide for Estimating the Magnitude of Variability from Expected Sources in Sampling Plans

The guide explains how to estimate the contributions of the variability of lot sampling units, laboratory sampling units, and specimens to the variation of the test result of a sampling plan. The guide explains how to combine the estimates of the variability from the three sources to obtain an estimate of the variability of the sampling plan results. The guide is applicable to all sampling plans that produce variables data. It is not applicable to plans that produce attribute data, since such plans do not take specimens in stages, but require that specimens be taken at random from all of the individual items in the lot.

D 4916 Standard Practice for Mechanical Auger Sampling

This practice describes procedures for the collection of an increment, partial sample, or gross sample of material using mechanical augers. Reduction and division of the material by mechanical equipment at the auger also is covered.

D 5013 Standard Practices for Sampling Wastes from Pipes and Other Point Discharges

These practices provide guidance for obtaining samples of waste at discharge points from pipes, sluiceways, conduits, and conveyor belts. The following are included: Practice A – Liquid or Slurry Discharges, and Practice B – Solid or Semisolid Discharges. These practices are intended for situations in which there are no other applicable ASTM sampling methods for the specific industry. These practices do not address flow and time-proportional samplers and other automatic sampling devices. Samples are taken from a flowing waste stream or moving waste mass and, therefore, are descriptive only within a certain period. The length of the period for which a sample is descriptive will depend on the sampling frequency and compositing scheme.

D 5088 Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste Sites

This practice covers the decontamination of field equipment used in the sampling of soils, soil gas, sludges, surface water, and ground water at waste sites that are to undergo both physical and chemical analyses. This practice is applicable only at sites at which chemical (organic and inorganic) wastes are a concern and is not intended for use at radioactive or mixed (chemical and radioactive) waste sites. Procedures are included for the decontamination of equipment that comes into contact with the sample matrix (sample contacting equipment) and for ancillary equipment that has not contacted the portion of sample to be analyzed (nonsample contacting equipment). This practice is based on recognized methods by which equipment may be decontaminated. When collecting environmental matrix samples, one should become familiar with the site-specific conditions. Based on these conditions and the purpose of the sampling effort, the most suitable method of decontamination can be selected to maximize the integrity of analytical and physical testing results. This practice is applicable to most conventional sampling equipment constructed of metallic and synthetic materials. The manufacturer of a specific sampling apparatus should be contacted if there is concern regarding the reactivity of a decontamination rinsing agent with the equipment.

D 5092 Standard Practice for Design and Installation of Ground Water Monitoring Wells in Aquifers

This practice addresses the selection and characterization (by defining soil, rock types, and hydraulic gradients) of the target monitoring zone as an integral component of monitoring well design and installation. The development of a conceptual hydrogeologic model for the intended monitoring zone(s) is recommended prior to the design and installation of a monitoring well. The guidelines are based on recognized methods by which monitoring wells may be designed and installed for the purpose of detecting the presence or absence of a contaminant, and collecting representative ground water quality data. The design standards and installation procedures in the practice are applicable to both detection and assessment monitoring programs for facilities. The recommended monitoring well design, as presented in this practice, is based on the assumption that the objective of the program is to obtain representative ground-water information and water quality samples from aquifers. Monitoring wells constructed following this practice should produce relatively turbidity-free samples for granular aquifer materials ranging from gravels to silty sand and sufficiently permeable consolidated and fractured strata. Strata having grain sizes smaller than the recommended design for the smallest diameter filter pack materials should be monitored by alternative monitoring well designs not addressed by this practice.

D 5283 Standard Practice for Generation of Environmental Data Related to Waste Management Activities Quality Assurance and Quality Control Planning and Implementation

This practice addresses the planning and implementation of the sampling and analysis aspects of environmental data generation activities. It defines the criteria that must be considered to assure the quality of the field and analytical aspects of environmental data generation activities. Environmental data include, but are not limited to, the results from analyses of samples of air, soil, water, biota, waste, or any combinations thereof. DQOs should be adopted prior to application of this practice. Data generated in accordance with this practice are subject to a final assessment to determine whether the DQOs were met. For example, many screening activities do not require all of the mandatory quality assurance and quality control steps found in this practice to generate data adequate to meet the project DQOs. The extent to which all of the requirements must be met remains a matter of technical judgment as it relates to the established DQOs. This practice presents extensive management requirements designed to ensure high-quality environmental data.

D 5314 Standard Guide for Soil Gas Monitoring in the Vadose Zone

This guide covers information pertaining to a broad spectrum of practices and applications of soil atmosphere sampling, including sample recovery and handling, sample analysis, data interpretation, and data reporting. This guide can increase the awareness of soil gas monitoring practitioners concerning important aspects of the behavior of the soil-water-gas contaminant system in which this monitoring is performed, as well as inform them of the variety of available techniques of each aspect of the practice. Appropriate applications of soil gas monitoring are identified, as are the purposes of the various applications. Emphasis is placed on soil gas contaminant determinations in certain application examples. This guide suggests a variety of approaches useful in monitoring vadose zone contaminants with instructions that offer direction to those who generate and use soil gas data. This guide does not recommend a standard practice to follow in all cases, nor does it recommend definite courses of action. The success of any one soil gas monitoring methodology is strongly dependent upon the environment in which it is applied.

D 5358 Standard Practice for Sampling with a Dipper or Pond Sampler

This practice describes the procedure and equipment for taking surface samples of water or other liquids using a dipper. A pond sampler or dipper with an extension handle allows the operator to sample streams, ponds, waste pits, and lagoons as far as 15 feet from the bank or other secure footing. The dipper is useful in filling a sample bottle without contaminating the outside of the bottle.

D 5387 Standard Guide for Elements of a Complete Data Set for Non-Cohesive Sediments

This guide covers criteria for a complete sediment data set, and it provides guidelines for the collection of non-cohesive sediment alluvial data. This guide describes what parameters should be measured and stored to obtain a complete sediment and hydraulic data set that could be used to compute sediment transport using any prominently known sediment-transport equations.

D 5451 Standard Practice for Sampling Using a Trier Sampler

This practice covers sampling using a trier. A trier resembles an elongated scoop, and is used to collect samples of granular or powdered materials that are moist or sticky and have a particle diameter less than one-half the diameter of the trier. The trier can be used as a vertical coring device only when it is certain that a relatively complete and cylindrical sample can be extracted.

D 5495 Standard Practice for Sampling with a Composite Liquid Waste Sampler (COLIWASA)

This practice describes the procedure for sampling liquids with the composite liquid waste sampler (COLIWASA). The COLIWASA is an appropriate device for obtaining a representative sample from stratified or unstratified liquids. Its most common use is for sampling containerized liquids, such as tanks, barrels, and drums. It may also be used for pools and other open bodies of stagnant liquid. (A limitation of the COLIWASA is that the stopper mechanism may not allow collection of approximately the bottom inch of material, depending on construction of the stopper.) The COLIWASA should not be used to sample flowing or moving liquids.

D 5608 Standard Practice for Decontamination of Field Equipment Used at Low Level Radioactive Waste Sites

This practice covers the decontamination of field equipment used in the sampling of soils, soil gas, sludges, surface water, and ground water at waste sites known or suspected of containing low-level radioactive wastes. This practice is applicable at sites where low-level radioactive wastes are known or suspected to exist. By itself or in conjunction with Practice D 5088, this practice may also be applicable for the decontamination of equipment used in the vicinity of known or suspected transuranic or mixed wastes. Procedures are contained in this practice for the decontamination of equipment that comes into contact with the sample matrix (sample contacting equipment), and for ancillary equipment that has not contacted the sample, but may have become contaminated during use (noncontacting equipment). This practice is applicable to most conventional sampling equipment constructed of metallic and hard and smooth synthetic materials. Materials with rough or porous surfaces, or having a high sorption rate, should not be used in radioactive-waste sampling due to the difficulties with decontamination. In those cases in which sampling will be periodically performed, such as sampling of wells, consideration should be given to the use of dedicated sampling equipment if legitimate concerns exist for the production of undesirable or unmanageable waste byproducts, or both, during the decontamination of tools and equipment. This practice does not address regulatory requirements for personnel protection or decontamination, or for the handling, labeling, shipping, or storing of wastes, or samples. Specific radiological release requirements and limits must be determined by users in accordance with local, State and Federal regulations.

D 5633 Standard Practice for Sampling with a Scoop

This procedure covers the method and equipment used to collect surface and near-surface samples of soils and physically similar materials using a scoop. This practice is applicable to rapid screening programs, pilot studies, and other semi-quantitative investigations. The practice describes how a shovel is used to remove the top layers of soil to the appropriate sample depth and either a disposable scoop or a reusable scoop is used to collect and place the sample in the sample container.

D 5658 Standard Practice for Sampling Unconsolidated Waste from Trucks

This practice covers several methods for collecting waste samples from trucks. These methods are adapted specifically for sampling unconsolidated solid wastes in bulk loads using several types of sampling equipment.

D 5679 Standard Practice for Sampling Consolidated Solids in Drums or Similar Containers

This practice covers typical equipment and methods for collecting samples of consolidated solids in drums or similar containers. These methods are adapted specifically for sampling drums having a volume of 110 U.S. gallons (416 L) or less, and are applicable to a hazardous material, product, or waste.

D 5680 Standard Practice for Sampling Unconsolidated Solids in Drums or Similar Containers

This practice covers typical equipment and methods for collecting samples of unconsolidated solids in drums or similar containers. These methods are adapted specifically for sampling drums having a volume of 110 U.S. gallons (416 L) or less, and are applicable to a hazardous material, product, or waste.

D 5730 Standard Guide for Site Characterization for Environmental Purposes with Emphasis on Soil, Rock, the Vadose Zone and Ground Water

This guide covers a general approach to planning field investigations that is useful for any type of environmental investigation with a primary focus on the subsurface and major factors affecting the surface and subsurface environment. Generally, such investigations should identify and locate, both horizontally and vertically, significant soil and rock masses and ground-water conditions present within a given site area and establish the characteristics of the subsurface materials by sampling or *in situ* testing, or both. The extent of characterization and specific methods used will be determined by the environmental objectives and data quality requirements of the investigation. This guide focuses on field methods for determining site characteristics and collection of samples for further physical and chemical characterization. It does not address special considerations required for characterization of karst and fractured rock terrain.

D 5743 Standard Practice for Sampling Single or Multilayered Liquids, with or without Solids, in Drums or Similar Containers

This practice covers typical equipment and methods for collecting samples of single or multilayered liquids, with or without solids, in drums or similar containers. These methods are adapted specifically for sampling drums having a volume of 110 gallons (416 L) or less, and are applicable to a hazardous material, product, or waste.

D 5792 Standard Practice for Generation of Environmental Data Related to Waste Management Activities: Development of Data Quality Objectives

This practice covers the development of data quality objectives (DQOs) for the acquisition of environmental data. Optimization of sampling and analysis design is a part of the DQO Process. This practice describes the DQO Process in detail. The various strategies for design optimization are too numerous to include in this practice. Many other documents outline alternatives for optimizing sampling and analysis design, therefore, only an overview of design optimization is included. Some design aspects are included in the examples for illustration purposes.

D 5903 Standard Guide for Planning and Preparing for a Groundwater Sampling Event

This guide covers planning and preparing for a ground-water sampling event. It includes technical and administrative considerations and procedures. Example checklists are also provided as appendices. This guide may not cover every consideration and procedure that is necessary before all ground-water sampling projects. This guide focuses on sampling of ground water from monitoring wells; however, most of the guidance herein can apply to the sampling of springs as well.

D 5911 Standard Practice for Minimum Set of Data Elements to Identify a Soil Sampling Site

This practice covers what information should be obtained to uniquely identify any soil sampling or examination site where an absolute and recoverable location is necessary for quality control of the study, such as for a waste disposal project. The minimum set of data elements was developed considering the needs for informational data bases, such as geographic information systems. Other distinguishing details, such as individual site characteristics, help in singularly cataloging the site. For studies that are not environmentally regulated, such as for an agricultural or preconstruction survey, the data specifications established by a client and the project manager may be different from that of the minimum set. As used in this practice, a soil sampling site is meant to be a single point, not a geographic area or property, located by an X, Y, and Z coordinate position at land surface or a fixed datum. All soil data collected for the site are directly related to the coordinate position, e.g., a sample is collected from a certain number of feet (or meters) or sampled from a certain interval to feet (or meters) below the X, Y, and Z coordinate position. A soil sampling site can include a test well, augered or bored hole, excavation, grab sample, test pit, sidewall sample, stream bed, or any other site where samples of the soil can be collected or examined for the purpose intended. Samples of soil (sediment) filtered from the water of streams, rivers, or lakes are not in the scope of this practice.

D 5956 Standard Guide for Sampling Strategies for Heterogeneous Wastes

This guide is a practical nonmathematical discussion for heterogeneous waste sampling strategies. This guide is consistent with the particulate material sampling theory, as well as inferential statistics, and may serve as an introduction to the statistical treatment of sampling issues. This guide does not provide comprehensive sampling procedures, nor does it serve as a guide to any specification.

D 6001 Standard Guide for Direct-Push Water Sampling for Geoenvironmental Investigations

This guide reviews methods for sampling ground water at discrete points or in increments by insertion of sampling devices by static force or impact without drilling and removal of cuttings. By directly pushing the sampler, the soil is displaced and helps to form an annular seal above the sampling zone. Direct-push water sampling can be one-time or multiple-sampling events. Methods for obtaining water samples for water quality analysis and detection of contaminants are presented. Field test methods described in this guide include installation of temporary well points and insertion of water samplers using a variety of insertion methods. The insertion methods include (1) soil probing using combinations of impact, percussion, or vibratory driving with or without additions of smooth static force; (2) smooth static force from the surface using hydraulic penetrometer or drilling equipment and incremental drilling combined with direct-push water sampling events. Methods for borehole abandonment by grouting are also addressed.

D 6008 Standard Practice for Conducting Environmental Baseline Surveys

The purpose of this practice is to define good commercial and customary practice in the United States for conducting an environmental baseline survey (EBS). Such surveys are conducted to determine certain elements of the environmental condition of Federal real property, including excess and surplus property at closing and realigning military installations. This effort is conducted to fulfill certain requirements of the Comprehensive Environmental Response Compensation and Liability Act of 1980 (CERCLA) section 120(h), as amended by the Community Environmental Response Facilitation Act of 1992 (CERFA). As such, this practice is intended to help a user to gather and analyze data and information in order to classify property into seven environmental condition of property area types (in accordance with the Standard Classification of Environmental Condition of Property Area Types). Once documented, the EBS is used to support Findings of Suitability to Lease, or uncontaminated property determinations, or a combination thereof, pursuant to the requirements of CERFA. Users of this practice should note that it does not address (except where explicitly noted) requirements of CERFA. The practice also does not address (except where explicitly noted) requirements for appropriate and timely regulatory consultation or concurrence, or both, during the conduct of the EBS or during the identification and use of the standard environmental condition of property area types.

D 6009 Standard Guide for Sampling Waste Piles

This guide provides guidance for obtaining representative samples from waste piles. Guidance is provided for site evaluation, sampling design, selection of equipment, and data interpretation. Waste piles include areas used primarily for waste storage or disposal, including above-grade dry land disposal units. This guide can be applied to sampling municipal waste piles, and it addresses how the choice of sampling design and sampling methods depends on specific features of the pile.

D 6044 Standard Guide for Representative Sampling for Management of Waste and Contaminated Media

This guide covers the definition of representativeness in environmental sampling, identifies sources that can affect representativeness (especially bias), and describes the attributes that a representative sample or a representative set of samples should possess. For convenience, the term “representative sample” is used in this guide to denote both a representative sample and a representative set of samples, unless otherwise qualified in the text. This guide outlines a process by which a representative sample may be obtained from a population, and it describes the attributes of a representative sample and presents a general methodology for obtaining representative samples. It does not, however, provide specific or comprehensive sampling procedures. It is the user’s responsibility to ensure that proper and adequate procedures are used.

D 6051 Standard Guide for Composite Sampling and Field Subsampling for Environmental Waste Management Activities

This guide discusses the advantages and appropriate use of composite sampling, field procedures and techniques to mix the composite sample and procedures to collect an unbiased and precise subsample from a larger sample. Compositing and subsampling are key links in the chain of sampling and analytical events that must be performed in compliance with project objectives and instructions to ensure that the resulting data are representative. This guide discusses the advantages and limitations of using composite samples in designing sampling plans for characterization of wastes (mainly solid) and potentially contaminated media. This guide assumes that an appropriate sampling device is selected to collect an unbiased sample. It does not address where samples should be collected (depends on the objectives), selection of sampling equipment, bias introduced by selection of inappropriate sampling equipment, sample collection procedures or collection of a representative specimen from a sample, or statistical interpretation of resultant data and devices designed to dynamically sample process waste streams. It also does not provide sufficient information to statistically design an optimized sampling plan, or to determine the number of samples to collect or to calculate the optimum number of samples to composite to achieve specified data quality objectives. The mixing and subsampling described in this guide is expected to cause significant losses of volatile constituents. Specialized procedures should be used for compositing samples for determination of volatiles.

D 6063 Standard Guide for Sampling of Drums and Similar Containers by Field Personnel

This guide covers information, including flow charts, for field personnel to follow in order to collect samples from drums and similar containers. The purpose of this guide is to help field personnel in planning and obtaining samples from drums and similar containers, using equipment and techniques that will ensure that the objectives of the sampling activity will be met. It can also be used as a training tool.

D 6169 Standard Guide for Selection of Soil and Rock Sampling Devices Used With Drill Rigs for Environmental Investigations

This guide covers the selection of soil and rock sampling devices used with drill rigs for the purpose of characterizing *in situ* physical and hydraulic properties, chemical characteristics, subsurface lithology, stratigraphy, and structure, and hydrogeologic units in environmental investigations.

D 6232 Standard Guide for Selection of Sampling Equipment for Waste and Contaminated Media Data Collection Activities

This guide covers criteria that should be considered when selecting sampling equipment for collecting environmental and waste samples for waste management activities. This guide includes a list of equipment that is used and is readily available. Many specialized sampling devices are not specifically included in this guide, however, the factors that should be weighed when choosing any piece of equipment are covered and remain the same for the selection of any piece of equipment. Sampling equipment described in this guide include automatic samplers, pumps, bailers, tubes, scoops, spoons, shovels, dredges, and coring and augering devices. The selection of sampling locations is outside the scope of this guide.

D 6233 Standard Guide for Data Assessment for Environmental Waste Management Activities

This guide covers a practical strategy for examining an environmental project data collection effort and the resulting data to determine conformance with the project plan and impact on data usability. This guide also leads the user through a logical sequence to determine which statistical protocols should be applied to the data.

D 6250 Standard Practice for Derivation of Decision Point and Confidence Limit for Statistical Testing of Mean Concentration in Waste Management Decisions

This practice covers a logical basis for the derivation of a decision point and confidence limit when the mean concentration is used for making environmental waste management decisions. The determination of a decision point or confidence limit should be made in the context of the defined problem. The main focus of this practice is on the determination of a decision point. In environmental management decisions, the derivation of a decision point allows a direct comparison of a sample mean against this decision point. Similar decisions can be made by comparing a confidence limit against a concentration limit. This practice focuses on making environmental decisions using this kind of statistical comparison. Other factors, such as any qualitative information that also may be important to decision making, are not considered in the practice. This standard derives the decision point and confidence limit in the framework of a statistical test of hypothesis under three different presumptions. The relationship between decision point and confidence limit also is described.

D 6282 Standard Guide for Direct Push Soil Sampling for Environmental Site Characterizations

This guide addresses direct push soil samplers, which may be driven into the ground from the surface or through pre-bored holes. The samplers can be continuous or discrete interval units. The samplers are advanced to the depth of interest by a combination of static push, or impacts from hammers, or vibratory methods, or a combination thereof. Field methods described in this guide include the use of discreet and continuous sampling tools, split and solid barrel samplers and thin walled tubes with or without fixed piston style apparatus. Insertion methods described include static push, impact, percussion, other vibratory/sonic driving, and combinations of these methods using direct push equipment adapted to drilling rigs, cone penetrometer units, and specially designed percussion/direct push combination machines. Hammers described by this guide for providing force for insertion include drop style, hydraulically activated, air activated and mechanical lift devices. The guide does not cover open chambered samplers operated by hand such as augers, agricultural samplers operated at shallow depths, or side wall samplers.

D 6286 Standard Guide for Selection of Drilling Methods for Environmental Site Characterization

This guide provides descriptions of various drilling methods for environmental site characterization, along with the advantages and disadvantages associated with each method. This guide is intended to aid in the selection of drilling method(s) for environmental soil and rock borings and the installation of monitoring wells and other water-quality monitoring devices. This guide does not address methods of well construction, well development, or well completion.

D 6311 Standard Guide for Generation of Environmental Data Related to Waste Management Activities: Selection and Optimization of Sampling Design

This guide provides practical information on the selection and optimization of sample designs in waste management sampling activities, within the context of the requirements established by the data quality objectives or other planning process. Specifically, this document provides (1) guidance for the selection of sampling designs; (2) techniques to optimize candidate designs; and (3) descriptions of the variables that need to be balanced in choosing the final optimized design.

D 6323 Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities

This guide covers common techniques for obtaining representative subsamples from a sample received at a laboratory for analysis. These samples may include solids, sludges, liquids, or multilayered liquids (with or without solids). The procedures and techniques discussed in this guide depend upon the sample matrix, the type of sample preparation and analysis performed, the characteristic(s) of interest, and the project specific instructions or data quality objectives. This guide includes several sample homogenization techniques, including mixing and grinding, as well as information on how to obtain a specimen or split laboratory samples. This guide does not apply to air or gas sampling.

D 6418 Standard Practice for Using the Disposable EnCore™ Sampler for Sampling and Storing Soil for Volatile Organic Analysis

This practice provides a procedure for using the disposable EnCore™ sampler to collect and store a soil sample of approximately 5 grams or 25 grams for volatile organic analysis. The EnCore™ sampler is designed to collect and hold a soil sample during shipment to the laboratory. It consists of a coring body/storage chamber, O-ring sealed plunger, and O-ring sealed cap. In performing the practice, the integrity of the soil sample structure is maintained and there is very limited exposure of the sample to the atmosphere. Laboratory subsampling is not required; the sample is expelled directly from the sampler body into the appropriate container for analysis.

D 6538 Standard Guide for Sampling Wastewater With Automatic Samplers

This guide covers the selection and use of automatic wastewater samplers including procedures for their use in obtaining representative samples. Automatic wastewater samplers are intended for the unattended collection of samples that are representative of the parameters of interest in the wastewater body. While this guide primarily addresses the sampling of wastewater, the same automatic samplers may be used to sample process streams and natural water bodies.

D 6582 Standard Guide for Ranked Set Sampling: Efficient Estimation of a Mean Concentration in Environmental Sampling

This guide describes ranked set sampling, discusses its relative advantages over simple random sampling, and provides examples of potential applications in environmental sampling. Ranked set sampling is useful and cost-effective when there is an auxiliary variable, which can be inexpensively measured relative to the primary variable, and when the auxiliary variable has correlation with the primary variable. The resultant estimation of the mean concentration is unbiased, more precise than simple random sampling, and more representative of the population under a wide variety of conditions.

D 6771 Standard Practice for Low-Flow Purging and Sampling for Wells and Devices Used for Ground-Water Quality Investigations

This practice covers the method for purging and sampling wells and devices used for ground-water quality investigations and monitoring programs known as low-flow purging and sampling. The method is also known by the terms minimal drawdown purging or low-stress purging. The method could be used for other types of ground-water sampling programs but these uses are not specifically addressed in this practice. This practice applies only to wells sampled at the wellhead. This practice does not address sampling of wells containing either light or dense non-aqueous-phase liquids (LNAPLs or DNAPLs).

E 122 Standard Practice for Choice of Sample Size to Estimate the Average for a Characteristic of a Lot or Process

This practice covers methods for calculating the sample size (the number of units to include in a random sample from a lot of material) in order to estimate, with a prescribed precision, an average of some characteristic for that lot or process. The characteristic may be either a numerical value of some property or the fraction of nonconforming units with respect to an attribute. If sampling from a process, the process must be in a state of statistical control for the results to have predictive value.

E 178 Standard Practice for Dealing with Outlying Observations

This practice covers outlying observations in samples and how to test the statistical significance of them. An outlying observation, or "outlier," is an observation that appears to deviate markedly from other members of the sample in which it occurs. An outlying observation may be merely an extreme manifestation of the random variability inherent in the data. If this is true, the value should be retained and processed in the same manner as the other observations in the sample. On the other hand, an outlying observation may be the result of gross deviation from prescribed experimental procedure or an error in calculating or recording the numerical value. In such cases, it may be desirable to institute an investigation to ascertain the reason for the aberrant value. The observation may even actually be rejected as a result of the investigation, though not necessarily so. At any rate, in subsequent data analysis the outlier or outliers probably will be recognized as being from a different population than that of the other sample values. The procedures covered herein apply primarily to the simplest kind of experimental data; that is, replicate measurements of some property of a given material, or observations in a supposedly single random sample. Nevertheless, the tests suggested do cover a wide enough range of cases in practice to have broad utility.

E 300 Standard Practice for Sampling Industrial Chemicals

This practice covers procedures for sampling several classes of industrial chemicals, as well as recommendations for determining the number and location of such samples to ensure representativeness in accordance with accepted probability sampling principles. Although this practice describes specific procedures for sampling various liquids, solids, and slurries, in bulk or in packages, these recommendations only outline the principles to be observed. They should not take precedence over specific sampling instructions contained in other ASTM product or method standards.

E 1402 Standard Terminology Relating to Sampling

This standard includes those items related to statistical aspects of sampling. It is applicable to sampling in any matrix and provides definitions, descriptions, discussions, and comparisons of trends.

E 1727 Standard Practice for Field Collection of Soil Samples for Lead Determination by Atomic Spectrometry Techniques

This practice covers the collection of soil samples using coring and scooping methods. Soil samples are collected in a manner that will permit subsequent digestion and determination of lead using laboratory analysis techniques such as Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), and Graphite Furnace Atomic Absorption Spectrometry (GFAAS).

F 301 Standard Practice for Open Bottle Tap Sampling of Liquid Streams

This practice covers a general method to take samples of liquid streams in such a way so that the samples are representative of the liquid in the sampled stream and that the sample acquisition process does not interfere with any operations taking place in the stream. The practice is particularly applicable for sampling the feed and filtrate streams around a filter medium. The practice includes consideration of potential limits in the sample size or sample flow rate observation capability of the device used to measure particle content in the sample.

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Test: **VOC 8260 QSM TCLP** Updated 07/11/2016 AJZ
Method: **EPA 8260C**
Matrix: **TCLP**

Analyte	CAS #	DL /MDL mg/L	DOD LOD mg/L	DOD LOQ mg/L	RL mg/L	LCS % Recovery	MS % Recovery	RPD Limit	Surrogate Limit
Benzene	71-43-2	0.00019	0.0005	0.001	0.001	79-120	79-120	20	
Carbon tetrachloride	56-23-5	0.00023	0.0005	0.001	0.001	72-136	72-136	20	
Chlorobenzene	108-90-7	0.00024	0.0005	0.001	0.001	82-118	82-118	20	
Chloroform	67-66-3	0.00015	0.00025	0.0005	0.0005	79-124	79-124	20	
1,2-Dichloroethane	107-06-2	0.0003	0.0005	0.001	0.001	73-128	73-128	20	
1,1-Dichloroethene	75-35-4	0.00024	0.0005	0.001	0.001	77-125	77-125	20	
2-Butanone	78-93-3	0.0024	0.005	0.001	0.001	56-143	56-143	20	
Tetrachloroethene	127-18-4	0.0003	0.0005	0.001	0.001	74-129	74-129	20	
Trichloroethene	79-01-6	0.00021	0.0005	0.001	0.001	79-123	79-123	20	
Vinyl chloride	75-01-4	0.00018	0.0005	0.001	0.001	58-137	58-137	20	
1,2 Dichloroethane-d4	17060-07-0								81-118
Bromofluorobenzene	460-00-4								85-114
d8-Toluene	2037-26-5								89-112
Dibromofluoromethane	1868-53-7								80-119

updated 07/08/2016 AJZ

Test: **SVOC 8270 QSM TCLP**
Method: **EPA 8270C**
Matrix: **TCLP**

Analyte	CAS #	DL/MDL mg/L	DOD LOD mg/L	DOD LOQ mg/L	RL mg/L	% Recovery LCS	% Recovery MS	RPD Limit	Surrogate Limit
1,4-Dichlorobenzene	106-46-7	0.00019	0.0004	0.001	0.001	30-100	30-100	30	
2-Methylphenol	95-48-7	0.00086	0.002	0.005	0.005	40-110	40-110	30	
2,4,5-Trichlorophenol	95-95-4	0.0011	0.002	0.005	0.005	50-110	50-110	30	
2,4,6-Trichlorophenol	88-06-2	0.001	0.002	0.005	0.005	50-115	50-115	30	
2,4-Dinitrotoluene	121-14-2	0.00021	0.0004	0.001	0.001	50-120	50-120	30	
3 & 4-Methylphenol	1319-77-3	0.0014	0.0036	0.009	0.09	30-110	30-110	30	
Hexachlorobenzene	118-74-1	0.00027	0.0004	0.001	0.001	50-110	50-110	30	
Hexachlorobutadiene	87-68-3	0.00018	0.0004	0.001	0.001	25-105	25-105	30	
Hexachloroethane	67-72-1	0.00022	0.0004	0.001	0.001	30-95	30-95	30	
Nitrobenzene	98-95-3	0.00016	0.0004	0.001	0.001	45-110	45-110	30	
Pentachlorophenol	87-86-5	0.0011	0.002	0.005	0.005	40-115	40-115	30	
Phenol	108-95-2	0.00048	0.002	0.005	0.005	1-115	1-115	30	
Pyridine	110-86-1	0.00062	0.001	0.003	0.003	50-130	50-130	30	
Surr: 2,4,6-Tribromophenol	118-79-6								40-125
Surr: 2-Fluorobiphenyl	321-60-8								50-110
Surr: 2-Fluorophenol	367-12-4								20-110
Surr: Nitrobenzene-d5	4165-60-0								40-110
Surr: Phenol-d5	4165-62-2								10-115
Surr: Terphenyl-d14	1718-51-0								50-135

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Test: **ICP QSM TCLP** updated 07/11/2016
Method: **EPA 6010C**
Matrix: **TCLP**

Analyte	CAS #	DL /MDL mg/L	DOD LOD mg/L	DOD LOQ mg/L	Reporting Limit	LCS % Recovery	MS % Recovery	RPD Limit
Arsenic	7440-38-2	0.004	0.012	0.024	0.024	80-120	80-120	20
Barium	7440-39-3	0.00029	0.0009	0.0018	0.0018	80-120	80-120	20
Cadmium	7440-43-9	0.0003	0.001	0.002	0.002	80-120	80-120	20
Chromium	7440-47-3	0.0006	0.002	0.004	0.004	80-120	80-120	20
Lead	7439-92-1	0.0014	0.002	0.004	0.004	80-120	80-120	20
Selenium	7782-49-2	0.0022	0.0065	0.013	0.013	80-120	80-120	20
Silver	7440-22-4	0.0007	0.002	0.004	0.004	80-120	80-120	20
Mercury	7439-97-6	0.00003	0.00006	0.00012	0.00012	80-120	80-120	20

Test: PCB
Method: EPA 8082A updated 07/08/2016 AJZ
Matrix: SOIL

Analyte	CAS #	DL/MDL ug/kg	DOD LOD ug/kg	DOD LOQ ug/kg	RL ug/kg	MS % Recovery	MS % Recovery	RPD	Surrogate % Recovery
Aroclor-1016	12674-11-2	5	20	30	30	47-134	47-134	30	
Aroclor-1221	11104-28-2	7	20	30	30				
Aroclor-1232	11141-16-5	9	20	30	30				
Aroclor-1242	53469-21-9	7	20	30	30				
Aroclor-1248	12672-29-6	7	20	30	30				
Aroclor-1254	11097-69-1	9	20	30	30	67-135	67-135	30	
Aroclor-1260	11096-82-5	6	20	30	30	53-140	53-140	30	
Aroclor-1262	37324-23-5	7	20	30	30				
Aroclor-1268	11100-14-4	5	20	30	30				
Surr: DCBP	2051-24-3								60-125

Test: PCB
Method: EPA 8082A
Matrix: GROUND WATER

Analyte	CAS #	DL/MDL ug/L	DOD LOD ug/L	DOD LOQ ug/L	RL ug/L	MS % Recovery	MS % Recovery	RPD	Surrogate % Recovery
Aroclor-1016	12674-11-2	0.113	0.3	1	1	46-129	46-129	30	
Aroclor-1221	11104-28-2	0.085	0.3	1	1				
Aroclor-1232	11141-16-5	0.145	0.3	1	1				
Aroclor-1242	53469-21-9	0.096	0.3	1	1				
Aroclor-1248	12672-29-6	0.088	0.3	1	1				
Aroclor-1254	11097-69-1	0.094	0.3	1	1	34-127	34-127	30	
Aroclor-1260	11096-82-5	0.099	0.3	1	1	45-134	45-134	30	
Aroclor-1262	37324-23-5	0.28	0.3	1	1				
Aroclor-1268	11100-14-4	0.056	0.3	1	1				
Surr: DCBP	2051-24-3								40-135

Table 12-1– Measurement Performance Criteria – Compound (Matrix)

Matrix	**Groundwater – Soil - Waste**				
Analytical Group	**Volatile Organic Compounds (VOCs)**				
Concentration Level	**Low**				
Sampling Procedure SOPP	**Analytical Method/SOP[1]**	**Data Quality Indicators**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
	SW8260C / VO004	Overall Precision	RPD ≤ 20% when detected in both samples ≥ sample-specific LOQ	Field Duplicates	S
		Analytical Precision (laboratory)	RPD ≤ 20%	Laboratory Sample Duplicates	A
		Analytical Accuracy/Bias (laboratory)	Analyte-specific (QSM)	Laboratory Control Samples	A
		Analytical Accuracy/Bias (matrix interference)	Analyte-specific (QSM)	Matrix Spike Duplicates	S&A
		Overall accuracy/bias (contamination)	No target analyte concentrations ≥ 1/2 LOQ	Equipment Blanks	S
		Sensitivity	Recovery within ±25% of LOQ	LOQ verification sample (spiked at LOQ)	A
		Completeness	See Worksheet #34	See Worksheet #34	

Table 12-2– Measurement Performance Criteria – Compound (Matrix)

Matrix	**Groundwater - Soil**				
Analytical Group	**Semi-Volatile Organic Compounds (SVOCs)**				
Concentration Level	**Low**				
Sampling Procedure SOPP	**Analytical Method/SOP^1**	**Data Quality Indicators**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
	SW8270D / 8270 SIM / SV006, SV007	Overall Precision	RPD ≤ 20% when detected in both samples ≥ sample-specific LOQ	Field Duplicates	S
		Analytical Precision (laboratory)	RPD ≤ 20%	Laboratory Sample Duplicates	A
		Analytical Accuracy/Bias (laboratory)	Analyte-specific (QSM)	Laboratory Control Samples	A
		Analytical Accuracy/Bias (matrix interference)	Analyte-specific (QSM)	Matrix Spike Duplicates	S&A
		Overall accuracy/bias (contamination)	No target analyte concentrations ≥ 1/2 LOQ	Equipment Blanks	S
		Sensitivity	Recovery within ±25% of LOQ	LOQ verification sample (spiked at LOQ)	A
		Completeness	See Worksheet #34	See Worksheet #34	

Table 12-3– Measurement Performance Criteria – Compound (Matrix)

Matrix	**Groundwater - Soil**				
Analytical Group	**Pesticides / PCBs (SVOCs)**				
Concentration Level	**Low**				
Sampling Procedure SOPP	**Analytical Method/SOP[1]**	**Data Quality Indicators**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
	8081B/8082A / SV002, SV008	Overall Precision	RPD ≤ 30% when detected in both samples ≥ sample-specific LOQ	Field Duplicates	S
		Analytical Precision (laboratory)	RPD ≤ 30%	Laboratory Sample Duplicates	A
		Analytical Accuracy/Bias (laboratory)	Analyte-specific (QSM)	Laboratory Control Samples	A
		Analytical Accuracy/Bias (matrix interference)	Analyte-specific (QSM)	Matrix Spike Duplicates	S&A
		Overall accuracy/bias (contamination)	No target analyte concentrations ≥ 1/2 LOQ	Equipment Blanks	S
		Sensitivity	Recovery within ±25% of LOQ	LOQ verification sample (spiked at LOQ)	A
		Completeness	See Worksheet #34	See Worksheet #34	

Table 12-4– Measurement Performance Criteria – Compound (Matrix)

Matrix	**Groundwater - Soil**				
Analytical Group	**Metals**				
Concentration Level	**Low**				
Sampling Procedure SOPP	**Analytical Method/SOP¹**	**Data Quality Indicators**	**Measurement Performance Criteria**	**QC Sample and/or Activity Used to Assess Measurement Performance**	**QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)**
	6010C/7470A/7471B / MT009, MT012	Overall Precision	RPD ≤ 20% when detected in both samples ≥ sample-specific LOQ	Field Duplicates	S
		Analytical Precision (laboratory)	RPD ≤ 20%	Laboratory Sample Duplicates	A
		Analytical Accuracy/Bias (laboratory)	Analyte-specific (QSM)	Laboratory Control Samples	A
		Analytical Accuracy/Bias (matrix interference)	Analyte-specific (QSM)	Matrix Spike Duplicates	S&A
		Overall accuracy/bias (contamination)	No target analyte concentrations ≥ 1/2 LOQ	Equipment Blanks	S
		Sensitivity	Recovery within ±25% of LOQ	LOQ verification sample (spiked at LOQ)	A
		Completeness	See Worksheet #34	See Worksheet #34	

QAPP Worksheet #23 – Analytical SOP References Table – Laboratory

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix / Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WC001 Rev 0	Reactive Cyanide Screen	Definitive	Solids & Water/Inorganics	Colorimetric	CT Laboratories	N
WC002 Rev 0	Reactive Cyanide Distillation	Definitive	Solids & Water/Inorganics	Distillation	CT Laboratories	N
WC003 Rev 8	Cyanide, Total & Amenable to Chlorination	Definitive	Solids & Water/Inorganics	Lachat	CT Laboratories	N
WC005 Rev 2	Color	Definitive	Water/Inorganics	Colorimetric	CT Laboratories	N
WC006 Rev 2	Turbidity	Definitive	Water/Inorganics	Nephelometry	CT Laboratories	N
WC007 Rev 2	Corrosivity - Langelier & Aggressive Indices	Definitive	Solids & Water/Inorganics	Calculation	CT Laboratories	N
WC008 Rev 10	Alkalinity, Automated Colorimetric	Definitive	Water/Inorganics	Colorimetric	CT Laboratories	N
WC009 Rev 4	Alkalinity, Titrimetric	Definitive	Water/Inorganics	Titrimetric	CT Laboratories	N
WC010 Rev 4	Biological Oxygen Demand (BOD & CBOD)	Definitive	Water/Inorganics	DO	CT Laboratories	N
WC011 Rev 3	Chemical Oxygen Demand, Closed Reflux	Definitive	Water/Inorganics	Colorimetric	CT Laboratories	N
WC012 Rev 2	Specific Conductance	Definitive	Water/Inorganics	Meter	CT Laboratories	N
WC013 Rev 5	Ammonia-Nitrogen, Automated	Definitive	Solids & Water/Inorganics	Colorimetric	CT Laboratories	N
WC014 Rev 0	Ammonia In-line Distillation	Definitive	Water/Inorganics	Lachat	CT Laboratories	N
WC015 Rev 6	Nitrate+Nitrite, Automated Colormetric	Definitive	Water/Inorganics	Lachat	CT Laboratories	N
WC016 Rev 5	TKN & KN	Definitive	Solids & Water/Inorganics	Colorimetric	CT Laboratories	N
WC017 Rev 5	Phosphorus, Total	Definitive	Water/Inorganics	Lachat	CT Laboratories	N
WC018 Rev0	Phosphorus, Soil Cupric Digestion	Definitive	Solids/Inorganics	Lachat	CT Laboratories	N
WC019 Rev 5	Phenolics	Definitive	Solids & Water/Inorganics	Lachat	CT Laboratories	N
WC020 Rev 3	pH- Liquids	Definitive	Water/Inorganics	Probe	CT Laboratories	N
WC021 Rev 4	pH- Soils and Waste	Definitive	Solids/Inorganics	Probe	CT Laboratories	N
WC022 Rev 3	Free Liquids (Paint Filter)	Definitive	Solids & Water/Inorganics	Filter	CT Laboratories	N
WC023 Rev 3	Available Phosphorus	Definitive	Solids/Inorganics	Lachat	CT Laboratories	N
WC024 Rev 7	Solids, Dissolved	Definitive	Water/Inorganics	Gravimetric	CT Laboratories	N
WC025 Rev 5	Solids, TSS	Definitive	Water/Inorganics	Gravimetric	CT Laboratories	N
WC026 Rev 4	Solids, Total	Definitive	Water/Inorganics	Gravimetric	CT Laboratories	N
WC027 Rev, 3	Solids, T Volatile	Definitive	Water/Inorganics	Gravimetric	CT Laboratories	N

QAPP Worksheet #23 – Analytical SOP References Table – Laboratory

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix / Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
WC028 Rev 5	Reactive Sulfide Screen	Definitive	Solids & Water/Inorganics	Colorimetric	CT Laboratories	N
WC029 Rev 0	Reactive Sulfide Distillation	Definitive	Solids & Water/Inorganics	Distillation	CT Laboratories	N
WC030 Rev 5	Sulfide	Definitive	Water/Inorganics	Titrimetric	CT Laboratories	N
WC031 Rev 2	Residual Chlorine	Definitive	Water/Inorganics	Spec	CT Laboratories	N
WC032 Rev 2	Hex Chrom, Automated Colormetric	Definitive	Water/Inorganics	Lachat	CT Laboratories	N
WC033 Rev 1	Hex Chrom, Alkaline Dig - Colormetric Analysis	Definitive	Solids/Inorganics	Spec	CT Laboratories	N
WC034 Rev 2	Flash-Point by Pensky-Martens Closed Cup Tester	Definitive	Solids & Water/Inorganics	Flash Point Tester	CT Laboratories	N
WC036 Rev 0	Specific Gravity	Definitive	Solids/Inorganics	Calculation	CT Laboratories	N
WC037 Rev 5	Ion Chromatography	Definitive	Solids & Water/Inorganics	IC	CT Laboratories	N
WC038 Rev 0	Ion Chromatography Analysis of Volatile Fatty Acids	Definitive	Water/Inorganics	IC	CT Laboratories	N
WC039 Rev 3	Total Organic Carbon in Water	Definitive	Water/Inorganics	Oxidation Combustion	CT Laboratories	
WC040 Rev 2	Total Organic Carbon in Soil	Definitive	Solids/Inorganics	Oxidation Combustion	CT Laboratories	N
WC043 Rev 0	Salinity	Definitive	Water/Inorganics	Salinity Meter	CT Laboratories	N
WC044 Rev 0	Cyanide Automated Colorimetric In-Line Distillation	Definitive	Water/Inorganics	Lachat	CT Laboratories	N
WC045 Rev 1	Percent Lipids	Definitive	Tissue/Inorganics	Gravimetric	CT Laboratories	N
PR002 Rev 2	TCLP / SPLP Extraction, Volatile Fraction (ZHE)	Definitive	TCLP	Extraction Vessel	CT Laboratories	N
PR003 Rev 4	TCLP / SPLP Extraction, Non-Volatile Fraction	Definitive	TCLP	Extraction Vessel	CT Laboratories	N
MT003 Rev 5	Acid Digestion of Waters for Dissolved or Total Recoverable Metals by ICP & GFAA	Definitive	Water/Metals Prep	ICP/GFAA	CT Laboratories	N
MT004 Rev 8	Acid Digestion of Waters for Total Metals by ICP	Definitive	Water/Metals Prep	ICP	CT Laboratories	N
MT005 Rev 2	Acid Digestion of Waters for Arsenic and Selenium by GFAA	Definitive	Water/Metals Prep	GFAA	CT Laboratories	N
MT009 Rev 2	Inductively Coupled Plasma (ICP) Emission – ICP-OES 6000	Definitive	Solids & Water/Metals	ICP	CT Laboratories	N
MT011 Rev 0	Graphite Furnace Atomic Absorption (GFAA)	Definitive	Solids & Water/Metals	GFAA	CT Laboratories	N

QAPP Worksheet #23 – Analytical SOP References Table – Laboratory

Lab SOP Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Matrix / Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
MT012 Rev 8	Mercury Cold Vapor Atomic Absorption (CV)	Definitive	Solids & Water/Metals	CetacM-6000A Mercury Analyzer	CT Laboratories	N
SV001 Rev 10	Diesel Range Organics by GC	Definitive	Solids & Water/Organics	GC	CT Laboratories	N
SV002 Rev 9	Organochlorine Pesticide by GC w/extended list	Definitive	Solids & Water/Organics	GC	CT Laboratories	N
SV004 Rev 11	Polychlorinated byphenyls (PCBs) as Aroclors by GC	Definitive	Solids & Water/Organics	GC	CT Laboratories	N
SV005 Rev 9	Semi-volatile Organic Compounds by 8270C	Definitive	Solids & Water/Organics	GC-MS	CT Laboratories	N
SV006 Rev 0	Semi-volatile Organic Compounds by 8270D	Definitive	Solids & Water/Organics	GC-MS	CT Laboratories	N
SV007 Rev 0	Semi-volatile Organic Compounds by GC/MS SIM	Definitive	Solids & Water/Organics	GC-MS SIM	CT Laboratories	N
SV008 Rev 14	Polynuclear Aromatic Hydrocarbons by HPLC	Definitive	Solids & Water/Organics	HPLC	CT Laboratories	N
SV009 Rev 10	Explosives by Modified Method 8330A	Definitive	Solids & Water/Organics	HPLC	CT Laboratories	N
SV010 Rev 5	Explosives by Modified Method 8330B w/ Extended Analyte List	Definitive	Solids & Water/Organics	HPLC	CT Laboratories	N
SV015 Rev 2	HEM - Oil and Grease	Definitive	Water/Inorganics	SPE	CT Laboratories	N
SV016 Rev 3	Ethylene Glycol and Propylene Glycol by GC	Definitive	Solids & Water/Organics	GC	CT Laboratories	N
SV017 Rev 3	Ethylene dibromide & Dibromochloropropane by ECD/GC	Definitive	Solids & Water/Organics	ECD-GC	CT Laboratories	N
SV018 Rev 0	Thiodiglycol by HPLC	Definitive	Water/Organics	HPLC	CT Laboratories	N
VO001 Rev 2	Analysis of Dissolved Methane, Ethene, Ethane & Carbon dioxide in Water	Definitive	Water/Organics	GC-FID	CT Laboratories	N
VO002 Rev 2	Analysis of Volatile Organic Compounds by GC/MS for Safe Drinking Water	Definitive	Drinking Water/Organics	GC-MS	CT Laboratories	N
VO003 Rev 9	Analysis of Volatile Organic Compounds by GC/MS (8260B)	Definitive	Solids & Water/Organics	GC-MS	CT Laboratories	N
VO004 Rev0	Analysis of Volatile Organic Compounds by GC/MS (8260C)	Definitive	Solids & Water/Organics	GC-MS	CT Laboratories	N
VO005 Rev 4	Analysis of BTEX and GRO by GC	Definitive	Solids & Water/Organics	GC	CT Laboratories	N

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QAPP Worksheet #24 – Analytical Instrument Calibration Table – Laboratory

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
ICP / 6010C	Initial calibration (ICAL)	Daily prior to sample analysis. Minimum one high standard and a calibration blank.	Linear regression - correlation coefficient ≥0.995	Correct problem, repeat ICAL	Analyst / Supervisor	MT009 Rev 2
ICP / 6010C	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	Percent recovery 90 to 110 percent	Correct problem. Rerun ICV. If rerun fails, repeat ICAL.	Analyst / Supervisor	MT009 Rev 2
ICP / 6010C	Continuing Calibration Verification (CCV)	After every 10 sample injections and at the end of the run	Within ± 10% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst / Supervisor	MT009 Rev 2
ICP / 6010C	Low-level Calibration Check Standard (Low-level ICV)	Daily.	All reported analytes within ± 20% of true value.	Correct problem and repeat ICAL.	Analyst / Supervisor	MT009 Rev 2
ICP / 6010C	Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run, after every 10 field samples, and at the end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst / Supervisor	MT009 Rev 2
ICP / 6010C	Interference Check Solutions (ICS)	After ICAL and prior to sample analysis.	ICS-A: Absolute value of concentration for all non-spiked project analytes < LOD (unless they are a verified trace impurity from one of the spiked analytes); ICS-AB: Within ± 20% of true value.	Terminate analysis; locate and correct problem; reanalyze ICS, reanalyze all samples.	Analyst / Supervisor	MT009 Rev 2
CVAA / 7470A/7471A	Initial calibration (ICAL)	Daily prior to sample analysis. Minimum 5 standards and a calibration blank.	Linear regression - correlation coefficient ≥0.995	Correct problem, repeat ICAL	Analyst / Supervisor	MT012 Rev 8
CVAA / 7470A/7471A	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of the true value.	Correct problem. Rerun ICV. If rerun fails, repeat ICAL.	Analyst / Supervisor	MT012 Rev 8

QAPP Worksheet #24 – Analytical Instrument Calibration Table – Laboratory

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
CVAA / 7470A/7471A	Continuing Calibration Verification (CCV)	After every 10 field samples and at the end of the analysis sequence.	All reported analytes within ± 10% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst / Supervisor	MT012 Rev 8
CVAA / 7470A/7471A	Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample rune, after every 10 field samples, and at the end of the analysis sequence.	No analytes detected > LOD.	Correct problem and repeat ICAL. All samples following the last acceptable calibration blank must be reanalyzed.	Analyst / Supervisor	MT012 Rev 8
HPLC / 8330A/B	Initial Calibration (ICAL)	At instrument setup and after ICV or CCV failure, prior to sample analysis. Minimum of 5 levels for linear and 6 levels for quadratic.	ICAL must meet one of the three options below: <u>Option 1</u>: RSD for each analyte ≤ 15%; <u>Option 2</u>: linear least squares regression for each analyte: $r^2 \geq 0.99$; <u>Option 3</u>: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst / Supervisor	SV009 Rev 10, SV010 Rev 5
HPLC / 8330A/B	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes and surrogates within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL. No samples will be analyzed until the problem has been corrected.	Analyst / Supervisor	SV009 Rev 10, SV010 Rev 5
HPLC / 8330A/B	Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All reported analytes and surrogates within ± 20% of the true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst / Supervisor	SV009 Rev 10, SV010 Rev 5

QAPP Worksheet #24 – Analytical Instrument Calibration Table – Laboratory

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC / 8015C(GRO/DRO)/ 8082A/8081B	Breakdown check (Endrin/DDT Method 8081 only)	Before sample analysis and at the beginning of each 12-hour shift.	Degradation of DDT and Endrin must each be ≤ 15%.	Correct problem, then repeat breakdown checks. No samples shall be run until degradation of DDT and Endrin is each ≤ 15%.	Analyst / Supervisor	SV002 Rev 9
GC / 8015C(GRO/DRO)/ 8082A/8081B	Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up and after ICV or CCV failure, prior to sample analysis. Minimum 5 levels for linear and 6 levels for quadratic. Quantitation for multi-component analytes such as chlordane, toxaphene, and Araclors must be performed using a 5-point calibration. Results may not be quantitated using a single point.	ICAL must meet one of the three options below: Option 1: RSD for each analyte ≤ 20%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$.	Correct problem then repeat ICAL. No samples shall be analyzed until ICAL has passed.	Analyst / Supervisor	SV001 Rev 10, SV002 Rev 9, SV004 Rev 11, VO005 Rev 4
GC / 8015C(GRO/DRO)/ 8082A/8081B	Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence. Calculate for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N/A.	Analyst / Supervisor	SV001 Rev 10, SV002 Rev 9, SV004 Rev 11, VO005 Rev 4
GC / 8015C(GRO/DRO)/ 8082A/8081B	Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change). Calculate for each analyte and surrogate.	RT width is ± 3 times standard deviation for each analyte RT from the 72-hour study.	N/A.	Analyst / Supervisor	SV001 Rev 10, SV002 Rev 9, SV004 Rev 11, VO005 Rev 4
GC / 8015C(GRO/DRO)/ 8082A/8081B	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 20% of true value.	Correct problem, rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	Analyst / Supervisor	SV001 Rev 10, SV002 Rev 9, SV004 Rev 11, VO005 Rev 4
GC / 8015C(GRO/DRO)/ 8082A/8081B	Continuing Calibration Verification (CCV)	Before sample analysis, after every 10 field samples, and at the end of the analysis sequence with the exception of CCVs for Pesticides multi-component analytes (i.e. Toxaphene, Chlordane), which are only required before sample analysis.	All reported analytes and surrogates within established RT windows. All reported analytes and surrogates within ± 20% of true value.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst / Supervisor	SV001 Rev 10, SV002 Rev 9, SV004 Rev 11, VO005 Rev 4

QAPP Worksheet #24 – Analytical Instrument Calibration Table – Laboratory

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS / 8260C/8270D	Tune Check	Prior to ICAL and prior to each 12-hour period of sample analysis.	Specific ion abundance criteria of BFB or DFTPP from method.	Retune instrument and verify. No samples shall be analyzed without a valid tune.	Analyst / Supervisor	SV006 Rev 0, SV007 Rev 0, V004 Rev 0
GC/MS / 8260C/8270D	Performance Check (Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation ≤ 20% for DDT. Benzidine and pentachlorophenol shall be present at their normal responses, and shall not exceed a tailing factor of 2.	Correct problem, then repeat performance checks.	Analyst / Supervisor	SV006 Rev 0
GC/MS / 8260C/8270D	Initial Calibration (ICAL) for all analytes (including surrogates)	At instrument set-up, prior to sample analysis. Minimum 5 levels for linear and 6 levels for quadratic.	Each analyte must meet one of the three options below: Option1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: $r^2 \geq 0.99$; Option 3: non-linear least squares regression (quadratic) for each analyte: $r^2 \geq 0.99$. *If specific version of a method requires addition evaluation (e.g., RFs or low calibration standard analysis and recovery criteria) these additional requirements must also be met.	Correct problem then repeat ICAL. No samples shall be analyzed until ICAL has passed.	Analyst / Supervisor	SV006 Rev 0, SV007 Rev 0, V004 Rev 0,
GC/MS / 8260C/8270D	Retention Time window position establishment	Once per ICAL and at the beginning of the analytical sequence.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	N/A.	Analyst / Supervisor	SV006 Rev 0, SV007 Rev 0, V004 Rev 0,
GC/MS / 8260C/8270D	Evaluation of Relative Retention Times (RRT)	With each sample. RRTs may be updated based on the daily CCV.	RRT of each reported analyte within ± 0.06 RRT units. RRTs shall be compared with the most recently updated RRTs.	Correct problem, then rerun ICAL.	Analyst / Supervisor	SV006 Rev 0, SV007 Rev 0, V004 Rev 0,
GC/MS / 8260C/8270D	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 20% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL. No samples shall be analyzed until calibration has been verified with a second source.	Analyst / Supervisor	SV006 Rev 0, SV007 Rev 0, V004 Rev 0,

QAPP Worksheet #24 – Analytical Instrument Calibration Table – Laboratory

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
GC/MS / 8260C/8270D	Continuing Calibration Verification (CCV)	Daily before sample analysis; after every 12 hours of analysis time; and at the end of the analytical batch run.	All reported analytes and surrogates within ± 20% of true value. All reported analytes and surrogates within ± 50% for end of analytical batch CCV. *If the specific version of a method requires additional evaluation (e.g., average RFs) these additional requirements must also be met.	Recalibrate, and reanalyze all affected samples since the last acceptable CCV; or Immediately analyze two additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst / Supervisor	SV006 Rev 0, SV007 Rev 0, V004 Rev 0,
GC/MS / 8260C/8270D	Internal Standards (IS)	Every field sample, standard, and QC sample.	Retention time within ± 10 seconds from retention time of the midpoint standard in the ICAL; IS area within – 50% to +100% of ICAL midpoint standard.	Inspect mass spectrometer and GC for malfunctions and correct problem. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	Analyst / Supervisor	SV006 Rev 0, SV007 Rev 0, V004 Rev 0,
Spectrophotometer (soil) / Lachat (water) (Hexavalent Chromium) / 7196	Initial Calibration (ICAL)	Daily ICAL prior to sample analysis.	>$r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Analyst / Supervisor	WC032 Rev 2, WC033 Rev 1
Spectrophotometer (soil) / Lachat (water) (Hexavalent Chromium) / 7196	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source standard prior to sample analysis.	All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst / Supervisor	WC032 Rev 2, WC033 Rev 1
Spectrophotometer (soil) / Lachat (water) (Hexavalent Chromium) / 7196	Continuing Calibration Verification (CCV)	Daily before sample analysis, after every 15 field samples and at the end of the analysis sequence.	Within ± 10% of true value.	Recalibrate and reanalyze all affected samples since last acceptable CCV; or Immediately analyze 2 additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since last acceptable CCV.	Analyst / Supervisor	WC032 Rev 2, WC033 Rev 1
Lachat (Cyanide) / 9012A/335.4	Initial Calibration (ICAL)	Daily ICAL prior to sample analysis.	>$r^2 \geq 0.99$.	Correct problem, then repeat ICAL.	Analyst / Supervisor	WC044 Rev 0, WC003 Rev 8
Lachat (Cyanide) / 9012A/335.4	Distillation Verification	Once after each ICAL, with 2 distilled ICAL standards; prior to sample analysis. Not required if all ICAL standards are distilled.	Within ± 10% of non-distilled std value.	Correct problem, rerun distilled standards or repeat ICAL	Analyst / Supervisor	WC044 Rev 0, WC003 Rev 8

QAPP Worksheet #24 – Analytical Instrument Calibration Table – Laboratory

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
Lachat (Cyanide) / 9012A/335.4	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of second source std prior to sample analysis.	Within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst / Supervisor	WC044 Rev 0, WC003 Rev 8
Lachat (Cyanide) / 9012A/335.4	Continuing Calibration Verification (CCV)	After every 10 field samples and at end of the analysis sequence.	Within ± 10% of true value.	Recalibrate and reanalyze all affected samples since last acceptable CCV; or Immediately analyze 2 additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since last acceptable CCV.	Analyst / Supervisor	WC044 Rev 0, WC003 Rev 8
Lachat (Cyanide) / 9012A/335.4	Initial and Continuing Calibration Blank (ICB/CCB)	Before beginning a sample run; After every 10 field samples; At end of the analysis sequence (after ICV and each CCV).	No cyanide detected >LOD.	Correct problem and reanalyze all samples analyzed since last acceptable calibration blank.	Analyst / Supervisor	WC044 Rev 0, WC003 Rev 8
IC (Anions, VFAs, Nitrocellulose / 9056/9056M	Initial Calibration (ICAL) for all analytes	ICAL prior to sample analysis.	>r^2 ≥ 0.99.	Correct problem, then repeat ICAL.	Analyst / Supervisor	WC037 Rev 5, WC038 Rev 0, VO008, Rev 1
IC (Anions, VFAs, Nitrocellulose / 9056/9056M	Retention Time window position establishment	Once per multipoint calibration.	Position shall be set using the midpoint std of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used	NA	Analyst / Supervisor	WC037 Rev 5, WC038 Rev 0, VO008, Rev 1
IC (Anions, VFAs, Nitrocellulose / 9056/9056M	Retention Time (RT) window width	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 time std deviation for each analyte RT over a 24-hour period.	NA	Analyst / Supervisor	WC037 Rev 5, WC038 Rev 0, VO008, Rev 1
IC (Anions, VFAs, Nitrocellulose / 9056/9056M	Initial Calibration Verification (ICV)	Once after each ICAL, analysis of a second source std prior to sample analysis.	All reported analytes within established RT windows. All reported analytes within ± 10% of true value.	Correct problem. Rerun ICV. If that fails, repeat ICAL.	Analyst / Supervisor	WC037 Rev 5, WC038 Rev 0, VO008, Rev 1

QAPP Worksheet #24 – Analytical Instrument Calibration Table – Laboratory

Instrument/ Method	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference[1]
IC (Anions, VFAs, Nitrocellulose / 9056/9056M	Continuing Calibration Verification (CCV)	Before sample analysis; after every 10 field samples; and at the end of the analysis sequence.	All reported analytes within established retention time windows. All reported analytes with ± 10% of true value.	Recalibrate and reanalyze all affected samples since last acceptable CCV; or Immediately analyze 2 additional consecutive CCVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CCV.	Analyst / Supervisor	WC037 Rev 5, WC038 Rev 0, VO008, Rev 1

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QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Identify all analytical instrumentation that requires maintenance, testing, or inspection and provide the SOP reference number for each. In addition, document the frequency, acceptance criteria, and corrective action requirements on the worksheet.

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
GC/MS	Replace septa, clean injection port, clip column, clip or replace pre-column check auto sampler, clean source	SVOC	Detector, injection port,column, autosampler	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	SV006
GC/MS	Replace septa, clean injection port, clip column, check auto sampler, clean source	VOC	Detector, injection port,column, autosampler	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	VO004
GC	Replace septa, clean injection port, clip column, clip or replace pre-column, check auto sampler	Pesticides / PCBs DRO/TPH	Detector, injection port,column, autosampler	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	SV002, SV004, SV001
GC	Replace septa, clean injection port, clip column, clip or replace pre-column, check auto sampler	VOC / GRO Dissolved Gases	Detector, injection port,column, autosampler	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	VO005, VO001
ICP-AES	Clean torch assembly, nebulizer, and spray chamber as needed. Check argon gas, vacuum, waste container, and reagent water levels daily. Replace pump tubing as needed.	Metals	Torch, nebulizer chamber, pump and pump tubing, vacuum source, waste container	Daily prior to calibration	Acceptable calibration	Correct problem and recalibrate	Analyst / Supervisor	MT009
CVAA	Check lamp voltage, check autosampler, make necessary pump tube changes.	Metals	Autosampler, gases, pump tubing.	Daily prior to calibration	Acceptable calibration	Correct problem and recalibrate	Analyst / Supervisor	MT0012
Spectrophoto-meter	Check the accuracy of the light source.	Inorganics	Detector, Light source	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	WC033

QAPP Worksheet #25 – Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference[1]
HPLC	Fill solvent bottles, change precolumn, column frits, flush column, clean pump head	Explosives & Propellants	Autosampler, column flow, detector, column and associated parts	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	SV009, SV010
Lachat	Replace pump tubing and o-rings. Clean out instrument lines.	Inorganics	Detector, flow rate, autosampler	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	WC013, WC014, WC015, WC016, WC017
IC	Replace ultrapure filters and column bed support assemblies.	Inorganics	Column flow, column and associated parts	As needed	Must meet initial and/or continuing calibration criteria	Repeat maintenance activity or remove from service	Lab Section Supervisor	WC037, WC038, VO008

Appendix B
Accident Prevention Plan/Site Safety and Health Plan

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ACCIDENT PREVENTION PLAN

FY16 RECYCLING OF MATERIALS AT THE ATLAS SCRAP YARD AREA OF CONCERN AND SETUP OF TEMPORARY STORAGE FACILITY

Former Ravenna Army Ammunition Plant Restoration Program
Portage and Trumbull Counties, Ohio

December 20, 2016

Contract No.: W912QR-12-D-0011
Delivery Order 0017

December 2016

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Attachment 3	Activity Hazard Analysis
Attachment 4	Camp Ravenna First Responder Reporting Form

Acronyms and Abbreviations

AHA	Activity Hazard Analysis
AOC	Area of Concern
APP	Accident Prevention Plan
AR	Administrative Record
ARNG	U.S. Army National Guard
CELRL	U.S. Army Corps of Engineers, Louisville District
CFR	Code of Federal Regulations
CHEMTREC	Chemical Transportation Emergency Center
COR	Contracting Officer's Representative
CPR	cardiopulmonary resuscitation
CSP	Certified Safety Professional
DO	Delivery Order
DoD	Department of Defense
EM	engineer manual
EMR	experience modification rate
ENG	engineer form
ERT	ERT, Inc.
ES&H	environmental, safety and health
HAZCOM	Hazard Communication
HAZWOPER	Hazardous Waste Operations and Emergency Response
hr	hour
IDW	investigative-derived waste
IRP	Installation Restoration Program
ODH	Ohio Department of Health
OHARNG	Ohio U.S. Army National Guard
OSHA	Occupational Safety and Health Administration
PG	Professional Geologist
PM	Project Manager
PMP	Project Management Professional
REIMS	Ravenna Environmental Information Management System
RVAAP	Former Ravenna Army Ammunition Plant
RVAAP-50	Atlas Scrap Yard, Former Ravenna Army Ammunition Plant
PPE	personal protective equipment
SDS	Safety Data Sheet
SHM	Safety and Health Manager
SI	Site Investigation
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
TCE	Trichloroethylene
TM	Technical Manager
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency
USP&FO	U.S. Property and Fiscal Officer
WWII	World War II

1. SIGNATURE SHEET

ACCIDENT PREVENTION PLAN APPROVAL

By their specific signature, the undersigned certify that they approve this APP for utilization during field activities in support of Contract No. W912QR-12-D-0011, Delivery Order (DO) 0017. This APP has been prepared by a Certified Safety Professional (CSP).

Prepared by:

By signing the APP, the ERT Division SHM certifies that J.T. Nolan has completed the required occupational safety and health courses and is qualified, by both training and experience to serve as the Site Safety and Health Officer (SSHO), as well as the "Occupational Safety and Health Administration (OSHA) Competent Person for overall site health and safety," for each phase of field work associated with the FY16 Recycling and Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility at the Former Ravenna Army Ammunition Plant (RVAAP) Camp Ravenna Joint Military Training Center, Portage and Trumbull Counties, Ohio.

11/15/2016

Michael Barsa, CSP
Board of Certified Safety Professionals No. 24437
301-323-1447
ERT, Inc. (ERT) Division Safety and Health Manager (SHM)

Date

Plan Approval:

11/15/2016

Sean Carney, PMP
Project Manager/Senior Technical Reviewer (ERT)

Date

9/22/2016

Jennifer Harlan, PMP
Division Manager (ERT)

Date

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2. BACKGROUND INFORMATION

a. Contractor Name

The contractor for the performance of this project is ERT. As the contractor for this project, ERT will be responsible for its successful completion and for the management of all resources necessary to meet the requirements of the Performance Work Statement.

b. Contract Number

The contract number for this project is W912QR-12-D-0011, Delivery Order 0017 issued to ERT by the U.S. Army Corps of Engineers (USACE), Louisville District (CELRL).

c. Project Name

This APP applies to intrusive field investigation activities occurring at the Former Ravenna Army Ammunition Plant in Portage and Trumbull Counties, OH.

d. Brief Project Description; Description of Work to be Performed

The former RVAAP was utilized as a load, assemble, and pack facility for munitions. At the onset of World War II (WWII), the RVAAP was built to produce large-caliber artillery projectiles and bombs. Although RVAAP downsized after WWII, plant production lines were reactivated during the Korean War and the Vietnam conflicts. Additionally, the plant conducted nearly continuous demilitarization of war stocks, refurbishment of inventoried ammunition, and minor research and development projects through 1992.

In 1992, the installation's status changed from "inactive but maintained" to that of "caretaker." Administrative control of the facility acreage was transferred from the Base Realignment and Closure Division to the U.S. Property and Fiscal Officer (USP&FO) for Ohio in a series of transfers from 1999 to 2013. As of September 2013, administrative accountability for the entire 21,683-acre facility has been transferred to the USP&FO for Ohio and subsequently licensed to the OHARNG for use as a military training site, Camp Ravenna. The RVAAP restoration program involves cleanup of former production/operational areas throughout the facility related to former activities conducted under the RVAAP. The RVAAP Installation Restoration Program (IRP), managed by the U.S. Army National Guard (ARNG) and the OHARNG, encompasses investigation and cleanup of past activities over the entire 21,683 acres of the former RVAAP (USACE, 2016).

The Atlas Scrap Yard AOC, formerly known as the construction camp, is located on approximately 73 acres in the central portion of Camp Ravenna south of Newton Falls Road and west of Paris Windham Road. There is no fence around the AOC as a perimeter boundary, but the AOC is bound by Newton Falls Road to the north and Paris Windham Road to the east. Load Line 4 is located to the south of the AOC. The Atlas Scrap Yard has served many operational functions over the history of the former RVAAP, but the AOC was never used for munitions productions. From 1940-1945 the AOC operated as a construction camp to house workers and their families while the facility was being constructed. By the end of WWII the majority of buildings and structures at the AOC had been demolished or relocated to other areas. The facilities that remained were used to support roads and grounds maintenance activities. The remaining structures were demolished after the Vietnam War, and the AOC became a stockpile storage area for bulk materials including gravel, railroad ballasts, sand and culverts (Leidos, 2015). The AOC contains several piles of debris consisting of railroad ties, telephone poles, and concrete (USACE, 2016).

The Administrative Record contains the former RVAAP restoration program records and historical maps for the former RVAAP/Camp Ravenna. It is currently stored at multiple locations at Camp Ravenna, including the Camp Ravenna Environmental Office (1438 State Route 534 SW, Newton Falls, Ohio 44444) and Buildings 1038 and 1047 (closest address 8451 State Route 5, Ravenna Ohio 44266). The Administrative Record is to be moved from its current locations and consolidated to a temporary storage facility within the Camp Ravenna property boundary (USACE, 2016).

This project will involve the loading, transportation, and recycling/disposal of stockpiled railroad ties, concrete, and telephone poles located at the Atlas Scrap Yard AOC. Additionally, the project will include the installation of a temporary storage facility suitable for temperature controlled storage of former RVAAP administrative records, and the re-location of records, documents, maps, shelving, and map cases from three current locations to the temporary storage facility.

The goal of this project is to preform recycling of stockpiled material at the Atlas Scrap Yard (RVAAP-50) Area of Concern (AOC) and setup temporary storage facility for a new information repository at the Former Ravenna Army Ammunition Plant (RVAAP) Camp Ravenna Joint Military Training Center, Portage and Trumbull Counties, Ohio.

e. Location of the Project

The Atlas Scrap Yard AOC, formerly known as the construction camp, is located on approximately 73 acres in the central portion of Camp Ravenna south of Newton Falls Road and west of Paris Windham Road (**Figure 1**). There is no fence around the AOC as a perimeter boundary, but the AOC is bound by Newton Falls Road to the north and Paris Windham Road to the east.

f. Contractor Safety Information

The Experience Modification Rate (EMR) is a factor that is calculated by measuring the difference between ERT actual claims experience in worker's compensation (including frequency and severity of the losses) as compared to the average expected claims experience for the entire class code(s) assigned to the company. An EMR is calculated using a three-year rolling period. ERT's EMR for 2015 is 0.91, and ERT's projected EMR for 2016 is 0.77. A summary of ERT's Work-Related Injuries and illnesses for the past three years is included below (Occupational Safety and Health Administration [OSHA] Form 300A Logs are provided in **Attachment 1**):

- 2015 - 3 recordable, 1 involving lost time (1 day total);
- 2014 - 1 recordable, no lost time; and
- 2013 - 1 recordable, no lost time.

g. Phases of Work/Hazardous Activities Requiring an Activity Hazard Analysis

The major phases of work involved in this project include:

- Site visits and inspections;

- Mobilize/demobilize;
- Site preparation;
- Complete the loading, transportation, and recycling/disposal of stockpiled railroad ties, concrete, and telephone poles located at the Atlas Scrap Yard AOC;
- Install a temporary storage facility suitable for temperature controlled storage of former RVAAP administrative records. Re-locate records, documents, maps, shelving, and map cases from three current locations to the temporary storage facility;
- Waste management; and
- Restoration of disturbed areas.

Included in this APP are the Site Safety and Health Plan (SSHP), presented as **Attachment 2**, and the Activity Hazard Analysis (AHA), presented as **Attachment 3**. The attachments are prepared in accordance with USACE Engineer Manual (EM) 385-1-1, November 2014 (USACE, 2014).

As required per EM 385-1-1, the phases of work require AHAs that assess specific hazards, risk levels, and risk mitigation controls. During the course of work, ERT's personnel and subcontractors will be involved in activities that will potentially expose them to chemical, physical, and biological hazards. Exposure to these hazards will be controlled through the use of engineering, operational, administrative controls, and personal protective equipment (PPE).

This APP will be ERT's overall project environmental, safety and health (ES&H) document (parent document) while the SSHP will be used to present the site-specific ES&H hazard and exposure mitigation information anticipated for the project tasks.

Prior to site mobilization, the ERT project team will receive a site-specific health and safety briefing. The field team will ensure that all equipment and materials required for safely completing the project objectives are on hand, tested, configured, and setup prior to deployment. ERT will confirm that all on-site personnel have the proper training records and are under medical surveillance.

The location of any underground hazards will be identified prior to intrusive work. If any potential hazards are identified, controls will be added to the intrusive activities-specific AHA.

At least 72 hours prior to intrusive field activities, ERT will coordinate through the Camp Ravenna Department of Public Works and local utility companies (via Ohio Utilities Protection Services or 800-362-2764) to mark-out and identify all underground utilities in the investigation areas. If necessary, sample locations will be offset. If needed, boring/sample locations to be advanced through concrete/asphalt surfaces will start with air knifing, concrete hammer drilling, and/or hand digging. ERT also has within its in-house equipment a variety of underground utility locators and underground metallic/electrical/pipe/cable utility locators that can be made available as a backup, if needed.

ERT and subcontractor field vehicles will act as mobile field offices throughout the duration of the field mobilization. All equipment brought to the site for the field effort will be removed from the property at the end of each field effort during demobilization (and whenever equipment is no longer needed on-site). The specific AHA associated with mobilization and demobilization is included in **Attachment 3**.

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3. STATEMENT OF SAFETY AND HEALTH POLICY

At ERT, the safety and health of our employees and subcontractors is the first consideration in the operation of this business. Safety and health is part of every operation and business line, and is, without question, the responsibility of every ERT employee.

It is the policy and intent of ERT to comply with all laws and regulations. To do this, ERT must constantly be aware of conditions in all work areas and activities that can produce injuries. No employee is required to work at a job that he or she knows is not safe or healthy. Identification of hazards and controlling them is the responsibility of everyone.

The personal safety and health of each ERT employee is of primary importance. The prevention of occupationally-induced injuries and illnesses is of such consequence that it will be given precedence over project operations, whenever necessary.

ERT maintains a safety and health program that conforms to the best management practices possible. To be successful, such a program must embody the proper attitudes towards injury and illness prevention, not only on the part of managers and employees, but also between each employee and his/her co-workers. Only through such a cooperative effort can a safety and health program that is in the best interest of the entire company be established and preserved.

ERT’s safety program goals and objectives are to provide a safe and healthy workplace. ERT’s accident experience goal is to have zero safety incidents. In the event an accident does occur, a formal internal audit will take place and all problems/issues related to the incident will be resolved immediately by the ERT Division Safety and Health Manager (SHM).

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4. RESPONSIBILITY OF AUTHORITY

a. Identification and Accountability of Personnel Responsible for Safety

ERT's Safety and Health Program specifies that all ERT personnel and subcontractors are responsible for their safety and the safety of those working with them. However, it is also stated that the ultimate ES&H responsibility begins with the President of ERT and this responsibility radiates outward to all management, administrative, operations, and field personnel. To achieve this philosophy, ERT empowers all personnel with stop work authority regarding known or potential ES&H issues. In addition, all ERT personnel are held accountable for performing their assigned tasks in a manner that promotes continuous, active hazard evaluation and safe task performance. Ultimately, it is the responsibility of the ERT Division SHM to ensure that the ES&H program is properly implemented.

The key personnel at ERT that are responsible for safe project performance at the corporate and project level include:

- The President of ERT
- ERT Division SHM/CSP
- ERT Division Manager
- ERT PM
- ERT SSHO/Site Superintendent
- ERT Field Team Members

The ES&H responsibilities of the personnel filling the roles listed above are presented in detail in Section 3.0 of the SSHP (**Attachment 1** of this APP). All on-site personnel will have 40-hour (hr) OSHA Hazardous Waste Operations and Emergency Response (HAZWOPER) training and current 8-hr refresher training. The SSHO/Site Superintendent will have completed the 8-hr OSHA HAZWOPER Supervisor Training. The SSHO/Site Superintendent will also have completed the 30-hr OSHA Construction Safety Training. In addition, for each field event, between ERT, its subcontractors, and any Government representatives on-site, a minimum of two on-site site personnel will also have current First Aid and cardiopulmonary resuscitation (CPR) training. All current training certifications for potential ERT field staff, including First Aid/CPR certifications are included in **Enclosure C** of the SSHP. Certifications will be added and/or updated as appropriate prior to mobilization of the field effort.

b. Lines of Authority

As a part of its corporate structure, ERT has developed a system whereby the lines of authority for personnel responsible for operations and ES&H are separate. All issues related to on-site operations regarding production and resources are handled by the PM. Issues that cannot be handled by the PM are delegated to the Division Manager. The SSHO/Site Superintendent reports directly to the ERT Division SHM for ES&H issues and is responsible for ensuring overall compliance with this APP by site personnel. A detailed list of responsibilities for all field staff working at the site is included in Section 3.0 of the SSHP.

As part of ERT's subcontractor agreement, subcontractors agree to conduct their operations in accordance with ERT's site plans and applicable federal, state, and local ES&H requirements.

All subcontractors are required to provide to ERT record of OSHA HAZWOPER training (40-hr) and current 8-hr refresher training as applicable per 29 Code of Federal Regulations (CFR) 1910.120(e)(3) for general site workers or 29 CFR 1910.120(e)(3)(ii) for site-specific workers, dependent upon the task being completed. No subcontractors will be allowed to access the site unless the OSHA Competent Person is also on-site. Attendance at Daily Safety Briefings is required of all subcontractors and provided by the SSHO/Site Superintendent.

All subcontractors are responsible for the safety and health of their employees and for complying with the standards established in this APP/SSHP. Specific responsibilities of subcontractors include:

- Complying with the requirements of their Statement of Work;
- Full compliance with ERT Standard Operating Procedures and Safety Guidelines;
- Understanding the AHA for their work activities;
- Maintaining a safe and healthy work environment;
- Compliance with contract requirements, laws, and regulations;
- Reviewing the APP/SSHP to ensure that the ES&H requirements of their specific tasks are satisfied;
- Performing all work in accordance with the APP/SSHP requirements;
- Providing trained and experienced workers for the specific work activities;
- Participating in the Daily Safety Briefings;
- Enforcing company- and project-specific rules and procedures during work activities;
- Reporting all incidents and participate in the investigations;
- Participating in routine site inspection activities; and
- Ensuring all equipment brought to the site is routinely inspected and maintained in safe working order.

It is the responsibility of the ERT Division SHM to review and accept the subcontractors' safety programs and to ensure that they comply with the requirements of 29 CFR 1910.120. It is the responsibility of the SSHO/Site Superintendent to ensure subcontractors exclusively follow this APP/SSHP and EM 385-1-1 while on-site. This APP/SSHP will be the ES&H document for the project.

ERT is in charge at the project site for those activities described within this APP (those activities specifically related to the scope of ERT's Delivery Order) and is responsible for the daily coordination of tasks and site personnel, and for ensuring Daily Safety Briefings. As a means of controlling and coordinating subcontractor/supplier activities, no subcontractors/suppliers will be allowed to access the site without signing in/out and meeting with the SSHO/Site Superintendent and/or Competent Person for the activity. Pre-operational safety briefings will be required of all subcontractors and will be provided by the SSHO/Site Superintendent.

5. TRAINING

a. Subjects for New Hire Orientation Training at the Time of Initial Hire of Each New Employee

ERT has established an ES&H training program for those staff within the Environmental Division who may conduct field activities at sites that could involve hazardous substances or hazardous, toxic, and/or radioactive waste. This training program establishes minimum training requirements for field workers, SSHO/Site Superintendents, and PMs, and includes:

- Requirements and responsibilities for accident prevention and the maintenance of safe and healthful work environments;
- General ES&H policies and procedures;
- Employee and supervisor responsibilities for reporting all accidents;
- Provisions for medical facilities and emergency response and procedures for obtaining medical treatment or emergency assistance;
- Procedures for reporting and correcting unsafe conditions or practices;
- Job hazards and the means to control/eliminate those hazards; and
- Specific training as required and appropriate to the role and responsibility level of the employee position.

Upon hire, all ERT staff members are required to undergo training appropriate to their role and responsibility level per ERT's existing ES&H program. Any new hire that has previously undergone such training is required to provide sufficient evidence of completion that satisfies the Division SHM. Refresher training will also be documented for new hires. General training and certification required for all staff necessary for successful and safe completion of field activities for this project are listed below:

PROJECT MANAGER

- OSHA 40-hour HAZWOPER Training (with up-to-date annual refresher) with a minimum of 3 days of supervised hazardous waste work experience.

SSHO

- OSHA 40-hour HAZWOPER Training (with up-to-date annual refresher) with a minimum of 3 days of supervised work experience;
- OSHA 8-hour HAZWOPER Supervisor Training;
- OSHA 30-hour Construction Safety Training, or as an equivalent, 30 hours of formal construction safety and health training covering the subjects of the OSHA 30-hour course; and
- At least 5 years of experience in the type of project assigned.

HEAVY EQUIPMENT OPERATOR

- OSHA 40-hour HAZWOPER Training (with up-to-date annual refresher) with a minimum of 3 days of supervised work experience; and
- Heavy Equipment Operations and Safety Training.

SITE SUPERINTENDENT

- USACE Construction Quality Management for Contractors
- OSHA 40-hour HAZWOPER Training (with up-to-date annual refresher) with a minimum of 3 days of supervised work experience; and
- Certified First Aid/CPR training (minimum of 2 on-site personnel at all times).

FIELD PERSONNEL/LABORERS

- OSHA 40-hour HAZWOPER Training (with up-to-date annual refresher) with a minimum of 3 days of supervised work experience; and

Certified First Aid/CPR training (minimum of 2 on-site personnel at all times).

In addition to the above training, each new hire in the ERT Environmental Services Division is required by the ERT Division Manager, assisted by the ERT Program SHM, to: (1) participate in the ERT medical surveillance program; and (2) review the ERT Corporate Safety and Health Plan, and if their position requires fieldwork, the ERT Field Activities Safety and Health Plan. Upon review, each new hire must sign the Corporate Safety and Health Plan review log, acknowledging that they have read and understand all components of the plans.

Copies of all training certifications and medical clearance forms for ERT personnel are maintained by the Human Resources Department.

b. Requirements for Mandatory Training/Certifications Applicable to this Project

In addition to the general ES&H training required for all employees listed in Section 5a above, all site workers will be provided site-specific hazard information training, as required by OSHA in 29 CFR 1910.120(i), and EM 385-1-1. This training will be based upon general hazards of the site, the specific tasks to be performed, and the hazards associated with the tasks. The SSHO/Site Superintendent will provide site-specific training at the safety indoctrination. Topics will include:

- Emergency procedures and Hazard Communication (HAZCOM) training, including evacuation routes;
- Areas of restricted access;
- Responsibilities for personnel safety;
- Identification of First Aid/CPR-qualified personnel;
- Site-specific physical, chemical, and biological hazards;
- Task specific equipment use;
- Task specific PPE beyond standard PPE training included in HAZWOPER 40-hr training;

- Location and use of the APP/SSHP (including SDSs);
- Evacuation area; and
- Route to nearest medical support facility.

All training will be recorded using the ERT Daily Safety Briefing form and will ensure that all site personnel have read and understand the APP and SSHP, and must have signed the SSHP Review Record (contained in the SSHP and available on-site). These documents will be transferred to the ERT PM for record keeping when site work is completed.

c. Requirements for Emergency Response Training

All ERT personnel involved with responding to an on-site emergency will be briefed in their roles and responsibilities as part of the initial indoctrination training discussed above. During this training, ERT personnel will be briefed on the HAZCOM program, emergency equipment, and First Aid/CPR procedures as described in the SSHP. ERT personnel will also be briefed on emergency response and contingency procedures presented in Section 11.0 of the SSHP, which include:

- Procedures and tests;
- Personnel injury/medical emergency;
- Firefighting;
- Emergency telephone numbers; and
- Medical support.

This training will be documented and will also involve a rehearsal of the emergency response procedures prior to the start of site activities. During this training the route to, and location of, the evacuation area and the location of medical support facility will be discussed with each staff member. The rally point locations and evacuation route are pre-determined as detailed in the site layout plan presented within **Figure 2**. If site conditions warrant changing the location of the evacuation area, the SSHO/Site Superintendent will make the determination and inform the field team. Prior to the initial start of work, all site personnel will familiarize themselves with the route to the nearest medical support facility.

The HAZCOM program, provided in Section 8.3 of the SSHP, will be reviewed with each site worker including:

- Chemicals that are at the site and where they are stored;
- Hazards associated with those chemicals, where the SDSs can be found and how to use them;
- Appropriate emergency response; and
- Emergency contact information.

ERT will conduct a Daily Safety Briefing, as discussed in Section 4.2.2 of the SSHP, to address potential site and task hazards prior to the deployment of personnel each day. This briefing will be conducted by the SSHO/Site Superintendent, during which all ERT and subcontractor personnel will be briefed on the tasks to be conducted that day, the hazards associated with the tasks, and the mitigation methods that will be employed by site personnel to reduce or eliminate their risk of exposure. The briefing will also include review of the previous day's observances, lessons learned, and/or current site-specific relevant topics of interest.

d. Procedures for Periodic Safety and Health Training for Supervisors and Employees

Periodic training as required in EM 385-1-1, Section 01.B.05, will be performed for all site personnel who are on-site for a length of time greater than one week. This project-specific training is intended to review past activities/lessons learned, plan for new or changed operations, review safety hazards and safe working procedures, and provide ES&H training and motivation. In addition to the Daily Safety Briefing, periodic training will also be conducted consistent with the intervals below:

- Upon the first working day of each month, as appropriate, for supervisors, provided by the SSHO/Site Superintendent; and
- Upon the first working day of each week for all site workers, provided by the SSHO/Site Superintendent.

Periodic training will be documented via the Daily Safety Briefing form, including dates, persons in attendance, subjects discussed, and persons who conducted the meetings. Documentation will be maintained on-site by the SSHO/Site Superintendent and provided to the ERT PM.

6. SAFETY AND HEALTH INSPECTIONS

a. Specific Assignment of Responsibilities

Daily and Weekly Safety Inspections and Audits: Daily inspections will be conducted by the SSHO/Site Superintendent to ensure that site operations and personnel are complying with this APP and SSHP, and other applicable regulatory requirements. At least once per week during site operations, the SSHO/Site Superintendent will conduct a compliance audit of the site using the Site-Specific Health and Safety Checklist (provided in **Enclosure B** to the SSHP).

Periodic Corporate Safety and Health Inspections: During the course of this project, it is possible that the ERT Division SHM may make an inspection of the project to ensure initial and continued compliance of the project with applicable safety, and health regulations. ERT views the possibility of audits conducted by the ERT Division SHM to be essential to the ES&H performance of site operations.

b. Inspector Qualifications

The SSHO/Site Superintendent and/or the Division SHM will be conducting inspections. Qualifications are provided in the resumes included as **Enclosure B** of the SSHP and in the training certificates provided in **Enclosure D** of the SSHP.

c. Frequency of Inspections

Per EM 385-1-1, an inspection must occur at least once every two weeks during active field work. Daily inspections and weekly audits will be conducted by the SSHO/Site Superintendent; therefore, this requirement will already be addressed.

d. Documentation of Safety Inspections

The results of daily inspections will be documented on the Daily Safety Briefing form maintained by the SSHO/Site Superintendent. Weekly audits will be recorded and documented in the Site-Specific Health and Safety Checklist (provided in **Enclosure B** to the SSHP). Any site or operational discrepancies identified, and immediate corrective actions taken will be noted on this form, and the results of the inspection will be reported to the ERT PM.

e. Deficiency Corrective Action Tracking and Follow-Up

Any deficiencies noted during a site inspection or audit will be reported to the ERT Division SHM and noted on the Site-Specific Health and Safety Checklist (provided in **Enclosure B** to the SSHP). The SSHO/Site Superintendent, in consultation with the ERT PM and Division SHM, will develop and implement the necessary corrective actions and monitor the progress until all issues are resolved and a follow-up evaluation conducted. These steps include:

- Reviewing and defining the specific procedure or activity found to be deficient;
- Finding the cause of the deficiency;
- Developing a plan to correct the deficiency;
- Implementing the corrective action plan; and
- Evaluation of the effectiveness of the corrective action.

The SSHO/Site Superintendent will provide all documentation of the deficiency and corrective action to the ERT Division SHM. The SHM will make a determination of whether any changes need to be incorporated into any site-specific plans or the ERT Corporate Safety and Health Plan.

f. The names of Competent and/or Qualified Person(s) and Proof of Competency/Qualification to Meet Specific OSHA Competent/Qualified Person(s)

The primary SSHO/Site Superintendent for this project will be J.T. Nolan. Proof of competency is provided in the resume for the SSHO/Site Superintendent provided in **Enclosure A** of the SSHP and in the training certificates provided in **Enclosure C** of the SSHP. The SSHO, or his authorized representative and/or alternate SSHO, will be considered the OSHA Competent Person on-site for overall site health and safety pertaining to the activities being performed. The Site Superintendent will also be considered the OSHA Competent Person during site preparation, during loading/transporting/recycling operations, during temporary storage facility installation, and during site restoration.

g. External Inspections

There will be no external inspections or certifications required for this project, but in the event that a representative from a regulatory agency arrives on-site to conduct an inspection, the ERT PM and/or ERT Division SHM will be contacted immediately.

7. SAFETY AND HEALTH EXPECTATIONS, INCENTIVE PROGRAMS, AND COMPLIANCE

a. Safety Program Goals, Objectives, and Accident Experience Goals

ERT's accident experience goal for this project is to perform this project without accident or defect. To facilitate this goal, ERT will implement the requirements of this APP, the SSHP, and the ERT Safety and Health Program. Additionally, ERT will make all project and site management personnel aware of this goal and will empower all site personnel with stop work authority for known or potential uncontrolled safety hazards.

b. Safety Incentive Programs

ERT organizes work teams and environment to work in a safe manner with no lost time, injury, or damage to government, ERT, or other customer-owned facilities or equipment. ERT realizes safety and health awareness must become inherent to the culture, and strives to engage all staff in active measures through awareness training, corporate and managerial attention and incentives for safe behaviors.

c. Noncompliance with Safety Requirements

General Requirements: As outlined previously in this APP, designated corporate and on-site personnel have been tasked with the overall responsibility of ensuring the safe and healthful conduct of site operations. Additionally, ERT has expended significant labor and resources towards the design and development of written programs and procedures used to safeguard site personnel from the hazards associated with this project. It is imperative that site personnel realize that their compliance with established safety and health procedures is of paramount importance in the prevention of accidents and emergencies that could compromise their safety and health, and also the well-being of other site personnel, the environment, and the public. Because violations of the safety and health procedures and programs outlined in either this APP or the SSHP can result in serious personal injury, illness, or environmental insult, personnel violating the safety or health requirements of this APP or the SSHP may be subject to disciplinary action.

Safety and Health Violations: It is the general policy of ERT that no personnel engage in any activity for which:

- they are not properly trained**;**
- the consequences of the activity are uncertain; or
- the activity hazards have not been assessed.

As deemed necessary, the SSHO/Site Superintendent and/or Competent Person for the activity may impose other prohibitions to ensure the safe conduct of operations. The prohibitions presented below are strictly forbidden at any time during any on-site operation, with violation of these possibly resulting in termination of employment.

- Horseplay or fighting;
- Use of alcohol prior to the mobilization to the site each day, while on-site, and until demobilization from the site each workday;

- Illegal use of drugs;
- Smoking in a work zone or in areas other than authorized designated smoking areas;
- Starting/maintaining an open flame of any kind;
- Use of equipment that has not been inspected and deemed safe for operation;
- Entry into a work site without prior approval of the SSHO/Site Superintendent;
- Working without the proper PPE;
- Initiation of work without the presence of a buddy; or
- Failure to report an incident that results in personal injury or property damage.

Disciplinary Actions: If an ERT Safety and Health Program, APP/SSHP, or AHA nonconformance occurs, appropriate disciplinary action will be taken. In all cases where a potential violation has been reported, the SSHO/Site Superintendent, in conjunction with the ERT PM, will conduct an investigation to validate the report and to determine the severity of the violation. Violations will be divided into two categories: major and minor. An example of a minor violation is reporting to work or conducting work without the proper PPE. A major violation is any violation of the APP/SSHP that could have resulted, or did result, in an accident involving personal injury to self or others, or property damage. A major violation will be investigated by the ERT SHM or their designee. **Table 1** below outlines the disciplinary actions and procedures to be followed if a noncompliance issue results from personnel actions.

Table 1. Disciplinary Action for Minor and Major Violations

Minor Violation Issues	
First Offense	A verbal warning will be given to the individual; the offense to be noted in individual's file and supervisor's project file; a discussion with the individual's supervisor or Field Team Leader will be conducted.
Second Offense	Written reprimand by the Division Manager will be entered in individual's file; discussion with individual and individual's supervisor.
Third Offense	Potential termination of employment as determined by the President of ERT.
Major Violation Issues	
Any Offense	Minimum penalty for a major violation will consist of a written reprimand being entered in individual's file and a discussion between the individual and the Division Manager. Depending upon the severity of the violation, the SSHO/Site Superintendent may temporarily dismiss the individual from the job site pending further investigation of the offense. Major violations will immediately be reported to the ERT PM and ERT Division SHM. Upon completion of a full investigation, the individual's employment may be terminated, if deemed appropriate by the President of ERT.

d. Procedures for Holding Managers and Supervisors Accountable for Safety

All ERT employees and subcontractors are responsible for conformance to safety procedures and policies on the job site. Supervisors and managers are responsible for ensuring that the proper safety procedures are documented in safety plans; that all staff have reviewed and acknowledged their understanding of the procedures; and ensuring that the procedures are followed. Should an incident occur resulting in illness or injury of an employee, an internal audit will be performed to assess whether the proper plans were in place and written procedures were followed. Should it be determined that managers or supervisors allowed hazardous work to be performed without proper procedures or worker acknowledgement of the written procedures, corrective actions will be implemented including disciplinary action when necessary.

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8. MISHAP REPORTING

a. Exposure Data (Man-Hours Worked)

The ERT PM will coordinate with the SSHO/Site Superintendent to ensure the acquisition of all exposure data. This will include the number of man-hours expended toward the contract and any reportable accidents that occurred during the project. This information will be relayed on a monthly basis to the CELRL Contracting Officer's Representative (COR)/Technical Manager (TM) and/or CELRL PM in a monthly report from the ERT PM.

b. Mishap Investigations, Reports, and Logs

Accidents/incidents will be reported to the CELRL COR/TM and/or CELRL PM. Initial reporting will be made via telephone to the ERT PM who will then report the incident to the CELRL COR/TM and/or CELRL PM, as soon as possible, but no later than 24 hours after the accident/incident. ERT will then initiate an accident investigation, with assistance from the on-site personnel, and will initiate the completion of the appropriate accident reporting forms, to include the Accident Report Forms (Engineer [ENG] Form 3394 and OSHA 301 forms), presented in **Enclosure B** to the SSHP.

The ERT PM and Division SHM will review the initial data presented on the accident report forms and will ensure they are complete and accurate prior to their submission to the CELRL COR/TM and/or CELRL PM. The initial draft of ENG 3394 and OSHA 301 forms, with supporting documentation and appropriate corrective actions will be submitted to the CELRL COR/TM and/or CELRL PM within five working days after the date the incident occurred. Corrective actions will be implemented as soon as reasonably possible.

c. Immediate Notification of Major Mishaps

In the event of an accident that requires off-site treatment, or any incident that could bring adverse attention or publicity to the U.S. Army, the ERT PM will notify Range Control immediately, followed second by notifying the CELRL COR/TM and/or CELRL PM. A draft copy of the ERT Incident Reporting Form will be completed and forwarded by ERT within 24 hours of the incident.

OSHA will also be notified of all major accidents, which would include: all work-related fatalities, all work-related inpatient hospitalizations of **one** or more employees, all work-related amputations, and all work-related losses of an eye. Any fatality having occurred within 30 days of a work-related incident is to be reported to OSHA within 8 hours of learning about it. For inpatient hospitalization, amputation, or eye loss happening within 24 hours of a work-related incident, the incident must be reported to OSHA within 24 hours of learning about it. The incident will be reported via the 24-hour OSHA hotline (1-800-321-OSHA [6742]) and an OSHA 301 form will be completed. Information to be reported to OSHA should include:

- Establishment name
- Location of the work-related incident
- Time of the work-related incident
- Type of reportable event (i.e., fatality, inpatient hospitalization, amputation or loss of an eye)
- Number of employees who suffered the event

- Names of the employees who suffered the event
- Contact person and his or her phone number
- Brief description of the work-related incident

Additionally, ENG 3394 forms will be completed and forwarded to USACE within 24 hours. Types of accidents that would be considered major (Class A and Class B) under DoD guidance (DoD, 2009) would include:

Class A:
- A fatal injury;
- A permanent total disability; and
- Property damage of $2,000,000 or more.

Class B:
- A permanent partial disability;
- The hospitalization of three or more people resulting from a single occurrence; and/or
- Property damage of $500,000 or more (but less than $2,000,000).

Minor accidents (Class C and Class D) (USACE, 2010) will also be reported via ENG 3394 forms to USACE within 24 hours, and these would include:

Class C:
- A non-fatal injury or occupational illness;
- One or more days away from work or training beyond the day or shift on which it occurred, or disability at any time (not meeting definition of Class A or B); and/or
- Property damage of $50,000 or more (but less than $500,000).

Class D:
- A non-fatal injury or occupational illness;
- Restricted work, transfer to another job, medical treatment greater than First Aid, needle stick injuries and cuts from sharps that are contaminated from another person's blood or other potentially infectious material, medical removal under medical surveillance requirements of an OSHA standard, occupational hearing loss that meets OSHA recordability criteria, or a work-related tuberculosis case, and/or
- Property damage of $5,000 or more (but less than $50,000).

ERT's accident experience goal for this project is to perform this project without accident or defect. To facilitate this goal, the ERT will implement the requirements of this APP, the SSHP, and the ERT Safety and Health Program. Additionally, the ERT Team will make all project and site management personnel aware of this goal and will empower all site personnel with stop work authority for known or potential uncontrolled safety hazards.

9. PLANS (PROGRAMS AND PROCEDURES) REQUIRED BY EM 385-1-1

The majority of applicable plans, programs, and procedures are addressed in the SSHP. Where a specific element does not apply to this project, a statement to that effect has been made.

a. Fatigue Management Plan (01.A.20)

Work hours for any one individual on-site will not exceed 10 hours per day for more than four consecutive days, or 50 hours within a seven-day work week, or 12 hours per day for more than three consecutive days; therefore, a fatigue management plan is not required.

b. Emergency Response Plans

1. Procedures and Tests (01.E.01)

During each morning's Daily Safety Briefing, the SSHO/Site Superintendent will identify emergency alarms, evacuation routes, and procedures to be followed in the event of an emergency (i.e., fire, lightning storms, etc.). Additionally, internal emergency notification procedures, hospital routes, external emergency support contact information, and notification procedures will be reviewed each day.

An initial review of the Emergency Action Plan will be conducted prior to the start of work. Periodic exercises may also be conducted to ensure that site personnel remain aware of immediate actions to be taken in the event of an emergency.

2. Spill Plans (01.E.01, 06.A.02)

The Government representatives on-site will be responsible for sample collection; however, ERT representatives should be aware that sampling events include the use of sample preservative and decontamination fluids. It is possible these fluids may spill during these events. Every effort will be made to ensure that no spills occur; and if a spill does occur it will be contained and cleaned immediately. All sampling materials will be carefully handled and placed in appropriate sample ware, then inside sealed plastic bags and labeled clearly. PPE, including latex or nitrile gloves and protective eyewear, must be used while collecting environmental samples and performing equipment decontamination.

There is a possibility of fuel spills during equipment/vehicle refueling. Any spills no matter the size must be reported to Range Control. Fuel spills represent both a potential respiratory hazard and a potential fire hazard. Spill kits will be maintained on-site. Fuel will be stored only in Department of Transportation compliant containers. A fuel spill kit or absorbent will be on hand when fueling of vehicles and/or equipment is taking place. In the event a spill occurs during refueling, every effort will be made to contain the spill and clean it up immediately. The SDSs for equipment/vehicle fuel (gasoline and/or diesel) is included in **Enclosure D** of the SSHP and will be available on-site. Spoils resulting from any spill will be disposed of in accordance with the requirements for that item. No cigarette smoking or open flames will be allowed while on-site.

Currently, it is not anticipated that any hazardous materials will be used for this project; however, if any hazardous materials are required on-site they will be properly labeled, the site workers will be made aware of the specific hazards, this APP/SSHP will be amended to reflect the additional hazard, and the appropriate SDSs will be added to **Enclosure D** of the SSHP.

Any spill of petroleum products over 25 gallons, or a spill of any amount into a body of water, must be reported to the OHARNG Environmental Office immediately. All other spills will be reported in writing on the OHARNG Spill Report Form (included within **Attachment 4** of this APP) to the OHARNG within 24 hours.

3. Firefighting Plan (01.E.01, 19.A)

All site personnel will be trained in the proper use of site fire extinguishers. In the event of a small fire that can easily be controlled with a fire extinguisher, site personnel may extinguish the fire. If the fire continues after initial firefighting efforts by site personnel, then the situation will be immediately recognized as an emergency. The SSHO/Site Superintendent will initiate the process to evacuate to the initial applicable rally point, then to the evacuation point, and then off-site. The SSHO/Site Superintendent will:

- Account for all employees at the rally point;
- Notify local emergency services. A list of emergency contact numbers is provided in **Table 2** of this APP and in the back cover of the SSHP. The emergency contact phone numbers, map to local hospital, and SDSs will also be kept on-site in the front cab of each field vehicle;
- Notify the ERT PM.

Site personnel will only perform the rescue and medical duties that each is trained and qualified to perform. All other necessary rescue and medical duties will be performed by qualified emergency personnel. Other than small fires, local emergency response services will be notified to handle the emergency. The SSHO/Site Superintendent will take measures to reduce injury and illness, primarily by evacuating personnel as quickly as possible, and then notify the ERT PM. Cleanup after such events may require specialized services. Work will not resume until the SSHO/Site Superintendent declares the incident closed.

Procedures or tests associated with fire emergency response activities will involve a rehearsal of the Emergency Action Plan prior to the start of site activities. During this briefing, the route to and location of the evacuation point will be determined by the SSHO/Site Superintendent and discussed with each field staff member.

4. Posting of Emergency Telephone Numbers (01.E.05)

Emergency telephone numbers are included below in **Table 2** and on the back cover of the SSHP. As previously noted, for this project, the ERT field vehicles will function as mobile field offices, and each vehicle will contain a full copy of the APP/SSHP.

Table 2. Emergency Contact Phone Numbers

Service/Contact	Agency/Position	Telephone No.
Range Control	Emergency	(614) 336-6041
University Hospitals Portage Medical Center	Emergency Room	(330) 297-2850
University Hospitals Portage Medical Center	Non-emergency medical	(330) 297-0811 (non-emergency)
Spill Response	Chemical Transportation	(800) 424-9300

Table 2. Emergency Contact Phone Numbers		
Service/Contact	**Agency/Position**	**Telephone No.**
	Emergency Center (CHEMTREC)	
Project Coordinator	OEPA	(517) 284-5084
Poison Control	Poison Control Center	(800) 222-1222
City of Twinsburg Animal Control	Animal Control	(330) 425-1234
Mark Leeper	ARNG Restoration Program Manager	(703) 607-7955
Kevin Sedlak	ARNG	(614) 336-2053
Katie Tait	OHARNG	(614) 336-6136
Craig Coombs	CELRL PM	(502) 315-6324
Matt Burg	CELRL Chief of Safety	(502) 315-7061
Nat Peters	CELRL COR	(502) 315-2624
Kevin Mieczkowski	CELRL TM	(502) 315-7447
Michael Barsa	ERT Division SHM	(301) 323-1447 (o) (410) 703-6213 (c)
Jennifer Harlan	ERT Division Manager	(301) 323-1394 (o)
Sean Carney	ERT PM	(607) 259-7017) (c)
J.T. Nolan	ERT SSHO/Site Superintendent/OSHA Competent Person for Overall Site Health and Safety	(301) 412-7301
Robert Koroncai	Alternate ERT SSHO/OSHA Competent Person for Overall Site Health and Safety	(301) 323-1445 (o) (267) 481-5567 (c)

5. Man Overboard/Abandon Ship (19.A.04)

This project does not involve any marine activities; therefore, this plan is not required.

6. Medical Support (03.A.02, 03.D)

The Emergency Action Plan is included in Section 11.0 of the SSHP. This includes details regarding on-site medical support, off-site medical support, and a map and turn-by-turn directions to the nearest hospital.

c. Prevention of Alcohol and Drug Abuse (Defense Federal Acquisition Regulation Supplement Subpart 252.223-7004) (01.C.02)

Introduction: The Drug-Free Workplace Act of 1988 set as a goal the elimination of the effects of illegal drugs in the workplace. Because of the inherently hazardous nature of the work performed by ERT personnel, the importance of creating and maintaining a safe, drug-free working environment is paramount. The performance of every employee must, at all times, support the company's mission to conduct site operations with a high level of productivity, reliability, judgment, and safety.

The management of ERT is thoroughly committed to providing a drug-free workplace for all employees. Drug and/or alcohol use and abuse are incompatible with ERT's high standards of performance, safety, and quality. As a term of employment, all employees agree to refrain from the use, distribution, possession, manufacture, or dispensing of a controlled substance, and drug and/or alcohol abuse. Violation of this policy may result in administrative action to include termination of employment.

Substance Use and Abuse Policy: Employee drug or substance use or abuse testing/screening conducted by ERT in support of this policy will be conducted at no expense to the employee, and, except for drug/substance use testing conducted for pre-employment, employees will receive reasonable compensation for the time required for participation in any drug or substance testing/screening.

As a matter of policy, ERT will strictly implement and enforce the policies listed below:

- No employee will report for work, or will work, impaired by any unauthorized or controlled substance;
- No employee will use any alcohol or a controlled substance at the site;
- Applicants for employment are subject to substance abuse screening as part of their baseline or pre-assignment physical examinations;
- Substance use or abuse screening may be conducted randomly and/or when an employee is involved in either a job-related accident or incident; and
- The SSHO/Site Superintendent has full authority to prevent/halt the work of an individual suspected to be impaired. If this occurs, the ERT PM will be notified immediately.

Prescription Medications: ERT project personnel may possess and use prescription medications and "over-the-counter" medications provided that all of the following apply:

- The prescription medication has been prescribed by an authorized medical practitioner for the current use (within the past 12 months) of the employee, and the medication is in its original container with a valid pharmacy label;
- The employee does not consume the prescribed, or over-the-counter, medication in quantities greater than, or more frequently than that directed on the label;
- Employees in possession of prescribed medications will not allow any other person to consume any amount of their prescribed medication; and
- In the event that the prescribed medication could cause adverse side effects, or where the medication indicates warnings relevant to side effects affecting the operation of equipment or machinery, the employee will inform the SSHO/Site Superintendent prior to engaging in project operations that are prohibited by the medication warnings.

While the on-site use of prescription and over-the-counter medications is authorized, under the requirements listed above, ERT reserves the right to have a licensed physician determine if the employee's use of the medication could adversely affect the individual or could increase the potential for injury or illness to the employee or other site personnel. If consumption of the medication could lead to adverse safety or health effects, the SSHO/Site Superintendent, and/or the ERT Division SHM may, on the advice of the licensed physician, limit or suspend the employee's work activities.

Suspicion Inspection and Testing: For the purposes of ensuring compliance with the prohibition against the unauthorized possession of controlled substances, employees may be subject to random and reasonable suspicion inspections and testing. An employee's company clothing, locker, closet, work area, desk files, company motor vehicle, and similar areas are subject to inspection. With the exception of ERT owned/rented/leased property, no person or property search, urine drug test, or breathalyzer test will be conducted without the employee’s consent. Refusal to submit to a legal inspection, or request for testing, may result in employee removal from site activities until further inspection or testing can determine the potential for prohibited drug or substance use or abuse.

d. Site Sanitation Plan (02)

ERT requires that all site personnel practice good housekeeping in both common areas and at the work site. The SSHO/Site Superintendent will conduct periodic inspections to ensure that good housekeeping practices are be followed by site personnel.

Field vehicles will be available to transport workers to nearby toilet facilities (as the site is located in a public, urban area, public restrooms of local businesses can be used, i.e., McDonald’s located just south of the site on Route 31 at the State Street intersection). Additionally, non-toxic hand soap or similar cleansing agents will be available for use within the designated support area and in each field vehicle. Site personnel will also be made aware that certain cleansing agents, such as hand sanitizers, may contain iodopropynyl butylcarbamate, which may cause allergic reactions to the skin. All work areas will be kept clean, and waste receptacles will be available. Drinking water will be readily available to all workers, and will be provided via bottles. No potable water supplies will be mixed with non-potable water supplies.

e. Medical Support Plan (03.A.01; 03.A.06; 03.D)

The Emergency Action Plan is included in Section 11.0 of the SSHP. This includes details regarding on-site medical support, off-site medical support, and a map and turn-by-turn directions to the nearest hospital.

f. Bloodborne Pathogen Plan (03.A.05)

As this effort will involve the use of staff trained in Frist Aid/CPR, although unlikely, there exists a potential for exposure to bloodborne pathogens (BBPs), if the administration of First Aid is necessary. Information related to BBPs is included in Section 11.0 of the SSHP.

g. Exposure Control Plan (03.A.05)

The Exposure Control Plan to minimize exposure to BBPs is included in Section 11.0 of the SSHP. Work practice controls and engineering controls are presented to minimize exposure.

h. Site Layout Plan (04.A)

The general site map showing access routes to the each of the investigation areas of the site and the evacuation route are shown in **Figure 2**. No other temporary construction buildings or structures will be required; therefore, the following items will not be required:

- Fencing;
- Construction of access routes;
- Spacing requirements of 09.A.19;

- Power distribution requirements; and
- Temporary ramp, trestle, scaffold, and platform approval.

i. Access and Haul Road Plan (04.B)

This plan is not required because no access and/or haul road construction is planned during this project.

j. Hearing Conservation Program (05.C)

If workers are subjected to noise exceeding an 8-hour (hr) time-weighted average sound level of 85 decibels on the A-weighted scale (dBA), hearing protection will be provided with an appropriate noise reduction rating to comply with 29 Code of Federal Regulations (CFR) 1910.95 and reduce noise levels to or below 85 dBA. If ear insert devices are used, the SSHO/Site Superintendent will ensure they are properly fitted and inserted. If ear muffs are used, the SSHO/Site Superintendent will ensure proper coverage of the ear by the muff. Hearing protection will be utilized during all heavy machinery operation.

k. Respiratory Protection Plan (05.G)

Due to the low probability of exposure, no requirement for respiratory protection is anticipated for this project. Any changes necessary to work activities based on monitoring would be documented via amendment to the APP/SSHP.

l. Health Hazard Control Program (06.A)

The Health Hazard Control Program is presented in Section 2.0 of the SSHP.

h. Written Hazard Communications Program (06.B.01)

The Written Hazard Communications Program is presented in Section 8.3 of the SSHP.

m. Process Safety Management Plan (06.B.04)

This plan is not required. No processes involving highly hazardous chemicals are anticipated for this project.

n. Lead Abatement Plan (06.C and Specifications)

This plan is not required. No work with lead is anticipated for this project.

o. Asbestos Abatement Plan (06.C and Specifications)

This plan is not required. No work with asbestos is anticipated for this project.

p. Radiation Safety Program (06.F)

This plan is not required. No contact with radioactive materials is anticipated for this project.

q. Abrasive Blasting Plan (06.I)

This plan is not required. No abrasive blasting is planned during this project.

r. Heat/Cold Stress Monitoring Plan (06.J)

The Heat/Cold Stress Monitoring Plan is presented as Section 8.0 in the SSHP.

s. Indoor Air Quality Management Plan (06.L)

This plan is not required. No indoor field work is planned during this project.

t. Mold Remediation Plan (06.L.04)

This plan is not required. Because no indoor field work is planned during this project, it is not expected that mold will be encountered.

u. Chromium (VI) Exposure Evaluation (06.M)

This plan is not required. No operations resulting in the generation of chromium (VI), i.e., welding, painting, paint removal, heating stainless steel, and/or handling anti-corrosive substances.

v. Crystalline Silica Assessment (06.N)

This plan is not required. No crystalline silica exposure is anticipated during this project.

w. Lighting Evaluation (07.A)

This plan is not required. All work will be performed outside during daylight hours.

x. Lighting Plan for Night Operations (07.A.09)

This plan is not required. No work will be performed during night hours.

y. Traffic Control Plan (08.C.05)

Due to proximity of a limited number of stockpiles adjacent to Paris Windham Road, field personnel may also be exposed to hazards associated working near moving vehicles during the project. To control these hazards, the following safety requirements will be strictly enforced:

- The work area will be cordoned off with safety cones, and a sign will be placed, per the example (or equivalent) provided in Enclosure B of the SSHP;
- In the unlikely event that road closure is necessary, ERT will work with Fort Benning personnel;
- Class II Safety Vests (fluorescent yellow-green) will be worn by site personnel when the following applies:
  - Exposure to vehicular traffic at speeds up to 45 mph;
  - Limited visibility of workers due to mobile heavy equipment operations, vehicles, load handling, or other hazardous activities;
  - Reduced visibility conditions due to inclement weather; and/or
  - No protective barriers when near vehicular traffic.

z. Fire Prevention Plan (09.A.01)

Explosion and fire hazards may be present at the work site due to the unexpected ignition of chemicals or fuels, the sudden release of materials under pressure, or due to the possibility of careless, unauthorized smoking in work areas. Site operations will be conducted in accordance with local fire codes and regulations.

Site personnel will be advised of all potential ignition sources and be reminded to practice good housekeeping and minimize fuel sources. Personnel will be observant of site conditions and ensure that no operations are conducted that could result in the ignition of a fire. Smoking will not be permitted on-site. The SSHO/Site Superintendent will ensure that fire safety practices are adhered to by site personnel.

Each field vehicle will be equipped with a fire extinguisher. Site personnel will be trained in the proper use of fire suppression equipment. Fires beyond the incipient stage, not able to be controlled with a fire extinguisher, will be handled by professionals of the local fire department. In the event of a fire that cannot be controlled by a fire extinguisher the Emergency Action Plan will be implemented. The SSHO/Site Superintendent will initiate making appropriate notifications. Per the Emergency Action Plan, site workers will immediately report to the primary rally point unless this location has become dangerous due to the existing emergency, in which case, personnel will proceed to a secondary rally point, to be determined in the field by the SSHO/Site Superintendent. Site personnel will always be on the alert for unexpected events, and be prepared to act in these emergencies.

aa. Wild Land Fire Management Plan (09.L)

This plan is not required as this project is not being executed in wild lands.

bb. Arc Flash Hazard Analysis (11.B)

This plan is not required as no work will be completed near energized parts.

cc. Assured Equipment Grounding Control Program (AEGCP) (11.D.05, App D)

It is not anticipated that temporary electrical power will be necessary; however, if needed, all temporary electrical receptacle outlets will be equipped with a ground-fault circuit interrupter.

dd. Hazardous Energy Control Plan (12.A.01)

This plan is not required as no hazardous energy will be involved for this project.

ee. Standard Pre-Lift Plan (LHE) (16.H)

This plan is not required. No lifts are planned during this project.

ff. Critical Lift Plan - LHE (16.H)

This plan is not required. No critical lifts are planned during this project.

gg. Naval Architectural Analysis (16.L)

This plan is not required as this project will not involve the use of floating cranes/derricks, crane barges, and/or auxiliary shipboard-mounted cranes.

hh. Contingency Plan for Severe Weather (19.A.03)

The contingency plan for severe weather is presented in Section 2.1.7 of the SSHP.

ii. Man Overboard/Abandon Ship (19.A.04)

This project does not involve any marine activities; therefore, this plan is not required.

jj. Float Plan (19.F.04)

This plan is not required. No marine work is planned for this project.

kk. Fall Protection Plan (21.D)

This plan is not required. No elevated work is planned for this project.

ll. Demolition/Renovation Plan (to include engineering survey) (23.A.02)

This plan is not required. No building and/or structure demolition is anticipated for this project.

mm. Rope Access Program (24.H.02)

This plan is not required. No climbing will be performed during the course of this project.

nn. Excavation/Trenching Plan (25.A.01)

This plan is not required. No excavation and/or trenching is anticipated for this project.

oo. Underground Construction Fire Prevention and Protection Plan (26.D.01)

This plan is not required. No underground construction is anticipated for this project.

pp. Compressed Air Plan (26.I.01)

This plan is not required. No compressed air operations are anticipated for this project.

qq. Formwork and Shoring Erection and Removal Plans (27.C)

This plan is not required. No formwork or shoring erection is planned during this project.

rr. Pre-Cast Concrete Plan (27.D)

This plan is not required. No pre-cast concrete will be used during this project.

ss. Lift Slab Plans (27.E)

This plan is not required. No slab work that would require lifting is planned during this project.

uu. Steel Erection Plan (28.B)

This plan is not required. No steel erection is anticipated for this project.

vv. Explosives Safety Site Plan (ESSP) (29.A)

This plan is not required. No work with explosives is anticipated for this project.

ww. Blasting Plan (29.A.01)

This plan is not required. No commercial or industrial blasting is anticipated for this project.

xx. Underwater Dive Operations Plan (30.A.14, 16)

This plan is not required. No diving operations are anticipated for this project.

yy. Tree Felling/Maintenance Program (31.A)

This plan is not required. No trees will be removed or maintained during the course of this project (only small brush will be cleared as necessary).

zz. Aircraft/Airfield Construction Safety & Phasing Plan (32.A.02)

This plan is not required. No operations will occur in the vicinity of an airfield for this project.

aaa. Site Safety and Health Plan for Hazardous, Toxic and Radioactive Waste (HTRW) (33.B)

The SSHP is included as **Attachment 2** of this APP.

bbb. Confined Space Entry Program (34.A.06, 07)

This plan is not required. No confined spaces will be entered during this project.

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10. RISK MANAGEMENT PROCESS

An analysis of safety and health hazards has been performed and is included in the AHA found in **Attachment 3** of this APP. Risk levels of each hazard and risk mitigation controls are included as part of the AHA.

Prior to initiating any site work, all staff are required to read the APP, SSHP, and AHAs, and sign the Review Record located within the SSHP acknowledging that they have read, understand, and accept responsibility for any activities they perform as part of this project. Pre-operational activity site-specific safety briefings will be performed prior to the initiation of field activities, with sign-off of this briefing by all staff. Additionally, safety briefings will be conducted by the SSHO/Site Superintendent/Competent Person for the activity on a daily basis. Safety briefings will also be conducted for temporary site workers and/or visitors entering the site. Personnel will sign an attendance roster, via the Daily Safety Briefing form in **Enclosure C** of the SSHP, to acknowledge receipt of the briefing.

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11. REFERENCES

DoD, 2009. *Memorandum from Under Secretary of Defense for Secretaries of the Military Departments, Regarding: Revision to Cost Thresholds for Accident Severity Classification,* July.

Leidos, 2015. *Remedial Investigation Report for Soil, Sediment, and Surface Water at RVAAP-50 Atlas Scrap Yard, Former Ravenna Army Ammunition Plant, Portage and Trumbull Counties, Ohio*. June.

U.S. Army Corps of Engineers (USACE), 2014. *Safety and Health Requirements Manual, EM 385-1-1*, 30 November 2014.

USACE, 2010. *USACE Accident Investigation and Reporting, ER 385-1-99*, 15 March 2010.

USACE, 2014. *USACE Safety and Health Requirements –EM 385-1-1.*

USACE, 2016. *Performance Work Statement, FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility, Former Ravenna Army Ammunition Plant Restoration Program, Camp Ravenna, Portage and Trumbull Counties, Ohio*. May.

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FIGURES

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US Army Corps of Engineers®
BUILDING STRONG®

ERT

Legend

━ Site Boundary

▢ Stockpiles

▣ Camp Ravenna Boundary

N
W E
S

0 350 700

Feet

Figure 1
Site Location Map
Atlas Scrap Yard Area of Concern
Former Ravenna
Army Ammunition Plant
Ravenna, Ohio

Revision Date: November 2016

Data Source: RVAPP, ESRI Street Map

Document Path: N:\GIS\Midwest\Ohio\RVAAP\MXD\QAPP\SiteLocation.mxd

US Army Corps of Engineers
BUILDING STRONG

ERT

Legend

★ Rally Point

🟩 Camp Ravenna Boundary

→ Evacuation Route

🟥 Work Site

N
W E
S

0 350 700
Feet

Figure 2
Site Layout Map with Evacuation Route

Atlas Scrap Yard Area of Concern
Former Ravenna Army
Ammunition Plant
Ravenna, Ohio

Revision Date: November 2016

Data Source: RVAPP, ESRI Street Map

Document Path: N:\GIS\Midwest\Ohio\RVAAP\MXD\APP_SSHP\EvacuationRoute.mxd

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ATTACHMENT 1

OSHA 300A Forms, 2013 - 2015

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OSHA's Form 300A (Rev. 01/2004)

Summary of Work-Related Injuries and Illnesses

Year 2015

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no 1218-0176

All establishments covered by Part 1904 must complete this Summary page, even if no injuries or illnesses occurred during the year. Remember to review the Log to verify that the entries are complete

Using the Log, count the individual entries you made for each category. Then write the totals below, making sure you've added the entries from every page of the log. If you had no cases write "0."

Employees former employees, and their representatives have the right to review the OSHA Form 300 in its entirety. They also have limited access to the OSHA Form 301 or its equivalent. See 29 CFR 1904.35, in OSHA's Recordkeeping rule, for further details on the access provisions for these forms.

Number of Cases

Total number of deaths	Total number of cases with days away from work	Total number of cases with job transfer or restriction	Total number of other recordable cases
0	1	0	2
(G)	(H)	(I)	(J)

Number of Days

Total number of days away from work	Total number of days of job transfer or restriction
1	0
(K)	(L)

Injury and Illness Types

Total number of...
(M)

(1) Injury	1	(4) Poisoning	0
(2) Skin Disorder	1	(5) Hearing Loss	0
(3) Respiratory Condition	0	(6) All Other Illnesses	1

Post this Summary page from February 1 to April 30 of the year following the year covered by the form

Establishment information

Your establishment name ERT, Inc

Street 14401 Sweitzer Lane, Suite 300

City Laurel State MD Zip 20707

Industry description (e.g., Manufacture of motor truck trailers)
IT, Earth Science, Geophysics, Environmental, Remote Sensing

Standard Industrial Classification (SIC), if known (e.g., SIC 3715)
951 871 874 873 495

OR North American Industrial Classification (NAICS), if known (e.g., 336212)
__ __ __ __ __ __

Employment information

Annual average number of employees 417

Total hours worked by all employees last year 901,370

Sign here

Knowingly falsifying this document may result in a fine.

I certify that I have examined this document and that to the best of my knowledge the entries are true, accurate, and complete.

Company executive	President Title
301-323-1411 Phone	14-Jan-16 Date

OSHA's Form 300A (Rev. 01/2004)

Summary of Work-Related Injuries and Illnesses

Year 2014

U.S. Department of Labor
Occupational Safety and Health Administration
Form approved OMB no. 1218-0176

All establishments covered by Part 1904 must complete this Summary page, even if no injuries or illnesses occurred during the year. Remember to review the Log to verify that the entries are complete

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Employees former employees, and their representatives have the right to review the OSHA Form 300 in its entirety. They also have limited access to the OSHA Form 301 or its equivalent. See 29 CFR 1904.35, in OSHA's Recordkeeping rule, for further details on the access provisions for these forms.

Number of Cases

Total number of deaths	Total number of cases with days away from work	Total number of cases with job transfer or restriction	Total number of other recordable cases
0	0	0	1
(G)	(H)	(I)	(J)

Number of Days

Total number of days away from work	Total number of days of job transfer or restriction
0	0
(K)	(L)

Injury and Illness Types

Total number of...
(M)

(1) Injury	1	(4) Poisoning	0
(2) Skin Disorder	0	(5) Hearing Loss	0
(3) Respiratory Condition	0	(6) All Other Illnesses	0

Post this Summary page from February 1 to April 30 of the year following the year covered by the form

Public reporting burden for this collection of information is estimated to average 58 minutes per response, including time to review the instruction, search and gather the data needed, and complete and review the collection of information. Persons are not required to respond to the collection of information unless it displays a currently valid OMB control number. If you have any comments about these estimates or any aspects of this data collection, contact: US Department of Labor, OSHA Office of Statistics, Room N-3644, 200 Constitution Ave. NW, Washington, DC 20210. Do not send the completed forms to this office.

Establishment Information

Your establishment name ERT, Inc.

Street 14401 Sweitzer laneFrost Place, Suite 300

City Laurel State MD Zip 20707

Industry description (e.g., Manufacture of motor truck trailers)
IT, Earth Science, Geophysics, Environmental, Remote Sensing

Standard Industrial Classification (SIC), if known (e.g., SIC 3715)
951 871 874 871 179 495

OR North American Industrial Classification (NAICS), if known (e.g., 336212)

Employment Information

Annual average number of employees 404.33

Total hours worked by all employees last year 825,048.46

Sign here

Knowingly falsifying this document may result in a fine.

I certify that I have examined this document and that to the best of my knowledge the entries are true, accurate, and complete.

Peter Li
Company executive

President
Title

301-361-0658
Phone

1/30/2015
Date

OSHA's Form 300A (Rev. 01/2004)
Summary of Work-Related Injuries and Illnesses

Year 2013

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

All establishments covered by Part 1904 must complete this Summary page, even if no injuries or illnesses occurred during the year. Remember to review the Log to verify that the entries are complete.

Using the Log, count the individual entries you made for each category. Then write the totals below, making sure you've added the entries from every page of the log. If you had no cases write "0."

Employees, former employees, and their representatives have the right to review the OSHA Form 300 in its entirety. They also have limited access to the OSHA Form 301 or its equivalent. See 29 CFR 1904.35, in OSHA's Recordkeeping rule, for further details on the access provisions for these forms.

Number of Cases

Total number of deaths	Total number of cases with days away from work	Total number of cases with job transfer or restriction	Total number of other recordable cases
0	0	0	1
(G)	(H)	(I)	(J)

Number of Days

Total number of days away from work	Total number of days of job transfer or restriction
0	0
(K)	(L)

Injury and Illness Types

Total number of...
(M)

(1) Injury	1	(4) Poisoning	0
(2) Skin Disorder	0	(5) Hearing Loss	0
(3) Respiratory Condition	0	(6) All Other Illnesses	0

Post this Summary page from February 1 to April 30 of the year following the year covered by the form

Public reporting burden for this collection of information is estimated to average 58 minutes per response, including time to review the instructions, search and gather the data needed, and complete and review the collection of information. Persons are not required to respond to the collection of information unless it displays a currently valid OMB control number. If you have any comments about these estimates or any aspects of this data collection, contact: US Department of Labor, OSHA Office of Statistics, Room N-3644, 200 Constitution Ave, NW, Washington, DC 20210. Do not send the completed forms to this office.

Establishment information

Your establishment name ERT, Inc.
Street 14401 Sweitzer Lane/Frost Place, Suite 300
City Laurel State MD Zip 20707
Industry description (e.g., Manufacture of motor truck trailers)
IT, Earth Science, Geophysics, Environmental, Remote Sensing
Standard Industrial Classification (SIC), if known (e.g., SIC 3715)
951 871 874 873 179 495
OR North American Industrial Classification (NAICS), if known (e.g., 336212)
___ ___ ___ ___ ___ ___

Employment information

Annual average number of employees 4185
Total hours worked by all employees last year 831207 60

Sign here

Knowingly falsifying this document may result in a fine.

I certify that I have examined this document and that to the best of my knowledge the entries are true, accurate, and complete.

Peter Li
Company executive

President
Title

301-361-0658
Phone

1/10/2014
Date

OSHA's Form 300A (Rev. 01/2004)

Summary of Work-Related Injuries and Illnesses

Year 2012

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

All establishments covered by Part 1904 must complete this Summary page, even if no injuries or illnesses occurred during the year. Remember to review the Log to verify that the entries are complete

Using the Log, count the individual entries you made for each category. Then write the totals below, making sure you've added the entries from every page of the log. If you had no cases write "0."

Employees former employees, and their representatives have the right to review the OSHA Form 300 in its entirety. They also have limited access to the OSHA Form 301 or its equivalent. See 29 CFR 1904.35, in OSHA's Recordkeeping rule, for further details on the access provisions for these forms.

Number of Cases

Total number of deaths	Total number of cases with days away from work	Total number of cases with job transfer or restriction	Total number of other recordable cases
0	0	0	1
(G)	(H)	(I)	(J)

Number of Days

Total number of days away from work	Total number of days of job transfer or restriction
0	0
(K)	(L)

Injury and Illness Types

Total number of...
(M)

(1) Injury	1	(4) Poisoning	0
(2) Skin Disorder	0	(5) Hearing Loss	0
(3) Respiratory Condition	0	(6) All Other Illnesses	0

Post this Summary page from February 1 to April 30 of the year following the year covered by the form

Public reporting burden for this collection of information is estimated to average 58 minutes per response, including time to review the instruction, search and gather the data needed, and complete and review the collection of information. Persons are not required to respond to the collection of information unless it displays a currently valid OMB control number. If you have any comments about these estimates or any aspects of this data collection, contact: US Department of Labor, OSHA Office of Statistics, Room N-3644, 200 Constitution Ave, NW, Washington, DC 20210. Do not send the completed forms to this office.

Establishment information

Your establishment name ERT, Inc

Street 6100 Frost Place, Suite A

City Laurel State MD Zip 20707

Industry description (e.g., Manufacture of motor truck trailers)
IT, Earth Science, Geophysics, Environmental, Remote Sensing

Standard Industrial Classification (SIC), if known (e.g., SIC 3715)
951 871 874 873 179 495

OR North American Industrial Classification (NAICS), if known (e.g., 336212)

Employment information

Annual average number of employees 487

Total hours worked by all employees last year 857,321.40

Sign here

Knowingly falsifying this document may result in a fine.

I certify that I have examined this document and that to the best of my knowledge the entries are true, accurate, and complete.

Peter Li
Company executive

President
Title

301-361-0658
Phone

1/31/2012
Date

OSHA's Form 300A (Rev. 01/2004)
Summary of Work-Related Injuries and Illnesses

Year 2011

U.S. Department of Labor
Occupational Safety and Health Administration

Form approved OMB no. 1218-0176

All establishments covered by Part 1904 must complete this Summary page, even if no injuries or illnesses occurred during the year. Remember to review the Log to verify that the entries are complete

Using the Log, count the individual entries you made for each category. Then write the totals below, making sure you've added the entries from every page of the log. If you had no cases write "0."

Employees former employees, and their representatives have the right to review the OSHA Form 300 in its entirety. They also have limited access to the OSHA Form 301 or its equivalent. See 29 CFR 1904.35, in OSHA's Recordkeeping rule, for further details on the access provisions for these forms.

Number of Cases

Total number of deaths	Total number of cases with days away from work	Total number of cases with job transfer or restriction	Total number of other recordable cases
0	2	0	3
(G)	(H)	(I)	(J)

Number of Days

Total number of days away from work	Total number of days of job transfer or restriction
36	0
(K)	(L)

Injury and Illness Types

Total number of...
(M)

(1) Injury	2	(4) Poisoning	0
(2) Skin Disorder	3	(5) Hearing Loss	0
(3) Respiratory Condition	0	(6) All Other Illnesses	0

Post this Summary page from February 1 to April 30 of the year following the year covered by the form

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Establishment information

Your establishment name ERT, Inc

Street 6100 Frost Place, Suite A

City Laurel State MD Zip 20707

Industry description (e.g., Manufacture of motor truck trailers)
IT, Earth Science, Geophysics, Environmental, Remote Sensing

Standard Industrial Classification (SIC), if known (e.g., SIC 3715)
951 871 874 873 179 495

OR North American Industrial Classification (NAICS), if known (e.g., 336212)

Employment information

Annual average number of employees 481

Total hours worked by all employees last year 959,702.21

Sign here

Knowingly falsifying this document may result in a fine.

I certify that I have examined this document and that to the best of my knowledge the entries are true, accurate, and complete.

Peter Li	President
Company executive	Title
301-361-0658	1/31/2012
Phone	Date

ATTACHMENT 2

Site Safety and Health Plan

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SITE SAFETY AND HEALTH PLAN
(ATTACHMENT 2 TO THE ACCIDENT PREVENTION PLAN)

FY16 RECYCLING OF MATERIALS AT THE ATLAS SCRAP YARD AREA OF CONCERN AND SETUP OF TEMPORARY STORAGE FACILITY
FORMER RAVENNA ARMY AMMUNITION PLANT RESTORATION PROGRAM

Former Ravenna Army Ammunition Plant
Portage and Trumbull Counties, Ohio

December 20, 2016

Contract No.: W912QR-12-D-0011
Delivery Order 0017

December 2016

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CERTIFICATION

This Site-Specific Safety and Health Plan (SSHP) has been prepared by a Certified Safety Professional (CSP).

Prepared by:

By signing the SSHP, the ERT Division SHM certifies that J.T. Nolan has completed the required occupational safety and health courses and is qualified, by both training and experience to serve as the Site Safety and Health Officer (SSHO), as well as the "Occupational Safety and Health Administration (OSHA) Competent Person for overall site health and safety," for each phase of field work associated with the FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility at the Former Ravenna Army Ammunition Plant, Camp Ravenna, Portage and Trumbull Counties, Ohio.

11/15/2016

Michael Barsa, CSP
Board of Certified Safety Professionals No. 24437
301-323-1447
ERT, Inc. (ERT) Division Safety and Health Manager
(SHM)

Date

Plan Approval:

11/15/2016

Sean Carney, PMP
Project Manager/Senior Technical Reviewer (ERT)

Date

9/22/2016

Jennifer Harlan, PMP
Division Manager (ERT)

Date

SITE SAFETY AND HEALTH PLAN REVIEW RECORD

Site Name: Former Ravenna Army Ammunition Plant (RVAAP)

Work Location Address: The Atlas Scrap Yard Area of Concern (AOC), formerly known as the construction camp, is located on approximately 73 acres in the central portion of Camp Ravenna south of Newton Falls Road and west of Paris Windham Road. There is no fence around the AOC as a perimeter boundary, but the AOC is bound by Newton Falls Road to the north and Paris Windham Road to the east.

I have read, understood, and agree to abide by the information set forth in this SSHP and discussed in the initial Daily Safety Briefing.

NAME	SIGNATURE	DATE
Name	Signature	Date
Name	Signature	Date
Name	Signature	Date
Name	Signature	Date
Name	Signature	Date
Name	Signature	Date
Name	Signature	Date
Name	Signature	Date
Name	Signature	Date

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ACRONYMS AND ABBREVIATIONS

ACGIH	American Conference of Governmental Industrial Hygienists
AHA	Activity Hazard Analysis
APP	Accident Prevention Plan
AR	Administrative Record
ARNG	U.S. Army National Guard
BBP	bloodborne pathogens
CCOHS	Canadian Centre for Occupational Health and Safety
CDC	Centers for Disease Control
CELRL	U.S. Army Corps of Engineers, Louisville District
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CHEMTREC	Chemical Transportation Emergency Center
COC	constituents of concern
COR	Contracting Officer’s Representative
CPR	cardiopulmonary resuscitation
CSP	Certified Safety Professional
dBA	decibels (A-weighted)
EM	engineer manual
ENG	engineer form
ERT	ERT, Inc.
HAZCOM	Hazard Communication
HAZWOPER	Hazardous Waste Operations and Emergency Response
hr	hour
LDA	Lyme Disease Association
ODH	Ohio Department of Health
ODNR	Ohio Department of Natural Resources
OEL	Occupational Exposure Limit
OHARNG	Ohio Army National Guard
OSHA	Occupational Safety and Health Administration
PM	Project Manager
PMP	Project Management Professional
PPE	personal protective equipment
ppm	parts per million
REIMS	Ravenna Environmental Information Management System
RMSF	Rocky Mountain spotted fever
RVAAP	Former Ravenna Army Ammunition Plant
RVAAP-50	Atlas Scrap Yard, Former Ravenna Army Ammunition Plant
SDS	Safety Data Sheet
SHM	Safety and Health Manager
SSHO	Site Safety and Health Officer
SSHP	Site Safety and Health Plan
TLV	threshold limit value
TM	Technical Manager
USACE	U.S. Army Corps of Engineers
USEPA	U.S. Environmental Protection Agency

USP&FO	Base Realignment and Closure Division to the U.S. Property and Fiscal Officer
VOC	volatile organic compound
WBGT	wet bulb globe temperature
WWII	World War II
°F	degrees Fahrenheit

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1.0 SITE DESCRIPTION AND CONTAMINATION CHARACTERIZATION

The goal of this project is to preform recycling of stockpiled material at the Atlas Scrap Yard (RVAAP-50) Area of Concern (AOC) and setup temporary storage facility for a new information repository at the Former Ravenna Army Ammunition Plant (RVAAP) Camp Ravenna Joint Military Training Center, Portage and Trumbull Counties, Ohio.

This SSHP is **Attachment 2** the Accident Prevention Plan (APP) for the Former Ravenna Army Ammunition Plant (RVAAP) project being conducted by ERT, Inc. (ERT) under the U.S. Army Corps of Engineers, Louisville District (CELRL). This SSHP covers the implementation of field activities described in contract W912QR-12-D-0011, Delivery Order 0017 at RVAAP in Portage and Trumble Counties, OH. This SSHP addresses the required components from U.S. Army Corps of Engineers (USACE) Engineer Manual (EM) 385-1-1 (USACE, 2014).

The major phases of work involved in this project include:

- Site visits and inspections;
- Mobilize/demobilize;
- Site preparation;
- Complete the loading, transportation, and recycling/disposal of stockpiled railroad ties, concrete, and telephone poles located at the Atlas Scrap Yard AOC;
- Install a temporary storage facility suitable for temperature controlled storage of former RVAAP administrative records. Re-locate records, documents, maps, shelving, and map cases from three current locations to the temporary storage facility;
- Waste management; and
- Restoration of disturbed areas.

Physical, chemical, and biological hazards are associated with these field activities and are discussed in Section 2.0.

1.1 Site Description

The former RVAAP was utilized as a load, assemble, and pack facility for munitions. At the onset of World War II (WWII), the RVAAP was built to produce large-caliber artillery projectiles and bombs. Although RVAAP downsized after WWII, plant production lines were reactivated during the Korean War and the Vietnam conflicts. Additionally, the plant conducted nearly continuous demilitarization of war stocks, refurbishment of inventoried ammunition, and minor research and development projects through 1992.

In 1992, the installation's status changed from "inactive but maintained" to that of "caretaker." Administrative control of the facility acreage was transferred from the Base Realignment and Closure Division to the U.S. Property and Fiscal Officer (USP&FO) for Ohio in a series of transfers from 1999 to 2013. As of September 2013, administrative accountability for the entire 21,683-acre facility has been transferred to the USP&FO for Ohio and subsequently licensed to the OHARNG for use as a military training site, Camp Ravenna. The RVAAP restoration program involves cleanup of former production/operational areas throughout the facility related to former activities conducted under the RVAAP. The RVAAP Installation Restoration Program (IRP), managed by the U.S. Army National Guard (ARNG) and the OHARNG, encompasses investigation and cleanup of past activities over the entire 21,683 acres of the former RVAAP (USACE, 2016)

The overall objectives of the field effort covered under the purview of this SSHP is the loading, transportation, and recycling/disposal of stockpiled railroad ties, concrete, and telephone poles located at the Atlas Scrap Yard AOC. Additionally, the field effort will include the installation of a temporary storage facility suitable for temperature controlled storage of former RVAAP administrative records, and the re-location of records, documents, maps, shelving, and map cases from three current locations to the temporary storage facility.

1.2 Constituents of Concern

The railroad ties are suspected to potentially contain arsenic, creosote, and/or other preservative chemicals that may have been present during the time of their manufacture (1940's). Additionally, soil in and around the Atlas Scrap Yard may contain semi-volatile organic compounds, polycyclic aromatic hydrocarbons, and/or lead, due to historical usage of the area. As site operations will not involve digging into the soil, there will not be any exposure to constituents of concern (COCs) present in soil during the course of this project. As railroad tie material will be handled via heavy machinery and handled in such a way that debris remains in-tact, any COCs present in the railroad tie material will not become friable, and as such, there will not be any exposure to COCs from handling railroad ties.

2.0 HAZARD/RISK ANALYSIS

An activity hazard analysis (AHA) has been completed and is included in **Attachment 3** of the APP. The AHA addresses the task-specific activity hazards associated with the remedial action activities that are to be conducted by ERT and its subcontractors, and the appropriate control measures and response actions. Equipment, inspection, and training requirements for each activity are also identified in the AHA. Health and safety equipment, such as personal protective equipment (PPE), is described in Section 5.0 of this SSHP.

The AHA is an ongoing process initiated with the preparation of the SSHP and continuing through the implementation and completion of field activities. If conditions change and/or new hazards are identified, appropriate AHAs will be prepared or amended, and necessary changes will be made to the SSHP.

2.1 Physical Hazards

Physical hazards, to be discussed in depth in the following sub-sections, will potentially be present at the site during field activities. These physical hazards include the following:

- General Physical Hazards;
- Fire/Explosion Hazards;
- Noise Hazards;
- Electrical Hazards;
- Utility Hazards;
- Weather Hazards;
- Material Handling/Moving/Lifting;
- Equipment Use Hazards;

2.1.1 General Physical Hazards

The work site may include many basic physical safety hazards, such as:

- Steep grades and/or uneven terrain, posing slip, trip, and fall hazards;
- Holes, ditches, etc., posing fall, cave-in and other hazards;
- Potential vegetation and/or brush that may reduce or obstruct visibility, pose trip or fall hazards, or cause cuts or other injuries
- Precariously positioned objects that may cause injuries
- Sharp objects (i.e., nails, metal shards, glass), which may cause cuts, injections, or other injuries;
- Slippery surfaces, posing slip and fall hazards; and
- Unstable surfaces which may cause falls or other injuries.

Basic safety hazards can directly injure workers and create additional hazards. For example, a person could trip due to uneven terrain, causing him/her to fall and be cut on rusty metal shards, and as a result become inoculated with materials adhering to the metal.

Each investigation area within the site will be visually inspected for the presence of general physical hazards (i.e., trip/slip hazards, unstable surfaces or steep grades, sharp objects) prior to beginning work. If hazards are present, these hazards will be either removed or recorded and precautionary measures will be taken to prevent injury.

Site personnel will look constantly, closely, and carefully for these general physical hazards and immediately inform the SSHO/Site Superintendent of conditions that they feel may be hazardous.

If hazards are present, other than those discussed in this SSHP, these hazards will be recorded by the SSHO/Site Superintendent and precautionary measures will be taken to prevent injury. If the newly identified hazards require engineering controls, implementation will be discussed with the ERT PM and CELRL Technical Manager (TM), Project Manager (PM), and Contracting Officer's Representative (COR).

2.1.2 Fire/Explosion Hazards

Explosion and fire hazards may be present at the work site due to the unexpected ignition of chemicals or fuels, the sudden release of materials under pressure, or due to possible careless unauthorized smoking in work areas. Site operations will be conducted in accordance with local fire codes and regulations. In consideration of preemptive fire prevention, smoking will only be allowed in designated areas, vehicle engines will be powered down when not in use to prevent tall grasses from catching fire, and flammable materials will be properly labeled and stored in well-ventilated areas.

Each field vehicle will be equipped with a fire extinguisher. Employees will be trained in the proper use of fire suppression equipment. Fires beyond the incipient stage, uncontrollable with fire extinguishers, will be handled by local fire department professionals (Ravenna Fire Department). In the event of a fire, employees will contact the fire department by calling Ravenna Fire Department Emergency Services Dispatch (911 from landline phone or mobile phone for emergencies; (330) 297-5738 for non-emergencies), and all staff will report to the pre-determined rally point for evacuation (as presented in **Figure 2** of the APP) unless this location has become dangerous due to the existing emergency. Otherwise, personnel will proceed to a secondary rally point established between the field personnel and site personnel during Daily Safety Briefings. ERT staff will always be on the alert for unexpected events, and be prepared to act in these emergencies.

2.1.3 Noise Hazards

Working around equipment often creates excessive noise. Noise can cause workers to be startled, annoyed, or distracted, and can cause physical damage to the ear, pain, and temporary and/or permanent hearing loss. Lastly, working around equipment can interfere with communication. If workers are subjected to noise exceeding an 8-hour (hr) time-weighted average sound level of 85 decibels on the A-weighted scale (dBA), hearing protection will be provided with an appropriate noise reduction rating to comply with 29 Code of Federal Regulations (CFR) 1910.95 and reduce noise levels to or below 85 dBA. If ear insert devices are used, the SSHO/Site Superintendent will ensure they are properly fitted and inserted. If ear muffs are used, the SSHO/Site Superintendent will ensure proper coverage of the ear by the muff. For this project, it is anticipated that the phases of work where workers will have greatest probability of exposure to sound levels greater than 85 dBA would be the loading, transportation, and recycling of waste and railroad ties.

2.1.4 Electrical Hazards

Overhead power lines, electrical wiring, electrical equipment, and buried cables pose risks to workers of electric shock, burns, muscle twitches, heart fibrillation, and other physical injuries, as well as fire and explosion hazards.

The location of any underground hazards will be identified prior to intrusive work. If any potential hazards are identified, controls will be added to the intrusive activities-specific AHA.

At least 72 hours prior to intrusive field activities, ERT will coordinate with local utility companies (via Ohio Utilities Protection Services or 800-362-2764) to mark-out and identify all underground utilities in the investigation areas. If necessary, sample locations will be offset.

Lightning may be an electrical hazard during outdoor operations, particularly for workers in open areas and handling metal equipment. Weather conditions will be monitored and work suspended by the SSHO/Site Superintendent during electrical storms. A lightning monitor will be maintained on-site and used to determine lightning hazard. If no lightning monitor is available, the SSHO/Site Superintendent will count the number of seconds between the lightning strike and the thunder clap, and then divide the number of seconds by 5 to determine the distance away in miles. Work will be halted and personnel will take shelter (for at least 30 minutes after the last audible thunder or visual flash of lightning) when lightning is located within 10 miles of the site (USACE, 2014).

2.1.5 Utility Hazards

Overhead utilities may exist but are not anticipated to be a hazard. Underground utility avoidance measures are discussed in Section 2.1.4 above.

2.1.6 Ionizing Radiation Hazards

Work around ionizing radiation is not anticipated for this project.

2.1.7 Weather Hazards

Weather conditions will be taken into consideration during site activities. Heavy rains, snowfall, freezing conditions, electrical storms, high winds, and extreme temperatures may create dangerous situations for workers. Inclement weather may also impair equipment performance. Whenever unfavorable conditions arise, the SSHO/Site Superintendent will evaluate both the safety hazards and the ability of the employees to effectively perform their tasks under such conditions. Activities will be halted by the SSHO/Site Superintendent during unfavorable conditions. In the event of lightning/thunder, work will be ceased immediately by the SSHO/Site Superintendent until 30 minutes after the last lightning strike is visually observed or the last thunder clap has been heard.

2.1.8 Material Handling/Moving/Lifting

Material handling at the subject site may include manually moving/lifting items which could potentially result in physical injury. Injuries to the back and abdominal muscles from the improper lifting of loads are the most common occupational injuries reported. Such injuries can range from relatively mild strains to major, permanently disabling injuries. Before lifting a load, personnel will consider the overall weight, distribution of weight, awkwardness of the load, distance to be carried, obstacles to be negotiated, site conditions, and visibility.

When using equipment to move materials, proper work practices will be followed; equipment used will be designed for the task to be performed. Equipment will be inspected regularly by the qualified operator and/or SSHO/Site Superintendent, and damaged or defective equipment will be removed for service. Planning is critical when handling materials. The SSHO/Site Superintendent will assist in the planning of material movement, taking into consideration the current location of such materials and hazards associated with moving them. Routes for moving materials will be clearly communicated to site personnel, with paths cleared of obstructions.

Loads will be lifted using the strength of the leg muscles rather than the back, stomach, or arm muscles. The item will be approached so that when lifted, the load will be balanced evenly. Backs will be kept straight and the arms nearly parallel with the body. The knees will be bent to grasp the load. Lifting will be done by straightening the legs without bending the body, holding the load as close to the body as possible and the back remaining as straight as possible. Bulky, heavy loads in excess of 50 pounds will be handled by at least two people, ensuring that the load is level and evenly distributed between personnel helping to carry it. Carriers will know the destination and path for the load.

2.1.9 Equipment Use Hazards

All equipment and machinery will be operated in accordance with the manufacturer's recommendations. Staff working in the area will wear appropriate PPE and maintain a minimum safe distance from the equipment and will be made aware of the pending action prior to operation of any heavy machinery.

Only qualified personnel will be permitted to operate equipment. Equipment will be inspected daily by the qualified operator; unsafe equipment will not be used. All equipment will be operated at safe speeds and in a safe manner. Personnel working near the heavy machinery will wear high visibility reflective vests and hard hats.

Proper and safe operation of equipment is defined as operation in accordance with the manufacturer's recommendation and in accordance with Engineer Manual (EM) 385-1-1. Standard Level D plus safety glasses with side shields, hearing protection, and cut-resistant work gloves (i.e., leather or heavy cotton) will be required at all times when using equipment.

If deemed necessary in the field, a temporary fence no less than three feet in height may be placed to restrict access to specific work locations. The fence will have a sign stating, "Danger! Stay Out" (or similar) posted on it.

2.1.10 Environmental Sampling Support Hazards

Environmental sampling of contaminated media (e.g., soil, groundwater) will not occur during this effort. Therefore, it is not expected that any hazards will exist with regards to environmental sampling. However, ERT team will submit one composite soil sample of the railroad tie material and telephone pole material scheduled for offsite recycling/disposal.

2.1.11 Chemical Hazards

This project may involves coming into contact with gasoline and/or diesel fuel as part of equipment fueling operations. Any potential skin and/or eye irritation will be mitigated by on-site personnel wearing the appropriate PPE (e.g., gloves, safety glasses or goggles, long sleeve clothing). Any daily site-specific conditions warranting site-specific procedures will be discussed during the Daily Safety Briefing.

2.2 Biological Hazards

2.2.1 Poisonous Plants

All field work will be occurring outside, and as such, exposure to poisonous plants is a possibility. Workers will wear protective clothing to protect against biological hazards including both insects and poisonous plants. Personnel are trained to recognize poisonous plants, such as poison ivy, poison oak, or poison sumac, and will avoid them. According to the Center for Disease Control (CDC), poison ivy and poison sumac may be found in Ohio (CDC, 2016a). If discovered, these plants will not be removed or burned. If incidental dermal contact occurs, the affected areas will be washed with soap and water immediately. If extreme contamination has occurred, the worker will wash his/her hands, face, and other exposed skin areas and change clothes at the project site. Refer to the photographs below to aid in identification of poisonous plants.

The compound leaves of poison ivy (**Figure 1**) consist of three pointed leaflets; the middle leaflet has a much longer stalk than the two side ones. The leaflet edges can be smooth or toothed but are rarely lobed. The leaves vary greatly in size, from 8 to 55 millimeters (0.31 to 2.16 inches) in length. They are reddish when they emerge in the spring, turn green during the summer, and become various shades of yellow, orange or red in the autumn.

Figure 1. Poison Ivy

Poison sumac (**Figure 2**) is a woody shrub or small tree growing to 7 meter (20 feet) tall. It typically has 7 to 13 (always an odd number) oval leaflets which alternate on the stem. Stems are often red, which is a good distinguishing feature. It bears small grayish white shiny berries which grow in clusters. All parts of the plant contain poisonous oils that cause rash on contact.

Figure 2. Poison Sumac

2.2.2 Insect Bites and Stings

Insect bite symptoms may include redness, rash, swelling, chills, fever, diarrhea, and vomiting. A worker who has been bitten or stung and shows symptoms of a severe reaction will immediately inform the SSHO/Site Superintendent and seek medical assistance. Workers with allergies to insects (e.g., bees) should advise the SSHO/Site Superintendent prior to field activities and should carry an antidote kit, if necessary.

Efforts will be made to avoid disturbing insect nests encountered on the job site if possible, as an insect swarm may develop. If an insect nest (e.g., a bee hive) is encountered in an area that is likely to be disturbed during site activities, the nest will be eradicated prior to site activities to ensure employee safety.

2.2.2.1 Fleas and Ticks

To prevent contact with fleas or disease-carrying ticks known to be present in Ohio (i.e., dog ticks, deer ticks, or Lone star ticks) (Ohio Department of Health [ODH], 2016), workers will be encouraged to wear long-sleeved shirts, coveralls or long pants, and boots that extend above the ankle. Workers are encouraged to tape their pant cuffs to their boots, especially when working in wooded, overgrown, or high grass areas. Workers will thoroughly check clothing, skin, and hair for the presence of ticks at the end of each workday. If a tick attaches to the body, it will be removed by gently tugging with tweezers where the mouth-parts enter the skin. The tick should not be killed prior to removal. If a worker suspects he/she has been bitten by a tick, he/she will immediately inform the SSHO/Site Superintendent and the bite will be documented. The tick will be saved if possible. The bite area will be monitored for several days/weeks for the development of a noticeable bulls-eye shaped skin rash at the affected area or other tick bite-related symptoms, including:

- Fever/chills: With all tick-borne diseases, patients can experience fever at varying degrees and time of onset;
- Aches and pains: Tick-borne disease symptoms include headache, fatigue, and muscle aches. With Lyme disease patients may also experience joint pain. The severity and time of onset of these symptoms can depend on the disease and the patient's personal tolerance level.
- Rash: Lyme disease, Rocky Mountain spotted fever (RMSF), and ehrlichiosis can result in distinctive rashes:
    - In Lyme disease, the rash may appear within 3-30 days, typically before the onset of fever. The Lyme disease rash is the first sign of infection and is usually a circular rash called erythema migraines. This rash occurs in approximately 70-80 percent of infected persons and begins at the site of a tick bite. The rash may be warm, but is not usually painful. Some patients develop additional erythema migrant lesions in other areas of the body several days after the bite.
    - The rash seen with RMSF varies greatly from person to person in appearance, location, and time of onset. Approximately 10 percent of people with RMSF never develop a rash. Most often, the rash begins 2 to 5 days after the onset of a fever taking the appearance of small, flat, pink, non-itchy spots (macules) on the wrists, forearms, ankles, and spreads to the trunk of the body. Sometimes the rash may spread to the palms of the hands and/or soles of the feet. The red to purple, spotted

(petechial) rash of RMSF is usually not seen until the sixth day or later after onset of symptoms and occurs in 35-60 percent of patients with the infection.

- In about 30 percent of patients (and up to 60 percent of children), ehrlichiosis and/or anaplasmosis can cause a rash. The appearance of the rash ranges from macular to maculopapular to petechial, and may appear after the onset of fever. Symptoms can also include mild to severe fever, headache, muscle pain, vomiting, and general discomfort.

If any of these symptoms develop, seek immediate medical attention for a possible infection of tick-related disease.

Insect repellant containing *DEET®* or permethrin is an effective means of tick control. Permethrin should only be applied to clothing and allowed to dry before wearing the clothing; do not apply it directly to skin. Do not use petroleum jelly, hot matches, nail polish, or other products to assist in tick removal. **Figure 4** presents a photograph of the dog tick life cycle: from left to right are larva, nymph, adult male, and adult female. **Figure 5** presents a photograph of the deer tick life cycle on a centimeter scale: from left to right are adult female, adult male, nymph, and larva. **Figure 6** presents a photograph of a lone star tick.

The Lyme Disease Association (LDA) reported 119 cases of Lyme Disease in 2014 (1367 cases since 1990) in all of Ohio (LDA, 2016).

Figure 3. Dog tick (*Dermacentor variabilis*)

Figure 4. Deer tick (*Ixodes scapularis*)

Figure 5. Lone star tick (*Amblyomma americanum*)

2.2.2.2 Mosquitoes

Mosquitoes may also carry disease, therefore workers will wear long-sleeved shirts, long pants, and boots that extend above the ankle; workers will be encouraged to tape their pant cuffs to their boots, especially when working in wooded, overgrown, or high grass areas. General guidelines for prevention of exposure to mosquitoes and the potential diseases that they carry (i.e., encephalitis and West Nile Virus) include the following:

- Wear long-sleeved shirts;
- Spray exposed skin with an insect repellent containing 15-30 percent *DEET®*;
- Spray clothing with products containing *DEET®* or permethrin, as mosquitoes may bite through thin clothing. Permethrin should only be applied to clothing and allowed to dry before wearing the clothing; do not apply it directly to skin. Wash treated clothing before wearing it again;
- Do not apply repellent to skin that is under clothing;
- Wash treated skin with soap and water after returning indoors.

Encephalitis symptoms usually occur within 2 to 10 days after being bitten by an infected mosquito. These symptoms include high fever, stiff neck, headache, confusion, lethargy and swelling of the brain.

West Nile Virus symptoms include fever, headache, nausea, vomiting, and rash, which are mild symptoms to severe symptoms that include neck stiffness, stupor, disorientation, tremor, coma, vision loss, and paralysis. These severe symptoms could last weeks or could be permanent. The onset of symptoms usually begins 3 to 14 days after a mosquito bite. Medical attention should be sought as soon as any symptoms of mosquito related illness are noticed The Centers for Disease Control (CDC) reports a 0.01-0.24 incidence rate of West Nile Virus neuroinvasive diseased per 100,000 people in Ohio for 2015 (CDCb, 2016).

2.2.2.3 Spiders

During work outdoors, venomous spiders may also present a concern to workers. All spider bites need to be immediately reported to the SSHO/Site Superintendent and the incident/accident forms need to be completed. Venomous spiders common in Ohio include: Southern Black Widow Spider and Brown Recluse Spider (**Figure 6** and **Figure 7**) (Spiders.us, 2016).

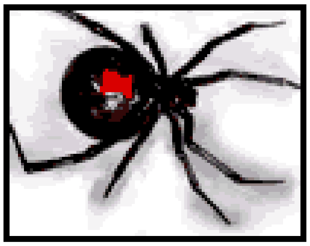

Figure 6. Southern Black Widow Spider (*Latrodectus mactans*)

Figure 7. Brown Recluse Spider (*Loxosceles reclusa*)

In general, most spiders are not aggressive unless agitated while guarding their egg sacs. They live in a variety of natural and domestic habitats such as under rocks and wooden boards, and in dense plant growth. Spider venom affects the nervous system and can cause pain in the lymph nodes. Other symptoms of a severe bite include nausea, elevated blood pressure, sweating, tremors, and increased white blood cell counts. The wound may appear as a bluish-red spot, surrounded by a whitish area. Victims of a spider bite may exhibit the following signs or symptoms:

- Sensation of a pinprick or minor burning at the time of the bite;
- Appearance of small punctures (but sometimes none are visible); and/or
- After 15 to 60 minutes, intense pain is felt at the site of the bite which spreads quickly, and is followed by profuse sweating, rigid abdominal muscles, muscle spasms, breathing difficulty, slurred speech, poor coordination, dilated pupils, and generalized swelling of face and extremities.

2.2.3 Animal Bites

The majority of field work will be occurring outside, and as such, there exists a possibility that rodents, snakes, stray dogs and cats, raccoons or other animals may be encountered on the job site. The only effective measure to preclude animal bites is avoidance. Contact with wild animals will be avoided. Personnel will not reach into an object such as a pipe that may contain a rodent or other animals, or attempt to remove the animal. If possible, delay activity in this area until the animal leaves or has been removed by an animal control unit. If it is necessary to use or move an object where an animal is hiding, the SSHO/Site Superintendent may have to notify the Range Control. Range Control should be contacted to aid or subdue an animal that may cause a risk to workers (i.e., a raccoon). Do not approach an animal, as this may cause the animal to become aggressive. Persons bitten by an animal will seek medical assistance immediately, especially if it is suspected that the animal may be rabid. Aggressive or disoriented behavior, as well as foaming at the mouth, can be signs of rabid animals. Until medical assistance can be reached, bitten persons will watch for symptoms of severe swelling, nausea, and shock. The local animal control agency will be contacted if a stray dog is observed on the property. Workers will not attempt to leash a stray dog (no matter how harmless it may appear); doing so would greatly increase the risk of an animal bite.

According to the Ohio Department of Natural Resources Division of Wildlife (ODNR), venomous snakes known to be located in Ohio include: the Northern Copperhead, the Eastern Timber Rattlesnake, and the Eastern Massasauga Rattlesnake (**Figure 8** through **Figure 10**) (ODNR, 2016).

Figure 8. Eastern Massasauga Rattlesnake (*Sistrurus catenatus catenatus*)

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Figure 9. Northern Copperhead (*Agkintrodon contortrix mokasen*)

Figure 10. Eastern Timber Rattlesnake (*Crotalus horridus*)

All personnel should be aware that site activities may have the potential for encountering or disturbing snakes. Areas with heavy undergrowth or shrubs are of special concern. Prompt First Aid measures are extremely important. If an individual is bitten by a snake, the basic rule is -- **TREAT ALL SNAKEBITES AS VENOMOUS**. **ANY** snake encountered on-site will be assumed to be venomous. If an encounter with a snake occurs, the field team will immediately leave the area. If it is necessary to return to the area, shin guards may be employed, especially if the area has limited visibility.

A probability exists that all snakes may be potential carriers of tetanus (lockjaw); if bitten by any snake, whether venomous or not, seek medical attention immediately. If bitten, identify and/or kill the snake (if it can be done quickly and safely) and take it to the hospital for identification. This information is valuable to medical personnel when treating snakebites. Call for emergency assistance immediately if someone has been bitten by a snake. Responding quickly in this type of emergency is crucial. While waiting for emergency assistance:

- Wash the bite with soap and water;
- Immobilize the bitten area and keep it lower than the heart;
- Cover the area with a clean, cool compress or a moist dressing to minimize swelling and discomfort; and
- Monitor vital signs.

If a victim is unable to reach medical care within 30 minutes, the American Red Cross recommends applying a bandage, wrapped 2 to 4 inches above the bite, to help slow the venom. This should not cut off the flow of blood from a vein or artery - the band should be loose enough to slip a finger under it. The emergency procedures for snakebites will be reviewed during the initial site safety briefing (during discussion of the emergency action plan, which is further described in Section 11.0 of this SSHP).

2.2.4 Bacteria

Adherence to PPE requirements, personal hygiene measures, and work site rules will minimize exposures. Bacteria are not expected to be a hazard based on the location of field activities.

2.2.5 Humans

While unauthorized personnel are not expected to be present or pose a threat to personnel working at the site, precautions will be taken to prohibit the presence of unauthorized personnel in the work site. Onlookers will be asked to maintain a safe distance from the work site (as determined by the SSHO/Site Superintendent to limit public exposure to operations and noise). Any obstinate or malicious behavior will be dealt with by calling the police, via Emergency Services Dispatch (911 from landline phone or mobile phone). In addition, the ERT PM and the CELRL COR/TM will be notified.

2.3 Vehicle Traffic Hazards

Field personnel may be exposed to vehicle accident hazards associated with the operation of vehicles during the project. Additionally, many of the areas to be investigated are located next to active roadways or in parking lots where vehicular traffic may be present. To control these hazards while operating a vehicle under ERT control, the following safety requirements will be strictly enforced in accordance with ERT's Corporate Health and Safety Manual:

- Seat belts will be worn anytime a vehicle is in motion, regardless of speed or distance to be traveled;
- The speed limit will be followed at all times;
- Vehicles will never be operated at a speed that is unsafe for the conditions (i.e., road surface, traffic, visibility, weather, etc.);
- Talking on hand-held cell phones and texting are prohibited while driving;
- Hand signals will be used with a signal person during backing if a back-up alarm is not present; and
- Smoking is not permitted in ERT vehicles.

Due to proximity of a limited number of stockpiles adjacent to Paris Windham Road, field personnel may also be exposed to hazards associated working near moving vehicles during the project. To control these hazards, the following safety requirements will be strictly enforced:

- The work area will be cordoned off with safety cones, and a sign will be placed, per the example (or equivalent) provided in **Enclosure B**;
- In the unlikely event that road closure is necessary, ERT will work with Fort Benning personnel;
- Class II Safety Vests (fluorescent yellow-green) will be worn by site personnel when the following applies:
  - Exposure to vehicular traffic at speeds up to 45 mph;
  - Limited visibility of workers due to mobile heavy equipment operations, vehicles, load handling, or other hazardous activities;

  - Reduced visibility conditions due to inclement weather; and/or
  - No protective barriers when near vehicular traffic.

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3.0 STAFF ORGANIZATION, QUALIFICATIONS, AND RESPONSIBILITIES

Ensuring safe performance of site operations and maintenance of a safe and healthy work site is the responsibility of everyone assigned to the site. Project personnel assigned to this remedial action are identified in **Table 1**; resumes for ERT key safety personnel are included in **Enclosure A**.

Table 1. Project Personnel

Service/Contact	Agency/Position	Telephone No.
Mark Leeper	ARNG Restoration Program Manager	(703) 607-7955
Kevin Sedlak	ARNG	(614) 336-2053
Katie Tait	OHARNG	(614) 336-6136
Gail Harris	RVAAP AR	(330) 872-8003
Pat Ryan	REIMS	(865) 481-4664
Craig Coombs	CELRL PM	(502) 315-6324
Matt Burg	CELRL Chief of Safety	(502) 315-7061
Nat Peters	CELRL COR	(502)315-2624
Kevin Mieczkowski	CELRL TM	(502) 315-7447
Michael Barsa	ERT Division SHM	(301) 323-1447 (o) (410) 703-6213 (c)
Jennifer Harlan	ERT Division Manager	(301) 323-1394 (o)
Sean Carney	ERT PM	(607) 259-7017) (c)
J.T. Nolan	ERT SSHO/Site Superintendent/OSHA Competent Person for Overall Site Health and Safety	(301) 412-7301
Robert Koroncai	Alternate ERT SSHO/OSHA Competent Person for Overall Site Health and Safety	(301) 323-1445 (o) (267) 481-5567 (c)

3.1 ERT Project Manager

The ERT PM is responsible for the overall direction, implementation, and enforcement of health and safety requirements on this project.

3.1.1 Qualifications

The ERT PM has OSHA 40-hr Hazardous Waste Operations and Emergency Response (HAZWOPER) Training (with up-to-date annual 8-hr refresher) with a minimum of 3 days of supervised work experience.

3.1.2 Responsibilities

Other ERT PM responsibilities include:

- Ensure that the project is being performed in a manner consistent with the ERT's Corporate Health and Safety Program;
- Ensure that an SSHP is prepared and approved;

- Provide the field team with project information related to health and safety matters and the development of the SSHP;
- Monitor compliance with the SSHP by ERT and subcontractor personnel:
- Ensure adequate resources are provided to the safety and health staff, so that they may carry out their duties;
- Ensure that all ERT and subcontractor personnel designated to work at the project sites are qualified according to ERT's medical surveillance and training requirements;
- Determine and implement personnel disciplinary actions for safety violations;
- Maintain communication with CELRL;
- Approve the appointment of the SSHO/Site Superintendent and any replacement personnel;
- Direct personnel to change a work practice if it is determined to be hazardous to the health and safety of site personnel;
- Remove personnel from the project if their actions endanger their health and safety, or the health and safety of co-workers; and
- Report exposure hours.

3.2 ERT Division Safety and Health Manager

3.2.1 Qualifications

The SHM is a Certified Safety Professional, and has over 6 years of experience managing safety and occupational health at hazardous waste site cleanup operations and over 12 years of experience in environmental safety and health.

3.2.2 Responsibilities

The SHM is responsible for the following actions:

- Develop, maintain, and oversee implementation of the SSHP;
- Visit the site as needed to audit the effectiveness of the SSHP;
- Remain available for site emergencies;
- Develop modifications to the SSHP as needed; and
- Approve the SSHP by signature.

3.3 Site Safety and Health Officer

The SSHO will report directly to the ERT SHM and is the primary person responsible for day-to-day health and safety during all field activities.

3.3.1 Qualifications

The SSHO meets all qualification requirements of EM 385-1-1. The SSHO will have completed the OSHA 40-hr HAZWOPER Training (with up-to-date annual refresher as needed) will have a minimum of 3 days of supervised work experience, will have at least 1 year of experience implementing safety and occupational health procedures at hazardous, toxic and radioactive waste sites, will have experience selecting PPE, will have completed the OSHA 8-hr HAZWOPER Supervisor Training, and will have completed the OSHA 30-hr Construction Safety Training, and First Aid and cardiopulmonary resuscitation (CPR) training.

3.3.2 Responsibilities

The SSHO will have the following responsibilities:

- Provide implementation/oversight of ERT safety and health matters;
- Confirm suitability for work of ERT and subcontractor personnel, based on OSHA and site-specific medical and training requirements;
- Conduct visitor orientation;
- Conduct on-site safety orientation and operational review on the first working day;
- Ensure copies of all forms listed in the SSHP are readily accessible by site personnel and visitors (these will be on-site in hard copy form when practicable and feasible);
- Conduct daily safety briefings;
- Conduct and document daily safety inspections, weekly safety audits, and self-assessments;
- Maintain safety, training, and visitor logs;
- Consult with the ERT SHM as needed to ensure all potential hazards are addressed appropriately;
- Enforce the ERT Alcohol/Drug Abuse Policy;
- Train personnel on the emergency action plan requirements for the site;
- Ensure prominent display of descriptions and maps associated with local hospital and emergency evacuation routes;
- Enforce the use of the “buddy” system;
- Ensure First Aid kits are on-site and adequately stocked (includes consideration of the blood borne pathogens standard);
- Provide oversight and serve as the interface for subcontractor health and safety representatives;
- Conduct injury/illness/incident/near miss reporting and investigation if needed;
- Stop work that is not in compliance with the contract;
- Work with field crew to identify, evaluate, and control hazards;
- Ensure all site activities are performed in a manner consistent with the ERT’s Corporate Health and Safety Program and the SSHP;
- Oversee on-site implementation of the SSHP;
- Ensure that all ERT personnel and subcontractors working at the Site have fulfilled appropriate medical surveillance and training requirements (via on-site maintenance of workers’ current training/surveillance documentation);

- Report all incidents, accidents, and near misses to the ERT PM and the CELRL COR/TM if needed;
- Maintain health and safety equipment on-site;
- Inspect ongoing activities, and report any health and safety deficiencies to the ERT PM;
- Accompany or maintain communication with each work crew;
- Conduct initial site-specific safety training and regular safety briefings for site personnel;
- Account for all personnel in the event of fire, explosion, severe storm, or other incident;
- Monitor weather conditions;
- Stop site activities if an "imminently dangerous" situation exists. The emergency situation will be immediately reviewed with the ERT PM;
- Direct personnel to change a work practice if it is determined to be hazardous to the health and safety of site personnel;
- Temporarily suspend an individual from site activities for infractions of the SSHP, pending discussion with the ERT PM; and
- Report to the ERT PM all exposure hours for ERT and subcontractor personnel.

During an emergency, the SSHO will be responsible for initiating and coordinating responses including:

- Initiating the evacuation of the work site when needed, communicating with off-site emergency responders, and coordinating activities of on-site and off-site emergency responders; and
- Determining if hazardous conditions are adequately alleviated prior to allowing resumption of work operations after an emergency.

3.4 Site Superintendent/Field Personnel/Laborers/Equipment Operators

3.4.1 <u>Qualifications</u>

All field personnel have completed the OSHA 40-hr HAZWOPER Training (with up-to-date annual refresher) and will have a minimum of 3 days of supervised work experience.

The Site Superintendent will also possess current USACE Construction Quality Management for Contractors training certification.

Heavy equipment operators will also possess training records showing participation in Heavy Equipment Operations and Safety Training.

3.4.2 <u>Responsibilities</u>

Field personnel responsibilities include:

- Following this SSHP and applicable safety and health rules, regulations, and procedures;
- Using required controls and safety devices, including PPE;
- Inspecting PPE before use for noticeable flaws;
- Notifying the SSHO of suspected safety or health hazards.

3.5 Subcontractors

All subcontractors will fully comply with the provisions of the APP/SSHP. The SSHO/Site Superintendent will provide oversight and ensure subcontractor compliance.

3.6 Visitors and Authorized Entrants

All visitors to site work areas must be approved by the SSHO/Site Superintendent. Visitors will receive a briefing in site health and safety, and visitors will then be escorted by the SSHO/Site Superintendent or his/her designee. The Site Control Log (located within **Enclosure B**) will be utilized to document any visitor access to the site.

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4.0 TRAINING

4.1 General

Hazardous waste workers are trained to meet 29 CFR 1910.120 requirements. At a minimum, site personnel have the following training certifications:

- 40 hrs of OSHA initial HAZWOPER training for general site workers and at least 3 days of field experience under the direct supervision of a trained, experienced supervisor;
- If more than 12 months have passed since initial training, an 8-hr OSHA HAZWOPER Annual Refresher Training must be completed;
- SSHO/Site Superintendent must have completed the 8-hr HAZWOPER supervisory training and OSHA 30-hr Construction Safety Training.

In addition to the required training, a minimum of two site workers will be current in First Aid and CPR training. The SSHO/Site Superintendent will be current in First Aid and CPR training.

4.2 On-site Training

Employees, subcontractors, and site visitors will read this SSHP and will indicate their understanding of the requirements by signing the SSHP Review Record, which is included in this SSHP on page ii, prior to the Table of Contents. The SSHO/Site Superintendent will brief employees on the potential hazards at the site and protective measures to be implemented, both prior to entry and daily during the work. **Figure 2** of the APP shows the evacuation routes to be used in the event of an emergency; this will be communicated to personnel during the daily safety brief.

4.2.1 Pre-Entry Briefing

The SSHO/Site Superintendent will provide site-specific training on the contents of this SSHP, including: emergency procedures; areas of restricted access; responsibilities for safety of personnel, and property; physical, chemical, and biological hazards; PPE; and location of the Safety Data Sheets (SDSs). Pre-entry briefings will be held prior to personnel working on-site. Following review of this document and receipt of the pre-entry briefing, personnel will indicate their understanding of the site-specific hazards and appropriate emergency response by signing the Review Record at the front of this SSHP.

4.2.2 Daily Safety Briefings

The SSHO/Site Superintendent is responsible for conducting daily safety briefings during field activities to discuss status of site health and safety, the day's activities specific safety concerns, and to identify the designated primary and secondary rally points during an emergency. These briefings will also address employees' concerns regarding on-site safety and hazard control practices and procedures.

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5.0 PERSONAL PROTECTIVE EQUIPMENT

5.1 PPE Usage

All personnel performing operations on-site will be required to use the appropriate level of protection. The minimum level of protection required to begin each activity of this project is Level D, modified to include long pants and shirt with long sleeves (as needed), and steel-toe work boots, composite toe work boots, or other appropriate footwear. Additional PPE required for specific tasks are described in the **Table 2**. Effective use of PPE requires that the equipment be properly used, maintained, and inspected prior to use and periodically during the day. Site-specific issues and standard procedures will be reiterated during pre-entry training.

Table 2. Minimum Level of Protection Requirements

Activity	Level of Protection
Site Visits and Inspections	Level D, modified to include safety-toe work boots
Mobilization/Demobilization	Level D, modified to include safety-toe work boots
Site Preparation	Level D, modified to include safety-toe work boots, safety glasses with side shields (as needed), and leather work gloves (as needed)
Complete the loading, transportation, and recycling of waste material	Level D, modified to include safety-toe work boots, hard hats, safety vests, safety glasses with side shields (as needed), and leather work gloves (as needed)
Install temporary storage facility	Level D, modified to include safety-toe work boots, safety glasses with side shields (as needed), and leather work gloves (as needed)
Waste management	Level D, modified to include safety-toe work boots, safety glasses with side shields (as needed), and leather work gloves (as needed)
Restoration of disturbed area	Level D, modified to include safety-toe work boots, safety glasses with side shields (as needed), and leather work gloves (as needed)

Hearing protection is required during use of equipment and other activities that produce sound pressure levels that exceeds 85 dBA steady state expressed as the time weighted average as specified in Table 5-4, EM 385-1-1, or 140 dBA impulse.

Any site personnel conducting activities that may involve exposure to Poison Ivy will be afforded the opportunity to wear an additional, light weight coverall over-garment to help mitigate the effects of exposure. Additionally, Poison Ivy treatments such as Ivy Dry, Cortaid, Benadryl, or Caladryl have proven to be effective treatment for exposure. The best treatment is avoidance.

The SSHO/Site Superintendent will conduct hazard assessments when site conditions change and hazards are identified requiring a change in the level of protection; the SSHO/Site Superintendent will re-evaluate the SSHP and the level of protection may be upgraded or downgraded prior to re-entry to the site.

The SSHO/Site Superintendent will ensure PPE users are trained to know when PPE is necessary and what level, how to properly wear and adjust the PPE, limitations of the PPE, and the proper care, inspection, testing, maintenance, useful life, storage, and disposal of the PPE. The SSHO/Site Superintendent will also review PPE usage in the field to determine whether employees have the proper understanding of PPE importance, and if not, will re-train the employee.

5.2 Written Certification of PPE Training

By signature and acceptance of this SSHP (as well as the parent APP), the ERT Division SHM certifies that all site workers whose certificates appear in **Enclosure C** of this SSHP have been trained in the proper use of PPE via their respective 40-hr OSHA HAZWOPER training and their 8-hr HAZWOPER refresher training on the dates listed on the completion certificates. Applicable certificates not included in **Enclosure C** for site workers will be furnished to the SSHO/Site Superintendent upon mobilization to the work site. In addition, any applicable certifications that may expire during the execution of this project will be refreshed prior to their expiration date(s) and updated in this SSHP, **Enclosure C**.

6.0 MEDICAL SURVEILLANCE

Field team members must have satisfactorily completed a comprehensive physical examination within 12 months (or 24 months if approved by an occupational physician) prior to the start of the field activities. Non-hazardous waste site workers will be medically examined to meet OSHA requirements specific to their job. Medical examinations and consultations will comply with the protocols of 29 CFR 1910.120 and will be provided according to the following schedule:

- Prior to fieldwork assignment;
- At least annually for employees covered by the program;
- At termination of employment or reassignment to an area where the employee had not been examined within the past 6 months; and
- As soon as possible after an identified overexposure to hazardous substances or health hazards.

A physician's clearance letter/form will be available for review by USACE upon request.

Medical examinations will be conducted more frequently if the physician deems such examinations as necessary to maintain employee health. Documentation for compliance with 29 CFR 1910.120 will be maintained in ERT's Division SHM Office and the records will be kept on file for at least 30 years after termination of employment. A minimum of the following information will be kept:

- Name and social security number;
- Physician's written opinions, recommendations, limitations, and test results;
- Employee medical complaints related to hazardous waste operations; and
- Information provided to the physician by the employee concerning possible exposures, accidents, etc.

Up-to-date certification of medical surveillance for field personnel is included in **Enclosure C**.

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7.0 EXPOSURE MONITORING/AIR SAMPLING

As stated in Section 1.2, no historical constituents of concern will be encountered during the course of operations; therefore, no exposure monitoring/air sampling will be necessary.

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8.0 HEAT AND COLD STRESS

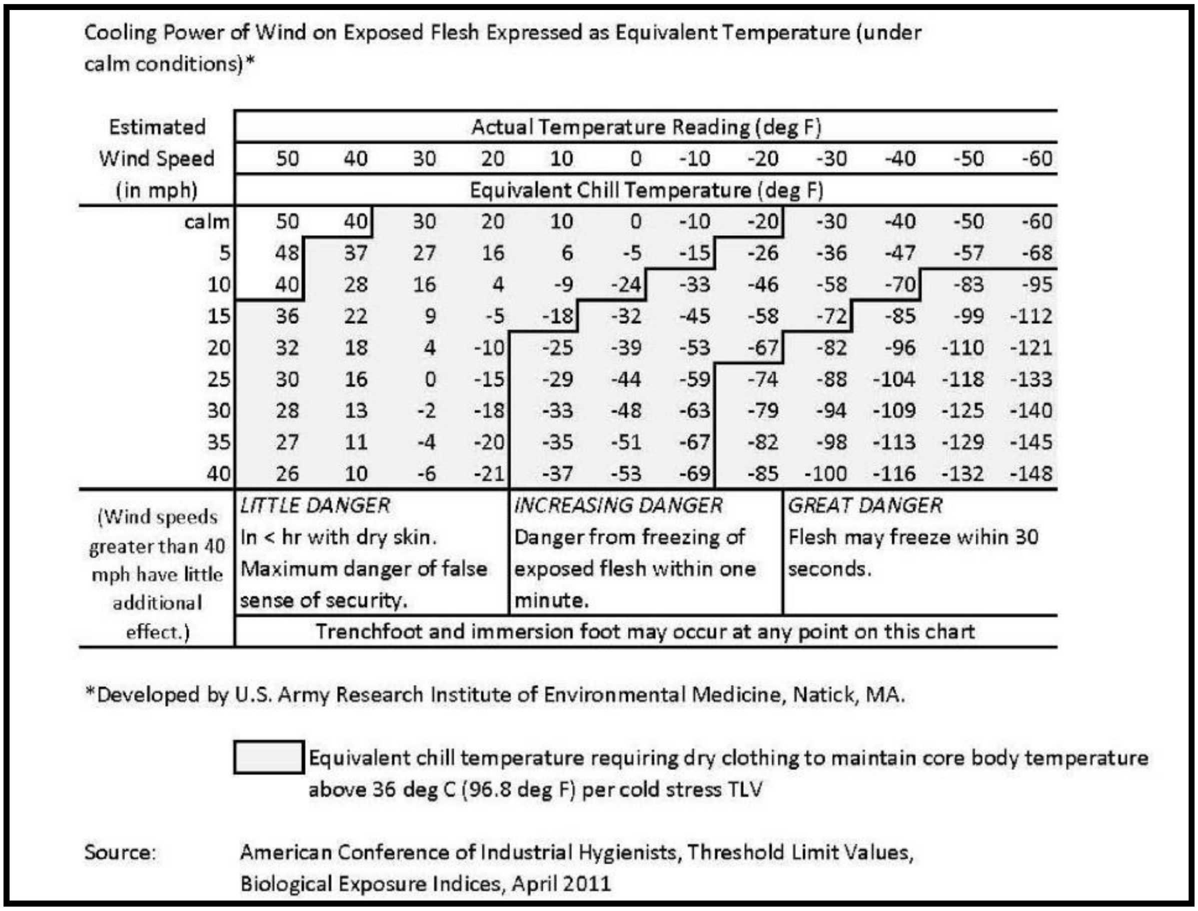
ERT will monitor the temperature at the work site using a wet bulb globe temperature (WBGT) 8758 digital psychrometer (or equivalent), which monitors wet bulb globe temperature, globe temperature, air temperature, and relative humidity. In addition to the WBGT, per EM 385-1-1 06.I.01, ERT will also monitor the weather via wireless internet connection to the National Weather Service website, smart phone application, and/or local radio, depending on availability. In the event of severe weather, work will be terminated and all site workers will seek shelter.

8.1 Cold Stress

Cold stress hazards are most likely to occur at low temperatures or low wind chill factors, with wet, windy conditions contributing to the risk. Site personnel will respect the OSHA Cold Stress Equation while working in cold conditions:

LOW TEMPERATURE + WIND SPEED + WETNESS = INJURIES and ILLNESS

Site personnel will be made familiar with the signs and symptoms of cold stress, which include hypothermia and frostbite. **Figure 11** below will be used to determine the degree of cold stress hazard:



Cooling Power of Wind on Exposed Flesh Expressed as Equivalent Temperature (under calm conditions)*

Estimated Wind Speed (in mph)	Actual Temperature Reading (deg F)											
	50	40	30	20	10	0	-10	-20	-30	-40	-50	-60
	Equivalent Chill Temperature (deg F)											
calm	50	40	30	20	10	0	-10	-20	-30	-40	-50	-60
5	48	37	27	16	6	-5	-15	-26	-36	-47	-57	-68
10	40	28	16	4	-9	-24	-33	-46	-58	-70	-83	-95
15	36	22	9	-5	-18	-32	-45	-58	-72	-85	-99	-112
20	32	18	4	-10	-25	-39	-53	-67	-82	-96	-110	-121
25	30	16	0	-15	-29	-44	-59	-74	-88	-104	-118	-133
30	28	13	-2	-18	-33	-48	-63	-79	-94	-109	-125	-140
35	27	11	-4	-20	-35	-51	-67	-82	-98	-113	-129	-145
40	26	10	-6	-21	-37	-53	-69	-85	-100	-116	-132	-148
(Wind speeds greater than 40 mph have little additional effect.)	*LITTLE DANGER* In < hr with dry skin. Maximum danger of false sense of security.				*INCREASING DANGER* Danger from freezing of exposed flesh within one minute.				*GREAT DANGER* Flesh may freeze wihin 30 seconds.			
	Trenchfoot and immersion foot may occur at any point on this chart											

*Developed by U.S. Army Research Institute of Environmental Medicine, Natick, MA.

☐ Equivalent chill temperature requiring dry clothing to maintain core body temperature above 36 deg C (96.8 deg F) per cold stress TLV

Source: American Conference of Industrial Hygienists, Threshold Limit Values, Biological Exposure Indices, April 2011

Figure 11. Wind Chill Chart

8.1.1 Hypothermia

Hypothermia is caused by a cold-induced decrease of the core body temperature that produces shivering, numbness, drowsiness, and muscular weakness. If severe enough, it can lead to unconsciousness and eventual death.

8.1.2 Frostbite

Frostbite can occur when constriction of blood vessels in the extremities decreases the efficient, ready supply of warming blood. This may result in formation of ice crystals in the tissues, causing tissue damage. Conditions may range from frostnip, which is a numbing of extremities, to deep-freezing tissue beneath the skin. Symptoms include white or grayish skin, blisters, numbness, mental confusion, failing eyesight, fainting, shock, and cessation of breathing. Death may occur from heart failure.

8.1.3 Monitoring, Prevention, and Response

Pain in the extremities may be the first warning of cold stress, and precautions will be taken to reduce exposure. Severe shivering will be taken as a sign of immediate danger to the worker, and exposure to cold will be immediately terminated. Personnel exhibiting signs and symptoms of cold stress will be removed from the site and decontaminated (if necessary). Decontamination will only occur if no further injury will be caused to the victim; first aid will be administered as appropriate. Emergency medical services will be contacted if symptoms are severe (e.g., more than numbness of the extremities or shivering). If decontamination is not possible due to injury, emergency medical services will be informed that the victim(s) is contaminated and appropriate PPE is required. When air temperatures are less than 36ºF (including wind chill), workers who become immersed in water or whose clothing becomes wet will be immediately provided a change of clothing, transported to a heated area, and be treated for hypothermia (if necessary) (Mylar blankets and towels will be included as part of first aid kits, and employees will insure they have a change of clothing available).

ERT will mitigate these risks in the investigation areas on-site through the use of appropriate PPE, such as climate appropriate clothing and boots, layered clothing, head covers, and gloves. The SSHO/Site Superintendent will monitor site personnel for signs of cold stress and/or general fatigue. Field personnel will receive breaks as outlined in the Cold Work-Rest Regimen Table (**Table 3**). Personnel not appropriately dressed for the elements will not be allowed within the investigation area until proper attire is donned.

As a precautionary measure, employees should wear layers of loose-fitting clothing including insulated coveralls, head cover (e.g., a wool cap), and boots when temperatures fall below 40°F, including wind chill. Protection of the hands, feet, and head is particularly important because these are likely to be injured first by cold. For light work occurring at 40°F and below, thermally protective gloves will be required. However, actual injury to hands, feet, and head is not likely to occur without prior development of early signs, such as numbing and shivering. Bare skin contact with cold surfaces (below 32°F) will be avoided. If wind chill becomes a factor at the work location, personnel will wear wind-resistant outer shell to decrease wind chill effects. Less than one minute of exposure to cold is permitted when the air speed and temperature results in an equivalent chill temperature of -26ºF to -72ºF (this scenario is highly unlikely, however).

At air temperatures below 45°F, the temperature will be monitored a minimum of every eight hours. At temperatures below 45°F and above 30°F, the temperature and wind speed will be monitored every 4 hours. At air temperatures below 30 °F, the temperature and wind speed will be measured and recorded at least every 4 hours or more frequently if it begins to lower.

A temperature-dependent work regimen limiting lengthy periods of outdoor activity may be necessary; **Table 3** (CCOHS, 2008) provides guidance for working in severe cold weather. This table should be used as guidance; however, the SSHO/Site Superintendent will make determinations on the work schedule based on site conditions and worker feedback. Additionally, when possible, work will be scheduled during the warmer part of the day if cold stress is a concern on the day work is to occur. Workers entering heated shelters will remove the outer layer of clothing and loosen remaining clothing to permit the evaporation of perspiration. Workers will avoid dehydration by drinking water or other decaffeinated beverages, including warm drinks and soups, as necessary and available.

Table 3. Threshold Limit Values Work/Warm-up Schedule for Four-Hour Shift

Air Temperature/ Sunny Sky	No Noticeable Wind		5 mph Wind		10 mph Wind		15 mph Wind		20 mph Wind	
˚F	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks	Max. Work Period	No. of Breaks
-15 to -19	(Norm breaks) 1		(Norm breaks) 1		75 min	2	55 min	3	40 min	4
-20 to -24	(Norm breaks) 1		75 min	2	55 min	3	40 min	4	30 min	5
-25 to -29	75 min	2	55 min	3	40 min	4	30 min	5	Cease non-emergency work	
-30 to -34	55 min	3	40 min	4	30 min	5	Cease non-emergency work			
-35 to -39	40 min	4	30 min	5	Cease non-emergency work					
-40 to -44	30 min	5	Cease non-emergency work							
-45 and below	Cease non-emergency work									

8.2 Heat Stress

Heat stress hazards can occur even in temperatures not commonly considered “hot” due to the level of physical activity, the level of PPE the worker is wearing, or the physical condition of the worker. Illness resulting from exposure to extreme heat is possible during field operations. Factors affecting heat stress include high temperatures and humidity, direct sun or heat, limited air movement, physical exertion and poor physical condition. Personnel will be familiar with the signs and symptoms of heat stress, including heat cramps, heat exhaustion, and heat stroke.

8.2.1 Heat Cramps

Symptoms of heat cramps include muscle spasms in the abdomen or limbs. Frequent rest periods and fluid intake are appropriate measures to prevent or reduce heat cramps.

8.2.2 Heat Exhaustion

Symptoms of heat exhaustion include severe dehydration; pale, clammy skin; profuse sweating; dizziness, light-headedness; slurred speech; rapid pulse; confusion; fainting; fatigue; cool skin; and nausea. Affected personnel will be escorted from the site to rest in a cool, shaded area, and given fluids slowly.

8.2.3 Heat Stroke

Heat stroke is a life-threatening condition occurring when the body's temperature-regulating system improperly functions. The symptoms are: hot dry skin; rapid, deep breathing; lack of perspiration; delirium; high fever (often 106°F or more); nausea; and unconsciousness. Brain damage and/or death may occur if body temperature is not reduced. Provide fluids, use cooling devices (hose-down or shower), call emergency medical services, or transport to hospital immediately.

Some preventive measures to avoid heat stress include:

- Frequent resting in cool or shaded areas; and
- Staying hydrated by consuming large quantities of fresh potable water (more than amount needed to simply “quench thirst”). Drink at least 8 ounces of water or diluted electrolyte sports drink beverages every 2 hours when temperatures exceed 75ºF – do not consume alcoholic beverages to combat dehydration or heat stress.

8.2.4 Monitoring, Prevention, and Response

Heat stress monitoring is typically conducted in a manner that anticipates and prevents the onset of heat stress symptoms. Non-acclimated workers, workers having had previous heat stress injuries, and workers wearing full body impermeable chemical protective clothing shall be monitored when the work area temperature is greater than 70°F. The worker’s heart rate and blood pressure will be measured at the start of a rest break, and the work period will be decreased so that after 1 minute of rest, a worker’s heart rate does not exceed 110 beats per minute. In the field, heart rate can be monitored manually (i.e., index finger on pressure point while looking at a watch). The SSHO/Site Superintendent will assess the condition of the employees, specific weather conditions, work tasks, and any other environmental factors and conditions to determine when to begin monitoring. **Table 4** (MSU, 1999) provides general guidance for working in hot weather conditions.

Table 4. ACGIH TLV for Hot Environments/Suggested Hot Work-Rest Regimen

Work-Rest Regimen	Work Load		
	Light	Moderate	Heavy
Continuous	86°F	80°F	77°F
75% Work, 25% Rest each hour	87°F	82°F	78°F
50% Work, 50% Rest each hour	89°F	85°F	82°F
25% Work, 75% Rest each hour	90°F	88°F	86°F

If a worker's heart rate is greater than 110 beats per minute, the next work period will be shortened by 33 percent, while the length of the rest period stays the same. If the heart rate is 110 beats per minute at the beginning of the next rest period, the following work cycle will be shortened by 33 percent. When ambient temperatures are expected to exceed 75°F, the resting heart rate of each worker will be measured prior to the start of on-site activities.

Other factors, such as a worker's level of acclimation, level of physical fitness, and age, may increase or decrease his susceptibility to heat stress. Before assigning a task to an individual worker, these factors will be taken into account to ensure that the task will not endanger the worker's health.

If a heat-related illness is suspected or observed, the affected person will be moved to a cool or shaded area and given plenty of liquids. If symptoms of a heat stroke are observed, the victim will be cooled. Another worker will immediately call University Hospitals Portage Medical Center at (330) 297-0811 **(if the affected person is able to be transported)**, or 911 from a landline phone or mobile phone if an ambulance is needed.

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9.0 STANDARD OPERATING SAFETY PROCEDURES, ENGINEERING CONTROLS, AND WORK PRACTICES

The following applies to all ERT work performed at the site.

9.1 Site Rules/Prohibitions

Site entry will not be allowed until staff have read and acknowledged understanding of the APP and this SSHP and received required site-specific safety training. All work will be performed using the "buddy system," meaning that no work can be performed by a single employee or subcontractor working alone. Smoking is not permitted in work areas. Designated smoking areas will be established by the SSHO/Site Superintendent in an area that is free and clear of potential fire hazards. Cigarette butts will be collected and disposed of along with all trash at an appropriate receptacle near the work site. Eating is only permitted at designated break areas (usually where vehicles are parked). Drinking water will be made available to field staff.

9.2 Work Permit Requirements

Additionally, at least 72 hours prior to any intrusive field activities (e.g., digging with excavator), ERT will coordinate with local utility companies (via Ohio Utility Protection Services or 1-800-362-2764) to mark-out and identify all underground utilities in the investigation areas.

9.3 Material Handling Procedures/Hazard Communication (HAZCOM) Program

There is a possibility of fuel spills during equipment/vehicle refueling. Any spills no matter the size must be reported to Range Control. Fuel spills represent both a respiratory and fire hazard. Any refueling will be performed within a designated area, with spill kits or absorbent maintained on-site by the SSHO/Site Superintendent. Fuel will be stored only in U.S. Department of Transportation (DOT) compliant containers. A fuel spill kit or absorbent will be on hand when fueling of vehicles and/or equipment is taking place. In the event a spill occurs during refueling, every effort will be made to contain the spill and clean it up immediately. Spoils resulting from any spill will be disposed of in accordance with the requirements for that item. No cigarette smoking or open flames will be allowed within 50 feet of the refueling area.

SDSs are included as **Enclosure D** and will be available on-site for chemicals to be used. Employees and subcontractors will be informed by the SSHO/Site Superintendent of the location of SDSs and all chemical, physical, and biological hazards associated with site work. Currently, it is not expected that any other hazardous materials will be used for this project (outside of the SDSs presented in **Enclosure D**); however, if any other materials are brought onsite they will be properly labeled and the site workers will be made aware of the specific hazards.

All site workers will receive the following training during the initial startup of site operations:

- Requirements and use of the HAZCOM Program at the site;
- The location of all hazardous or toxic agents at the site;
- Identification and recognition of hazardous or toxic agents at the site;
- Physical and health hazards of the hazardous or toxic agents pertinent to site activities, and

- Protective measures employees can implement when working with site-specific hazardous or toxic agents.

The inventory listed below in **Table 5** constitutes hazardous substances that could be brought on-site. Fuel used for fueling power equipment that may be used during work activities will be stored in approved containers off-site.

Table 5. Inventory of Hazardous Chemicals

Chemical	MSDS on Site	Quantity On Site	Storage Location	Use on Site
Diesel Fuel[1]	Yes	NA	NA	To fuel equipment
Gasoline[1]	Yes	NA	NA	To fuel equipment

Legend:
MSDS = Material Safety Data Sheet
NA = not applicable
< = less than
[1] = Fuel will be brought to the site on an as-needed basis for refueling of equipment; no storage of fuel will occur.

Hazardous or Toxic Agent Labeling: EM-385-1-1 specifies that procedures for assuring that containers used to store and transport hazardous or toxic agents around the project site are appropriately labeled to communicate the physical and health hazards associated with the agents in the containers. Further, in accordance with 49 CFR 1910.1200(f), the chemical manufacturer, importer, or distributor must ensure that each container of hazardous chemicals leaving the workplace is labeled, tagged or marked with the following information: identity of the hazardous chemical(s); appropriate hazard warnings; and name and address of the chemical manufacturer, importer, or other responsible party.

For the items specified for use at the site, ERT will ensure that the required information is contained on the labeled chemicals as supplied by the manufacturer. Further, the SSHO will ensure through daily inspection that the labels remain affixed and legible. ERT has no plan to transport smaller quantities of these items around the site; however, in the event it becomes necessary, in accordance with 1910.1200(f), no labeling will be required because the SSHO will ensure that the amounts will only be for the immediate use of the employee who performs the transfer.

9.4 Drum/Container/Tank Handling

A minimal number of U.S. Department of Transportation Type H metal drums may be required for IDW. These will be stored on pallets at the in an area determined by ERT, USACE, and/or USEPA. Drummed IDW will be properly labeled with contents (i.e., decontamination fluids, soil cuttings, and/or drilling fluid/purge water) to ensure personnel awareness.

9.5 Comprehensive AHA of Treatment Technologies Employed at the Site

No treatment technologies are being employed at the site.

9.6 Site Control Measures

Site access will be coordinated among the CELRL COR/TM, and the ERT PM along with on-site Government personnel. Only personnel essential to the task at hand will be permitted within the safe working area. Field vehicles, used as mobile offices, support equipment, emergency supplies, and First Aid equipment will be located within 30 seconds of walking from the safe working area whenever possible.

9.7 Site Sanitation and Personal Hygiene

The work site will be kept as clean and neat as possible; regular cleaning shall be conducted in order to maintain safe and sanitary conditions with all work areas. An adequate supply of bottled drinking water will be provided, to be used for both drinking and personal cleansing, if needed. Field vehicles will be available to transport workers to nearby toilet facilities (as the site is located within an active military training center, toilet facilities will be available within a nearby building). Non-toxic hand soap or similar cleansing agents will also be available in site vehicles. Site personnel will be made aware that certain cleansing agents, such as hand sanitizers, may contain iodopropynyl butylcarbamate, which may cause allergic reactions to the skin.

9.8 Emergency Equipment and First Aid

Field crews will have cellular telephones as a means of communication to effectively care for injured workers. Emergency telephone numbers and a highly visible map delineating the best route to the nearest emergency medical facility, University Hospitals Portage Medical Center, Ravenna will be contained in each field vehicle. This figure (map) also appears on the back cover of this SSHP.

First Aid kits will be readily accessible to all workers and protected from the weather. The individual contents of the First Aid kit will be kept sterile. First Aid kit locations will be clearly marked and distributed throughout the site, as appropriate. The contents of a First Aid kit will be checked prior to each mobilization to ensure serviceability and completeness. The contents of the First Aid kit will, at a minimum, contain the items listed in Table 3-1, EM 385-1-1.

Additionally, an emergency eyewash station will be available for use. The eye wash station will be portable and American National Standards Institute Z358.1 compliant. The eyewash station will be located in close proximity to the work site for easy access. Site workers will be reminded daily of the location of the eyewash station.

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10.0 EQUIPMENT DECONTAMINATION

The ERT team will decontaminate the excavator at the conclusion of the excavation activities. The decontamination pad will consist of a 20-40 mil HDPE liner and plywood sheets. The ERT team will manually remove soils adhered to excavator and undercarriage of the excavator to a visually clean standard. All soils will be removed, brushed, and scraped from the machine. The ERT team does not anticipate having to power wash the machine; thereby not creating liquid waste.

Each truck will undergo a dry decontamination via brushing to remove soil from the tailgate and side-boards of the trailer that may have dropped during load-out. This will occur in the designated loading zone. The loading zone will consist of a 20-40 mil HDPE liner and woven geo-textile on which each trailer will be staged during loading. The ERT team’s laborer will brush the soil away from the tires of the trailer so that the excavated material does not come into contact with the tires as the truck pulls away. All residues will be collected so that the loading zone is clean to a visual standard before being occupied by the next trailer. Each truck will be covered with a tarp after loading and prior to leaving the site. The ERT team does not anticipate having to power wash the trucks; thereby not creating liquid waste.

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11.0 EMERGENCY ACTION PLAN AND CONTINGENCY PROCEDURES

Prior to work start-up, personnel will be made familiar with this Emergency Action Plan. The SSHO/Site Superintendent will make this plan available for inspection and copying by subcontractors and will review the location of evacuation areas exit routes and notification procedures. The rally point will be established at the pre-determined location at the site (see **Figure 2** of the APP). The SSHO/Site Superintendent will identify the location of the rally point and discuss evacuation procedures during daily safety meetings.

11.1 Emergency Incident Procedures

In the event of an emergency, the information available at that time will be properly evaluated and the appropriate steps will be taken to implement the Emergency Action Plan. The SSHO/Site Superintendent will assume command of the situation and will call the appropriate emergency services, evacuate personnel to the designated evacuation location as needed, and take other steps necessary to gain control over the emergency.

11.2 Emergency Notification Procedures

Field crews will utilize cellular telephones as a means of communications within the workforce and with the SSHO/Site Superintendent. The SSHO/Site Superintendent will notify site workers when the Emergency Action Plan is being implemented.

Emergency telephone numbers of the nearest hospital are provided in Section 11.4, **Table 6**, and on the back cover of this SSHP; a map showing the route and direction to the nearest hospital is provided in Section 10.6 and on the back cover of this SSHP. The field personnel will immediately stop work and report to the SSHO/Site Superintendent under the following potential emergency situations:

- Medical emergency;
- Discovery of unanticipated hazards (e.g., drums, heavily contaminated materials, etc.);
- Overexposure of personnel to on-site contamination; and
- Cold/heat-related injury or heat stress.

In a medical emergency, the SSHO/Site Superintendent will stop all work. Personnel currently trained in First Aid/CPR will evaluate the nature of the injury, decontaminate the victim if necessary and will not result in further injury, move the victim if necessary, and initiate First Aid assistance immediately. At least two personnel trained in First Aid and CPR will be on-site at all times. A First Aid kit will be located in each field vehicle.

For any injury requiring care beyond First Aid, the Range Control will be notified immediately; contact numbers are provided in **Table 6**. Victims who are heavily contaminated with toxic or dangerous materials will be decontaminated, only if decontamination will not result in further injury, before being transported from the site. If decontamination is not possible, emergency medical services will be informed that the victim(s) is contaminated and appropriate PPE is required. A fellow worker will accompany injured workers to the hospital to inform the admitting clerk that the injury is work related and to assist in completing the insurance forms.

The SSHO/Site Superintendent will complete an Engineer (ENG) Form 3394 Incident Report Form (included in **Enclosure B**) which will be submitted to the ERT PM and Division SHM within 24 hours (as forwarded to USACE within 5 days of the incident) of the following types of incidents:

- Job-related injuries and illnesses;
- Accidents resulting in significant property damage;
- Accidents involving vehicles and/or vessels; and/or
- Accidents in which there may have been no injury or property damage, but which have a high probability of recurring with at least a moderate risk to personnel or property

Work being performed at the time of the accident should be temporarily suspended and a preliminary investigation/assessment conducted to determine cause and ensure mitigation measures are employed to prevent reoccurrence prior to work resuming.

Accidents will be reported according to procedures described in Section 8.0 of the project APP.

Bloodborne pathogen (BBP) awareness is a subject of First Aid/CPR annual recertification and a subject taught in annual 8-hr HAZWOPER refresher training. As part of the ERT Corporate Safety and Health Program, it is recognized that employees trained in First Aid/CPR may be exposed to blood, and as such, ERT, via its corporate health and safety provider, has made voluntarily available the hepatitis B vaccine and vaccination series to all employees who may have an occupational exposure. ERT also makes available via its corporate healthcare provider post-exposure evaluation and follow-up to all employees who have had an exposure incident.

For this project, personnel will not be involved in activities reasonably expected to result in exposure to blood. Additionally, personnel will be wearing all appropriate protective equipment, including eye and hand protection. In the rare situation that First Aid needs to be administered, supplies present in the kits (e.g., gloves, barriers, etc.) will be sufficient to prevent exposure to any BBPs.

ERT personnel will not be providing medical assistance as a primary job duty at the site; however, these BBP exposure control procedures will be applicable to designated potential First Aid providers on this project. ERT personnel expected to administer First Aid must have a basic understanding of BBPs in order to protect themselves effectively from any hazards.

ERT field personnel, who possess current certification to do so, may find themselves in a rare situation where they must deliver First Aid and/or CPR in a nonclinical setting. First Aid/CPR duties are often performed in uncontrolled environments, which, due to a lack of time and other factors, do not allow for application of a complex decision-making process to the emergency at hand. Only minor injuries will be treated via First Aid, and only small volume of bodily fluids/soiled supplies will be cleaned up by the First Aid provider. Any injury requiring medical support beyond basic First Aid, however, will be handled in full to by emergency responders (to include cleanup of blood).

Via the provision of First Aid/CPR training to employees, ERT provides information on BBPs and the Occupational Exposure to BBPs Standard to all field personnel with special emphasis on those employees who may be certified and called upon to perform First Aid.

This training is designed to eliminate or minimize employee exposure to BBPs through information and training, engineering controls, administrative controls, use of PPE, safe handling procedures, decontamination, and proper disposal methods.

When treating a victim for an injury, conducting CPR, or handling potentially infectious waste, the use of universal precautions is the recommended approach to infection control. Universal precautions assume all human blood and certain human body fluids are infectious for Human Immunodeficiency Virus, Hepatitis B Virus, and other BBPs. Other body substances, including feces, urine, or vomit are not included, unless they contain visible blood. Under circumstances in which differentiation between body fluid types is difficult or impossible, all body fluids shall be considered potentially infectious materials. Work practice controls to minimize exposure to BBPs are as follows:

- Work practice controls reduce the likelihood of exposure by formalizing the manner in which a task is performed.
- All First Aid procedures involving blood or other potentially infectious materials shall be performed in a manner that minimizes splashing, spraying, spattering, and generation of droplets of these substances.
- Mouth suctioning of blood or other infectious materials is prohibited.
- When handling red bag waste, hold the top end of the bag rather than the bottom.
- Containers of potentially infectious waste should be labeled with a biohazard label.
- All PPE should be inspected prior to use. PPE should not be worn if the PPE barrier is compromised.
- Hands and other skin surfaces should be washed with soap immediately and thoroughly if contaminated with blood, other body fluids to which universal precautions apply, or their potentially contaminated articles. Hands should always be washed after gloves are removed even if the gloves appear intact.
- Where hand washing facilities are not readily accessible, antiseptic hand cleaner along with clean cloth/paper towels or antiseptic towelettes should be used. When antiseptic hand cleaners or towelettes are used hands shall be washed with soap and running water as soon as feasible.

Engineering controls isolate or remove the BBP hazard from the workplace:

- Proper containerizing, labeling, and disposal of contaminated items are required for all potentially infectious waste.
- Limiting access or close off areas which contain potentially infectious materials.
- Assessing each work area for potential sharp edge hazards, and covering them with a tarp (or equivalent).

Administrative controls reduce or eliminate BBP hazards from the workplace by program development, auditing to ensure these programs are in place and implemented and providing information and training.

Where BBP hazards cannot be mitigated via engineering controls or administrative controls, PPE should be utilized. Work gloves should be worn to protect the hands from potential cuts, scrapes, and/or abrasions.

For protection against BBPs, for an employee administering First Aid, minimum PPE would require nitrile gloves. For an employee administering CPR, a face shield should be employed to prevent possible exposure to blood in the mouth. First-aid kits will be supplemented with BBP kits or supplies and will be readily accessible at all times.

If the chance of being exposed to blood is high, the caregiver should put on protective attire before beginning CPR or First Aid. Protective barriers should be used in accordance with the level of exposure encountered.

Under rare or extraordinary circumstances, a responding employee may decide, based on his or her judgment, that use of PPE would prevent delivery of care or pose an increased hazard to safety of the employee or co-worker. When this judgment has been made, an investigation of the event will be initiated and documented in order to determine what changes in procedures or protective equipment is needed.

In the event that off-site medical care becomes necessary, the location of the closest local medical facility from the site is provided. Turn by turn directions are also included. In addition, although not anticipated, should work locations change significantly requiring updated hospital maps, ERT will generate new site maps including routes to the hospital from each work location. The maps will be reviewed with site personnel prior to beginning work as well as placed within the front cab of each field vehicle.

On-site emergencies may ultimately be handled by off-site emergency support personnel. Initial response and First Aid treatment will be available through on-site personnel. In case of a hazardous materials emergency, the SSHO/Site Superintendent will assume control and insure that the site remains clear of personnel until the arrival of off-site emergency personnel.

Emergency response services will be activated by Range Control from landline phone or from a mobile phone. Within RVAAP, there are designated medical transfer points (see map provided in **Enclosure E**). The closest medical transfer point to the project site is located at the intersection of Old Newton Falls Road and Paris Windham Road. University Hospitals Portage Medical Center will be able to provide the quickest response to an incident, and also ensure that off-site emergency support will be expedited as appropriate.

When possible, the following information will be provided when reporting an emergency:

1. Name and location of person reporting
2. Location of accident/incident (global positioning system coordinates, if available)
3. Name and affiliation of injured party
4. Description of injuries, fire, spill, or explosion
5. Status of medical aid and/or other emergency control efforts
6. Details of chemicals involved
7. Summary of accident, including suspected cause and time it occurred
8. Temporary control measures taken to minimize further risk

Once emergency response agencies have been notified, the ERT PM and CELRL COR/TM will be notified immediately.

11.3 Fire/Explosion Emergencies

In the unlikely event of a fire or explosion, the SSHO/Site Superintendent will immediately implement the Emergency Action Plan. Once at the predetermined evacuation location, the SSHO/Site Superintendent will:

- Account for all employees at the evacuation location;
- Notify local emergency services; a list of the contact numbers is provided in **Table 6**. The emergency contact phone numbers and map to local hospital that appear in Section 11.6 will be also kept on-site in each field vehicle; and
- Notify the ERT Division SHM and ERT PM and follow the guidance provided.

The SSHO/Site Superintendent will determine if the situation requires evacuation. If no evacuation is required, then personnel properly trained in fire suppression and other response procedures will attempt to deal with the situation. The SSHO/Site Superintendent and field crew will only perform the rescue and medical duties that each is trained and qualified to perform. All other necessary rescue and medical duties will be performed by qualified emergency personnel. Other than small fires or spills, local emergency response services will be notified to handle the emergency. The SSHO/Site Superintendent will take measures to reduce injury and illness, primarily by evacuating personnel as quickly as possible when necessary. Cleanup after such events may require specialized services. Work will not resume until the SSHO/Site Superintendent declares the incident closed.

11.4 Emergency Contacts

The following emergency telephone numbers will be posted in field vehicles:

Table 6. Emergency Contact Phone Numbers

Service/Contact	Agency/Position	Telephone No.
Emergency Services Dispatch (fire, medical, police)	Emergency	911 (from landline phone or mobile phone)
University Hospitals Portage Medical Center	Emergency Room	(330) 297-2850
University Hospitals Portage Medical Center	Non-emergency medical	(330) 297-0811 (non-emergency)
Spill Response	Chemical Transportation Emergency Center (CHEMTREC)	(800) 424-9300
U.S. Environmental Protection Agency (USEPA) National Response Center	24-hour hotline	(800) 424-8802
OEPA	Regulator (24-hour Emergency Hotline)	(800) 282-9378
Poison Control	Poison Control Center	(800) 222-1222
City of Twinsburg Animal Control	Animal Control	(330) 425-1234
Mark Leeper	ARNG Restoration Program Manager	(703) 607-7955
Katie Tait	OHARNG	(614) 336-6136

Table 6. Emergency Contact Phone Numbers

Service/Contact	**Agency/Position**	**Telephone No.**
Gail Harris	RVAAP AR	(330) 872-8003
Pat Ryan	REIMS	(865) 481-4664
Craig Coombs	CELRL PM	(502) 315-6324
Matt Burg	CELRL Chief of Safety	(502) 315-7061
Nat Peters	CELRL COR	(502)315-2624
Kevin Mieczkowski	CELRL TM	(502) 315-7447
Michael Barsa	ERT Division SHM	(301) 323-1447 (o) (410) 703-6213 (c)
Jennifer Harlan	ERT Division Manager	(301) 323-1394 (o)
Sean Carney	ERT PM	(607) 259-7017) (c)
JT Nolan	ERT SSHO/Site Superintendent/OSHA Competent Person for Overall Site Health and Safety	(301) 412-7301
Robert Koroncai	Alternate ERT SSHO/Site Superintendent/OSHA Competent Person for Overall Site Health and Safety	(301) 323-1445 (o) (267) 481-5567 (c)

11.6 Hospital Routes

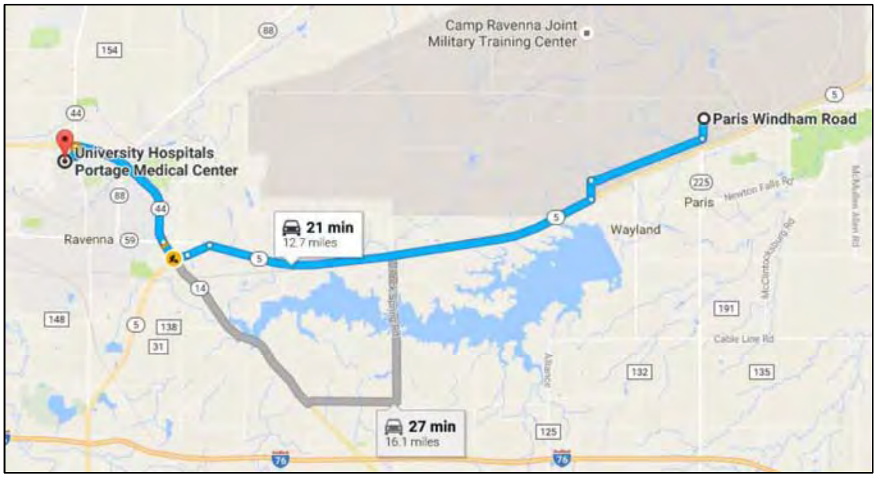
The nearest emergency hospital to the site is University Hospitals Portage Medical Center. The nearest Level I trauma center is Akron General Medical Center located at 1 Akron General Avenue, Akron, OH 44307 which is approximately a 45-minute drive from the site.

In the event that a patient requires care beyond the capabilities of University Hospitals Portage Medical Center, emergency services will dispatch a helicopter to transport the patient to Akron General Medical Center, Akron, OH. Helicopter dispatch can either happen at the scene of the accident, or at University Hospitals Portage Medical Center (should a patient be transported there first). The need for helicopter dispatch will be made by emergency medical services. In the event emergency medical attention is needed, site workers should activate emergency medical services and allow them to assess the level of care needed.

Figure 2 of the APP shows routes from each work area to various rally points, leading to the evacuation route off-site. The route from the site to University Hospitals Portage Medical Center, Ravenna is shown in **Figure 11** and on the back cover of this SSHP. The turn-by-turn directions are provided below. Turn-by-turn directions (including street names and distances) will be verified by driving the route prior to beginning field work. If necessary, pen and ink changes to this SSHP can be made to copies being used in the field.

University Hospitals Portage Medical Center
6847 N Chestnut St,
Ravenna, OH 44266
Phone number: (330) 297-0811

University Hospitals Portage Medical Center
6847 N Chestnut St,
Ravenna, OH 44266
Phone number: (330) 297-0811



Directions:

From Camp Ravenna, Ohio

1. Head south on Paris Windham Rd., 0.3 mi

2. Turn Right onto first cross street, 1.9 mi

3. Turn Right onto Pa St. to exit the Installation via George St. 1, 0.3 mi

4. Turn Right onto OH-5 W, 6.4 mi

5. Turn Left onto OH-5W/OH-5 Bypass, 0.4 mi

6. Use Right lane to take OH-14 ramp, 0.2 mi

7. Turn Right onto OH-14 W/OH-44 N/Cleveland – East Liverpool Rd., 2.4 mi

8. Arrive at University Hospitals Portage Medical Center, 0.2 mi

Distance from site to University Hospitals Portage Medical Center = 12.7 miles (21 minutes)

Figure 12. Hospital (Emergency) Directions

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12.0 REFERENCES

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ENCLOSURE A

Safety Personnel Resumes

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14401 Sweitzer Lane, Suite 300
Laurel, MD 20707
Phone: 301-361-0620
Fax: 301-361-0659
www.ERTCorp.com

Michael W. Barsa, Certified Safety Professional
Division Safety and Health Manager – ERT, Inc.

Education	Summary of Qualifications and Experience
B.S. Environmental Science and Policy, University of Maryland College Park, 2004	▪ 12 years of environmental H&S experience overseeing field sampling, chemical analysis, chemical/hazardous waste remediation and subcontractor H&S on federal, state, and private sector projects. ▪ 10 years of total experience with H&S roles on various environmental and construction field projects, with over 4 years being direct experience participating in field efforts. ▪ 6 years of total experience assisting with management of and/or managing Division Safety and Health program. ▪ 10 years of experience performing environmental investigation and remediation through Resource Conservation and Recovery Act (RCRA)/CERCLA/National Contingency Plan (NCP) and Military Munitions Response Program (MMRP) support. ▪ 11 years of experience working with nine USACE districts over three USACE divisions. ▪ Experience with 2 USEPA regions.

Professional Registrations/Training

- Certified Safety Professional; Board of Certified Safety Professionals: 24437
- OSHA 40-Hour HAZWOPER Training (2004) and 8-hr Annual Refresher (current through January 2017)
- OSHA 8-Hour HAZWOPER Supervisor Training (2010)
- OSHA 30-Hour Construction Safety Training; May 2009
- Adult CPR and First Aid; January 2014 (current through January 2018)
- Bloodborne Pathogens Standard Training, January 2016
- Fleet Safety Management; OSHAcademy; May 2013
- Fire Prevention Plans; OSHAcademy; May 2013
- Emergency Action Plans; OSHAcademy; May 2013
- Electrical Safety Basics; OSHAcademy; May 2013
- Fall Protection Program; OSHAcademy; May 2013
- Energy Control Program-Lockout/Tagout; OSHAcademy; May 2013
- Effective Safety Committee Meetings; OSHAcademy; May 2013
- Ergonomics Program Management; OSHAcademy; May 2013
- Preventing Workplace Violence; OSHAcademy; May 2013
- Confined Space Program; OSHAcademy; May 2013

- Safety Supervision and Leadership; OSHAcademy; May 2013
- OSHA 300 Recordkeeping; OSHAcademy; May 2013
- Personal Protective Equipment; OSHAcademy; May 2013
- Introduction to Industrial Hygiene; OSHAcademy; May 2013
- Conducting Occupational Safety and Health Training; OSHAcademy; May 2013
- Developing Occupational Safety and Health Training; OSHAcademy; May 2013
- Safety Management System Evaluation; OSHAcademy; May 2013
- Introduction to Ergonomics; OSHAcademy; May 2013
- Conducting a Job Hazard Analysis; OSHAcademy; May 2013
- Hazard Communication Program; OSHAcademy; May 2013
- Hazard Analysis and Control; OSHAcademy; May 2013
- Introduction to Occupational Safety and Health Training; OSHAcademy; May 2013
- Effective Accident Investigation; OSHAcademy; May 2013
- Bloodborne Pathogens Program Management; OSHAcademy; May 2013
- Effective Safety Committee Operations; OSHAcademy; May 2013
- Introduction to Safety Management; OSHAcademy; May 2013
- Exposure Assessments Strategies and Statistics, American Industrial Hygiene Association; January 2013
- Slips, Trips, Falls Training; ERT; October 2012
- Battery Handling Training; ERT; October 2012
- Biological Hazard Training; ERT; October 2012
- Hearing Conservation Training; ERT; January 2014
- Fire Extinguisher Training; ERT; January 2014
- Confined Space Operations Training; November 2010
- Sampling for Defensible Environmental Decisions; EnviroStat, Inc.; February 2009
- Respiratory Protection Training; Cabrera Services, Inc.; January 2007
- Introduction to the Incident Command System (ICS) for Public Works Personnel; Federal Emergency Management Agency (FEMA) Independent Study Program; May 2006
- Incident Command System (ICS) for Single Resources and Initial Action Incidents; FEMA Independent Study Program; May 2006
- Radiological Emergency Management; FEMA Independent Study Program; May 2006
- Radiological Emergency Response; FEMA Independent Study Program; May 2006
- Radiation Site Worker Training; Cabrera Services, Inc.; June 2006 through July 2010
- Construction Quality Management for Contractors, U.S. Army Corps of Engineers; March 2007 through March 2012
- Department of Transportation/International Air Transport Association General Awareness and Function Specific Training and Testing; Cabrera Services, Inc.; January 2008 through December 2009

Experience Record

Mr. Barsa has 11 years of experience performing remedial investigation and remedial action work for both federal, state, and private sector clients, with 10 years being involved in environmental/construction health and safety (H&S) roles (4 years of which being directly involved in field efforts). He has served as Site Safety and Health Officer (SSHO)/Safety Team Lead/Project Health and Safety (H&S) Officer for multiple remedial investigation and remedial action sites regulated under statutes governed by CERCLA, NYSDEC, MDE, NJDEP, and PADEP.

Mr. Barsa has served in safety roles, cognizant of project-specific H&S issues for multiple hazardous, toxic, and radioactive waste (HTRW) remedial investigations and remedial action sites regulated under statutes governed by the U.S. Environmental Protection Agency (USEPA), Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA), and various state-and-locally-governed environmental agencies. Mr. Barsa has also served as SSHO on numerous construction projects, particularly with regards to drilling and/or excavating with heavy machinery. With his experience enforcing project-specific health and safety (H&S) standards on numerous environmental regulatory compliance, environmental site assessments, remedial investigations, and remedial action projects Mr. Barsa demonstrates his competence and proficiency as an H&S technical leader. His H&S experience includes authoring/reviewing project H&S plans, managing project specific H&S controls, managing subcontractor activities and appropriate equipment usage in accordance with project H&S plans, ensuring the performance of project tasks in accordance with project H&S plans, activity hazard analyses (AHAs), and H&S-specific standard operating procedures (SOPs).

Mr. Barsa has experience with implementing/managing the selection and proper usage of personal protective equipment (PPE), and several other articles of H&S equipment, including steel-toed shoes, safety glasses, hearing protection, respirators (both supplied and air purifying), and hard hats. He also has experience with management of on-site workers via site control log, monthly exposure reports, conducting daily H&S briefings, managing H&S training for subcontractors, performing H&S air monitoring via photoionization detector (PID)/organic vapor monitor (OVM), ambient air temperature monitoring for heat and cold stress, and ensuring all site workers were familiar with First Aid kits and routes to hospital. Involvement in the various projects has included participation in project design, execution and management of field operations (with regards to H&S), and providing H&S oversight of construction and remediation contractors, as well as performing field audits.

He assisted with Division Safety and Health program management, working under numerous Certified Industrial Hygienists and Certified Safety Professionals for 4 years before assuming the role of Division Safety and Health Manager. He has served in the role of Division Safety and Health Manager for 2 years ensuring employee compliance with ERT Corporate Health and Safety Manual, 29 CFR1910.120 (Hazardous Waste Operations and Emergency Response [HAZWOPER], Occupational Safety and Health Administration (OSHA) Standards for General Construction, and EM 385-1-1 U.S. Army Corps of Engineers (USACE) Safety and Health Requirements Manual.

Jesse T. Nolan, Site Safety and Health Officer/Site Superintendent

Education:

- B.S., 2006, Environmental Science, University of Maryland, College Park, MD

Professional Registration/Certification(s):

- OSHA 40-Hour Hazardous Waste Operations, February 2006
- OSHA 8-Hour HAZWOPER Supervisor Refresher Training, May 2016
- OSHA 30-Hour Construction Safety, February 2012
- OSHA Fall Protection for Competent Person, November 2014
- OSHA Excavation and Trenching for Competent Person, January 2016
- VA Responsible Land Disturber, October 2015
- Adult First Aid and CPR/AED, September 2016 (current through September 2018)
- Confined Space Entry Training, 2011
- U.S. Army Corps of Engineers Construction Quality Management for Contractors, January 2011, April 2016

Project Construction Safety Experience:

Project Name and Location: Yorktown FISC Demo Cut and Cover Tanks – Yorktown, Virginia

Project Date: August 2015 – Current (12 months safety experience)

***Position title, Relevant Duties/Responsibilities*:**

As the Project Superintendent, Mr. Nolan provided onsite management of staff and subcontractors involved in the demolition of (6) 1.1 million-gallon underground fuel tanks. Key tasks completed in association with tank demolitions included abatement and removal of all associated asbestos coated pipeline, Installation of new firewater lines, construction of 21,000 square feet of new paved roadway, management of contaminated soil and tank/pipeline fluids, and final grading and restoration. Mr. Nolan served as Project Superintendent during this project, but was ultimately responsible for the health and safety of all field personnel and subcontractors involved in every aspect of the project. During this project, Mr. Nolan focuses approximately 80 percent of his time (12 months safety experience) as the Project Superintendent.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site meetings and supplementing daily H&S pre-work meeting, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations are performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. Assisted the SSHO in ensuring that all subcontractors and staff have obtained adequate safety training and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included respirators, nitrile gloves, safety-toed shoes, safety glasses, hard hat, hearing protection, and task-specific gloves.

Project Name and Location: Atlantic Wood Industries, Western Berm Extension – Portsmouth, Virginia

Project Date: February 2015 – November 2015 (5 months safety experience)

***Position title, Relevant Duties/Responsibilities*:**

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff and subcontractors involved in the construction of a 300 foot earth berm built as an addition to an existing containment berm at the Atlantic Wood Superfund Site. Construction of the core of the berm involved the use of onsite impacted soil. Site soil was impacted by creosote and pentachlorophenol (PCP). Mr. Nolan performed daily equipment safety inspections and site safety oversight for field personnel and subcontractors involved in every aspect of the project. During this project, Mr. Nolan focuses approximately 50 percent of his time (5 months safety experience) as the Project H&S Officer.

Jesse T. Nolan, Site Safety and Health Officer/Site Superintendent

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations are performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensures that all subcontractors and staff have obtained adequate safety training and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included respirators, nitrile gloves, safety-toed shoes, safety glasses, hard hat, hearing protection, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:
- conducting daily H&S meetings
- management of site workers via site control log
- briefing site personnel on recognition of hazards in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)

Implementation of applicable PPE included:
- ensuring First Aid kits were readily available to all site personnel
- monitoring areas for noise and provided hearing protection as necessary
- ensuring appropriate PPE selected for hazardous tasks (i.e., respirator cartridge selection)

Equipment and Work Area Inspections included:
- ensuring the equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, and mechanical components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer’s guidelines
- ensuring equipment and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of equipment and ensured that proper fire extinguisher was present.

H&S monitoring of the work area included:
- Personnel were using proper PPE and used properly.
- Marking areas and advising personnel of exclusion zones.
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via photoionization detector (PID)/organic vapor monitor (OVM)
- monitoring the weather and advised site personnel of impending inclement weather

monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Portsmouth Public School District Maintenance Facility Demolition – Portsmouth, Virginia

Project Date: September 2014 – February 2015 (3 months safety experience)

***Position title, Relevant Duties/Responsibilities*:**

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff and subcontractors conducting demolition of 10 buildings on the facility. Activities conducted in conjunction with the building demolitions included abatement of asbestos containing materials, inspection and removal of all hazardous materials, removal of multiple underground petroleum storage tanks, and cut & caps of all utilities servicing the facility. Mr. Nolan performed daily equipment safety inspections and site safety oversight for field personnel and subcontractors involved in every aspect of the project. During this project, Mr. Nolan focuses approximately 50 percent of his time (3 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations are

Jesse T. Nolan, Site Safety and Health Officer/Site Superintendent

performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensures that all subcontractors and staff have obtained adequate safety training (i.e., Asbestos Awareness, hazardous material handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included tyvek suits, respirators, nitrile gloves, safety-toed shoes, safety glasses, hearing protection, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- briefing site personnel on recognition of hazards in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- monitoring areas for noise and provided hearing protection as necessary
- ensuring appropriate PPE selected for hazardous tasks (i.e., respirator cartridge selection)

Equipment and Work Area Inspections included:

- ensuring the equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, and mechanical components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring equipment and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of equipment and ensured that proper fire extinguisher was present.

H&S monitoring of the work area included:

- Personnel were using proper PPE and used properly.
- Marking areas and advising personnel of exclusion zones.
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via photoionization detector (PID)/organic vapor monitor (OVM)
- monitoring the weather and advised site personnel of impending inclement weather

monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: West Virginia Ordinance Works – Point Pleasant, West Virginia

Project Date: February 2014 – Current (6 months safety experience)

***Position title, Relevant Duties/Responsibilities*:**

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing operation and maintenance of two USACE groundwater treatment plants at the 2,700-acre site. Treatment plants process over 300,000 gallons per day of groundwater impacted with nitroaromatic compounds from former TNT manufacturing operations conducted at the site. Mr. Nolan performed daily equipment safety inspections and site safety oversight for field personnel conducting groundwater sampling from a network of over 75 groundwater monitoring wells. During this ongoing project, Mr. Nolan focuses approximately 20 percent of his time (6 months safety experience) as the Project H&S Officer.

Jesse T. Nolan, Site Safety and Health Officer/Site Superintendent

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations are performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensures that all subcontractors and staff have obtained adequate safety awareness training (i.e., water treatment system operations, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:
- conducting daily H&S meetings
- management of site workers via site control log
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:
- ensuring First Aid kits were readily available to all site personnel
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, Tyvek, etc.) when needed;

Equipment and Work Area Inspections included:
- ensuring the equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, and mechanical components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring equipment and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of equipment and ensured that proper fire extinguisher was present.

H&S monitoring of the work area included:
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via photoionization detector (PID)/organic vapor monitor (OVM)
- monitoring the weather and advised site personnel of impending inclement weather

monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Waste Disposal Cell Installation and Management – Atlantic Wood Industries, Portsmouth, Virginia

Project Date: January 2013 – June 2013 (3 months safety experience)

***Position title, Relevant Duties/Responsibilities*:**

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing installation and management of a waste disposal cell in Portsmouth, Virginia. Mr. Nolan performed daily equipment safety inspections and site safety oversight for field crews constructing waste cell facility using heavy equipment (excavators, end-dumps, bulldozers, skid-loaders, etc.). On this project, Mr. Nolan focused 50 percent of his time (3 months safety

Jesse T. Nolan, Site Safety and Health Officer/Site Superintendent

experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the drill rig and associated equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment, augers, rods, and tools were properly secured during transport
- ensuring safe movement of drill rig (i.e., mast down, level foundation, use of a guide)
- ensuring drill rig and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of drill rig, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring soil stability/proper grading/proper base/use of outriggers when setting drill rig
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the drill rig

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. DPT rods and sampler, etc.)
- ensuring proper handling and transport of DPT rods and sampler
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via photoionization detector (PID)/organic vapor monitor (OVM)

- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Environmental Site Investigation – Arlington National Cemetery, Arlington, Virginia

Project Date: October – November 2012 and September – October 2013 (2 months safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing installation and sampling of groundwater monitoring wells in Arlington, Virginia. Mr. Nolan performed daily equipment safety inspections and site safety oversight for field crews installing and sampling groundwater monitoring wells. On this project, Mr. Nolan focused 50 percent of his time (2 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the drill rig and associated equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment, augers, rods, and tools were properly secured during transport
- ensuring safe movement of drill rig (i.e., mast down, level foundation, use of a guide)
- ensuring drill rig and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of drill rig, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring soil stability/proper grading/proper base/use of outriggers when setting drill rig
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the drill rig

H&S monitoring of the work area included:

Jesse T. Nolan, Site Safety and Health Officer/Site Superintendent

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. DPT rods and sampler, etc.)
- ensuring proper handling and transport of DPT rods and sampler
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Avtex Fibers Superfund Site– Extraction Well Installation – Front Royal, Virginia

Project Date: June – August 2013, November – January 2014 (3 months safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing installation of extraction wells via hollow stem auger(HSA) drilling methods in Front Royal, Virginia. On this project, Mr. Nolan focused 50 percent of his time (3 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the drill rig and associated equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment, augers, rods, and tools were properly secured during transport
- ensuring safe movement of drill rig (i.e., mast down, level foundation, use of a guide)
- ensuring drill rig and other vehicles backed up safely (i.e. use of a guide/flag)

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- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of drill rig, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring soil stability/proper grading/proper base/use of outriggers when setting drill rig
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the drill rig

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. HSA rods and sampler, etc.)
- ensuring proper handling and transport of HSA augers and sampler
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Rip Rap Excavation and Disposal – Craney Island Facility, Portsmouth, Virginia

Project Date: December 2012 - January 2013 (1 month safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing the excavation and removal of petroleum impacted material using track excavators and other heavy construction equipment in Portsmouth, Virginia. On this project, Mr. Nolan focused 50 percent of his time (1 month safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the drill rig and associated equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment, augers, rods, and tools were properly secured during transport
- ensuring safe movement of drill rig (i.e., mast down, level foundation, use of a guide)
- ensuring drill rig and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of drill rig, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring soil stability/proper grading/proper base/use of outriggers when setting drill rig
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the drill rig

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. DPT rods and sampler, etc.)
- ensuring proper handling and transport of DPT rods and sampler
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Interim Removal Action, Area I – Former Frankford Arsenal, Philadelphia, Pennsylvania

Project Date: May – June 2012 (1 month safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing the excavation and disposal of contaminated soil via mechanized heavy construction equipment in Philadelphia, PA. On this project, Mr. Nolan focused 50 percent of his time (1 month safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors

- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the mechanized heavy construction equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all mechanized heavy construction equipment was properly secured during transport
- ensuring safe movement of mechanized heavy construction equipment (i.e., mast down, level foundation, use of a guide)
- ensuring mechanized heavy construction equipment and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of mechanized heavy construction equipment, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the mechanized heavy construction equipment

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. mechanized heavy construction equipment, etc.)
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Jet Fuel and Fuel Oil Pipeline Removal – Former Floyd Bennett Field, Naval Air Station, Brooklyn, New York

Project Date: September – October 2010 and August – September 2011 (2 months safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing the excavation and disposal of contaminated soil via mechanized heavy construction equipment in Philadelphia, PA. On this project, Mr. Nolan focused 50 percent of his time (2 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:
- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:
- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:
- ensuring the mechanized heavy construction equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer’s guidelines
- ensuring all mechanized heavy construction equipment was properly secured during transport
- ensuring safe movement of mechanized heavy construction equipment (i.e., mast down, level foundation, use of a guide)
- ensuring mechanized heavy construction equipment and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of mechanized heavy construction equipment, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the mechanized heavy construction equipment

H&S monitoring of the work area included:
- ensuring site personnel kept safe distance from mechanized moving parts (i.e. mechanized heavy construction equipment, etc.)
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Jesse T. Nolan, Site Safety and Health Officer/Site Superintendent

Project Name and Location: RW-06 Remediation – Kirtland Air Force Base, Albuquerque, New Mexico

Project Date: February – May 2011 (2 months safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing the final removal action of residual waste at the site via mechanized heavy construction equipment in Albuquerque, New Mexico. On this project, Mr. Nolan focused 50 percent of his time (2 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the mechanized heavy construction equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer’s guidelines
- ensuring all mechanized heavy construction equipment was properly secured during transport
- ensuring safe movement of mechanized heavy construction equipment (i.e., mast down, level foundation, use of a guide)
- ensuring mechanized heavy construction equipment and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of mechanized heavy construction equipment, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the mechanized heavy construction equipment

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. mechanized heavy construction equipment, etc.)

- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed
-

Project Name and Location: SM-1A Reactor Survey – Ft. Greely Army Base, Delta Junction, Alaska

Project Date: July – August 2011 (3 month safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing environmental sampling via DPT and HSA drilling methods in Delta Junction, Alaska. On this project, Mr. Nolan focused 50 percent of his time (1 month safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the drill rigs were routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer’s guidelines
- ensuring all drill rig equipment was properly secured during transport
- ensuring safe movement of drill rig equipment (i.e., mast down, level foundation, use of a guide)
- ensuring drill rigs and other vehicles backed up safely (i.e. use of a guide/flag)

- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of drill rigs, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the drill rigs

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. drill rigs, etc.)
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Linde/Praxair Formerly Used Site Remedial Action Program (FUSRAP) CSX Property Investigation – Tonawanda, New York

Project Date: October 2010 – January 2011 and November – December 2011 (3 months safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing an investigation of potential contamination and environmental sampling via DPT and HSA drilling methods in Tonawanda, New York. On this project, Mr. Nolan focused 50 percent of his time (3 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the drill rigs were routinely inspected and maintained in a proper functioning condition (i.e.,

locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all drill rig equipment was properly secured during transport
- ensuring safe movement of drill rig equipment (i.e., mast down, level foundation, use of a guide)
- ensuring drill rigs and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of drill rigs, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the drill rigs

H&S monitoring of the work area included:
- ensuring site personnel kept safe distance from mechanized moving parts (i.e. drill rigs, etc.)
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Patapsco Waste Water Treatment Plant ENR Construction – Baltimore, Maryland

Project Date: November 2009 – September 2010 (6 months safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing an investigation of potential contamination and environmental sampling to delineate areas for removal and disposal via DPT and HSA drilling methods in Baltimore, Maryland. On this project, Mr. Nolan focused 50 percent of his time (6 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:
- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:
- ensuring First Aid kits were readily available to all site personnel

- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the drill rigs and mechanized heavy equipment were routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all drill rigs and mechanized heavy equipment was properly secured during transport
- ensuring safe movement of drill rigs and mechanized heavy equipment (i.e., mast down, level foundation, use of a guide)
- ensuring drill rigs, mechanized heavy equipment, and vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of drill rigs and mechanized heavy equipment, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the drill rigs and mechanized heavy equipment

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. drill rigs, mechanized heavy equipment, etc.)
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Silver Spring Metro Center – Sediment Control Pond – Silver Spring, Maryland

Project Date: July – October 2009 (2 months safety experience)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing the excavation and installation of sediment control ponds in petroleum impacted soils via mechanized heavy equipment (track excavator, bull dozer, etc.) in Silver Spring, Maryland. On this project, Mr. Nolan focused 50 percent of his time (2 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the mechanized heavy equipment were routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all mechanized heavy equipment was properly secured during transport
- ensuring safe movement of mechanized heavy equipment (i.e., mast down, level foundation, use of a guide)
- ensuring mechanized heavy equipment and vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of mechanized heavy equipment, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the mechanized heavy equipment

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. mechanized heavy equipment, etc.)
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Project Name and Location: Zurich Fuel Release – Baltimore, Maryland

Project Date: May 2008 – June 2009 (6 months)

Position title, Relevant Duties/Responsibilities:

As the Project H&S Officer, Mr. Nolan provided H&S oversight of staff performing the installation of extraction wells and a dual phase vapor extraction (DPVE) system via air rotary and HSA drilling methods in Baltimore, Maryland. On this project, Mr. Nolan focused 50 percent of his time (6 months safety experience) as the Project H&S Officer.

Safety tasks performed by Mr. Nolan included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all

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subcontractors and staff had obtained adequate safety awareness training (i.e., drill rig, chemical handling, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Project H&S Officer, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, kevlar, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the drill rigs were routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all drill rigs were properly secured during transport
- ensuring safe movement of drill rigs (i.e., mast down, level foundation, use of a guide)
- ensuring drill rigs and vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of drill rigs, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the drill rigs

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts (i.e. drill rigs, etc.)
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

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Education

- B.S., GeoEnvironmental Studies, Shippensburg University, 2010
- Graduate Coursework, GeoEnvironmental Studies, Shippensburg University, 2011-2013

Professional Registrations/Training

- OSHA 40-Hr HAZWOPER Training (2013)
- OSHA 8-Hr HAZWOPER Refresher Training (current through December 2016)
- HAZWOPER 8-Hr Site Supervisor Training, January 2015
- OSHA 30-Hr Construction Safety & Health Training, January 2015
- First Aid/CPR/AED Training; January 2016 (current through January 2018)
- Bloodborne Pathogen Training; January 2015

Profile

Mr. Koroncai has served as a Team Safety Lead, cognizant of project-specific Health and Safety (H&S) issues for multiple hazardous, toxic, and radioactive waste (HTRW) remedial investigations and remedial action sites regulated under statutes governed by the U.S. Environmental Protection Agency, Comprehensive Environmental Response, Compensation, and Liability Act, and various state-and-locally-governed environmental agencies. With his almost five years of experience enforcing project-specific environmental health and safety (H&S) standards on numerous environmental regulatory compliance, environmental site assessments, remedial investigations, remedial action projects, and other various work sites Mr. Koroncai demonstrates his competence and proficiency as an H&S technical leader. His H&S experience includes reviewing/enforcing project H&S plans, managing project specific H&S controls, managing subcontractor activities and appropriate equipment usage in accordance with project H&S plans, ensuring the performance of project tasks in accordance with project H&S plans, activity hazard analyses (AHAs), and H&S-specific standard operating procedures (SOPs).

Mr. Koroncai has experience with implementing/managing the selection and proper usage of personal protective equipment (PPE), including steel-toed shoes, safety glasses, hearing protection, respirators (air purifying) and hard hats. He also has experience with keeping track of on-site workers via site control log, conducting daily H&S briefings, ensuring H&S training for subcontractors, performing H&S air monitoring via photoionization detector (PID)/organic vapor monitor (OVM), ambient air temperature monitoring for heat and cold stress, and ensuring all site workers were familiar with First Aid kits and routes to hospital.

Experience and Qualifications

Employer: *ERT, Inc.*	***Client/Project Name:*** *U.S. Army Corps of Engineers (USACE) Baltimore District* *Frankford Arsenal*
Safety Experience: *March 2014 to September 2014 (4 months safety experience)*	

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As the Field Team Lead and Team Safety Lead working under the SSHO, Mr. Koroncai assisted with H&S oversight of staff performing direct-push technology (DPT) and Hollow Stem Augur (HSA) soil characterization and monitoring well installation, as well as groundwater sampling. On this project, Mr. Koroncai focused 50 percent of his time (4 months total safety experience) towards H&S.

Mr. Koroncai was responsible for assisting with evaluating and implementing project-specific H&S standards (i.e., daily inspections and regular maintenance prior to use) during use of a DPT rig and HSA drill rig. Safety tasks performed by Mr. Koroncai included assistance with conducting daily pre-work site H&S briefing, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff utilized the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

Staff training and topics of daily H&S briefings included:

- management of site workers via site control log
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, etc.) when needed;

Equipment and work area inspections included:

- ensuring the DPT and HSA drill rigs and associated equipment were routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer’s guidelines
- ensuring all equipment, augers, rods, and tools were properly secured during transport
- ensuring safe movement of DPT and HSA drill rigs (i.e., mast down, level foundation, use of a guide)
- ensuring DPT and HSA drill rigs, and other vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of DPT and HSA drill rigs, ensured that proper fire extinguisher was present, and

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that fuel was stored appropriately
- ensuring soil stability/proper grading/proper base/use of outriggers when setting DPT and HSA drill rigs
- ensuring that buried and overhead utilities in the vicinity of the drilling locations were properly marked out prior to utilization of the DPT and HSA drill rigs

H&S monitoring of the work area included:
- ensuring site personnel kept safe distance from mechanized moving parts (i.e. DPT and HSA rods and sampler, etc.)
- ensuring proper handling and transport of DPT and HSA rods and samplers
- ensuring site personnel handled drums properly (i.e., volatile materials, bulging drums, compromised integrity)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather, including use of lightning meter
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Employer: *ERT, Inc.*	***Client/Project Name:*** *U.S. Army Corps of Engineers (USACE) Louisville District, Former Scioto Ordnance Plant*
Safety Experience: *December 2014 (0.5 month safety experience)*	

As a member of the Field Team working under the SSHO, Mr. Koroncai assisted with oversight of staff performing Digital Geophysical Mapping of 15 scared/disturbed areas at the Former Scioto Ordnance Plant. On this project, Mr. Koroncai focused 50 percent of his time (0.5 months safety experience) towards environmental H&S.

Mr. Koroncai helped to evaluate and implement project-specific H&S standards (i.e., daily inspections and regular maintenance of equipment prior to use). Safety tasks performed by Mr. Koroncai included assisting conducting daily pre-work site H&S briefing, keeping track of site personnel exposure, and ensuring all operations were performed in accordance with project H&S plans, AHAs, and H&S-specific SOPs. He helped to ensure that all subcontractors and staff employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included level D items, including but not limited to; safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

Staff training and topics of daily H&S briefings included:
- briefing site personnel on recognition factors of potential MEC in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e.

insects, animals)
- ensuring personnel were cognizant of exposure to weather.

Implementation of applicable PPE included:
- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., insulated, leather, etc.) when needed;

Equipment and work area inspections included:
- operating and maintaining equipment in accordance with manufacturer’s guidelines
- ensuring all equipment, and tools were properly secured during transport
- ensuring all vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity or on the property of the school, ensured that properly inspected fire extinguishers were present, and that fuel was stored appropriately

H&S monitoring of the work area included:
- ensuring site personnel handled environmental samples properly (double or triple bagged based on suspected contaminant)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Employer: *ERT, Inc.* ***Safety Experience:*** *December 2014 (0.5 Month)*	***Client/Project Name:*** *USACE, Louisville District, Savanna Army Depot*

As the Environmental Team Lead and Team Safety Lead, Mr. Koroncai assisted with oversight of staff performing Land Use Control Implementation for 21 Lower Post and Plant Area Sites (part of the Savanna Army Depot Activity site). H&S tasks included enforcing the abbreviated Accident Prevention Plan (AAPP), performing oversight to ensure subcontractor compliance with the AAPP, and perform other duties, which included: keeping track of site workers via site control log, conducting daily health and safety briefings, ambient air temperature monitoring, ensuring proper use of equipment, ensuring correct personal protective equipment is being used, ensuring all site workers were familiar with First Aid kits and routes to hospital. On this project, Mr. Koroncai focused 90 percent of his time 0.5 months safety experience) towards H&S tasks.

Mr. Koroncai was responsible for evaluating and implementing project-specific H&S standards. Safety tasks performed by Mr. Koroncai included conducting daily pre-work site H&S and daily

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tailgate briefings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training, and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE included safety-toed shoes, safety glasses, and task-specific gloves.

H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and topics of daily H&S briefings included:
- conducting daily H&S briefings
- management of site workers via site control log
- managing H&S training for subcontractors
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals, and passerby people)
- ensuring personnel were cognizant of exposure to the weather.

Implementation of applicable PPE included:
- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- providing proper gloves (i.e., insulated, etc.) when needed;

H&S monitoring of the work area included:
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Employer: *ERT, Inc.*	***Client/Project Name:*** *CB&I Federal Services, Wright-Patterson Air Force Base, Ohio*
Safety Experience: *October 2014 (1 month safety experience)*	

As the Geophysical Team Lead and Team Safety Lead working under the SSHO, Mr. Koroncai provided H&S oversight of staff performing geophysical activities associated with a munitions response site on the active Wright-Patterson Air Force Base airfield. H&S tasks included ensuring site worker safety around air traffic and heavy equipment (i.e., mini-excavator,) around excavated trenches with potential unexploded ordnance (UXO) hazards; participation in daily site safety briefings. On this project, Mr. Koroncai focused 90 percent of his time (1 month safety experience) towards H&S.

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Mr. Koroncai was responsible for evaluating and implementing project-specific H&S standards. Safety tasks performed by Mr. Koroncai included assistance with conducting daily pre-work site H&S and daily tailgate briefings, keeping track of site personnel via site control log, and ensuring all staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all staff had obtained adequate safety awareness training (i.e., mini-excavator, unexploded ordnance, air traffic, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, and task-specific gloves.

H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and topics of daily H&S briefings included:

- conducting daily H&S briefings
- management of site workers via site control log
- briefing site personnel on recognition factors of particular ordnance used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e., insects, animals)
- ensuring personnel were adequately trained and cognizant of air traffic hazards
- ensuring personnel were cognizant of exposure to the weather

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- providing proper gloves (i.e., insulated, etc.) when needed;

Equipment and work area inspections included:

- ensuring the heavy equipment (i.e., mini-excavator) was routinely inspected and maintained in a proper functioning condition (i.e., locking out all motors, electrical, mechanical, and hydraulic components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer’s guidelines
- ensuring all heavy equipment (i.e., mini-excavator) and vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of heavy equipment (i.e., mini-excavator), ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring soil stability/proper grading/proper base/use of outriggers when setting heavy equipment (i.e., mini-excavator)
- ensuring that buried and overhead utilities in the vicinity of were properly marked out prior to utilization of the heavy equipment (i.e., mini-excavator)
- ensuring all equipment were properly secured during transport
- ensuring safe movement of equipment and vehicles (i.e., movement around and across

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runway and air traffic)

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from mechanized moving parts of heavy equipment (i.e., mini-excavator, air traffic)
- ensuring proper handling and transport of heavy equipment (i.e., mini-excavator)
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- ensuring site personnel kept safe distances from suspected unexploded ordnance
- monitoring air traffic and communication from air traffic control officers
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Employer: *ERT, Inc.*	***Client/Project Name:*** *Straughan Environmental, NASA Goddard Space Flight Center*
Safety Experience: *July 2014 (0.5 months safety experience)*	

As the Environmental Team Lead and Team Safety Lead working under the SSHO, Mr. Koroncai provided H&S oversight of staff performing environmental groundwater sampling activities related to an identified trichloroethylene (TCE) plume at the National Aeronautics and Space Administration (NASA) Goddard Space Flight Center (GSFC). H&S tasks included: coordinating the fieldwork with the client; conducting daily H&S briefings; ensuring proper use of equipment; making the field team aware of and monitoring the possible hazards on the jobs sites (i.e., vehicular traffic, rough terrain, working in partially enclosed areas [culverts, ditches, etc], sound levels) implementing and overseeing field controls used to help mitigate traffic issues (i.e., flagging crews, traffic cones, amber safety lights on vehicles, wearing high visibility safety gear, and adequate hearing protection). On this project, Mr. Koroncai focused 50 percent of his time (0.5 month safety experience) as the Team Safety Lead.

Mr. Koroncai was responsible for evaluating and implementing project-specific H&S standards (i.e., daily inspections and regular maintenance prior to use) during the investigations. Safety tasks performed by Mr. Koroncai included conducting daily pre-work site H&S and daily tailgate briefings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all staff had obtained adequate safety awareness training and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Team Safety Lead, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

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Staff training and H&S briefings included:

- conducting daily H&S briefings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- providing proper gloves (i.e., nitrile, etc.) when needed;

Equipment and work area inspections included:

- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment, and tools were properly secured during transport
- ensuring all vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all generators were shut off during refueling, ensured no smoking occurred in vicinity of the generators, ensured that proper fire extinguishers were present, and that fuel was stored appropriately

H&S monitoring of the work area included:

- ensuring site personnel handled drums properly
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via PID/OVM
- monitoring the weather and advised site personnel of impending inclement weather

Employer: *ERT, Inc.*	***Client/Project Name:*** *Geophysical Investigations for Various Commercial Clients (i.e. Maryland Transportation Authority, T.L.B. Associates, Inc., etc.)*
Safety Experience: *December 2013 through December 2014 (1 month safety experience)*	

As a member of the Geophysical Field Team/Team Safety Lead, Mr. Koroncai has provided H&S oversight of staff performing numerous investigations (i.e., seismic refraction and/or ground-penetrating radar surveys). H&S tasks have included: coordinating the fieldwork with the client; conducting daily H&S meetings; ensuring proper use of equipment; making the field team aware of and monitoring the possible hazards on the jobs sites (i.e., vehicular traffic, rough terrain, working in partially enclosed areas [culverts, ditches, etc], sound levels) implementing and overseeing field controls used to help mitigate traffic issues (i.e., flagging crews, traffic cones, amber safety lights on vehicles, wearing high visibility safety gear, and adequate hearing protection). On this project, Mr. Koroncai focused 10 percent of his time (1 month safety experience) as the Team Safety Lead.

Mr. Koroncai was responsible for evaluating and implementing project-specific H&S standards (i.e., daily inspections and regular maintenance of equipment prior to use) during the investigations. Safety tasks performed by Mr. Koroncai included conducting daily pre-work site H&S and daily tailgate meetings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, and task-specific gloves.

As the Team Safety Lead, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S meetings
- management of site workers via site control log
- managing H&S training for subcontractors
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, etc.) when needed;

Equipment and Work Area Inspections included:

- ensuring the geophysical equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer’s guidelines
- ensuring all geophysical equipment were properly secured during transport
- ensuring vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of vehicles, ensured that proper fire extinguisher was present, and that fuel was

stored appropriately

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from geophysical equipment during use
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- performing H&S air monitoring (and breathing zone monitoring) via photoionization detector (PID)/organic vapor monitor (OVM)
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed.

Employer: *United States Environmental Protection Agency*	***Client/Project Name:*** *U.S. EPA Region 3, National Coastal Condition Assessment*
Safety Experience: *May 2010 through August 2010 (1 month safety experience)*	

As a member of the Environmental Sample Team, Mr. Koroncai provided H&S oversight of staff performing water and sediment sampling activities associated with the National Coastal Condition Assessment for USEPA Region 3 on board the Ocean Survey Vessel (OSV) Bold. H&S tasks included: work plan preparation and review; ensuring worker safety around equipment (i.e., Rosette sampler, benthic grab sampler); ensuring worker safety onboard the vessel; and H&S monitoring for the environmental sampling teams. On this project, Mr. Koroncai focused 25 percent of his time (1 month safety experience) towards H&S.

Mr. Koroncai was responsible for evaluating and implementing project-specific H&S standards (i.e., daily inspections and regular maintenance prior to use) during use of environmental sampling equipment. Safety tasks performed by Mr. Koroncai included conducting daily pre-work site H&S and daily tailgate briefings, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all subcontractors and staff had obtained adequate safety awareness training (i.e., vessel operations, equipment use, etc.) and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included closed-toed shoes, life jacket, hard hats, and task-specific gloves.

H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and topics of daily H&S briefings included:

- conducting daily H&S briefings
- managing H&S training for subcontractors

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- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:
- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- ensuring properly fitting life jackets were worn and extras were readily available
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, etc.) when needed;

Equipment and work area inspections included:
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment, and tools were properly secured when not in use
- ensured no smoking occurred outside designated areas on the vessel

H&S monitoring of the work area included:
- ensuring site personnel kept safe distance from mechanized moving parts on vessel
- ensuring proper handling and storage of equipment and tools
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed

Employer: *H&H Chevrolet*	***Client/Project Name:*** *Not Applicable*
Safety Experience: *September 2009 through May 2010 and September 2011 through November 2013 (36 months safety experience)*	

As the Shift Supervisor and Team Safety Lead, Mr. Koroncai provided H&S oversight of staff performing automotive mechanical work. H&S tasks included: coordinating the work with the client; conducting daily H&S briefings; ensuring proper use of equipment; and making the team aware of and monitoring the possible hazards of the work area (i.e., vehicular traffic, slippery and uneven terrain, working in close proximity to hazardous areas [gases under pressure, mechanical lifts, etc], sound levels, chemicals). On this project, Mr. Koroncai focused 90 percent of his time (36 months safety experience) as the Team Safety Lead.

Mr. Koroncai was responsible for evaluating and implementing project-specific H&S standards (i.e., daily inspections and regular maintenance prior to use) during the investigations. Safety tasks performed by Mr. Koroncai included conducting daily pre-work site H&S and daily tailgate

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briefings, keeping track of site personnel via personnel logs, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all staff had obtained adequate safety awareness training and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, face shields, air-purifying respirators, and task-specific gloves.

As the Team Safety Lead, H&S tasks included managing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S briefings
- management of site workers via personnel log
- managing H&S training for subcontractors
- briefing personnel on recognition factors of particular chemicals used in the area
- ensuring personnel were cognizant of their proximity to mechanical hazards

Implementation of applicable PPE included:

- ensuring correct usage of respirators in applicable work zones
- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, etc.) when needed
- providing safety glasses or face shields as necessary

Equipment and work area inspections included:

- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment, chemicals, and tools were properly secured when not in use
- ensuring all vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all vehicles were shut off during refueling, ensured no smoking occurred in vicinity of the generators, ensured that proper fire extinguishers were present, and that fuel was stored appropriately
- ensuring all compressed air lines were shut off and decompressed when not in use

H&S monitoring of the work area included:

- ensuring site personnel handled chemicals properly
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)

Employer: *Environmental Quality Resources, LLC.*	***Client/Project Name:*** *Clark Construction, Intercounty*
Safety Experience:	

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April 2011 through August 2011 (5 month safety experience)	*Connector (MD 200)*

As the Team Lead and Team Safety Lead, Mr. Koroncai provided H&S oversight of staff performing numerous commercial landscaping activities. H&S tasks included: coordinating the fieldwork with the client; conducting daily H&S briefings; ensuring proper use of equipment, ensuring site worker safety around heavy machinery (i.e., mini-excavator, skid-steer, telehandler, etc.), making the field team aware of and monitoring the possible hazards on the jobs sites (i.e., vehicular traffic, rough terrain, working in partially enclosed areas [culverts, ditches, etc], sound levels) implementing and overseeing field controls used to help mitigate traffic issues (i.e., flagging crews, traffic cones, amber safety lights on vehicles, wearing high visibility safety gear, and adequate hearing protection). Throughout various projects conducted, Mr. Koroncai focused approximately 90 percent of his time (5 months of time total) dedicated as the field Team Safety Lead.

Mr. Koroncai was responsible for evaluating and implementing project-specific H&S standards (i.e., daily inspections and regular maintenance prior to use) during the work. Safety tasks performed by Mr. Koroncai included conducting daily pre-work site H&S and daily tailgate briefings, keeping track of site personnel via site control log, and ensuring all subcontractor and staff operations were performed in accordance with H&S project plans, AHAs, and H&S-specific SOPs. He ensured that all staff had obtained adequate safety awareness training and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, hearing protection, hard hats, reflective vests, and task-specific gloves.

As the Team Safety Lead, H&S tasks included reviewing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting daily H&S briefings
- management of site workers via site control log
- managing H&S training for subcontractors
- briefing site personnel on recognition factors of particular hazards in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, etc.) when needed;

Robert Koroncai Jr., Site Safety and Health Officer for HTRW Operations

Equipment and work area inspections included:

- ensuring the equipment was routinely inspected and maintained in a proper functioning condition (i.e., locking out all components when making repairs or not in use)
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment were properly secured during transport
- ensuring vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of vehicles, ensured that proper fire extinguisher was present, and that fuel was stored appropriately
- ensuring that buried and overhead utilities in the vicinity of the excavation locations were properly marked out

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from heavy machinery and equipment during use
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed.

Employer: *Shippensburg Borough Public Works Department*	***Client/Project Name:*** *Not Applicable*
Safety Experience: *April 2009 to September 2009 (6 months safety experience)*	

As Team Safety Lead, Mr. Koroncai provided H&S oversight of numerous activities (i.e. park maintenance, leaf removal) at the Shippensburg Public Works Department, Pennsylvania. H&S tasks included: conducting weekly H&S briefs and daily tailgate safety meetings; ensuring proper use of equipment; making the field team aware of and monitoring the possible hazards on the jobs sites (i.e., vehicular traffic, rough terrain, working in partially enclosed areas [culverts, ditches, etc], biological (insects and wildlife), and public interference) implementing and overseeing field controls used to help mitigate traffic issues (i.e., flagging crews, traffic cones, amber safety lights on vehicles, and wearing high visibility safety gear). On this project, Mr. Koroncai focused 90 percent of is time (6 months safety experience) as the Team Safety Lead.

Mr. Koroncai was responsible for evaluating and implementing project-specific H&S standards (i.e., daily inspections and regular maintenance prior to use) during daily activities. Safety tasks performed by Mr. Koroncai included conducting daily pre-work site H&S and weekly tailgate briefings and ensuring all operations were performed within OSHA safety guidelines, AHAs, and

Robert Koroncai Jr., Site Safety and Health Officer for HTRW Operations

H&S-specific SOPs. He ensured that all staff had obtained adequate safety awareness training and employed the proper task-specific PPE during performance of their assigned tasks. Task specific PPE routinely included safety-toed shoes, safety glasses, and task-specific gloves.

As the Team Safety Lead, H&S tasks included reviewing project-specific staff trainings/briefings, staff PPE, equipment inspections/maintenance, and site H&S monitoring.

Staff training and H&S briefings included:

- conducting weekly H&S briefings
- briefing site personnel on recognition factors of particular hazards in the area
- ensuring personnel were adequately trained and cognizant of biological hazards (i.e. insects, animals)
- ensuring personnel were cognizant of exposure to the sun and provided proper sun blocking agents

Implementation of applicable PPE included:

- ensuring First Aid kits were readily available to all site personnel
- ensuring properly fitting clothing worn while operating equipment
- monitoring areas for noise and provided hearing protection as necessary
- providing proper gloves (i.e., nitrile, etc.) when needed;

Equipment and work area inspections included:

- ensuring the equipment was routinely inspected and maintained in a proper functioning condition
- operating and maintaining equipment in accordance with manufacturer's guidelines
- ensuring all equipment were properly secured during transport
- ensuring vehicles backed up safely (i.e. use of a guide/flag)
- ensuring all motors were shut off during refueling, ensured no smoking occurred in vicinity of vehicles, ensured that proper fire extinguisher was present, and that fuel was stored appropriately

H&S monitoring of the work area included:

- ensuring site personnel kept safe distance from equipment during use
- ensuring use of proper lifting techniques and glove usage to prevent physical injury from heavy lifting/pinch points
- advising personnel on slip, trip, and fall hazards and controls (i.e., good housekeeping, foot protection)
- monitoring the weather and advised site personnel of impending inclement weather
- monitoring site personnel for signs of heat/cold stress and enforced work/rest regimens as needed.

Mr. Sean Carney, PMP, Project Manager, ERT

Experience	Active Professional Licenses (license/number/state/year)
▪ 6 Years of experience as a project manager for USACE Baltimore and Louisville Districts. ▪ Total of 12 Years of experience performing and remedial action (RA) work with state, federal and private sector clients.	▪ Project Management Professional / No.1484852 / 2012
Education (degree/year/specialization)	**Additional Training/Certifications**
▪ MS, 2004, Environmental Science and Policy ▪ BS, 2001, Biology	▪ HAZWOPER 40-Hr and 8-Hr Annual Refresher Training ▪ HAZWOPER 8-Hr Site Supervisor Training ▪ OSHA 30-Hr Construction Safety Training, 2010, #1588828 ▪ USACE Construction Quality Control Management for Contractors ▪ OSHA 30-Hr Construction Industry Outreach Training Program ▪ USDOT Course 1-4 for Hazardous Materials Management, Packaging and Labeling, Shipping, and Loading and Storage.

Summary of Qualifications and Experience Relevant to Evaluation Criteria

- 12 years of environmental experience in a variety of fields including RCRA soil and groundwater characterization, remediation, and removal actions.
- 8 years of CERCLA and RCRA experience with federal and state regulators in EPA Regions 2, 3 and 5. Extensive experience with MDE , PADEP, and EPA Region 3 at active federal facilities.

Role and Assignment on this Project

Project Manager (PM)

Relevant Project Experience and Qualifications

Project Name and Location: SI/RI/FS/PP/DD for Area IV Groundwater at Former Frankford Arsenal, Philadelphia, PA

General Scope of Project:

Role and Description of the Duties Performed: Project Manager site-wide (Area IV) groundwater environmental investigation activities in support of ongoing CERCLA activities. Participated in project planning, investigation design, implementing and preparing SI, implementing and preparing RI, preparing FS, developing the remedial design (RD), completing the Proposed Plan (PP) and Decision Document (DD), and completing interim remedial actions (IRA). IRAs included . Directed all activities associated with the project which included management of 5 subcontractors and over 20 subcontractor personnel. Served as the point of contact (POC) for subcontractors ensuring they were aware of client schedule and deliverable expectations, and maintained regular and concise communication to ensure successful and on time delivery of services. Served as the POC for the client. Participated in project planning meetings and project review meetings with the client , property owners and regulatory agency.

Client POC (Name/Phone): Todd Beckwith/410-962-6784

Project Name and Location: RI/FS for Five Nike Sites, MD and PA

General Scope of Project: This project includes site characterization and remediation at five separate Nike Missile Launch Sites. This project demonstrates ERT's successful prime contractor experience in performing multiple concurrent CERCLA investigations and remedial actions at diverse geologically complex sites. ERT is responsible for carrying each site through to closure, including Site Inspection (SI), Remedial Investigation (RI), Feasibility Study (FS) through Decision Document (DD) approval. Activities relevant to the SOW include characterization of a variety of media including surface and subsurface soils, and groundwater. As a result of site characterization at one Nike site, ERT identified and delineated lead contamination in soil and then conducted lead impacted soil removal. Also relevant to ERT's approach to the SOW is the successful use of subcontractor personnel to take soil borings, conduct laboratory sample analysis and third-party validation of analytical results, waste disposal, and professional survey activities during the performance of this contract.

Role and Description of the Duties Performed: Project Manager for five separate former Nike Missile Sites in support of ongoing CERCLA activities. Participated in project planning, investigation design, implementing RI, preparing FS, developing the remedial design (RD) and completing remedial actions (RA). RAs include lead impacted soil excavation and disposal off-site at a

Treatment Storage and Disposal Facility and in situ groundwater remediation. Directed all activities associated with the project which included management of 10 subcontractors and over 50 subcontractor personnel. Served as the point of contact (POC) for subcontractors ensuring they were aware of client schedule and deliverable expectations, and maintained regular and concise communication to ensure successful and on time delivery of services. Participated in project planning meetings and project review meetings with the client and regulatory agency.

Client POC(Name/Phone): Hamid Rafiee /410-962-7546

Project Name and Location: Lake Ontario Ordnance Works (LOOW), Lewiston, NY

General Scope of Project: This project involved site characterization to identify a variety of contaminants from several potential sources. ERT sampled various media including soil, sediment, surface water, groundwater which were all analyzed for VOCs, SVOCs, metals, PCBs, pesticides, explosives (with MEC construction support), and radiological parameters. Through the site characterization, ERT successfully identified contaminants ranging from TNT production chemicals, and raw explosives, to metals, petroleum products, solvents, and radioactive waste and identified the correct source of contamination, despite the site's complex history and use. Historic activities at this site include a WWII TNT production plant, jet fuel production at a former Air Force facility, and multiple Nike Missile launch and control areas. The site is adjacent to the Niagara Falls Storage Site, where Manhattan Project radioactive waste is stored. In addition, the site is currently used as a large landfill and chemical waste treatment/storage facility. Following site characterization, ERT conducted asbestos cleanup and abatement on the dilapidated structure of an acid neutralization building at the waste water treatment facility on site.

Role and Description of the Duties Performed: Project manager responsible for managing complex, multimedia RI/FS activities. Provided technical expertise and project management for work plan and RI/FS, engineering evaluation/cost analysis development. Oversaw all field work including the collection and analysis of soil samples, and the planning and implementation of asbestos abatement activities in accordance with all safety and regulatory requirements. Served as the POC for subcontractors ensuring they were aware of client schedule and deliverable expectations, and maintained regular and concise communication to ensure successful and on time delivery of services. Reviewed subcontractor asbestos abatement plans and identified several missing documents including staff Asbestos Hazard Emergency Response Act (AHERA) certifications and transport permits, which were provided in a second draft before providing plans to USACE for review.

Facilitated the technical project planning process to ensure stakeholder support and approval of project deliverables. Responsible for schedule and cost control management. Provided monthly reports and any other support as needed to USACE.

Client POC(Name/Phone): Michael Senus /716-879-4309

Project Name and Location: Queens West Development Stage 2, Queens, NY

General Scope of Project: This project involved RI and RA activities at a ten acre former oil refinery. RI characterization activities included the advancement of over 250 soil borings, 10 sediment borings, 10 surface water samples, and the installation of over 20 shallow and deep groundwater monitoring wells. Included the collection and management of more than 500 environmental samples and associated QA/QC samples, analyzed for VOCs, SVOCs, metals, PCBs, pesticides and herbicides. RA activities included the excavation of more than 200,000 tons of VOC, SVOC and metals impacted soil and dual-phase extraction removal of more than 20,000 gallons of free-phase petroleum product. Bioremedial technologies were utilized to perform the final treatment of groundwater impacts. Subslab vapor systems were installed, operated and maintained, supporting each new residential and commercial structure erected on the site. The project involved the use of 10 subcontractors comprised of more than 100 subcontractor personnel.

Role and Description of the Duties Performed: Project Manager responsible for directing all subcontractor activities associated with this large scale project. Managed contractors to ensuring work remained on schedule and confirmed completion of all subcontractor activities including demolition, groundwater well installation, environmental data sampling and analysis, remediation system construction, operation and maintenance. Through successful subcontractor management, Mr. Carney identified construction subcontractor QC failures including work plan deviations in the field and ensured corrections were made to meet client expectations prior to subcontractor demobilization.

Explored alternative methods for remediation. This included managing a remedial pilot study targeting in situ remediation of free-phase product and sorbed-phase soil impacts. The pilot study resulted in an expedited RA, project schedule and monetary savings to the client. Enhanced bioremediation technologies were also utilized to perform the final treatment of groundwater contamination.

Participated in project planning meetings and project review meetings with the client and regulators and maintained community involvement program for this large redevelopment project.

Project Name and Location: First Avenue Waterside Steam Generation Plant, New York, NY

General Scope of Project: This project involved RI and RA at a $175M NYDEC Brownfield Voluntary Cleanup project. RI field investigations included taking more than 100 soil borings and more than 350 environmental samples. The RI identified significant soil and groundwater contamination. Remedial actions included the excavation and removal of more than 150,000 tons of contaminated material and the installation of a groundwater treatment system treating approximately 500,000 gallons of groundwater per day. The project consisted of the remediation and closure in place of five tunnels and the removal of more than 10,000 tons of PCB-contaminated sediments from within.

Role and Description of the Duties Performed: Construction Manager responsible for directing all activities of the project including oversight of more than 12 subcontractors and more than 150 subcontractor personnel ensuring proper field procedures were followed in accordance with client expectations. Reviewed subcontractor plans, and oversaw subcontractor excavation and removal of soils ensuring complete remediation. Participated in project planning meetings and project review meetings with the client and regulatory agency.

ENCLOSURE B

Health and Safety Report Forms

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SITE CONTROL LOG

Date/Day/Time: ____________________

Project Name: ____________________

Project Location: ____________________

TIME		NAME	ORGANIZATION
IN	OUT		

SITE SAFETY AND HEALTH PLAN REVIEW RECORD

Project: FY16 Recycling of Materials at the Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility, Former Ravenna Army Ammunition Plant Restoration Program
Site: Former Ravenna Army Ammunition Plant
ERT Project No.: 3369

I have read the Site-Specific Health and Safety Plan for this site and have been briefed on the nature, level, and degree of exposure likely as a result of participation in this project. I meet and agree to conform to all the requirements of this Plan.

Name	Signature	Affiliation	Date

INCIDENT/INJURY/ILLNESS REPORTING FORM (page 1)

Date: ____________________ **Project No:** ______________________________

Time: ____________________ **Project Name:** ______________________________

Employee's Name: ______________________________ **Employee No.:** ____________

Employee Office: ______________________________ **Employee Phone:** ____________

Incident/Injury/Illness Location: ______________________________

Incident/Injury/Illness Description:

____

____

____

____

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____

____

____

Extent of Injury or Damage:

____

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INCIDENT/INJURY/ILLNESS REPORTING FORM (page 2)

Actions Taken:

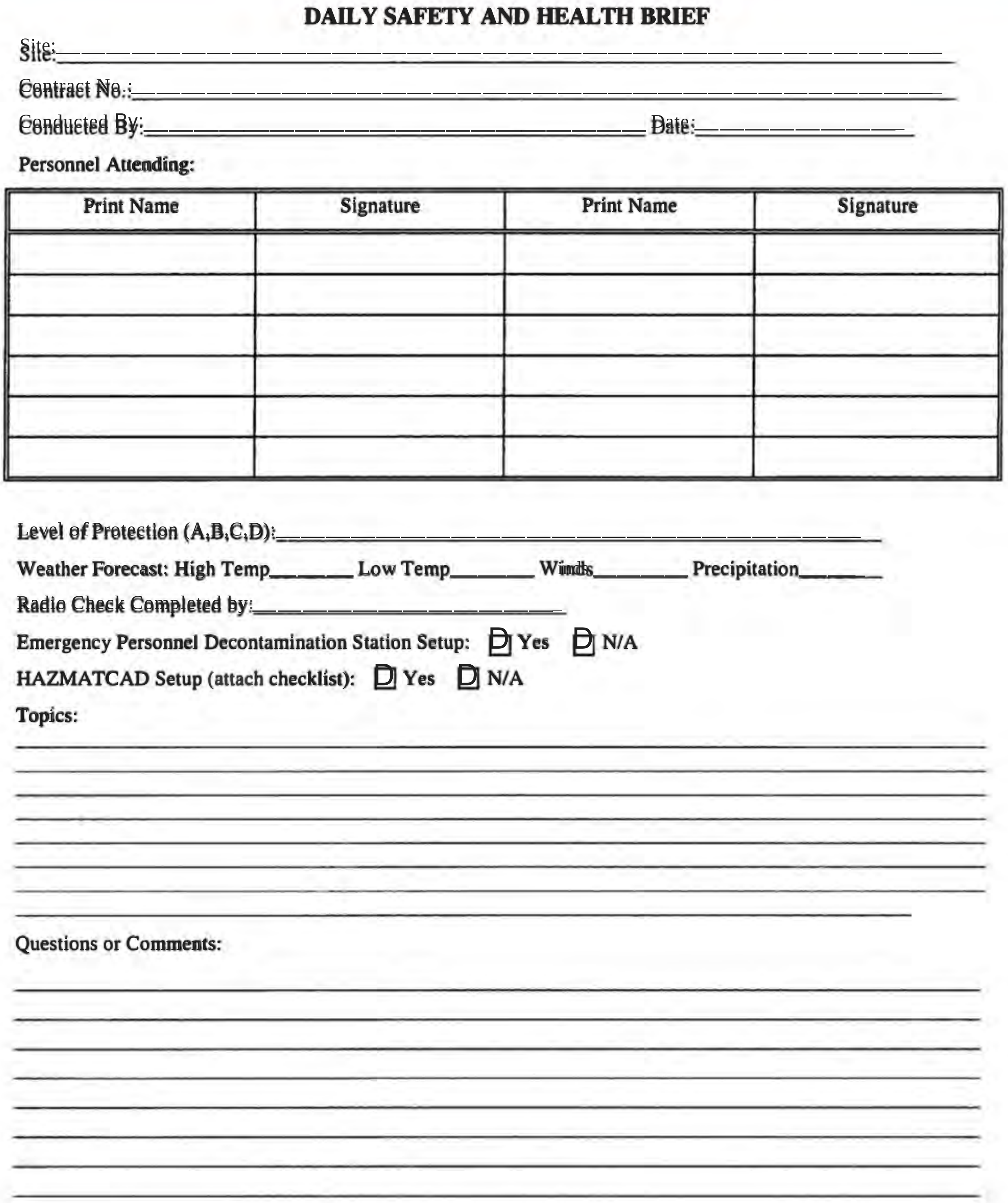
List of all personnel involved and their home phone numbers:

Describe any measures taken to prevent reoccurrence:

Other Notes:

Employee's Signature/Date:

Site Supervisor's Signature/Date:



DAILY SAFETY AND HEALTH BRIEF

Site:____

Contract No.:____

Conducted By:______________________________ Date:____________

Personnel Attending:

Print Name	Signature	Print Name	Signature

Level of Protection (A,B,C,D):______________________________________

Weather Forecast: High Temp_______ Low Temp_______ Winds________ Precipitation_______

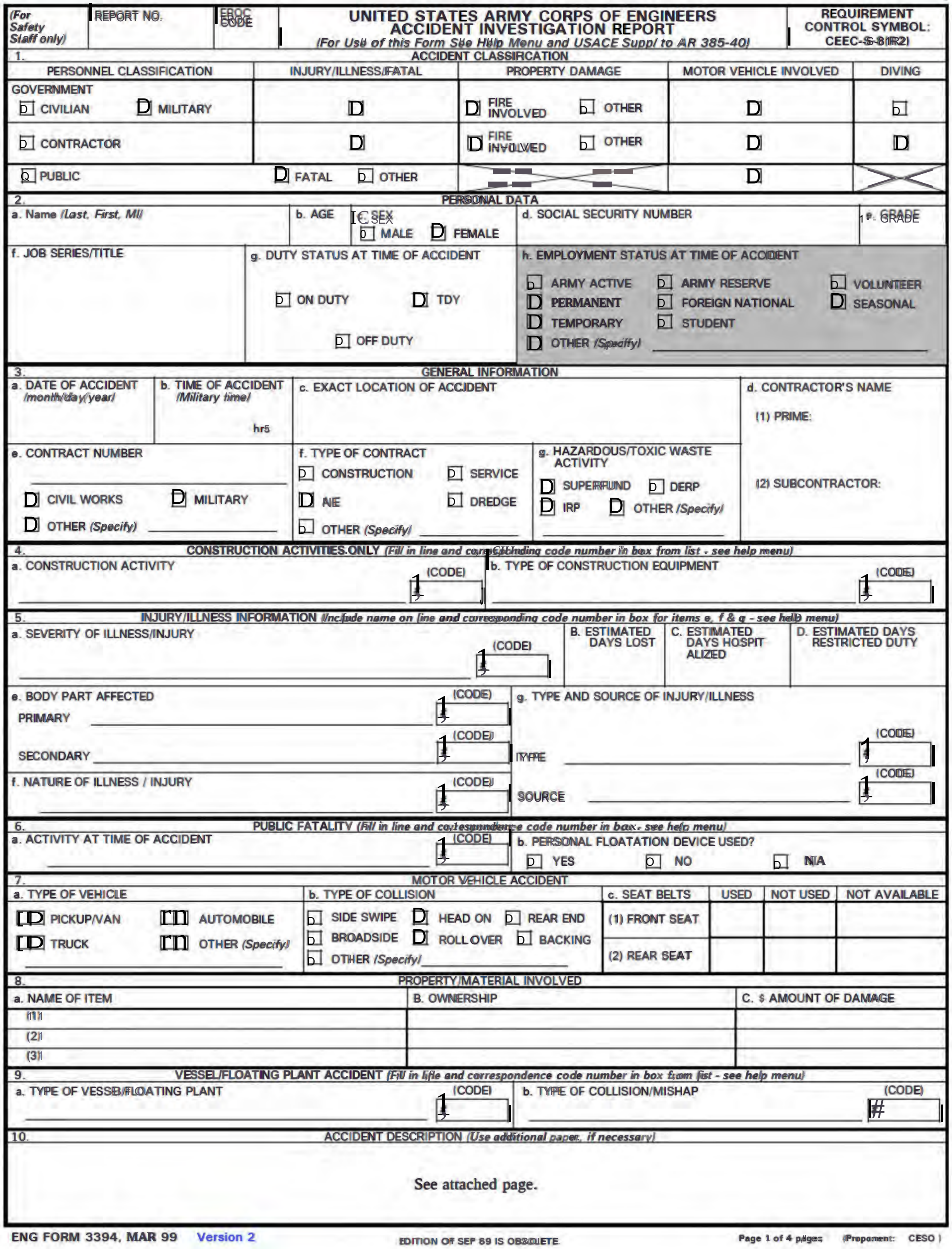
Radio Check Completed by:__________________________

Emergency Personnel Decontamination Station Setup: ☐ Yes ☐ N/A

HAZMATCAD Setup (attach checklist): ☐ Yes ☐ N/A

Topics:

Questions or Comments:



(For Safety Staff only)	REPORT NO.	EROC CODE	UNITED STATES ARMY CORPS OF ENGINEERS ACCIDENT INVESTIGATION REPORT *(For Use of this Form See Help Menu and USACE Suppl to AR 385-40)*	REQUIREMENT CONTROL SYMBOL: CEEC-S-8(R2)

1. ACCIDENT CLASSIFICATION

PERSONNEL CLASSIFICATION	INJURY/ILLNESS/FATAL	PROPERTY DAMAGE	MOTOR VEHICLE INVOLVED	DIVING
GOVERNMENT ☐ CIVILIAN ☐ MILITARY	☐	☐ FIRE INVOLVED ☐ OTHER	☐	☐
☐ CONTRACTOR	☐	☐ FIRE INVOLVED ☐ OTHER	☐	☐
☐ PUBLIC	☐ FATAL ☐ OTHER		☐	

2. PERSONAL DATA

a. Name *(Last, First, MI)*	b. AGE	c. SEX ☐ MALE ☐ FEMALE	d. SOCIAL SECURITY NUMBER	e. GRADE

f. JOB SERIES/TITLE	g. DUTY STATUS AT TIME OF ACCIDENT	h. EMPLOYMENT STATUS AT TIME OF ACCIDENT
	☐ ON DUTY ☐ TDY ☐ OFF DUTY	☐ ARMY ACTIVE ☐ ARMY RESERVE ☐ VOLUNTEER ☐ PERMANENT ☐ FOREIGN NATIONAL ☐ SEASONAL ☐ TEMPORARY ☐ STUDENT ☐ OTHER *(Specify)* ________

3. GENERAL INFORMATION

a. DATE OF ACCIDENT *(month/day/year)*	b. TIME OF ACCIDENT *(Military time)*	c. EXACT LOCATION OF ACCIDENT	d. CONTRACTOR'S NAME
	hrs		(1) PRIME:

e. CONTRACT NUMBER	f. TYPE OF CONTRACT	g. HAZARDOUS/TOXIC WASTE ACTIVITY	(2) SUBCONTRACTOR:
________ ☐ CIVIL WORKS ☐ MILITARY ☐ OTHER *(Specify)* ________	☐ CONSTRUCTION ☐ SERVICE ☐ A/E ☐ DREDGE ☐ OTHER *(Specify)* ________	☐ SUPERFUND ☐ DERP ☐ IRP ☐ OTHER *(Specify)*	

4. CONSTRUCTION ACTIVITIES ONLY *(Fill in line and corresponding code number in box from list - see help menu)*

a. CONSTRUCTION ACTIVITY	(CODE)	b. TYPE OF CONSTRUCTION EQUIPMENT	(CODE)

5. INJURY/ILLNESS INFORMATION *(Include name on line and corresponding code number in box for items e, f & g - see help menu)*

a. SEVERITY OF ILLNESS/INJURY	(CODE)	B. ESTIMATED DAYS LOST	C. ESTIMATED DAYS HOSPITALIZED	D. ESTIMATED DAYS RESTRICTED DUTY

e. BODY PART AFFECTED	(CODE)	g. TYPE AND SOURCE OF INJURY/ILLNESS	(CODE)
PRIMARY ________			
SECONDARY ________		TYPE ________	
f. NATURE OF ILLNESS / INJURY ________		SOURCE ________	

6. PUBLIC FATALITY *(Fill in line and correspondence code number in box - see help menu)*

a. ACTIVITY AT TIME OF ACCIDENT	(CODE)	b. PERSONAL FLOATATION DEVICE USED?
		☐ YES ☐ NO ☐ N/A

7. MOTOR VEHICLE ACCIDENT

a. TYPE OF VEHICLE	b. TYPE OF COLLISION	c. SEAT BELTS	USED	NOT USED	NOT AVAILABLE
☐ PICKUP/VAN ☐ AUTOMOBILE	☐ SIDE SWIPE ☐ HEAD ON ☐ REAR END	(1) FRONT SEAT			
☐ TRUCK ☐ OTHER *(Specify)* ________	☐ BROADSIDE ☐ ROLL OVER ☐ BACKING ☐ OTHER *(Specify)* ________	(2) REAR SEAT			

8. PROPERTY/MATERIAL INVOLVED

a. NAME OF ITEM	B. OWNERSHIP	C. $ AMOUNT OF DAMAGE
(1)		
(2)		
(3)		

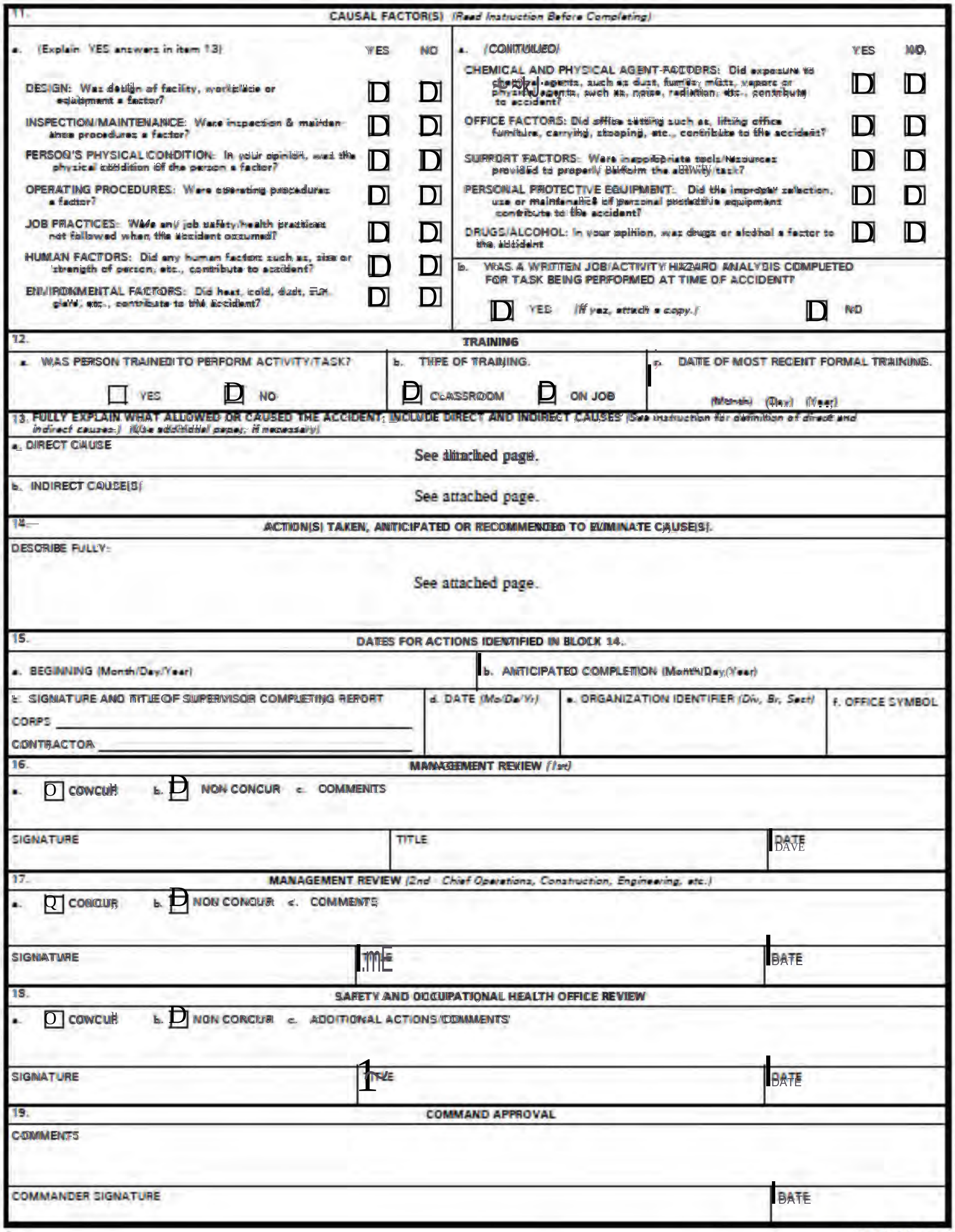
9. VESSEL/FLOATING PLANT ACCIDENT *(Fill in line and correspondence code number in box from list - see help menu)*

a. TYPE OF VESSEL/FLOATING PLANT	(CODE)	b. TYPE OF COLLISION/MISHAP	(CODE)

10. ACCIDENT DESCRIPTION *(Use additional paper, if necessary)*

See attached page.

ENG FORM 3394, MAR 99 Version 2 EDITION OF SEP 89 IS OBSOLETE Page 1 of 4 pages (Proponent: CESO)



11. CAUSAL FACTOR(S) *(Read Instruction Before Completing)*

a. (Explain YES answers in item 13) YES NO

DESIGN: Was design of facility, workplace or equipment a factor? ☐ ☐

INSPECTION/MAINTENANCE: Were inspection & maintenance procedures a factor? ☐ ☐

PERSON'S PHYSICAL CONDITION: In your opinion, was the physical condition of the person a factor? ☐ ☐

OPERATING PROCEDURES: Were operating procedures a factor? ☐ ☐

JOB PRACTICES: Were any job safety/health practices not followed when the accident occurred? ☐ ☐

HUMAN FACTORS: Did any human factors such as, size or strength of person, etc., contribute to accident? ☐ ☐

ENVIRONMENTAL FACTORS: Did heat, cold, dust, sun, glare, etc., contribute to the accident? ☐ ☐

a. *(CONTINUED)* YES NO

CHEMICAL AND PHYSICAL AGENT FACTORS: Did exposure to chemical agents, such as dust, fumes, mists, vapors or physical agents, such as, noise, radiation, etc., contribute to accident? ☐ ☐

OFFICE FACTORS: Did office setting such as, lifting office furniture, carrying, stooping, etc., contribute to the accident? ☐ ☐

SUPPORT FACTORS: Were inappropriate tools/resources provided to properly perform the activity/task? ☐ ☐

PERSONAL PROTECTIVE EQUIPMENT: Did the improper selection, use or maintenance of personal protective equipment contribute to the accident? ☐ ☐

DRUGS/ALCOHOL: In your opinion, was drugs or alcohol a factor to the accident? ☐ ☐

b. WAS A WRITTEN JOB/ACTIVITY HAZARD ANALYSIS COMPLETED FOR TASK BEING PERFORMED AT TIME OF ACCIDENT?

☐ YES *(If yes, attach a copy.)* ☐ NO

12. TRAINING

a. WAS PERSON TRAINED TO PERFORM ACTIVITY/TASK?

☐ YES ☐ NO

b. TYPE OF TRAINING.

☐ CLASSROOM ☐ ON JOB

c. DATE OF MOST RECENT FORMAL TRAINING.

(Month) (Day) (Year)

13. FULLY EXPLAIN WHAT ALLOWED OR CAUSED THE ACCIDENT; INCLUDE DIRECT AND INDIRECT CAUSES *(See instruction for definition of direct and indirect causes.) (Use additional paper, if necessary)*

a. DIRECT CAUSE

See attached page.

b. INDIRECT CAUSE(S)

See attached page.

14. ACTION(S) TAKEN, ANTICIPATED OR RECOMMENDED TO ELIMINATE CAUSE(S).

DESCRIBE FULLY:

See attached page.

15. DATES FOR ACTIONS IDENTIFIED IN BLOCK 14.

a. BEGINNING (Month/Day/Year)

b. ANTICIPATED COMPLETION (Month/Day/Year)

c. SIGNATURE AND TITLE OF SUPERVISOR COMPLETING REPORT

CORPS ____________________

CONTRACTOR ____________________

d. DATE *(Mo/Da/Yr)*

e. ORGANIZATION IDENTIFIER *(Div, Br, Sect)*

f. OFFICE SYMBOL

16. MANAGEMENT REVIEW *(1st)*

a. ☐ CONCUR b. ☐ NON CONCUR c. COMMENTS

SIGNATURE

TITLE

DATE

17. MANAGEMENT REVIEW *(2nd - Chief Operations, Construction, Engineering, etc.)*

a. ☐ CONCUR b. ☐ NON CONCUR c. COMMENTS

SIGNATURE

TITLE

DATE

18. SAFETY AND OCCUPATIONAL HEALTH OFFICE REVIEW

a. ☐ CONCUR b. ☐ NON CONCUR c. ADDITIONAL ACTIONS/COMMENTS

SIGNATURE

TITLE

DATE

19. COMMAND APPROVAL

COMMENTS

COMMANDER SIGNATURE

DATE

Page 2 of 4 pages

*U.S. GOVERNMENT PRINTING OFFICE:

10. ACCIDENT DESCRIPTION *(Continuation)*

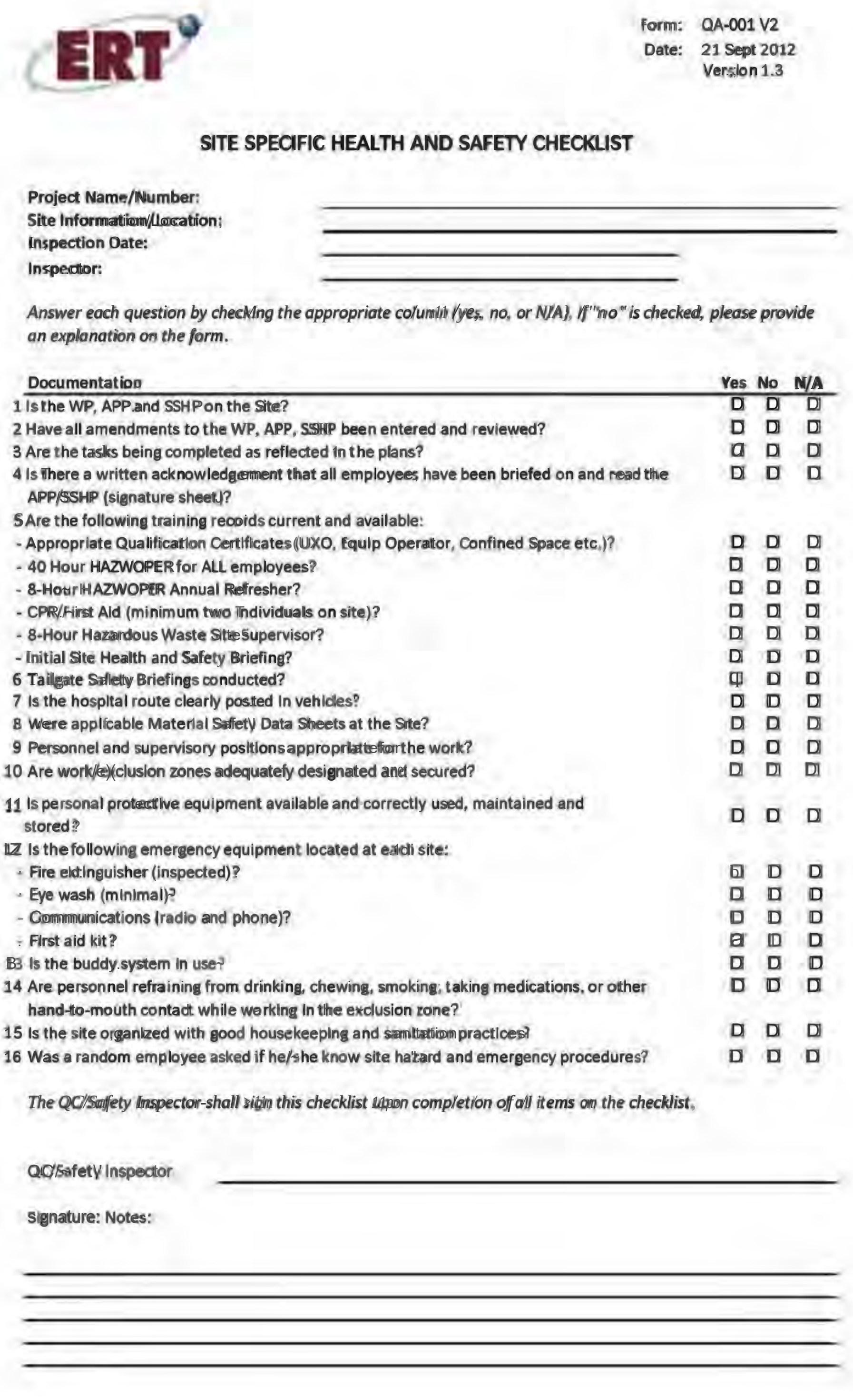
13a. DIRECT CAUSE *(Continuation)*

Page 3 of 4 pages

13b.	**INDIRECT CAUSES** *(Continuation)*

14.	**ACTION(S) TAKEN, ANTICIPATED, OR RECOMMENDED TO ELIMINATE CAUSE(S)** *(Continuation)*

Page 4 of 4 pages



Form: QA-001 V2
Date: 21 Sept 2012
Version 1.3

SITE SPECIFIC HEALTH AND SAFETY CHECKLIST

Project Name/Number: ____________________
Site Information/Location: ____________________
Inspection Date: ____________________
Inspector: ____________________

Answer each question by checking the appropriate column (yes, no, or N/A). If "no" is checked, please provide an explanation on the form.

	Documentation	**Yes**	**No**	**N/A**
1	Is the WP, APP and SSHP on the Site?	☐	☐	☐
2	Have all amendments to the WP, APP, SSHP been entered and reviewed?	☐	☐	☐
3	Are the tasks being completed as reflected in the plans?	☐	☐	☐
4	Is there a written acknowledgement that all employees have been briefed on and read the APP/SSHP (signature sheet)?	☐	☐	☐
5	Are the following training records current and available:			
	- Appropriate Qualification Certificates (UXO, Equip Operator, Confined Space etc.)?	☐	☐	☐
	- 40 Hour HAZWOPER for ALL employees?	☐	☐	☐
	- 8-Hour HAZWOPER Annual Refresher?	☐	☐	☐
	- CPR/First Aid (minimum two individuals on site)?	☐	☐	☐
	- 8-Hour Hazardous Waste Site Supervisor?	☐	☐	☐
	- Initial Site Health and Safety Briefing?	☐	☐	☐
6	Tailgate Safety Briefings conducted?	☐	☐	☐
7	Is the hospital route clearly posted in vehicles?	☐	☐	☐
8	Were applicable Material Safety Data Sheets at the Site?	☐	☐	☐
9	Personnel and supervisory positions appropriate for the work?	☐	☐	☐
10	Are work/exclusion zones adequately designated and secured?	☐	☐	☐
11	Is personal protective equipment available and correctly used, maintained and stored?	☐	☐	☐
12	Is the following emergency equipment located at each site:			
	- Fire extinguisher (inspected)?	☐	☐	☐
	- Eye wash (minimal)?	☐	☐	☐
	- Communications (radio and phone)?	☐	☐	☐
	- First aid kit?	☐	☐	☐
13	Is the buddy system in use?	☐	☐	☐
14	Are personnel refraining from drinking, chewing, smoking, taking medications, or other hand-to-mouth contact while working in the exclusion zone?	☐	☐	☐
15	Is the site organized with good housekeeping and sanitation practices?	☐	☐	☐
16	Was a random employee asked if he/she know site hazard and emergency procedures?	☐	☐	☐

The QC/Safety Inspector shall sign this checklist upon completion of all items on the checklist.

QC/Safety Inspector ____________________

Signature: Notes:


WORK
ZONE

ENCLOSURE C

Training Certificates

(Certifications will be renewed and updated prior to starting field efforts, as required)

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Board of Certified Safety Professionals

Upon the recommendation of the
Board of Certified Safety Professionals,
by virtue of the authority vested in it,
has conferred on

Michael W Barsa

the credential of

Certified Safety Professional

and has granted the title as evidence of meeting the qualifications and passing the required examination so long as this credential is not suspended or revoked and is renewed annually and meets all recertification requirements.

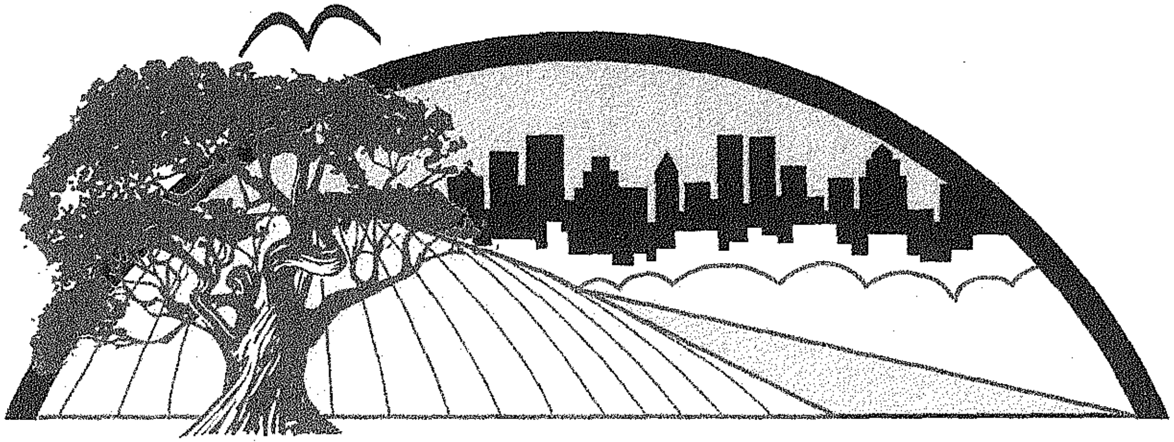
CSP®

October 30, 2013
DATE ISSUED

24437
CERTIFICATION NUMBER

BOARD PRESIDENT SIGNATURE

BOARD SECRETARY SIGNATURE



ALL AMERICAN ENVIRONMENTAL SERVICES, INC.

This is to certify that

MICHAEL W. BARSA

has successfully completed

"HAZARDOUS WASTE SITE WORKER" 40-HOUR COURSE
SATISFYING OSHA 29 CFR 1910.120 (e) (3) (i)

at

ALL AMERICAN SCHOOL OF OCCUPATIONAL SAFETY AND HEALTH
COLUMBIA, MARYLAND

School Director

OCTOBER 25-29, 2004
40S-04010B

Certificate of Completion

This certifies that

Michael Barsa

Has Successfully completed

8 Hour HAZWOPER Refresher Training

Refresher certification does NOT necessarily indicate initial 24 or 40 Hour HAZWOPER certification

In Accordance w/Federal OSHA Regulation 29 CFR 1910.120(e) & (p)

And all State OSHA and EPA Regulations As Well

This course (Version 3) is approved for 8 Contact Hours (0.8 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) issued by Safety Unlimited, Inc. (Accreditation # 044).

Julius P. Griggs

Julius P. Griggs
Instructor #892

160106540532
Certificate Number

1/6/2016
Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley, CA 93063
888 309-SAFE (7233) or 805 306-8027 866-869-7097 (fax)
www.safetyunlimited.com

Proof of initial certification and subsequent refresher training is NOT required to take refresher training
Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

OSHA 600230087

U.S. Department of Labor
Occupational Safety and Health Administration

Michael Barsa

has successfully completed a 30-hour Occupational Safety and Health Training Course in

Construction Safety & Health

M. Barton #278 5/14/2009

(Trainer) (Date)

Certificate of Completion

This certifies that

Michael Barsa

Has Successfully completed

8 Hour HAZWOPER Supervisor Training

This certificate does not in itself indicate initial 24 or 40 Hour HAZWOPER Training

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)(4)

And all State OSHA and EPA Regulations As Well

Julius P. Griggs	101228440532	12/28/2010
Julius P. Griggs Instructor #892	Certificate Number	Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

690A East Los Angeles Ave Suite 180 Simi Valley, CA 93065
888-309-7233 * 805-306-8027 * 866-869-7097 (F)
www.safetyunlimited.com

Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

Certification Card

BasicPlus

CPR, AED, and First Aid
For Adults

Michael Barsa

has successfully completed and competently performed the required knowledge and skill objectives for this program.

MEDIC First Aid

Scott Smith
Authorized Instructor (Print Name)

050703J2247
Registry No.

01/20/16 Class Completion Date	**01/20/18** Expiration Date
410-694-9471 Training Center Phone No.	**172382** Training Center I.D.

This card certifies the holder has demonstrated the required knowledge and skill objectives to a currently authorized MEDIC First Aid Instructor. Certification does not guarantee future performance, or imply licensure or credentialing. Course content conforms to the *2010 AHA Guidelines for CPR and ECC*, and other evidence-based treatment recommendations. Certification period may not exceed 24 months from class completion. More frequent reinforcement of skills is recommended.

ALL AMERICAN ENVIRONMENTAL SERVICES, INC.
CONSULTING ▪ FIELD SERVICES ▪ TRAINING

LETTER OF SATISFACTORY COMPLETION

All American Environmental Services, Inc. hereby certifies that Michael Barsa has satisfactorily completed a course of instruction titled "Medic First Aid®, Basic Plus CPR, AED, and First Aid for Adults" conducted on January 20, 2016.

This course satisfies the training requirements specified by the Department of Labor, Occupational Safety and Health Administration, as found in 29 CFR§1910.151 Subpart K "Medical Services and First Aid; Authority Secs. 4,6,8, of the Occupational Safety and Health Act of 1970" and 29 CFR§1910.266 App B Mandatory First Aid and CPR Training. It's also recognized, endorsed and or approved by the Department of Homeland Security, the United States Coast Guard (DHS/USCG), by state regulatory agencies and the National Registry of Emergency Medical Technicians (NREMT). All treatment guidelines follow the new 2010 American Heart Association Science Advisory "Hands-Only, Compression-Only" Resuscitation; The new 2010 American Heart Association for CPR and Emergency Cardiovascular Care (ECC); 2010 International Consensus on Cardiopulmonary Resuscitation and Emergency Cardiovascular Care Science with Treatment Recommendations, International Liaison Committee on Resuscitation (ILCOR), ASTM Standard F2171-02, ASTM International, and the National Standard Curriculum for Bystander Care, National Highway Traffic Safety Administration, U.S. Department of Transportation.

All American Environmental Services, Inc. certifies that this course satisfies the training requirements for those individuals who will be performing as a First Aid Provider in Occupational Settings.

All American Environmental Services, Inc. provides this letter of satisfactory completion based on this individual's demonstration of practical skills. Certification expires on January 20, 2018. Retraining is recommended within 24 months of issue.

All American Environmental Services, Inc. recommends that this letter be made a part of your employee's personnel file.

Scott A. Smith
Instructor

717 E. Ordnance Road ▪ Suite 207 ▪ Baltimore, MD 21226 ▪ Telephone: 410-694-9471
web page: www.aaesi.com ▪ e-mail: info@aaesi.com

ALL AMERICAN ENVIRONMENTAL SERVICES, INC.

This is to certify that

MICHAEL BARSA

has successfully completed

"BLOODBORNE PATHOGENS"
SATISFYING OSHA 29 CFR 1910.1030

at

ALL AMERICAN SCHOOL OF OCCUPATIONAL SAFETY AND HEALTH
BALTIMORE, MARYLAND

School Director

JANUARY 20, 2016
ERT-1601

PHYSICIAN'S CERTIFICATION/WRITTEN OPINION FOR EMPLOYEE MEDICAL CLEARANCE

Exam Date: 9/8/16 **Expiration Date:** 9/8/17

Employee Name: Michael [illegible] **Employer Name:** ERT, Inc.

Pre-Placement ____ *Annual* ✓ *Exit* ____

EVALUATION FOR HAZARDOUS WASTE OPERATIONS/EMERGENCY RESPONSE (29 CFR 1910.120)

✓ Based on a review of the information obtained from the medical questionnaire and medical exam conducted in accordance with 29 CFR 1910.120, no medical conditions have been detected which would place this individual at increased risk of material impairment of the employee's health from work in a hazardous waste operation or emergency response. There are no limitations upon the employee's assigned work.

____ *Not qualified for hazardous waste work** due to medical problems that restrict full participation at HAZWOPER sites

RESPIRATORY COMPLIANCE MEDICAL EXAMINATION (29 CFR 1910.134)

On the basis of the information obtained from the medical examination and/or a review of the medical questionnaire (including the OSHA Respirator Medical Evaluation Questionnaire), the above-named individual is qualified for:

✓ Full respirator use including: half-face/full-face, air-purifying, and/or SCBA in any work situation.

____ Limited respirator use on a temporary basis. Restrictions below.

____ *Not qualified to use respirator**. May specify other work restrictions.

Restrictions: ____

HEARING CONSERVATION PROGRAM

✓ Baseline and/or annual audiometric testing has been performed and above-named employee has been evaluated**

Examination conducted by:

K.S Winstead PA-C
C0002657 / MD 9/8/16

(Signature) *(Print Name)* *(Date)*

Physician ~~(MD only)~~ reviewing physical and medical results:

Jeffrey J. Hong MD Jeffrey Hong, MD
D33983/MD 9/8/16

(Signature) *(Print Name)* *(Date)*

Physician's Address/Phone Number:

Concentra Medical Center 811 Cromwell Park Drive
Suite 105 Glen Burnie MD 21061 (410)553-0110

Physician's State License Number (if applicable): ____

**If it is the opinion of the examining physician that an examinee is unqualified to perform hazardous waste site work or to wear a respirator, the physician should append a further report to this statement which details reasons for the opinion. Any attached medical information should be treated as Confidential Medical Information in accordance with Section 102C of the Americans with Disabilities Act.*

***As part of the ERT, Inc. Corporate Hearing Conservation Program*

CERTIFICATE OF COMPLETION

This is to certify that

JESSE T. NOLAN

Has successfully completed

40 HOUR OSHA HAZMAT SITEWORKER

Training

In accordance with OSHA 29CFR1910.120

At

Hillis-Carnes Engineering Associates, Inc.

February 27, 2006

Instructor

Hallie R. Goald III

Certificate of Completion

This certifies that

Jesse Nolan

Has Successfully completed

8 Hour HAZWOPER Supervisor Refresher Training

This certification alone does NOT indicate INITIAL 8 Hour OSHA Supervisor Training

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)(8)

And all State OSHA/EPA Regulations as well

This course (Version 1) is approved for 8 Contact Hours (0.8 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) issued by Safety Unlimited, Inc. (Accreditation # 044)

Julius P. Griggs
Julius P. Griggs
Training Director

1605025121712
Certificate Number

5/2/2016
Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley, CA 93063
888 309-SAFE (7233) or 805 306-8027 866-869-7097 (fax)
www.safetyunlimited.com

Proof of initial certification and subsequent refresher training is NOT required to take refresher training
Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

Certificate of Completion

This certifies that

Jesse Nolan

Has Successfully completed

8 Hour HAZWOPER Supervisor Training

This certificate does not in itself indicate initial 24 or 40 Hour HAZWOPER Training

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)(4)

And all State OSHA/EPA Regulations as well

Julius P. Griggs

Julius P. Griggs Instructor #892	120109455411 Certificate Number	1/9/2012 Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

690A East Los Angeles Ave Suite 180 Simi Valley, CA 93065
888-309-7233 * 805-308-8027 * 866-869-7097 (F)
www.safetyunlimited.com

Annual Refresher Training NOT Required

Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

American Red Cross

Jesse Nolan

has successfully completed requirements for

Adult First Aid/CPR/AED: valid 2 Years

Date Completed: 09/28/2016
conducted by: American Red Cross
Instructor: Melody Green

ID: 0Z2TD1
Scan code or visit:
redcross.org/confirm

AFP
AMHERST FAMILY PRACTICE
H. Nelson Gustin, MD • William J. Snyder, MD • Patricia M. Houser, MD • Magan L. Williams, DO

Employee: Jesse Nolan

Company: A-Zone Environmental

The employee listed above is

__✓__ Qualified for any work with no restrictions
_____ Qualified for restricted work

_____ Restricted lifting _______ lbs.
_____ Restricted bending
_____ Restricted climbing
_____ Restricted exposure to ____________________
_____ Restricted periods of standing
_____ Restricted to work suitable for individual of small stature
_____ Other restrictions ____________________

_____ Not qualified for any available work

Diagnosis ______________________________

Recommendations to Employee Stop smoking. Establish c̄ PCP.

____

Remarks ______________________________

____

Date: September 28, 2016

C. Blun PA-C

Jesse Nolan # 729960

1867 Amherst Street • Winchester Virginia 22601 • (540) 667-8724 • 800-892-1AFP • Fax 540-662-5638 • www.afpdoc.com

U.S. ARMY CORPS OF ENGINEERS

USACE LEARNING CENTER
HUNTSVILLE, ALABAMA

NAVFAC

CERTIFICATE

Jesse Nolan

NAO-06-16-00047

has completed the Corps of Engineers and Naval Facility Engineering Command Training Course

CONSTRUCTION QUALITY MANAGEMENT FOR CONTRACTORS - #784

Norfolk, VA	February 9, 2016	Norfolk 1CEU/0.8LU/8PDH	Robert B. Stewart, PE
Location	Training Date(s)	Instructional District/ NAVFAC	CQM-C Manager
Kenneth A. Newton, PE	kanewton@cox.net	757-478-4370	Kenneth A Newton
Facilitator/Instructor	Email	Telephone	Facilitator/Instructor Signature
			Jeffrey D. Dziedzic
			Chief, USACE Learning Center

THIS CERTIFICATE EXPIRES FIVE YEARS FROM DATE OF ISSUE

~~**CQM-C Recertification online course: https://www.myuln.net**~~

Certificate of Completion

Presented to:

JESSE NOLAN

On **1/19/2013**, JESSE **NOLAN** successfully completed the OSHA 30 Hour Outreach Training for the Construction Industry.

Taylor Alan Siker

OSHA Authorized Trainer

As an OSHA Authorized Trainer, I verify that I have conducted this OSHA outreach training class in accordance with OSHA Outreach Training Program requirements. I will document this class to my authorizing OSHA training organization. Upon successful review of my documentation, I will provide each student their completion card within 90 days of the end of the class.

OSHA TRAINING INSTITUTE EDUCATION CENTER

USF UNIVERSITY OF SOUTH FLORIDA

American Safety Council .com

eTraining, Inc.

Certificate of Completion

This certifies that

Jesse Nolan

has received the proper training for successfully completing the

Fall Protection for the Competent Person

OSHA 29 CFR 1926.500

November 11, 2014

Certificate Number: 41179

www.etraintoday.com

Niall O'Malley, President

Larry A. Baylor

Larry A. Baylor, VP Content Development

eTraining, Inc.

Certificate of Completion

This certifies that

Jesse Nolan

has received training and has successfully completed the

Excavation and Trenching for the Competent Person

OSHA 29 CFR 1926.650-652 Subpart P - 5 Hours

January 21, 2016 Certificate Number: 56439

www.etraintoday.com

Niall O'Malley, President

Larry A. Baylor

Larry A. Baylor, VP Content Development

COMMONWEALTH OF VIRGINIA

State Water Control Board

629 East Main Street, Richmond, Virginia 23219

RESPONSIBLE LAND DISTURBER

Jesse T. Nolan

CERTIFICATE NUMBER

RLD02618

EXPIRATION DATE

10/2/2018

This certificate is for your records and should be kept in a safe location. Please detach the above certificate and the two wallet size cards below. It is your responsibility to ensure that your certification is kept current and that you meet the requirements for re-certification before the expiration date.

COMMONWEALTH OF VIRGINIA
State Water Control Board
629 East Main Street, Richmond, Virginia 23219

RESPONSIBLE LAND DISTURBER

Jesse T. Nolan

Certificate Number
RLD02618

Expiration Date
10/2/2018

COMMONWEALTH OF VIRGINIA
State Water Control Board
629 East Main Street, Richmond, Virginia 23219

RESPONSIBLE LAND DISTURBER

Jesse T. Nolan

Certificate Number
RLD02618

Expiration Date
10/2/2018

Certificate of Completion

This certifies that

Robert Koroncai

Has Successfully completed

OSHA 40 Hour HAZWOPER Training

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)

And State OSHA/EPA Regulations as well including 29 CFR 1926.65(e)
This course is approved for 40 Contact Hours (4 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) issued by Safety Unlimited, Inc. (Accreditation# 044)

Julius P. Griggs
Julius P. Griggs
Instructor #892

131219196388
Certificate Number

12/19/2013
Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley, CA 93063
888 309-SAFE (7233) or 805 306-8027 866-869-7097 (fax)
www.safetyunlimited.com

Annual Refresher Training Required
Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

Certificate of Completion

This certifies that

Robert Koroncai

Has Successfully completed

8 Hour HAZWOPER Refresher Training

Refresher certification does NOT necessarily indicate initial 24 or 40 Hour HAZWOPER certification

In Accordance w/Federal OSHA Regulation 29 CFR 1910.120(e) & (p)

And all State OSHA and EPA Regulations As Well

This course (Version 2) is approved for 8 Contact Hours (0.8 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) issued by Safety Unlimited, Inc. (Accreditation# 044).

Julius P. Griggs
Julius P. Griggs
Instructor #892

151231596388
Certificate Number

12/31/2015
Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley, CA 93063
888 309-SAFE (7233) or 805 306-8027 866-869-7097 (fax)
www.safetyunlimited.com

Proof of initial certification and subsequent refresher training is NOT required to take refresher training
Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

Certificate of Completion

This certifies that

Robert Koroncai

Has Successfully completed

8 Hour HAZWOPER Supervisor Training

This certificate does not in itself indicate initial 24 or 40 Hour HAZWOPER Training

In Accordance With Federal OSHA Regulation 29 CFR 1910.120(e)(4)

And all State OSHA/EPA Regulations as well

This course is approved for 8 Contact Hours (0.8 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) issued by Safety Unlimited, Inc. (Accreditation# 044)

Julius P. Griggs

Julius P. Griggs
Instructor #892

150102496388
Certificate Number

1/2/2015
Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley, CA 93063
888 309-SAFE (7233) or 805 306-8027 866-869-7097 (fax)
www.safetyunlimited.com

Annual Refresher Training NOT Required
Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

OSHA Occupational Safety and Health Administration

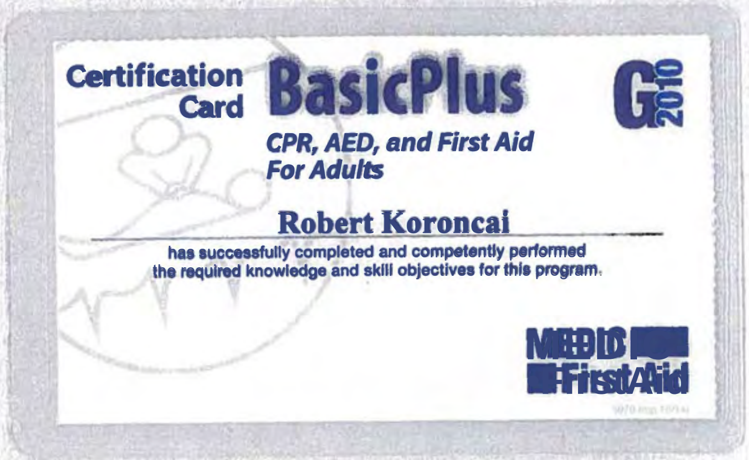
36-601270710

This card acknowledges that the recipient has successfully completed a 30-hour Occupational Safety and Health Training Course in
Construction Safety and Health

ROBERT KORONCAI

Marie Athey — 1/6/2015

(Trainer name – print or type) — (Course end date)



Certification Card **BasicPlus**

G 2010

CPR, AED, and First Aid For Adults

Robert Koroncai

has successfully completed and competently performed the required knowledge and skill objectives for this program.

MEDIC First Aid

Scott Smith

Authorized Instructor (Print Name)

05070312247

Registry No.

01/20/16	**01/20/18**
Class Completion Date	Expiration Date
410-694-9471	**172382**
Training Center Phone No.	Training Center I.D.

This card certifies the holder has demonstrated the required knowledge and skill objectives to a currently authorized MEDIC First Aid Instructor. Certification does not guarantee future performance, or imply licensure or credentialing. Course content conforms to the *2010 AHA Guidelines for CPR and ECC*, and other evidence-based treatment recommendations. Certification period may not exceed 24 months from class completion. More frequent reinforcement of skills is recommended.

ALL AMERICAN ENVIRONMENTAL SERVICES, INC.
CONSULTING ▪ FIELD SERVICES ▪ TRAINING

LETTER OF SATISFACTORY COMPLETION

All American Environmental Services, Inc. hereby certifies that Robert Koroncai has satisfactorily completed a course of instruction titled "Medic First Aid®, Basic Plus CPR, AED, and First Aid for Adults" conducted on January 20, 2016.

This course satisfies the training requirements specified by the Department of Labor, Occupational Safety and Health Administration, as found in 29 CFR§1910.151 Subpart K "Medical Services and First Aid; Authority Secs. 4,6,8, of the Occupational Safety and Health Act of 1970" and 29 CFR§1910.266 App B Mandatory First Aid and CPR Training. It's also recognized, endorsed and or approved by the Department of Homeland Security, the United States Coast Guard (DHS/USCG), by state regulatory agencies and the National Registry of Emergency Medical Technicians (NREMT). All treatment guidelines follow the new 2010 American Heart Association Science Advisory "Hands-Only, Compression-Only" Resuscitation; The new 2010 American Heart Association for CPR and Emergency Cardiovascular Care (ECC); 2010 International Consensus on Cardiopulmonary Resuscitation and Emergency Cardiovascular Care Science with Treatment Recommendations, International Liaison Committee on Resuscitation (ILCOR), ASTM Standard F2171-02, ASTM International, and the National Standard Curriculum for Bystander Care, National Highway Traffic Safety Administration, U.S. Department of Transportation.

All American Environmental Services, Inc. certifies that this course satisfies the training requirements for those individuals who will be performing as a First Aid Provider in Occupational Settings.

All American Environmental Services, Inc. provides this letter of satisfactory completion based on this individual's demonstration of practical skills. Certification expires on January 20, 2018. Retraining is recommended within 24 months of issue.

All American Environmental Services, Inc. recommends that this letter be made a part of your employee's personnel file.

Scott A. Smith
Instructor

717 E. Ordnance Road ▪ Suite 207 ▪ Baltimore, MD 21226 ▪ Telephone: 410-694-9471
web page: www.aaesi.com ▪ e-mail: info@aaesi.com

ALL AMERICAN ENVIRONMENTAL SERVICES, INC.

This is to certify that

ROBERT KORONCAI

has successfully completed

"BLOODBORNE PATHOGENS"
SATISFYING OSHA 29 CFR 1910.1030

at

ALL AMERICAN SCHOOL OF OCCUPATIONAL SAFETY AND HEALTH
BALTIMORE, MARYLAND

School Director

JANUARY 20, 2016
ERT-1601

PHYSICIAN'S CERTIFICATION/WRITTEN OPINION FOR EMPLOYEE MEDICAL CLEARANCE

Exam Date: 1/4/16 **Expiration Date:** 1/4/17

Employee Name: Robert Kerrance **Employer Name:** ERT, Inc.

Pre-Placement ____ ***Annual*** A ***Exit*** ____

EVALUATION FOR HAZARDOUS WASTE OPERATIONS/EMERGENCY RESPONSE (29 CFR 1910.120)

✓ Based on a review of the information obtained from the medical questionnaire and medical exam conducted in accordance with 29 CFR 1910.120, no medical conditions have been detected which would place this individual at increased risk of material impairment of the employee's health from work in a hazardous waste operation or emergency response. There are no limitations upon the employee's assigned work.

____ ***Not qualified for hazardous waste work**** **due to medical problems that restrict full participation at HAZWOPER sites**

RESPIRATORY COMPLIANCE MEDICAL EXAMINATION (29 CFR 1910.134)

On the basis of the information obtained from the medical examination and/or a review of the medical questionnaire (including the OSHA Respirator Medical Evaluation Questionnaire), the above-named individual is qualified for:

✓ Full respirator use including: half-face/full-face, air-purifying, and/or SCBA in any work situation.

____ **Limited respirator use on a temporary basis. Restrictions below:**

____ ***Not qualified to use respirator****. **May specify other work restrictions.**

Restrictions: ____

HEARING CONSERVATION PROGRAM

✓ Baseline and/or annual audiometric testing has been performed and above-named employee has been evaluated**

Examination conducted by:

KRUPA SHAH PA 1/4/16

(Signature) ***(Print Name)*** ***(Date)***

Physician (*MD only*) reviewing physical and medical results:

SAME

(Signature) ***(Print Name)*** ***(Date)***

Physician's Address/Phone Number:

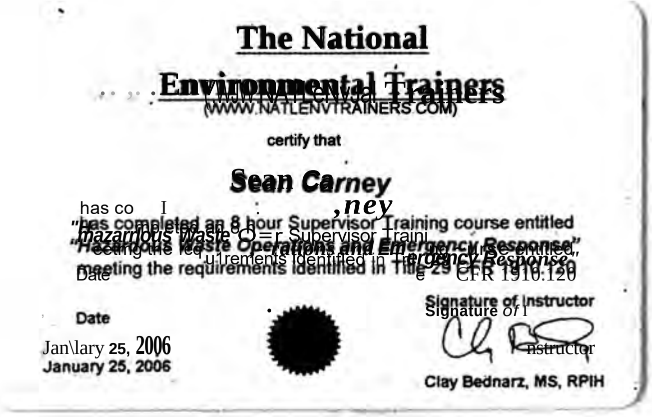
6654 Dobbin Road, Columbia, MD 21045- Concentra, Urgent Care

(410) 381-1330

Physician's State License Number (if applicable): D0038819 (MD

If it is the opinion of the examining physician that an examinee is unqualified to perform hazardous waste site work or to wear a respirator, the physician should append a further report to this statement which details reasons for the opinion. Any attached medical information should be treated as Confidential Medical Information in accordance with Section 102C of the Americans with Disabilities Act.

*****As part of the ERT, Inc. Corporate Hearing Conservation Program***



The National

Environmental Trainers

(WWW.NATLENVTRAINERS.COM)

certify that

Sean Carney

has completed an 8 hour Supervisor Training course entitled

"Hazardous Waste Operations and Emergency Response"

meeting the requirements identified in Title 29 CFR 1910.120

Date

January 25, 2006

Signature of Instructor

Clay Bednarz, MS, RPIH

40-HOUR HAZARDOUS MATERIALS TRAINING

Sean M. Carney

has successfully met the requirements of 29 CFR 1910.120(e)(3) on

March 7-11, 2005

UMD1 02888

Certificate #

Certificate of Completion

This certifies that

Sean Carney

Has Successfully completed

8 Hour HAZWOPER Refresher Training

Refresher certification does NOT necessarily indicate initial 24 or 40 Hour HAZWOPER certification

In Accordance w/Federal OSHA Regulation 29 CFR 1910.120(e) & (p)

And all State OSHA and EPA Regulations As Well

This course (Version 3) is approved for 8 Contact Hours (0.8 CEUs) of continuing education per the California Department of Public Health for Registered Environmental Health Specialist (REHS) issued by Safety Unlimited, Inc. (Accreditation # 044).

Julius P. Griggs
Julius P. Griggs
Instructor #892

160211514845
Certificate Number

2/11/2016
Issue Date

SAFETY UNLIMITED, Inc.
OSHA Compliant Safety Training Since 1993

2139 Tapo St., Suite 228 Simi Valley, CA 93063
888 309-SAFE (7233) or 805 306-8027 866-869-7097 (fax)
www.safetyunlimited.com

Proof of initial certification and subsequent refresher training is NOT required to take refresher training
Want to be sure this certificate is valid? Visit safetyunlimited.com/verification

Certificate of Completion

OSHAcampus.com™
powered by 360training.com

This Certifies That

Sean Carney

is awarded this certificate for

OSHA - 30 Hour Construction Industry Outreach Training Program

Credit Hours: 30

Completion Date: 06/13/2010 10:24 CST

Certificate Number: 1588828

Student Signature

AUTHORIZED IACET PROVIDER

Michael Millsap, Trainer C 0034819 and G 0021414

360training.com, Inc. has been approved as an Authorized Provider by the International Association for Continuing Education and Training (IACET), 1760 Old Meadow Road, Suite 500, McLean, VA 22102; (703) 506-3275.

360training.com ♦ 13801 North Mo pac, Suite 100 ♦ Austin, TX 78727 ♦ 888-360-TRNG ♦ www.360training.com

The National Environmental Trainers

(WWW.NATLENVTRAINERS.COM)

certify that

Sean Carney

has completed an 8 hour Supervisor Training course entitled
"Hazardous Waste Operations and Emergency Response"
meeting the requirements identified in Title 29 CFR 1910.120

Date

January 25, 2006

Signature of Instructor

Clay Bednarz, MS, RPIH

PHYSICIAN'S CERTIFICATION/WRITTEN OPINION FOR EMPLOYEE MEDICAL CLEARANCE

Exam Date: 2-8-16 Expiration Date: 2-8-17

Employee Name: Sean Carney Employer Name: ERT, Inc.

Pre-Placement ____ ***Annual*** X *Exit* ____

EVALUATION FOR HAZARDOUS WASTE OPERATIONS/EMERGENCY RESPONSE (29 CFR 1910.120)

✓ Based on a review of the information obtained from the medical questionnaire and medical exam conducted in accordance with 29 CFR 1910.120, no medical conditions have been detected which would place this individual at increased risk of material impairment of the employee's health from work in a hazardous waste operation or emergency response. There are no limitations upon the employee's assigned work.

____ *Not qualified for hazardous waste work** due to medical problems that restrict full participation at HAZWOPER sites

RESPIRATORY COMPLIANCE MEDICAL EXAMINATION (29 CFR 1910.134)

On the basis of the information obtained from the medical examination and/or a review of the medical questionnaire (including the OSHA Respirator Medical Evaluation Questionnaire), the above-named individual is qualified for:

✓ Full respirator use including: half-face/full-face, air-purifying, and/or SCBA in any work situation.

____ Limited respirator use on a temporary basis. Restrictions below.

____ *Not qualified to use respirator**. May specify other work restrictions.

Restrictions: ____________________

HEARING CONSERVATION PROGRAM

✓ Baseline and/or annual audiometric testing has been performed and above-named employee has been evaluated**

Examination conducted by:

[illegible] Dasgupta 2/9/16

(Signature) *(Print Name)* *(Date)*

Physician (*MD only*) reviewing physical and medical results:

2/9/16

(Signature) *(Print Name)* *(Date)*

Physician's Address/Phone Number:

Concentra Medical Center
10820 Abbotts Bridge Rd., Ste 3000
Duluth, Georgia 30097
770-441-0444 Phone
770-449-7962 Fax

Physician's State License Number (if applicable): ____________________

**If it is the opinion of the examining physician that an examinee is unqualified to perform hazardous waste site work or to wear a respirator, the physician should append a further report to this statement which details reasons for the opinion. Any attached medical information should be treated as Confidential Medical Information in accordance with Section 102C of the Americans with Disabilities Act.*

***As part of the ERT, Inc. Corporate Hearing Conservation Program*

ENCLOSURE D

Safety Data Sheets

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Safety Data Sheet

Material Name: Gasoline All Grades **SDS No. 9950**

US GHS

Synonyms: Hess Conventional (Oxygenated and Non-oxygenated) Gasoline; Reformulated Gasoline (RFG); Reformulated Gasoline Blendstock for Oxygenate Blending (RBOB); Unleaded Motor or Automotive Gasoline

* * * Section 1 - Product and Company Identification * * *

Manufacturer Information

Hess Corporation
1 Hess Plaza
Woodbridge, NJ 07095-0961

Phone: 732-750-6000 Corporate EHS
Emergency # 800-424-9300 CHEMTREC
www.hess.com (Environment, Health, Safety Internet Website)

* * * Section 2 - Hazards Identification * * *

GHS Classification:

Flammable Liquid - Category 2
Skin Corrosion/Irritation - Category 2
Germ Cell Mutagenicity - Category 1B
Carcinogenicity - Category 1B
Toxic to Reproduction - Category 1A
Specific Target Organ Toxicity (Single Exposure) - Category 3 (respiratory irritation, narcosis)
Specific Target Organ Toxicity (Repeat Exposure) - Category 1 (liver, kidneys, bladder, blood, bone marrow, nervous system)
Aspiration Hazard - Category 1
Hazardous to the Aquatic Environment – Acute Hazard - Category 3

GHS LABEL ELEMENTS

Symbol(s)

Signal Word

DANGER

Hazard Statements

Highly flammable liquid and vapour.
Causes skin irritation.
May cause genetic defects.
May cause cancer.
May damage fertility or the unborn child.
May cause respiratory irritation.
May cause drowsiness or dizziness.
Causes damage to organs (liver, kidneys, bladder, blood, bone marrow, nervous system) through prolonged or repeated exposure.
May be fatal if swallowed and enters airways.
Harmful to aquatic life.

Precautionary Statements

Prevention

Keep away from heat/sparks/open flames/hot surfaces. No smoking
Keep container tightly closed.
Ground/bond container and receiving equipment.
Use explosion-proof electrical/ventilating/lighting/equipment.
Use only non-sparking tools.
Take precautionary measures against static discharge.
Wear protective gloves/protective clothing/eye protection/face protection.
Wash hands and forearms thoroughly after handling.
Obtain special instructions before use.
Do not handle until all safety precautions have been read and understood.
Do not breathe mist/vapours/spray.
Use only outdoors or in well-ventilated area.
Do not eat, drink or smoke when using this product.
Avoid release to the environment.

Response

In case of fire: Use water spray, fog, dry chemical fire extinguishers or hand held fire extinguisher.
IF ON SKIN (or hair): Wash with plenty of soap and water. Remove/Take off immediately all contaminated clothing and wash before reuse. If skin irritation occurs, get medical advice/attention.
IF exposed or concerned: Get medical advice/attention.
IF INHALED: Remove victim to fresh air and keep at rest in a position comfortable for breathing. Call a poison center or doctor/physician if you feel unwell.
Get medical advice/attention if you feel unwell.
IF SWALLOWED: Immediately call a POISON CENTER or doctor/physician. Do not induce vomiting.

Storage

Store in a well-ventilated place.
Keep cool. Keep container tightly closed.
Store locked up.

Disposal

Dispose of contents/container in accordance with local/regional/national/international regulations.

* * * Section 3 - Composition / Information on Ingredients * * *

CAS #	Component	Percent
86290-81-5	Gasoline, motor fuel	100
108-88-3	Toluene	1-25
106-97-8	Butane	<10
1330-20-7	Xylenes (o-, m-, p- isomers)	1-15
95-63-6	Benzene, 1,2,4-trimethyl-	<6
64-17-5	Ethyl alcohol	0-10
100-41-4	Ethylbenzene	<3
71-43-2	Benzene	0.1-4.9

110-54-3	Hexane	0.5-4

A complex blend of petroleum-derived normal and branched-chain alkane, cycloalkane, alkene, and aromatic hydrocarbons. May contain antioxidant and multifunctional additives. Non-oxygenated Conventional Gasoline and RBOB do not have oxygenates (Ethanol). Oxygenated Conventional and Reformulated Gasoline will have oxygenates for octane enhancement or as legally required.

* * * Section 4 - First Aid Measures * * *

First Aid: Eyes

In case of contact with eyes, immediately flush with clean, low-pressure water for at least 15 min. Hold eyelids open to ensure adequate flushing. Seek medical attention.

First Aid: Skin

Remove contaminated clothing. Wash contaminated areas thoroughly with soap and water or with waterless hand cleanser. Obtain medical attention if irritation or redness develops.

First Aid: Ingestion

DO NOT INDUCE VOMITING. Do not give liquids. Obtain immediate medical attention. If spontaneous vomiting occurs, lean victim forward to reduce the risk of aspiration. Monitor for breathing difficulties. Small amounts of material which enter the mouth should be rinsed out until the taste is dissipated.

First Aid: Inhalation

Remove person to fresh air. If person is not breathing, provide artificial respiration. If necessary, provide additional oxygen once breathing is restored if trained to do so. Seek medical attention immediately.

* * * Section 5 - Fire Fighting Measures * * *

General Fire Hazards

See Section 9 for Flammability Properties.

Vapors may be ignited rapidly when exposed to heat, spark, open flame or other source of ignition. Flowing product may be ignited by self-generated static electricity. When mixed with air and exposed to an ignition source, flammable vapors can burn in the open or explode in confined spaces. Being heavier than air, vapors may travel long distances to an ignition source and flash back. Runoff to sewer may cause fire or explosion hazard.

Hazardous Combustion Products

Carbon monoxide, carbon dioxide and non-combusted hydrocarbons (smoke). Contact with nitric and sulfuric acids will form nitrocresols that can decompose violently.

Extinguishing Media

SMALL FIRES: Any extinguisher suitable for Class B fires, dry chemical, CO2, water spray, fire fighting foam, or gaseous extinguishing agent.

LARGE FIRES: Water spray, fog or fire fighting foam. Water may be ineffective for fighting the fire, but may be used to cool fire-exposed containers.

Firefighting foam suitable for polar solvents is recommended for fuel with greater than 10% oxygenate concentration.

Unsuitable Extinguishing Media

None

Fire Fighting Equipment/Instructions

Small fires in the incipient (beginning) stage may typically be extinguished using handheld portable fire extinguishers and other fire fighting equipment. Firefighting activities that may result in potential exposure to high heat, smoke or toxic by-products of combustion should require NIOSH/MSHA- approved pressure-demand self-contained breathing apparatus with full facepiece and full protective clothing. Isolate area around container involved in fire. Cool tanks, shells, and containers exposed to fire and excessive heat with water. For massive fires the use of unmanned hose holders or monitor nozzles may be advantageous to further minimize personnel exposure. Major fires may require withdrawal, allowing the tank to burn. Large storage tank fires typically require specially trained personnel and equipment to extinguish the fire, often including the need for properly applied fire fighting foam.

* * * Section 6 - Accidental Release Measures * * *

Recovery and Neutralization

Carefully contain and stop the source of the spill, if safe to do so.

Materials and Methods for Clean-Up

Take up with sand or other oil absorbing materials. Carefully shovel, scoop or sweep up into a waste container for reclamation or disposal. Caution, flammable vapors may accumulate in closed containers.

Emergency Measures

Evacuate nonessential personnel and remove or secure all ignition sources. Consider wind direction; stay upwind and uphill, if possible. Evaluate the direction of product travel, diking, sewers, etc. to confirm spill areas. Spills may infiltrate subsurface soil and groundwater; professional assistance may be necessary to determine the extent of subsurface impact.

Personal Precautions and Protective Equipment

Response and clean-up crews must be properly trained and must utilize proper protective equipment (see Section 8).

Environmental Precautions

Protect bodies of water by diking, absorbents, or absorbent boom, if possible. Do not flush down sewer or drainage systems, unless system is designed and permitted to handle such material. The use of fire fighting foam may be useful in certain situations to reduce vapors. The proper use of water spray may effectively disperse product vapors or the liquid itself, preventing contact with ignition sources or areas/equipment that require protection.

Prevention of Secondary Hazards

None

* * * Section 7 - Handling and Storage * * *

Handling Procedures

USE ONLY AS A MOTOR FUEL.
DO NOT SIPHON BY MOUTH

Handle as a flammable liquid. Keep away from heat, sparks, and open flame! Electrical equipment should be approved for classified area. Bond and ground containers during product transfer to reduce the possibility of static-initiated fire or explosion.

Special slow load procedures for "switch loading" must be followed to avoid the static ignition hazard that can exist when higher flash point material (such as fuel oil) is loaded into tanks previously containing low flash point products (such as this product) - see API Publication 2003, "Protection Against Ignitions Arising Out Of Static, Lightning and Stray Currents."

Storage Procedures

Keep away from flame, sparks, excessive temperatures and open flame. Use approved vented containers. Keep containers closed and clearly labeled. Empty product containers or vessels may contain explosive vapors. Do not pressurize, cut, heat, weld or expose such containers to sources of ignition.

Store in a well-ventilated area. This storage area should comply with NFPA 30 "Flammable and Combustible Liquid Code". Avoid storage near incompatible materials. The cleaning of tanks previously containing this product should follow API Recommended Practice (RP) 2013 "Cleaning Mobile Tanks In Flammable and Combustible Liquid Service" and API RP 2015 "Cleaning Petroleum Storage Tanks".

Incompatibilities

Keep away from strong oxidizers.

* * * Section 8 - Exposure Controls / Personal Protection * * *

Component Exposure Limits

Gasoline, motor fuel (86290-81-5)

ACGIH: 300 ppm TWA
500 ppm STEL

Toluene (108-88-3)

ACGIH: 20 ppm TWA
OSHA: 200 ppm TWA; 375 mg/m3 TWA
150 ppm STEL; 560 mg/m3 STEL
NIOSH: 100 ppm TWA; 375 mg/m3 TWA
150 ppm STEL; 560 mg/m3 STEL

Butane (106-97-8)

ACGIH: 1000 ppm TWA (listed under Aliphatic hydrocarbon gases: Alkane C1-4)
OSHA: 800 ppm TWA; 1900 mg/m3 TWA
NIOSH: 800 ppm TWA; 1900 mg/m3 TWA

Xylenes (o-, m-, p- isomers) (1330-20-7)

ACGIH: 100 ppm TWA
150 ppm STEL
OSHA: 100 ppm TWA; 435 mg/m3 TWA
150 ppm STEL; 655 mg/m3 STEL

Benzene, 1,2,4-trimethyl- (95-63-6)

NIOSH: 25 ppm TWA; 125 mg/m3 TWA

Ethyl alcohol (64-17-5)

ACGIH: 1000 ppm STEL
OSHA: 1000 ppm TWA; 1900 mg/m3 TWA
NIOSH: 1000 ppm TWA; 1900 mg/m3 TWA

Ethylbenzene (100-41-4)

ACGIH:	20 ppm TWA
OSHA:	100 ppm TWA; 435 mg/m3 TWA 125 ppm STEL; 545 mg/m3 STEL
NIOSH:	100 ppm TWA; 435 mg/m3 TWA 125 ppm STEL; 545 mg/m3 STEL

Benzene (71-43-2)

ACGIH:	0.5 ppm TWA 2.5 ppm STEL Skin - potential significant contribution to overall exposure by the cutaneous route
OSHA:	5 ppm STEL (Cancer hazard, Flammable, See 29 CFR 1910.1028, 15 min); 0.5 ppm Action Level; 1 ppm TWA
NIOSH:	0.1 ppm TWA 1 ppm STEL

Hexane (110-54-3)

ACGIH:	50 ppm TWA Skin - potential significant contribution to overall exposure by the cutaneous route
OSHA:	500 ppm TWA; 1800 mg/m3 TWA
NIOSH:	50 ppm TWA; 180 mg/m3 TWA

Engineering Measures

Use adequate ventilation to keep vapor concentrations of this product below occupational exposure and flammability limits, particularly in confined spaces.

Personal Protective Equipment: Respiratory

A NIOSH/MSHA-approved air-purifying respirator with organic vapor cartridges or canister may be permissible under certain circumstances where airborne concentrations are or may be expected to exceed exposure limits or for odor or irritation. Protection provided by air-purifying respirators is limited.

Use a positive pressure, air-supplied respirator if there is a potential for uncontrolled release, exposure levels are not known, in oxygen-deficient atmospheres, or any other circumstance where an air-purifying respirator may not provide adequate protection.

Personal Protective Equipment: Hands

Gloves constructed of nitrile, neoprene, or PVC are recommended.

PERSONAL PROTECTIVE EQUIPMENT

Personal Protective Equipment: Eyes

Safety glasses or goggles are recommended where there is a possibility of splashing or spraying.

Personal Protective Equipment: Skin and Body

Chemical protective clothing such as of E.I. DuPont TyChem®, Saranex® or equivalent recommended based on degree of exposure. Note: The resistance of specific material may vary from product to product as well as with degree of exposure. Consult manufacturer specifications for further information.

* * * Section 9 - Physical & Chemical Properties * * *

Appearance:	Translucent, straw-colored or light yellow	**Odor:**	Strong, characteristic aromatic hydrocarbon odor. Sweet-ether like
Physical State:	Liquid	**pH:**	ND
Vapor Pressure:	6.4 - 15 RVP @ 100 °F (38 °C) (275-475 mm Hg @ 68 °F (20 °C)	**Vapor Density:**	AP 3-4
Boiling Point:	85-437 °F (39-200 °C)	**Melting Point:**	ND
Solubility (H2O):	Negligible to Slight	**Specific Gravity:**	0.70-0.78
Evaporation Rate:	10-11	**VOC:**	ND
Percent Volatile:	100%	**Octanol/H2O Coeff.:**	ND
Flash Point:	-45 °F (-43 °C)	**Flash Point Method:**	PMCC
Upper Flammability Limit (UFL):	7.6%	**Lower Flammability Limit (LFL):**	1.4%
Burning Rate:	ND	**Auto Ignition:**	>530°F (>280°C)

* * * Section 10 - Chemical Stability & Reactivity Information * * *

Chemical Stability

This is a stable material.

Hazardous Reaction Potential

Will not occur.

Conditions to Avoid

Avoid high temperatures, open flames, sparks, welding, smoking and other ignition sources.

Incompatible Products

Keep away from strong oxidizers.

Hazardous Decomposition Products

Carbon monoxide, carbon dioxide and non-combusted hydrocarbons (smoke). Contact with nitric and sulfuric acids will form nitrocresols that can decompose violently.

* * * Section 11 - Toxicological Information * * *

Acute Toxicity

A: General Product Information

Harmful if swallowed.

B: Component Analysis - LD50/LC50

Gasoline, motor fuel (86290-81-5)

Inhalation LC50 Rat >5.2 mg/L 4 h; Oral LD50 Rat 14000 mg/kg; Dermal LD50 Rabbit >2000 mg/kg

Toluene (108-88-3)

Inhalation LC50 Rat 12.5 mg/L 4 h; Inhalation LC50 Rat >26700 ppm 1 h; Oral LD50 Rat 636 mg/kg; Dermal LD50 Rabbit 8390 mg/kg; Dermal LD50 Rat 12124 mg/kg

Butane (106-97-8)

Inhalation LC50 Rat 658 mg/L 4 h

Xylenes (o-, m-, p- isomers) (1330-20-7)
Inhalation LC50 Rat 5000 ppm 4 h; Inhalation LC50 Rat 47635 mg/L 4 h; Oral LD50 Rat 4300 mg/kg; Dermal LD50 Rabbit >1700 mg/kg

Benzene, 1,2,4-trimethyl- (95-63-6)
Inhalation LC50 Rat 18 g/m3 4 h; Oral LD50 Rat 3400 mg/kg; Dermal LD50 Rabbit >3160 mg/kg

Ethyl alcohol (64-17-5)
Oral LD50 Rat 7060 mg/kg; Inhalation LC50 Rat 124.7 mg/L 4 h

Ethylbenzene (100-41-4)
Inhalation LC50 Rat 17.2 mg/L 4 h; Oral LD50 Rat 3500 mg/kg; Dermal LD50 Rabbit 15354 mg/kg

Benzene (71-43-2)
Inhalation LC50 Rat 13050-14380 ppm 4 h; Oral LD50 Rat 1800 mg/kg

Hexane (110-54-3)
Inhalation LC50 Rat 48000 ppm 4 h; Oral LD50 Rat 25 g/kg; Dermal LD50 Rabbit 3000 mg/kg

Potential Health Effects: Skin Corrosion Property/Stimulativeness

Practically non-toxic if absorbed following acute (single) exposure. May cause skin irritation with prolonged or repeated contact. Liquid may be absorbed through the skin in toxic amounts if large areas of skin are repeatedly exposed.

Potential Health Effects: Eye Critical Damage/ Stimulativeness

Moderate irritant. Contact with liquid or vapor may cause irritation.

Potential Health Effects: Ingestion

Ingestion may cause gastrointestinal disturbances, including irritation, nausea, vomiting and diarrhea, and central nervous system (brain) effects similar to alcohol intoxication. In severe cases, tremors, convulsions, loss of consciousness, coma, respiratory arrest, and death may occur.

Potential Health Effects: Inhalation

Excessive exposure may cause irritations to the nose, throat, lungs and respiratory tract. Central nervous system (brain) effects may include headache, dizziness, loss of balance and coordination, unconsciousness, coma, respiratory failure, and death.

WARNING: the burning of any hydrocarbon as a fuel in an area without adequate ventilation may result in hazardous levels of combustion products, including carbon monoxide, and inadequate oxygen levels, which may cause unconsciousness, suffocation, and death.

Respiratory Organs Sensitization/Skin Sensitization

This product is not reported to have any skin sensitization effects.

Generative Cell Mutagenicity

This product may cause genetic defects.

Carcinogenicity

A: General Product Information

May cause cancer.

IARC has determined that gasoline and gasoline exhaust are possibly carcinogenic in humans. Inhalation exposure to completely vaporized unleaded gasoline caused kidney cancers in male rats and liver tumors in female mice. The U.S. EPA has determined that the male kidney tumors are species-specific and are irrelevant for human health risk assessment. The significance of the tumors seen in female mice is not known. Exposure to light hydrocarbons in the same boiling range as this product has been associated in animal studies with effects to the central and peripheral nervous systems, liver, and kidneys. The significance of these animal models to predict similar human response to gasoline is uncertain.

This product contains benzene. Human health studies indicate that prolonged and/or repeated overexposure to benzene may cause damage to the blood-forming system (particularly bone marrow), and serious blood disorders such as aplastic anemia and leukemia. Benzene is listed as a human carcinogen by the NTP, IARC, OSHA and ACGIH.

B: Component Carcinogenicity

Gasoline, motor fuel (86290-81-5)

ACGIH: A3 - Confirmed Animal Carcinogen with Unknown Relevance to Humans

Toluene (108-88-3)

ACGIH: A4 - Not Classifiable as a Human Carcinogen
IARC: Monograph 71 [1999]; Monograph 47 [1989] (Group 3 (not classifiable))

Xylenes (o-, m-, p- isomers) (1330-20-7)

ACGIH: A4 - Not Classifiable as a Human Carcinogen
IARC: Monograph 71 [1999]; Monograph 47 [1989] (Group 3 (not classifiable))

Ethyl alcohol (64-17-5)

ACGIH: A3 - Confirmed Animal Carcinogen with Unknown Relevance to Humans
IARC: Monograph 100E [in preparation] (in alcoholic beverages); Monograph 96 [2010] (in alcoholic beverages) (Group 1 (carcinogenic to humans))

Ethylbenzene (100-41-4)

ACGIH: A3 - Confirmed Animal Carcinogen with Unknown Relevance to Humans
IARC: Monograph 77 [2000] (Group 2B (possibly carcinogenic to humans))

Benzene (71-43-2)

ACGIH: A1 - Confirmed Human Carcinogen
OSHA: 5 ppm STEL (Cancer hazard, Flammable, See 29 CFR 1910.1028, 15 min); 0.5 ppm Action Level; 1 ppm TWA
NIOSH: potential occupational carcinogen
NTP: Known Human Carcinogen (Select Carcinogen)
IARC: Monograph 100F [in preparation]; Supplement 7 [1987]; Monograph 29 [1982] (Group 1 (carcinogenic to humans))

Reproductive Toxicity

This product is suspected of damaging fertility or the unborn child.

Specified Target Organ General Toxicity: Single Exposure

This product may cause drowsiness or dizziness.

Specified Target Organ General Toxicity: Repeated Exposure

This product causes damage to organs through prolonged or repeated exposure.

Aspiration Respiratory Organs Hazard

The major health threat of ingestion occurs from the danger of aspiration (breathing) of liquid drops into the lungs, particularly from vomiting. Aspiration may result in chemical pneumonia (fluid in the lungs), severe lung damage, respiratory failure and even death.

* * * Section 12 - Ecological Information * * *

Ecotoxicity

A: General Product Information

Very toxic to aquatic life with long lasting effects. Keep out of sewers, drainage areas and waterways. Report spills and releases, as applicable, under Federal and State regulations.

B: Component Analysis - Ecotoxicity - Aquatic Toxicity

Gasoline, motor fuel (86290-81-5)

Test & Species		Conditions
96 Hr LC50 Alburnus alburnus	119 mg/L [static]	
96 Hr LC50 Cyprinodon variegatus	82 mg/L [static]	
72 Hr EC50 Pseudokirchneriella subcapitata	56 mg/L	
24 Hr EC50 Daphnia magna	170 mg/L	

Toluene (108-88-3)

Test & Species		Conditions
96 Hr LC50 Pimephales promelas	15.22-19.05 mg/L [flow-through]	1 day old
96 Hr LC50 Pimephales promelas	12.6 mg/L [static]	
96 Hr LC50 Oncorhynchus mykiss	5.89-7.81 mg/L [flow-through]	
96 Hr LC50 Oncorhynchus mykiss	14.1-17.16 mg/L [static]	
96 Hr LC50 Oncorhynchus mykiss	5.8 mg/L [semi-static]	
96 Hr LC50 Lepomis macrochirus	11.0-15.0 mg/L [static]	
96 Hr LC50 Oryzias latipes	54 mg/L [static]	
96 Hr LC50 Poecilia reticulata	28.2 mg/L [semi-static]	
96 Hr LC50 Poecilia reticulata	50.87-70.34 mg/L [static]	
96 Hr EC50 Pseudokirchneriella subcapitata	>433 mg/L	
72 Hr EC50 Pseudokirchneriella subcapitata	12.5 mg/L [static]	
48 Hr EC50 Daphnia magna	5.46 - 9.83 mg/L [Static]	
48 Hr EC50 Daphnia magna	11.5 mg/L	

Xylenes (o-, m-, p- isomers) (1330-20-7)

Test & Species		Conditions
96 Hr LC50 Pimephales promelas	13.4 mg/L [flow-through]	

96 Hr LC50 Oncorhynchus mykiss	2.661-4.093 mg/L [static]	
96 Hr LC50 Oncorhynchus mykiss	13.5-17.3 mg/L	
96 Hr LC50 Lepomis macrochirus	13.1-16.5 mg/L [flow-through]	
96 Hr LC50 Lepomis macrochirus	19 mg/L	
96 Hr LC50 Lepomis macrochirus	7.711-9.591 mg/L [static]	
96 Hr LC50 Pimephales promelas	23.53-29.97 mg/L [static]	
96 Hr LC50 Cyprinus carpio	780 mg/L [semi-static]	
96 Hr LC50 Cyprinus carpio	>780 mg/L	
96 Hr LC50 Poecilia reticulata	30.26-40.75 mg/L [static]	
48 Hr EC50 water flea	3.82 mg/L	
48 Hr LC50 Gammarus lacustris	0.6 mg/L	

Benzene, 1,2,4-trimethyl- (95-63-6)

Test & Species		Conditions
96 Hr LC50 Pimephales promelas	7.19-8.28 mg/L [flow-through]	
48 Hr EC50 Daphnia magna	6.14 mg/L	

Ethyl alcohol (64-17-5)

Test & Species		Conditions
96 Hr LC50 Oncorhynchus mykiss	12.0 - 16.0 mL/L [static]	
96 Hr LC50 Pimephales promelas	>100 mg/L [static]	
96 Hr LC50 Pimephales promelas	13400 - 15100 mg/L [flow-through]	
48 Hr LC50 Daphnia magna	9268 - 14221 mg/L	
24 Hr EC50 Daphnia magna	10800 mg/L	
48 Hr EC50 Daphnia magna	2 mg/L [Static]	

Ethylbenzene (100-41-4)

Test & Species		Conditions
96 Hr LC50 Oncorhynchus mykiss	11.0-18.0 mg/L [static]	
96 Hr LC50 Oncorhynchus mykiss	4.2 mg/L [semi-static]	
96 Hr LC50 Pimephales promelas	7.55-11 mg/L [flow-through]	
96 Hr LC50 Lepomis macrochirus	32 mg/L [static]	
96 Hr LC50 Pimephales promelas	9.1-15.6 mg/L [static]	
96 Hr LC50 Poecilia reticulata	9.6 mg/L [static]	
72 Hr EC50 Pseudokirchneriella subcapitata	4.6 mg/L	
96 Hr EC50 Pseudokirchneriella subcapitata	>438 mg/L	
72 Hr EC50 Pseudokirchneriella subcapitata	2.6 - 11.3 mg/L [static]	

96 Hr EC50 Pseudokirchneriella subcapitata	1.7 - 7.6 mg/L [static]	
48 Hr EC50 Daphnia magna	1.8 - 2.4 mg/L	

Benzene (71-43-2)

Test & Species		Conditions
96 Hr LC50 Pimephales promelas	10.7-14.7 mg/L [flow-through]	
96 Hr LC50 Oncorhynchus mykiss	5.3 mg/L [flow-through]	
96 Hr LC50 Lepomis macrochirus	22.49 mg/L [static]	
96 Hr LC50 Poecilia reticulata	28.6 mg/L [static]	
96 Hr LC50 Pimephales promelas	22330-41160 µg/L [static]	
96 Hr LC50 Lepomis macrochirus	70000-142000 µg/L [static]	
72 Hr EC50 Pseudokirchneriella subcapitata	29 mg/L	
48 Hr EC50 Daphnia magna	8.76 - 15.6 mg/L [Static]	
48 Hr EC50 Daphnia magna	10 mg/L	

Hexane (110-54-3)

Test & Species		Conditions
96 Hr LC50 Pimephales promelas	2.1-2.98 mg/L [flow-through]	
24 Hr EC50 Daphnia magna	>1000 mg/L	

Persistence/Degradability

No information available.

Bioaccumulation

No information available.

Mobility in Soil

No information available.

* * * Section 13 - Disposal Considerations * * *

Waste Disposal Instructions

See Section 7 for Handling Procedures. See Section 8 for Personal Protective Equipment recommendations.

Disposal of Contaminated Containers or Packaging

Dispose of contents/container in accordance with local/regional/national/international regulations.

* * * Section 14 - Transportation Information * * *

Component Marine Pollutants

This material contains one or more of the following chemicals required by US DOT to be identified as marine pollutants.

Component	CAS #	
Gasoline, motor fuel	86290-81-5	DOT regulated marine pollutant

DOT Information

Shipping Name: Gasoline

UN #: 1203 **Hazard Class:** 3 **Packing Group:** II

Placard:

* * * Section 15 - Regulatory Information * * *

Regulatory Information

A: Component Analysis

This material contains one or more of the following chemicals required to be identified under SARA Section 302 (40 CFR 355 Appendix A), SARA Section 313 (40 CFR 372.65) and/or CERCLA (40 CFR 302.4).

Toluene (108-88-3)

SARA 313: 1.0 % de minimis concentration
CERCLA: 1000 lb final RQ; 454 kg final RQ

Xylenes (o-, m-, p- isomers) (1330-20-7)

SARA 313: 1.0 % de minimis concentration
CERCLA: 100 lb final RQ; 45.4 kg final RQ

Benzene, 1,2,4-trimethyl- (95-63-6)

SARA 313: 1.0 % de minimis concentration

Ethylbenzene (100-41-4)

SARA 313: 0.1 % de minimis concentration
CERCLA: 1000 lb final RQ; 454 kg final RQ

Benzene (71-43-2)

SARA 313: 0.1 % de minimis concentration
CERCLA: 10 lb final RQ (received an adjusted RQ of 10 lbs based on potential carcinogenicity in an August 14, 1989 final rule); 4.54 kg final RQ (received an adjusted RQ of 10 lbs based on potential carcinogenicity in an August 14, 1989 final rule)

Hexane (110-54-3)

SARA 313: 1.0 % de minimis concentration
CERCLA: 5000 lb final RQ; 2270 kg final RQ

SARA Section 311/312 – Hazard Classes

Acute Health	Chronic Health	Fire	Sudden Release of Pressure	Reactive
X	X	X	--	--

Component Marine Pollutants

This material contains one or more of the following chemicals required by US DOT to be identified as marine pollutants.

Component	CAS #	
Gasoline, motor fuel	86290-81-5	DOT regulated marine pollutant

State Regulations

Component Analysis - State

The following components appear on one or more of the following state hazardous substances lists:

Component	**CAS**	**CA**	**MA**	**MN**	**NJ**	**PA**	**RI**
Gasoline, motor fuel	86290-81-5	No	No	No	No	Yes	No
Toluene	108-88-3	Yes	Yes	Yes	Yes	Yes	No
Butane	106-97-8	Yes	Yes	Yes	Yes	Yes	No
Xylenes (o-, m-, p- isomers)	1330-20-7	Yes	Yes	Yes	Yes	Yes	No
Benzene, 1,2,4-trimethyl-	95-63-6	No	Yes	Yes	Yes	Yes	No
Ethyl alcohol	64-17-5	Yes	Yes	Yes	Yes	Yes	No
Ethylbenzene	100-41-4	Yes	Yes	Yes	Yes	Yes	No
Benzene	71-43-2	Yes	Yes	Yes	Yes	Yes	No
Hexane	110-54-3	No	Yes	Yes	Yes	Yes	No

The following statement(s) are provided under the California Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65):

WARNING! This product contains a chemical known to the state of California to cause cancer.
WARNING! This product contains a chemical known to the state of California to cause reproductive/developmental effects.

Component Analysis - WHMIS IDL

The following components are identified under the Canadian Hazardous Products Act Ingredient Disclosure List:

Component	CAS #	Minimum Concentration
Toluene	108-88-3	1 %
Butane	106-97-8	1 %
Benzene, 1,2,4-trimethyl-	95-63-6	0.1 %
Ethyl alcohol	64-17-5	0.1 %
Ethylbenzene	100-41-4	0.1 %
Benzene	71-43-2	0.1 %
Hexane	110-54-3	1 %

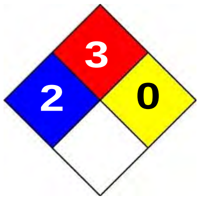
Additional Regulatory Information

Component Analysis - Inventory

Component	CAS #	TSCA	CAN	EEC
Gasoline, motor fuel	86290-81-5	No	DSL	EINECS
Toluene	108-88-3	Yes	DSL	EINECS
Butane	106-97-8	Yes	DSL	EINECS
Xylenes (o-, m-, p- isomers)	1330-20-7	Yes	DSL	EINECS
Benzene, 1,2,4-trimethyl-	95-63-6	Yes	DSL	EINECS
Ethyl alcohol	64-17-5	Yes	DSL	EINECS
Ethylbenzene	100-41-4	Yes	DSL	EINECS
Benzene	71-43-2	Yes	DSL	EINECS
Hexane	110-54-3	Yes	DSL	EINECS

* * * Section 16 - Other Information * * *

NFPA® Hazard Rating	Health	2	
	Fire	3	
	Reactivity	0	
HMIS® Hazard Rating	Health	2	Moderate
	Fire	3	Serious
	Physical	0	Minimal
			*Chronic



Key/Legend

EPA = Environmental Protection Agency; TSCA = Toxic Substance Control Act; ACGIH = American Conference of Governmental Industrial Hygienists; IARC = International Agency for Research on Cancer; NIOSH = National Institute for Occupational Safety and Health; NTP = National Toxicology Program; OSHA = Occupational Safety and Health Administration., NJTSR = New Jersey Trade Secret Registry.

Literature References

None

Other Information

Information presented herein has been compiled from sources considered to be dependable, and is accurate and reliable to the best of our knowledge and belief, but is not guaranteed to be so. Since conditions of use are beyond our control, we make no warranties, expressed or implied, except those that may be contained in our written contract of sale or acknowledgment.

Vendor assumes no responsibility for injury to vendee or third persons proximately caused by the material if reasonable safety procedures are not adhered to as stipulated in the data sheet. Additionally, vendor assumes no responsibility for injury to vendee or third persons proximately caused by abnormal use of the material, even if reasonable safety procedures are followed. Furthermore, vendee assumes the risk in their use of the material.

End of Sheet

Safety Data Sheet

Material Name: Diesel Fuel, All Types

SDS No. 9909
US GHS

Synonyms: Ultra Low Sulfur Diesel; Low Sulfur Diesel; No. 2 Diesel; Motor Vehicle Diesel Fuel; Non-Road Diesel Fuel; Locomotive/Marine Diesel Fuel

* * * Section 1 - Product and Company Identification * * *

Manufacturer Information

Hess Corporation
1 Hess Plaza
Woodbridge, NJ 07095-0961

Phone: 732-750-6000 Corporate EHS
Emergency # 800-424-9300 CHEMTREC
www.hess.com (Environment, Health, Safety Internet Website)

* * * Section 2 - Hazards Identification * * *

GHS Classification:

Flammable Liquids - Category 3
Skin Corrosion/Irritation – Category 2
Germ Cell Mutagenicity – Category 2
Carcinogenicity - Category 2
Specific Target Organ Toxicity (Single Exposure) - Category 3 (respiratory irritation, narcosis)
Aspiration Hazard – Category 1
Hazardous to the Aquatic Environment, Acute Hazard – Category 3

GHS LABEL ELEMENTS

Symbol(s)

Signal Word

DANGER

Hazard Statements

Flammable liquid and vapor.
Causes skin irritation.
Suspected of causing genetic defects.
Suspected of causing cancer.
May cause respiratory irritation.
May cause drowsiness or dizziness.
May be fatal if swallowed and enters airways.
Harmful to aquatic life.

Precautionary Statements

Prevention

Keep away from heat/sparks/open flames/hot surfaces. No smoking
Keep container tightly closed.
Ground/bond container and receiving equipment.

Use explosion-proof electrical/ventilating/lighting/equipment.
Use only non-sparking tools.
Take precautionary measures against static discharge.
Wear protective gloves/protective clothing/eye protection/face protection.
Wash hands and forearms thoroughly after handling.
Obtain special instructions before use.
Do not handle until all safety precautions have been read and understood.
Avoid breathing fume/mist/vapours/spray.

Response
In case of fire: Use water spray, fog or foam to extinguish.
IF ON SKIN (or hair): Wash with plenty of soap and water. Remove/Take off immediately all contaminated clothing and wash it before reuse. If skin irritation occurs: Get medical advice/attention.
IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a poison center/doctor if you feel unwell.
If swallowed: Immediately call a poison center or doctor. Do NOT induce vomiting.
IF exposed or concerned: Get medical advice/attention.

Storage
Store in a well-ventilated place. Keep cool.
Keep container tightly closed.
Store locked up.

Disposal
Dispose of contents/container in accordance with local/regional/national/international regulations.

* * * Section 3 - Composition / Information on Ingredients * * *

CAS #	Component	Percent
68476-34-6	Fuels, diesel, no. 2	100
91-20-3	Naphthalene	<0.1

A complex mixture of hydrocarbons with carbon numbers in the range C9 and higher.

* * * Section 4 - First Aid Measures * * *

First Aid: Eyes

In case of contact with eyes, immediately flush with clean, low-pressure water for at least 15 min. Hold eyelids open to ensure adequate flushing. Seek medical attention.

First Aid: Skin

Remove contaminated clothing. Wash contaminated areas thoroughly with soap and water or with waterless hand cleanser. Obtain medical attention if irritation or redness develops. Thermal burns require immediate medical attention depending on the severity and the area of the body burned.

First Aid: Ingestion

DO NOT INDUCE VOMITING. Do not give liquids. Obtain immediate medical attention. If spontaneous vomiting occurs, lean victim forward to reduce the risk of aspiration. Monitor for breathing difficulties. Small amounts of material which enter the mouth should be rinsed out until the taste is dissipated.

First Aid: Inhalation

Remove person to fresh air. If person is not breathing, provide artificial respiration. If necessary, provide additional oxygen once breathing is restored if trained to do so. Seek medical attention immediately.

* * * Section 5 - Fire Fighting Measures * * *

General Fire Hazards

See Section 9 for Flammability Properties.

Vapors may be ignited rapidly when exposed to heat, spark, open flame or other source of ignition. When mixed with air and exposed to an ignition source, flammable vapors can burn in the open or explode in confined spaces. Being heavier than air, vapors may travel long distances to an ignition source and flash back. Runoff to sewer may cause fire or explosion hazard.

Hazardous Combustion Products

Carbon monoxide, carbon dioxide and non-combusted hydrocarbons (smoke).

Extinguishing Media

SMALL FIRES: Any extinguisher suitable for Class B fires, dry chemical, CO2, water spray, fire fighting foam, and other gaseous agents.

LARGE FIRES: Water spray, fog or fire fighting foam. Water may be ineffective for fighting the fire, but may be used to cool fire-exposed containers.

Unsuitable Extinguishing Media

None

Fire Fighting Equipment/Instructions

Small fires in the incipient (beginning) stage may typically be extinguished using handheld portable fire extinguishers and other fire fighting equipment. Firefighting activities that may result in potential exposure to high heat, smoke or toxic by-products of combustion should require NIOSH/MSHA- approved pressure-demand self-contained breathing apparatus with full facepiece and full protective clothing. Isolate area around container involved in fire. Cool tanks, shells, and containers exposed to fire and excessive heat with water. For massive fires the use of unmanned hose holders or monitor nozzles may be advantageous to further minimize personnel exposure. Major fires may require withdrawal, allowing the tank to burn. Large storage tank fires typically require specially trained personnel and equipment to extinguish the fire, often including the need for properly applied fire fighting foam.

* * * Section 6 - Accidental Release Measures * * *

Recovery and Neutralization

Carefully contain and stop the source of the spill, if safe to do so.

Materials and Methods for Clean-Up

Take up with sand or other oil absorbing materials. Carefully shovel, scoop or sweep up into a waste container for reclamation or disposal. Caution, flammable vapors may accumulate in closed containers.

Emergency Measures

Evacuate nonessential personnel and remove or secure all ignition sources. Consider wind direction; stay upwind and uphill, if possible. Evaluate the direction of product travel, diking, sewers, etc. to confirm spill areas. Spills may infiltrate subsurface soil and groundwater; professional assistance may be necessary to determine the extent of subsurface impact.

Personal Precautions and Protective Equipment

Response and clean-up crews must be properly trained and must utilize proper protective equipment (see Section 8).

Environmental Precautions

Protect bodies of water by diking, absorbents, or absorbent boom, if possible. Do not flush down sewer or drainage systems, unless system is designed and permitted to handle such material. The use of fire fighting foam may be useful in certain situations to reduce vapors. The proper use of water spray may effectively disperse product vapors or the liquid itself, preventing contact with ignition sources or areas/equipment that require protection.

Prevention of Secondary Hazards

None

* * * Section 7 - Handling and Storage * * *

Handling Procedures

Handle as a combustible liquid. Keep away from heat, sparks, excessive temperatures and open flame! No smoking or open flame in storage, use or handling areas. Bond and ground containers during product transfer to reduce the possibility of static-initiated fire or explosion.

Special slow load procedures for "switch loading" must be followed to avoid the static ignition hazard that can exist when higher flash point material (such as fuel oil) is loaded into tanks previously containing low flash point products (such as this product) - see API Publication 2003, "Protection Against Ignitions Arising Out Of Static, Lightning and Stray Currents."

Storage Procedures

Keep away from flame, sparks, excessive temperatures and open flame. Use approved vented containers. Keep containers closed and clearly labeled. Empty product containers or vessels may contain explosive vapors. Do not pressurize, cut, heat, weld or expose such containers to sources of ignition.

Store in a well-ventilated area. This storage area should comply with NFPA 30 "Flammable and Combustible Liquid Code". Avoid storage near incompatible materials. The cleaning of tanks previously containing this product should follow API Recommended Practice (RP) 2013 "Cleaning Mobile Tanks In Flammable and Combustible Liquid Service" and API RP 2015 "Cleaning Petroleum Storage Tanks."

Incompatibilities

Keep away from strong oxidizers.

* * * Section 8 - Exposure Controls / Personal Protection * * *

Component Exposure Limits

Fuels, diesel, no. 2 (68476-34-6)

ACGIH: 100 mg/m3 TWA (inhalable fraction and vapor, as total hydrocarbons, listed under Diesel fuel)
Skin - potential significant contribution to overall exposure by the cutaneous route (listed under Diesel fuel)

Naphthalene (91-20-3)

ACGIH:	10 ppm TWA 15 ppm STEL Skin - potential significant contribution to overall exposure by the cutaneous route
OSHA:	10 ppm TWA; 50 mg/m3 TWA
NIOSH:	10 ppm TWA; 50 mg/m3 TWA 15 ppm STEL; 75 mg/m3 STEL

Engineering Measures

Use adequate ventilation to keep vapor concentrations of this product below occupational exposure and flammability limits, particularly in confined spaces.

Personal Protective Equipment: Respiratory

A NIOSH/MSHA-approved air-purifying respirator with organic vapor cartridges or canister may be permissible under certain circumstances where airborne concentrations are or may be expected to exceed exposure limits or for odor or irritation. Protection provided by air-purifying respirators is limited.

Use a positive pressure, air-supplied respirator if there is a potential for uncontrolled release, exposure levels are not known, in oxygen-deficient atmospheres, or any other circumstance where an air-purifying respirator may not provide adequate protection.

Personal Protective Equipment: Hands

Gloves constructed of nitrile, neoprene, or PVC are recommended.

Personal Protective Equipment: Eyes

Safety glasses or goggles are recommended where there is a possibility of splashing or spraying.

Personal Protective Equipment: Skin and Body

Chemical protective clothing such as of E.I. DuPont TyChem®, Saranex® or equivalent recommended based on degree of exposure. Note: The resistance of specific material may vary from product to product as well as with degree of exposure. Consult manufacturer specifications for further information.

* * * Section 9 - Physical & Chemical Properties * * *

Appearance:	Clear, straw-yellow.	**Odor:**	Mild, petroleum distillate odor
Physical State:	Liquid	**pH:**	ND
Vapor Pressure:	0.009 psia @ 70 °F (21 °C)	**Vapor Density:**	>1.0
Boiling Point:	320 to 690 °F (160 to 366 °C)	**Melting Point:**	ND
Solubility (H2O):	Negligible	**Specific Gravity:**	0.83-0.876 @ 60°F (16°C)
Evaporation Rate:	Slow; varies with conditions	**VOC:**	ND
Percent Volatile:	100%	**Octanol/H2O Coeff.:**	ND
Flash Point:	>125 °F (>52 °C) minimum	**Flash Point Method:**	PMCC
Upper Flammability Limit (UFL):	7.5	**Lower Flammability Limit (LFL):**	0.6
Burning Rate:	ND	**Auto Ignition:**	494°F (257°C)

* * * Section 10 - Chemical Stability & Reactivity Information * * *

Chemical Stability

This is a stable material.

Hazardous Reaction Potential

Will not occur.

Conditions to Avoid

Avoid high temperatures, open flames, sparks, welding, smoking and other ignition sources.

Incompatible Products

Keep away from strong oxidizers.

Hazardous Decomposition Products

Carbon monoxide, carbon dioxide and non-combusted hydrocarbons (smoke).

* * * Section 11 - Toxicological Information * * *

Acute Toxicity

A: General Product Information

Harmful if swallowed.

B: Component Analysis - LD50/LC50

Naphthalene (91-20-3)

Inhalation LC50 Rat >340 mg/m3 1 h; Oral LD50 Rat 490 mg/kg; Dermal LD50 Rat >2500 mg/kg; Dermal LD50 Rabbit >20 g/kg

Potential Health Effects: Skin Corrosion Property/Stimulativeness

Practically non-toxic if absorbed following acute (single) exposure. May cause skin irritation with prolonged or repeated contact. Liquid may be absorbed through the skin in toxic amounts if large areas of skin are repeatedly exposed.

Potential Health Effects: Eye Critical Damage/ Stimulativeness

Contact with eyes may cause mild irritation.

Potential Health Effects: Ingestion

Ingestion may cause gastrointestinal disturbances, including irritation, nausea, vomiting and diarrhea, and central nervous system (brain) effects similar to alcohol intoxication. In severe cases, tremors, convulsions, loss of consciousness, coma, respiratory arrest, and death may occur.

Potential Health Effects: Inhalation

Excessive exposure may cause irritations to the nose, throat, lungs and respiratory tract. Central nervous system (brain) effects may include headache, dizziness, loss of balance and coordination, unconsciousness, coma, respiratory failure, and death.

WARNING: the burning of any hydrocarbon as a fuel in an area without adequate ventilation may result in hazardous levels of combustion products, including carbon monoxide, and inadequate oxygen levels, which may cause unconsciousness, suffocation, and death.

Respiratory Organs Sensitization/Skin Sensitization

This product is not reported to have any skin sensitization effects.

Generative Cell Mutagenicity

This material has been positive in a mutagenicity study.

Carcinogenicity

A: General Product Information

Suspected of causing cancer.

Studies have shown that similar products produce skin tumors in laboratory animals following repeated applications without washing or removal. The significance of this finding to human exposure has not been determined. Other studies with active skin carcinogens have shown that washing the animal's skin with soap and water between applications reduced tumor formation.

B: Component Carcinogenicity

Fuels, diesel, no. 2 (68476-34-6)

ACGIH:	A3 - Confirmed Animal Carcinogen with Unknown Relevance to Humans (listed under Diesel fuel)

Naphthalene (91-20-3)

ACGIH:	A4 - Not Classifiable as a Human Carcinogen
NTP:	Reasonably Anticipated To Be A Human Carcinogen (Possible Select Carcinogen)
IARC:	Monograph 82 [2002] (Group 2B (possibly carcinogenic to humans))

Reproductive Toxicity

This product is not reported to have any reproductive toxicity effects.

Specified Target Organ General Toxicity: Single Exposure

This product is not reported to have any specific target organ general toxicity single exposure effects.

Specified Target Organ General Toxicity: Repeated Exposure

This product is not reported to have any specific target organ general toxicity repeat exposure effects.

Aspiration Respiratory Organs Hazard

The major health threat of ingestion occurs from the danger of aspiration (breathing) of liquid drops into the lungs, particularly from vomiting. Aspiration may result in chemical pneumonia (fluid in the lungs), severe lung damage, respiratory failure and even death.

* * * Section 12 - Ecological Information * * *

Ecotoxicity

A: General Product Information

Keep out of sewers, drainage areas and waterways. Report spills and releases, as applicable, under Federal and State regulations.

B: Component Analysis - Ecotoxicity - Aquatic Toxicity

Fuels, diesel, no. 2 (68476-34-6)

Test & Species		**Conditions**
96 Hr LC50 Pimephales promelas	35 mg/L [flow-through]	

Naphthalene (91-20-3)

Test & Species		**Conditions**
96 Hr LC50 Pimephales promelas	5.74-6.44 mg/L [flow-through]	
96 Hr LC50 Oncorhynchus mykiss	1.6 mg/L [flow-through]	
96 Hr LC50 Oncorhynchus mykiss	0.91-2.82 mg/L [static]	
96 Hr LC50 Pimephales promelas	1.99 mg/L [static]	

96 Hr LC50 Lepomis macrochirus	31.0265 mg/L [static]
72 Hr EC50 Skeletonema costatum	0.4 mg/L
48 Hr LC50 Daphnia magna	2.16 mg/L
48 Hr EC50 Daphnia magna	1.96 mg/L [Flow through]
48 Hr EC50 Daphnia magna	1.09 - 3.4 mg/L [Static]

Persistence/Degradability

No information available.

Bioaccumulation

No information available.

Mobility in Soil

No information available.

* * * Section 13 - Disposal Considerations * * *

Waste Disposal Instructions

See Section 7 for Handling Procedures. See Section 8 for Personal Protective Equipment recommendations.

Disposal of Contaminated Containers or Packaging

Dispose of contents/container in accordance with local/regional/national/international regulations.

* * * Section 14 - Transportation Information * * *

DOT Information

Shipping Name: Diesel Fuel

NA #: 1993 **Hazard Class:** 3 **Packing Group:** III

Placard:

* * * Section 15 - Regulatory Information * * *

Regulatory Information

Component Analysis

This material contains one or more of the following chemicals required to be identified under SARA Section 302 (40 CFR 355 Appendix A), SARA Section 313 (40 CFR 372.65) and/or CERCLA (40 CFR 302.4).

Naphthalene (91-20-3)

CERCLA: 100 lb final RQ; 45.4 kg final RQ

SARA Section 311/312 – Hazard Classes

Acute Health	Chronic Health	Fire	Sudden Release of Pressure	Reactive
X	X	X	--	--

SARA SECTION 313 - SUPPLIER NOTIFICATION

This product may contain listed chemicals below the de minimis levels which therefore are not subject to the supplier notification requirements of Section 313 of the Emergency Planning and Community Right- To-Know Act (EPCRA) of 1986 and of 40 CFR 372. If you may be required to report releases of chemicals listed in 40 CFR 372.28, you may contact Hess Corporate Safety if you require additional information regarding this product.

State Regulations

Component Analysis - State

The following components appear on one or more of the following state hazardous substances lists:

Component	CAS	CA	MA	MN	NJ	PA	RI
Fuels, diesel, no. 2	68476-34-6	No	No	No	Yes	No	No
Naphthalene	91-20-3	Yes	Yes	Yes	Yes	Yes	No

The following statement(s) are provided under the California Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65):

WARNING! This product contains a chemical known to the state of California to cause cancer.

Component Analysis - WHMIS IDL

No components are listed in the WHMIS IDL.

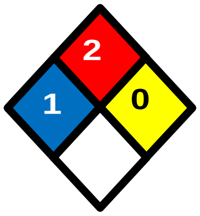
Additional Regulatory Information

Component Analysis - Inventory

Component	CAS #	TSCA	CAN	EEC
Fuels, diesel, no. 2	68476-34-6	Yes	DSL	EINECS
Naphthalene	91-20-3	Yes	DSL	EINECS

* * * Section 16 - Other Information * * *

NFPA® Hazard Rating		
	Health	1
	Fire	2
	Reactivity	0



HMIS® Hazard Rating			
	Health	1*	Slight
	Fire	2	Moderate
	Physical	0	Minimal
			*Chronic

Key/Legend

ACGIH = American Conference of Governmental Industrial Hygienists; ADG = Australian Code for the Transport of Dangerous Goods by Road and Rail; ADR/RID = European Agreement of Dangerous Goods by Road/Rail; AS = Standards Australia; DFG = Deutsche Forschungsgemeinschaft; DOT = Department of Transportation; DSL = Domestic Substances List; EEC = European Economic Community; EINECS = European Inventory of Existing Commercial Chemical Substances; ELINCS = European List of Notified Chemical Substances; EU = European Union; HMIS = Hazardous Materials Identification System; IARC = International Agency for Research on Cancer; IMO = International Maritime Organization; IATA = International Air Transport Association; MAK = Maximum Concentration Value in the Workplace; NDSL = Non-Domestic Substances List; NFPA = National Fire Protection Association; NOHSC = National Occupational Health & Safety Commission; NTP = National Toxicology Program; STEL = Short-term Exposure Limit; TDG = Transportation of Dangerous Goods; TLV = Threshold Limit Value; TSCA = Toxic Substances Control Act; TWA = Time Weighted Average

Literature References

None

Other Information

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Vendor assumes no responsibility for injury to vendee or third persons proximately caused by the material if reasonable safety procedures are not adhered to as stipulated in the data sheet. Additionally, vendor assumes no responsibility for injury to vendee or third persons proximately caused by abnormal use of the material, even if reasonable safety procedures are followed. Furthermore, vendee assumes the risk in their use of the material.

End of Sheet

ENCLOSURE E

Medical Transfer Point Map

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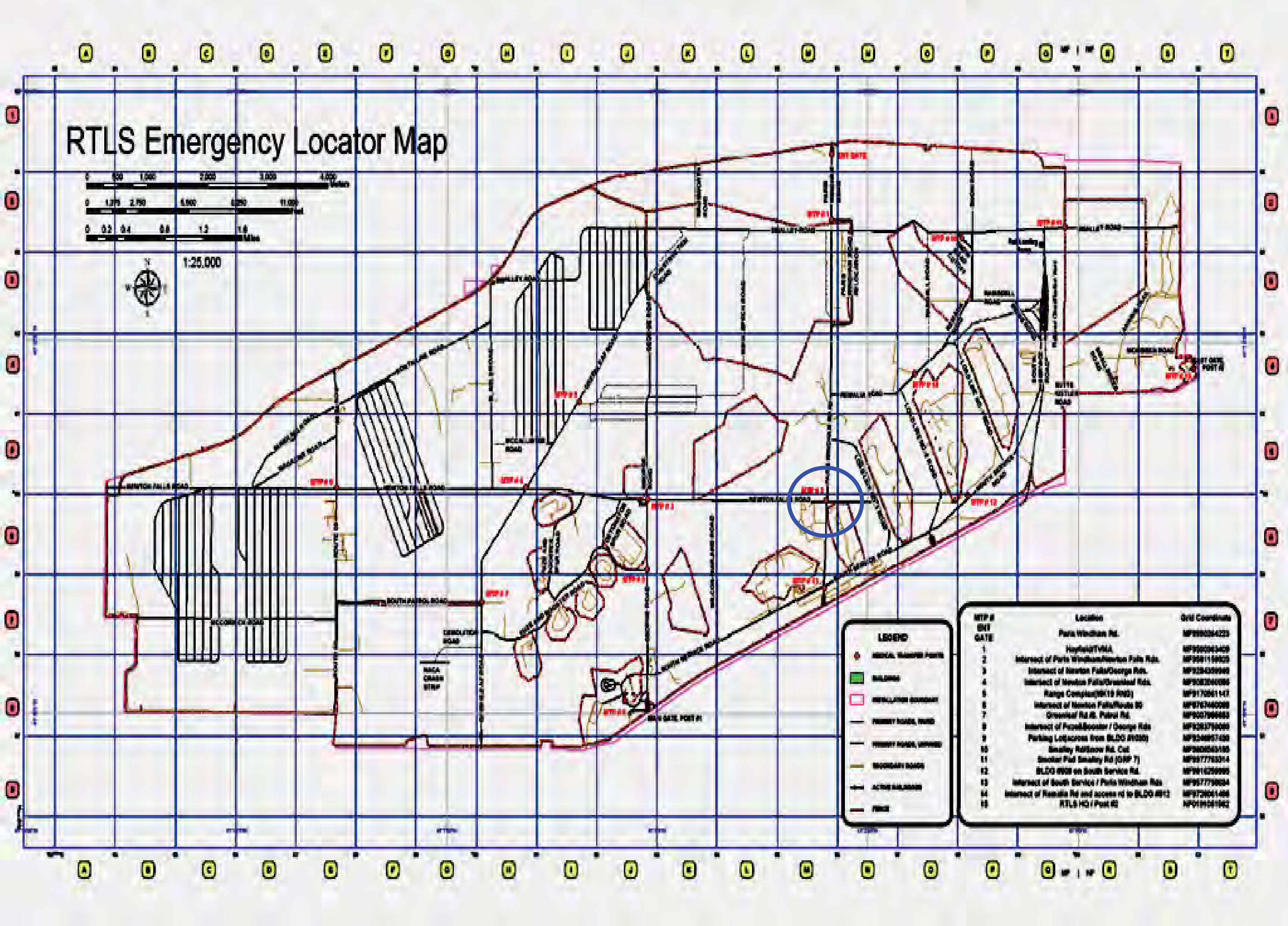
RTLS Emergency Locator Map
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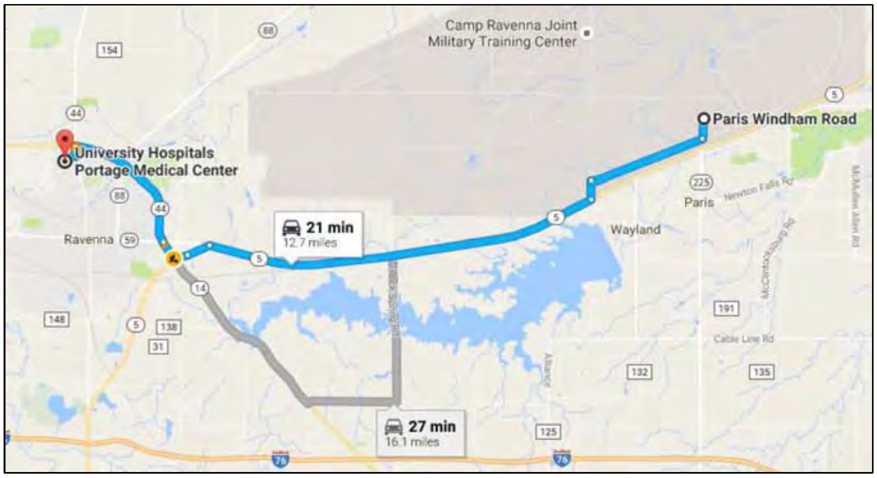
Meters
Feet
Miles
NEWTON FALLS ROAD
SMALLEY ROAD
SOUTH PATROL ROAD
MCCORMICK ROAD
REMALIA ROAD
SOUTH SERVICE ROAD
DEMOLITION ROAD
NACA CRASH STRIP
MAIN GATE, POST #1
ENT GATE
EAST GATE POST #2
LEGEND
MEDICAL TRANSFER POINTS
BUILDINGS
INSTALLATION BOUNDARY
PRIMARY ROADS, PAVED
PRIMARY ROADS, UNPAVED
SECONDARY ROADS
ACTIVE RAILROADS
FENCE
MTP # ENT GATE
Location
Grid Coordinate
Paris Windham Rd.
1 Hayfield/TVMA
2 Intersect of Paris Windham/Newton Falls Rds.
3 Intersect of Newton Falls/George Rds.
4 Intersect of Newton Falls/Greenleaf Rds.
5 Range Complex(MK19 RND)
6 Intersect of Newton Falls/Route 80
7 Greenleaf Rd.&S. Patrol Rd.
8 Intersect of Pozzi&Booster / George Rds
9 Parking Lot(across from BLDG #1036)
10 Smalley Rd/Snow Rd. Cut
11 Shooter Pad Smalley Rd (ORP 7)
12 BLDG #908 on South Service Rd.
13 Intersect of South Service / Paris Windham Rds
14 Intersect of Remalia Rd and access rd to BLDG #912
15 RTLS HQ / Post #2

Emergency Contact Phone Numbers		
Service/Contact	**Agency/Position**	**Telephone No.**
Range Control	Emergency	614-336-6041
University Hospitals Portage Medical Center	Emergency Room	(330) 297-2850
University Hospitals Portage Medical Center	Non-emergency medical	(330) 297-0811 (non-emergency)
Spill Response	Chemical Transportation Emergency Center (CHEMTREC)	(800) 424-9300
Poison Control	Poison Control Center	(800) 222-1222
City of Twinsburg Animal Control	Animal Control	(330) 425-1234
Mark Leeper	ARNG Restoration Program Manager	(703) 607-7955
Kevin Sedlak	ARNG	(614) 336-2053
Katie Tait	OHARNG	(614) 336-6136
Craig Coombs	CELRL PM	(502) 315-6324
Matt Burg	CELRL Chief of Safety	(502) 315-7061
Nat Peters	CELRL COR	(502)315-2624
Kevin Mieczkowski	CELRL TM	(502) 315-7447
Michael Barsa	ERT Division SHM	(301) 323-1447 (o) (410) 703-6213 (c)
Jennifer Harlan	ERT Division Manager	(301) 323-1394 (o)
Sean Carney	ERT PM	(607) 259-7017) (c)
J.T. Nolan	ERT SSHO/Site Superintendent/OSHA Competent Person for Overall Site Health and Safety	(301) 412-7301
Robert Koroncai	Alternate ERT SSHO/OSHA Competent Person for Overall Site Health and Safety	(301) 323-1445 (o) (267) 481-5567 (c)

University Hospitals Portage Medical Center

6847 N Chestnut St,
Ravenna, OH 44266
Phone number: (330) 297-0811



Directions:

From Camp Ravenna, Ohio

1. Head south on Paris Windham Rd., 0.3 mi

2. Turn Right onto first cross street, 1.9 mi

3. Turn Left onto Pa St. 1, 0.3 mi

4. Turn Right onto OH-5 W, 6.4 mi

5. Turn Left onto OH-5W/OH-5 Bypass, 0.4 mi

6. Use Right lane to take OH-14 ramp, 0.2 mi

7. Turn Right onto OH-14 W/OH-44 N/Cleveland – East Liverpool Rd., 2.4 mi

8. Arrive at University Hospitals Portage Medical Center, 0.2 mi

Distance from site to University Hospitals Portage Medical Center = 12.7 miles (21 minutes)

ATTACHMENT 3

Activity Hazard Analysis

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ACTIVITY HAZARDS ANALYSIS

Overall Risk Assessment Code (RAC)
(Use highest code)
L

Date: 12 September 2016

Project: RVAAP, Atlas Scrap Yard AOC

Activity: Site Visit and Inspection

Activity Location: RVAAP, Atlas Scrap Yard AOC

Prepared By: Michael Barsa, CSP

Risk Assessment Code Matrix

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

Add Identified Hazards

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Loading and Unloading Equipment	Lifting too heavy a load while loading/ unloading supplies or equipment, lifting a load for too long, and/or carrying a load for a farther distance than expected	Workers will be advised on: Proper lifting techniques such as keeping the back straight; Lifting with the legs; Avoid twisting the back ; Use mechanical help or assistance from others whenever possible; Break heavy loads down into smaller, lighter loads, if possible; Ensure the path of travel is clear prior to lifting and transporting the load. Anticipate distance and time necessary for transport. Wear leather gloves while moving equipment, if necessary.	L
X	Loading and Unloading Equipment	Not being aware of wet/slippery surfaces being present near transport vehicle, poor housekeeping, poor visibility due to lack of adequate lighting, being unaware of rough/ uneven terrain, not utilizing footwear for task at hand, and/or having line of sight be impaired due to size of load being carried	The immediate work area will be visually inspected continuously by the workers to mark, move, or barricade hazard areas to prevent mishaps. Work areas will be kept organized; workers should plan chosen route of travel; proper boots should be worn (safety toe) per the SSHP; work areas will be well lit (work will only occur during daylight hours); loads will be inspected prior to transport to ensure line of site is not obstructed due to size of load.	L
X	Transporting Equipment via Motor Vehicle	While operating motor vehicle, encountering traffic/unmindful drivers, encountering poor road conditions/ruts, snow, ice, puddles/poor traction, and/or lack of clear communication with spotter while maneuvering in tight areas	Workers will drive defensively at all time and slow vehicle speeds to match road conditions. Seat belts will be worn at all times while operating vehicle. A spotter will be used to maneuver into tight areas and back up vehicle; spotter and operator will ensure communication and signals are understood prior to operation of vehicle. Use of lights, hazards, brakes, and directional signals will be used as necessary.	L
X	Walking site areas, observing, and documenting (photographs and/or entries in logbooks)	Not being aware of wet/slippery surfaces being present near transport vehicle, poor housekeeping, poor visibility due to lack of adequate lighting, being unaware of rough/ uneven terrain, and/or not utilizing footwear for task at hand	The path of travel for site walking will be visually inspected continuously by workers to avoid obstacles, and proper footwear will be donned (safety toe boots) per the SSHP. Work areas will be kept organized; work areas will be well lit (work will only occur in daylight hours).	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Walking site areas, observing, and documenting (photographs and/or entries in logbooks)	Working in vicinity of moving vehicles while inspecting work areas, not being aware of surroundings, not using an understandable means of signaling to the vehicle operator, and/or not being visible to operators of moving vehicles	Workers will be cognizant of avoiding moving vehicles when conducting operations in close proximity to parking lots, streets, and/or driveways; High visibility apparel will be made available to workers conducting operations where moving vehicles may be present; barricades such as cones and/or tape may be used; if possible, prior to vehicle operation, operator and worker will ensure means of signaling is understood; if not, possible, worker will maintain distance from all moving vehicles .	L
X	Walking site areas, observing, and documenting (photographs and/or entries in logbooks)	Not being accustomed to humidity/temperature extremes associated with outdoor work; wearing incorrect clothing for the weather (i.e. short sleeves when its cold); working in direct sunlight, working in wet clothing, lack of training on monitoring of the WBGT, and/or lack of training in monitoring of heart rates.	Workers will be cognizant of heat and cold stress symptoms and associated First Aid procedures. Electrolyte/fluids replacement will be readily available to workers. Appropriate clothing/PPE as defined in the SSHP will be donned. Work/Rest regimens will be established per the SSHP as necessary. The SSHO will have a WBGT meter on site for monitoring temperature according to manufacturer's instructions, and the SSHO will check and record the radial pulse of any worker showing signs of potential heat stress. The radial artery can be found close to the inside part of the wrist near the thumb. The SSHO will use the middle and index fingers to find the radial artery, and the SSHO will count the radial pulse for a full minute (60 seconds) to determine heart rate in beats per minute. In the field logbook, the SSHO will note the date/time, the heart rate, relative strength of the beat, which wrist used, and anything anomalous. For individuals with normal cardiac performance, sustained (over several minutes) heart rate should remain below 180 beats per minute minus age (in years), recovery heart rate at one minute after a peak work effort should be below 110 bpm, and there should be no symptoms of sudden and severe fatigue, nausea, dizziness, or lightheadedness.	L
X	Walking site areas, observing, and documenting (photographs and/or entries in logbooks)	Continuing to work outside during thunderstorm; continuing to work in high wind conditions; working in especially cold/wet conditions	Work will cease when severe weather conditions prevail. Shelter will be sought in field vehicles, never under trees. The SSHO will monitor the weather and advise the workers with the approach of inclement weather. In the event of thunder/lightning, workers will stand down until at least 30 minutes after last lightning observed or thunder heard. Workers should be equipped with appropriate rain gear and dress in layers.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Walking site areas, observing, and documenting (photographs and/or entries in logbooks)	Walking through tall grass where ticks may be present; not being aware of bees, wasps, and/or hornets in work area; stepping on/running into nest that may be present in the work area; not being aware of potentially poisonous insects/ animals that may be encountered on site; being in area where worker should not be and disturbing insects/animals; not recognizing plants as poisonous and/or being in area where worker does not need to be where poisonous plants may exist	Workers will be briefed on the site specific biological hazards (as described in the SSHP) during the initial site orientation. Appropriate First Aid measures will be identified for these hazards; Workers will be advised to avoid contact with these hazards (i.e., walk around tall grass if possible, ensure long pants and long sleeves are worn) and to check themselves at end of the day for ticks. If tick are discovered on the skin, it must immediately be reported to the SSHO. Site personnel will be encouraged to wear light color clothing with cuffs and openings closed. If any worker is allergic to bee/wasp/hornet stings, the SSHO should be advised, and an epinephrine injector should be readily available for that individual(s). If any wild animals are encountered on site, workers will avoid the area until the animal moves and/or seek shelter in vehicles. The SSHP will be consulted so that workers can properly identify poisonous plants potentially encountered on site, and workers will avoid them. If these plants cannot be avoided, long pants, long sleeves, gloves, and/or barrier creams may be used.	L
X	Walking site areas, observing, and documenting (photographs and/or entries in logbooks)	Not wearing shirt with sleeves/long pants while being exposed to sunlight; not utilizing sunscreen; not utilizing shade where available; not shielding eyes from bright sun	Workers will be advised in the proper use of sun blocking agents and shaded areas will be sought out on site; Workers should avoid direct exposure to the sun for long periods of time. Pop up tents may be used to create shade, if available. Safety glasses may be worn with UVA/UVB protection.	L

Add Items

	EQUIPMENT	TRAINING	INSPECTION
X	Camera and/or Field Logbook	Per manufacturers' instructions and per site work plan/ QAPP	Ensuring camera and any data cards are functional prior to use on site; ensuring field logbook is utilized in accordance with the site work plan/QAPP
X	Safety supplies (Safety-toed boots, potable water, sunscreen, insect repellent, leather gloves, etc.)	40-hr HAZWOPER and Annual Refresher Per manufacturers' manuals and/or instructions	In accordance with manufacturers' instructions

Involved Personnel:

SSHO, Site Superintendent, Field Team

Relevant Competent Person(s): SSHO

PPE: Level D, modified to include safety-toe work boots

Acceptance Authority (digital signature):

Digitally signed by Michael W. Barsa, CSP
Date: 2016.09.22 12:05:42 -04'00'

ACTIVITY HAZARDS ANALYSIS

Overall Risk Assessment Code (RAC)
(Use highest code)
L

Date: 4 August 2016

Project: RVAAP, Atlas Scrap Yard AOC

Activity: Mobilization - Demobilization

Activity Location: RVAAP, Atlas Scrap Yard AOC

Prepared By: Michael Barsa, CSP

Risk Assessment Code Matrix

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

Severity	Frequent	Likely	Occasional	Seldom	Unlikely
Catastrophic	E	E	H	H	M
Critical	E	H	H	M	L
Marginal	H	M	M	L	L
Negligible	M	L	L	L	L

Add Identified Hazards

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Material Handling (manual) - muscle strain / back injury from manual lifting	Do not lift beyond your capabilities. Adhere to proper lifting techniques. Consider the following before engaging in a work activity that may cause back injury: • Know the weight to be lifted and postural requirements. • Check that the object will remain stable when moved. • Evaluate if a mechanical device should be used to move the object (e.g., hand truck, cart, dolly, pallet jack, etc.). • Determine number of people needed to move object safely. • Consider personnel medical limitations/restrictions. • Consider environmental conditions (e.g., slippery conditions, lighting, etc.). • Plan the lift and the travel path before starting activity. Implement one of the following controls for objects greater than 50 pounds: • Engineer the lift out of the task through work planning. • Use a mechanical device. • Use two or more people.	L
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Material Handling (manual) - injury from sharps (i.e., materials/equipment having corners, edges or projections capable of cutting or piercing the skin)	Wear cut-resistant work gloves when the possibility of lacerations or other injury may occur. Utilize mechanical device, if appropriate, to avoid directly handling sharps. Maintain all hand and power tools in a safe condition. Wear a face shield for protection from glass breakage.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Vehicle Accidents/Collisions	All equipment will be properly secured during transport. Use ground guide and back-up alarm during backing. Vehicle and equipment operators should look in the direction of travel; look before backing up. Arrange traffic flow to prevent foot traffic from crossing the routes of heavy equipment and moving loads. Ensure the operator acknowledges your presence before walking near equipment in operation. Use of cell phones by the driver/operator is prohibited while the vehicle/equipment is in motion.	L
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Heavy/Hauling Equipment Operation - vehicle traffic	Look in the direction of travel before backing up. Arrange traffic flow to prevent foot traffic from crossing the routes of heavy/hauling equipment and moving loads. Adhere to posted speed limits. Use reflective warning vests when exposed to vehicle traffic. Reroute traffic as necessary to minimize potential for an accident. Utilize dedicated flaggers when necessary at site roadway crossings. Set-up signs, traffic cones and/or barricades to define and protect the project area from the non project-related vehicle traffic.	L
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Slip/Trip/Fall	Use caution when working on uneven or wet ground. Practice good housekeeping Use caution when working on uneven or wet ground. Eliminate or clearly mark protrusions and obstructions.	L
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Not being accustomed to humidity/ temperature extremes associated with outdoor work; wearing incorrect clothing for the weather (i.e. short sleeves when its cold); working in direct sunlight, working in wet clothing, lack of training on monitoring of the WBGT, and/or lack of training in monitoring of heart rates.	Workers will be cognizant of heat and cold stress symptoms and associated First Aid procedures. Electrolyte/fluids replacement will be readily available to workers. Appropriate clothing/PPE as defined in the SSHP will be donned. Work/Rest regimens will be established per the SSHP as necessary. The SSHO will have a WBGT meter on site for monitoring temperature according to manufacturer's instructions, and the SSHO will check and record the radial pulse of any worker showing signs of potential heat stress. The radial artery can be found close to the inside part of the wrist near the thumb. The SSHO will use the middle and index fingers to find the radial artery, and the SSHO will count the radial pulse for a full minute (60 seconds) to determine heart rate in beats per minute. In the field logbook, the SSHO will note the date/ time, the heart rate, relative strength of the beat, which wrist used, and anything anomalous. For individuals with normal cardiac performance, sustained (over several minutes) heart rate should remain below 180 beats per minute minus age (in years), recovery heart rate at one minute after a peak work effort should be below 110 bpm, and there should be no symptoms of sudden and severe fatigue, nausea, dizziness, or lightheadedness.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Continuing to work outside during thunderstorm; continuing to work in high wind conditions; working in especially cold/wet conditions	Work will cease when severe weather conditions prevail. Shelter will be sought in field vehicles, never under trees. The SSHO will monitor the weather and advise the workers with the approach of inclement weather. In the event of thunder/lightning, workers will stand down until at least 30 minutes after last lightning observed or thunder heard. Workers should be equipped with appropriate rain gear and dress in layers.	L
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Walking through tall grass where ticks may be present; not being aware of bees, wasps, and/or hornets in work area; stepping on/running into nest that may be present in the work area; not being aware of potentially poisonous insects/animals that may be encountered on site; being in area where worker should not be and disturbing insects/animals; not recognizing plants as poisonous and/or being in area where worker does not need to be where poisonous plants may exist	Workers will be briefed on the site specific biological hazards (as described in the SSHP) during the initial site orientation. Appropriate First Aid measures will be identified for these hazards; Workers will be advised to avoid contact with these hazards (i.e., walk around tall grass if possible, ensure long pants and long sleeves are worn) and to check themselves at end of the day for ticks. If tick are discovered on the skin, it must immediately be reported to the SSHO. Site personnel will be encouraged to wear light color clothing with cuffs and openings closed. If any worker is allergic to bee/wasp/hornet stings, the SSHO should be advised, and an epinephrine injector should be readily available for that individual(s). If any wild animals are encountered on site, workers will avoid the area until the animal moves and/or seek shelter in vehicles. The SSHP will be consulted so that workers can properly identify poisonous plants potentially encountered on site, and workers will avoid them. If these plants cannot be avoided, long pants, long sleeves, gloves, and/or barrier creams may be used.	L
X	Mobilize/Demobilize Equipment • Travel to site • Deliver/ship heavy equipment • Unload equipment/supplies	Not wearing shirt with sleeves/long pants while being exposed to sunlight; not utilizing sunscreen; not utilizing shade where available; not shielding eyes from bright sun	Workers will be advised in the proper use of sun blocking agents and shaded areas will be sought out on site; Workers should avoid direct exposure to the sun for long periods of time. Pop up tents may be used to create shade, if available. Safety glasses may be worn with UVA/UVB protection.	L

Add Items

	EQUIPMENT	TRAINING	INSPECTION
X	Semi-tractor with lowboy	Proper use, inspection, and care of equipment prior to use.	Daily Inspection
X	Truck	Proper use, inspection, and care of equipment prior to use.	Daily Inspection
X	Safety supplies (Safety-toed boots, potable water, sunscreen, insect repellent, leather gloves, etc.)	40-hr HAZWOPER and Annual Refresher Per manufacturers' manuals and/or instructions	In accordance with manufacturers' instructions
X	Camera and/or Field Logbook	Per manufacturers' instructions and per site work plan/QAPP	Ensuring camera and any data cards are functional prior to use on site; ensuring field logbook is utilized in accordance with the site work plan/QAPP

ACTIVITY HAZARDS ANALYSIS

Involved Personnel:

SSHO, Site Superintendent, Field Team

Relevant Competent Person(s): SSHO

PPE: Level D, modified to include safety-toe work boots

Acceptance Authority (digital signature): Digitally signed by Michael W. Barsa, CSP
Date: 2016.09.22 12:06:34 -04'00'

ACTIVITY HAZARDS ANALYSIS

Overall Risk Assessment Code (RAC)
(Use highest code)
L

Date: 12 September 2016

Project: RVAAP, Atlas Scrap Yard AOC

Activity: Site Preparation

Activity Location: RVAAP, Atlas Scrap Yard AOC

Prepared By: Michael Barsa, CSP

Risk Assessment Code Matrix

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

Add Identified Hazards

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Loading and Unloading Equipment	Physical injury from heavy lifting and pinch points	Workers will be advised on; Proper lifting techniques such as keeping the back straight; Lifting with the legs; Avoid twisting the back; Use mechanical help or two-man lift whenever weight of item being lifted exceeds 50 pounds; Break heavy loads down into smaller, lighter loads, if possible; Ensure the path of travel is clear prior to lifting and transporting the load. Wear gloves while moving equipment. See Section 14A, EM 385-1-1 and NIOSH, Work Practices Guide for Manual Lifting.	L
X	Loading and Unloading Equipment	Slips, trips, and falls	The work area will be visually inspected continuously by the workers to mark, move, or barricade hazard areas to prevent mishaps. Work areas will be kept organized; work areas will be well lit.	L
X	Transporting Equipment	Operating Motor Vehicle: Traffic/unmindful drivers; Road conditions/ruts, snow, ice, puddles/poor traction; Maneuvering in tight areas/potential vehicle or personnel damage	Workers will drive defensively at all time and slow vehicle speeds to match road conditions. Seat belts will be worn at all times while operating vehicle. A spotter will be used to maneuver into tight areas and back up vehicle. Use of lights, hazards, brakes, and directionals will be used as necessary.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Transporting Equipment	Exposure to vehicular traffic	Workers will be trained and cognizant of managing/avoiding vehicular traffic when conducting operations in close proximity to streets and driveways; Workers conducting operations that expose them to traffic hazards will wear high visibility apparel.	L
X	General	Not being accustomed to humidity/temperature extremes associated with outdoor work; wearing incorrect clothing for the weather (i.e. short sleeves when its cold); working in direct sunlight, working in wet clothing, lack of training on monitoring of the WBGT, and/or lack of training in monitoring of heart rates.	Workers will be cognizant of heat and cold stress symptoms and associated First Aid procedures. Electrolyte/fluids replacement will be readily available to workers. Appropriate clothing/PPE as defined in the SSHP will be donned. Work/Rest regimens will be established per the SSHP as necessary. The SSHO will have a WBGT meter on site for monitoring temperature according to manufacturer's instructions, and the SSHO will check and record the radial pulse of any worker showing signs of potential heat stress. The radial artery can be found close to the inside part of the wrist near the thumb. The SSHO will use the middle and index fingers to find the radial artery, and the SSHO will count the radial pulse for a full minute (60 seconds) to determine heart rate in beats per minute. In the field logbook, the SSHO will note the date/time, the heart rate, relative strength of the beat, which wrist used, and anything anomalous. For individuals with normal cardiac performance, sustained (over several minutes) heart rate should remain below 180 beats per minute minus age (in years), recovery heart rate at one minute after a peak work effort should be below 110 bpm, and there should be no symptoms of sudden and severe fatigue, nausea, dizziness, or lightheadedness.	L
X	General	Continuing to work outside during thunderstorm; continuing to work in high wind conditions; working in especially cold/wet conditions	Work will cease when severe weather conditions prevail. Shelter will be sought in field vehicles, never under trees. The SSHO will monitor the weather and advise the workers with the approach of inclement weather. In the event of thunder/lightning, workers will stand down until at least 30 minutes after last lightning observed or thunder heard. Workers should be equipped with appropriate rain gear and dress in layers.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	General	Walking through tall grass where ticks may be present; not being aware of bees, wasps, and/or hornets in work area; stepping on/running into nest that may be present in the work area; not being aware of potentially poisonous insects/animals that may be encountered on site; being in area where worker should not be and disturbing insects/animals; not recognizing plants as poisonous and/or being in area where worker does not need to be where poisonous plants may exist	Workers will be briefed on the site specific biological hazards (as described in the SSHP) during the initial site orientation. Appropriate First Aid measures will be identified for these hazards; Workers will be advised to avoid contact with these hazards (i.e., walk around tall grass if possible, ensure long pants and long sleeves are worn) and to check themselves at end of the day for ticks. If tick are discovered on the skin, it must immediately be reported to the SSHO. Site personnel will be encouraged to wear light color clothing with cuffs and openings closed. If any worker is allergic to bee/wasp/hornet stings, the SSHO should be advised, and an epinephrine injector should be readily available for that individual(s). If any wild animals are encountered on site, workers will avoid the area until the animal moves and/or seek shelter in vehicles. The SSHP will be consulted so that workers can properly identify poisonous plants potentially encountered on site, and workers will avoid them. If these plants cannot be avoided, long pants, long sleeves, gloves, and/or barrier creams may be used.	L
X	General	Not wearing shirt with sleeves/long pants while being exposed to sunlight; not utilizing sunscreen; not utilizing shade where available; not shielding eyes from bright sun	Workers will be advised in the proper use of sun blocking agents and shaded areas will be sought out on site; Workers should avoid direct exposure to the sun for long periods of time. Pop up tents may be used to create shade, if available. Safety glasses may be worn with UVA/UVB protection.	L

Add Items

	EQUIPMENT	TRAINING	INSPECTION
X	None		

Involved Personnel:

SSHO, Site Superintendent, Field Team

Relevant Competent Person(s): SSHO and Site Superintendent

PPE: Level D, modified to include safety-toe work boots, safety glasses with side shields (as needed), and leather work gloves (as needed)

Acceptance Authority (digital signature):

Digitally signed by Michael W. Barsa, CSP
Date: 2016.09.22 12:07:33 -04'00'

ACTIVITY HAZARDS ANALYSIS

Overall Risk Assessment Code (RAC) (Use highest code): L

Date: 12 September 2016

Project: RVAAP, Atlas Scrap Yard AOC

Activity: Load, Transport, Recycle Railroad Ties

Activity Location: RVAAP, Atlas Scrap Yard AOC

Prepared By: Michael Barsa, CSP

Risk Assessment Code Matrix

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

Add Identified Hazards

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Load, Transport, and Recycle railroad ties	Material Handling (manual) - muscle strain / back injury from manual lifting	Do not lift beyond your capabilities. Adhere to proper lifting techniques. Consider the following before engaging in a work activity that may cause back injury: • Know the weight to be lifted and postural requirements. • Check that the object will remain stable when moved. • Evaluate if a mechanical device should be used to move the object (e.g., hand truck, cart, dolly, pallet jack, etc.). • Determine number of people needed to move object safely. • Consider personnel medical limitations/restrictions. • Consider environmental conditions (e.g., slippery conditions, lighting, etc.). • Plan the lift and the travel path before starting activity. Implement one of the following controls for objects greater than 50 pounds: • Engineer the lift out of the task through work planning. • Use a mechanical device. • Use two or more people.	L
X	Load, Transport, and Recycle railroad ties	Material Handling (manual) - injury from sharps (i.e., materials/equipment having corners, edges or projections capable of cutting or piercing the skin)	Wear cut-resistant work gloves when the possibility of lacerations or other injury may occur. Utilize mechanical device, if appropriate, to avoid directly handling sharps. Maintain all hand and power tools in a safe condition. Wear a face shield for protection from glass breakage.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Load, Transport, and Recycle railroad ties	Heavy/Hauling Equipment Operation - general hazards	Adhere to USACE EM 385-1-1, Section 18 (Machinery and Mechanized Equipment). Use only properly trained and qualified personnel. Operate equipment in strict accordance with manufacturer's requirements and wear seat belts at all times. Perform daily inspections of equipment and take out-of-service any equipment determined to be unsafe or with a deficiency that effects the safe operation of the equipment. Maintain minimum 10 feet clearance between overhead power lines (rated 50 kV or below) and any part of the equipment. Use 3-point contact when accessing/egressing any equipment that requires personnel to climb. Clean tracks to provide safe walking/working surface and avoid walking on tracks whenever possible. Avoid walking on machine tracks whenever possible and clean tracks for safe walking/working surfaces. Isolate equipment swing and turn radius. Prohibit equipment operation when there is heavy fog with limited visibility. Restrict entry to the work area to authorized personnel. Utilize a dedicated spotter when necessary. Understand and review the posted hand signals. Wear hearing protection during operation of open-cab equipment for elevated noise levels. Equipment operators may not use a cell/mobile phone while the equipment is operating.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Load, Transport, and Recycle railroad ties	Heavy/Hauling Equipment Operation - injury to ground personnel	Personnel shall not approach operating equipment (within 25 feet) without prior hand/eye/radio contact and permission from the operator of the equipment. Stop movement of equipment and lower booms/buckets when approached by ground personnel. Do not pass under overhead loads. Ensure dedicated spotters are readily and easily identifiable visually (e. g. orange hardhat or equivalent means). Approach operating equipment from front (in line of operator's vision) to get operator's attention while at a safe distance. Maintain a minimum 25 feet safe distance from operating equipment. Remain outside equipment swing and turn radius. Use caution when standing next to idle equipment. Avoid positioning between fixed objects and operating equipment and equipment pinch points. Maintain 25 feet upwind position during loading and dumping operations. Stay alert of equipment operation and listen for back-up alarms. Wear high visibility vest when required to work in close proximity to operating equipment. Wear high visibility vest with reflective material (stripe or panel) during work activities performed after daylight hours.	L
X	Load, Transport, and Recycle railroad ties	Fire/Explosion - fueling operations	Adhere to USACE EM 385-1-1, Section 9.B (Flammable and Combustible Liquids). Place portable containers on level ground during fueling operations. Items such as tables, tailgates, truck beds or other similar configurations are not to be considered as an equivalent to level ground during refueling activities. Check container/tank fuel levels only when the fill-nozzle is shut off. Avoid filling the container/tank completely full. Maintain enough air space (about 5-10% of container/tank capacity) to allow for expansion of the fuel. Shut off equipment engines prior to fueling. Allow all equipment to cool down prior to fueling (i.e., a minimum cool-down time of 20-minutes). Exception is vehicles/heavy equipment on which the fuel tank fill location is remote from all hot compartments or surfaces.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Load, Transport, and Recycle railroad ties	Heavy/Hauling Equipment Operation - vehicle traffic	Look in the direction of travel before backing up. Arrange traffic flow to prevent foot traffic from crossing the routes of heavy/hauling equipment and moving loads. Adhere to posted speed limits. Use reflective warning vests when exposed to vehicle traffic. Reroute traffic as necessary to minimize potential for an accident. Utilize dedicated flaggers when necessary at site roadway crossings. Set-up signs, traffic cones and/or barricades to define and protect the project area from the non project-related vehicle traffic.	L
X	Load, Transport, and Recycle railroad ties	Loading/unloading mobile equipment and/or "large" equipment on a trailer or truck	Use ramps of adequate design that are solid and substantial enough to bear the weight of the equipment with carrier - including tooling. • Load and unload on level ground. • Use the assistance of a ground level spotter. • Check the brakes on the vehicle or carrier before approaching loading ramps. • Distribute the weight of the vehicle or carrier, and tools on the trailer so that the center of weight is approximately on the center line of the trailer. Adhere to the trailer manufacturer's weight distribution recommendations. Secure the vehicle/equipment and tools to the hauling vehicle with ties, chains, and/or load binders of adequate capacity prior to unloading	L
X	Load, Transport, and Recycle railroad ties	Physical injury from heavy lifting and pinch points	Workers will be advised on: Proper lifting techniques such as keeping the back straight; Lifting with the legs; Avoid twisting the back; Use mechanical help or assistance from others whenever possible; Break heavy loads down into smaller, lighter loads, if possible; Ensure the path of travel is clear prior to lifting and transporting the load; wear gloves while moving equipment.	L
X	Load, Transport, and Recycle railroad ties	Slips, trips, and falls	The work area shall be visually inspected continuously by the members of the work crew to mark, move, or barricade to prevent mishaps. Work areas will be kept organized; work areas will be well lit; not to go below Level D PPE.	L
X	Load, Transport, and Recycle railroad ties	Exposure to potential back strains during manual lifting operations	Workers will be advised on; Proper lifting techniques such as keeping the back straight; Lifting with the legs; Avoid twisting the back; Use mechanical help or assistance from others whenever possible; Break heavy loads down into smaller, lighter loads, if possible; Ensure the path of travel is clear prior to transporting the load.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Load, Transport, and Recycle railroad ties	Not being accustomed to humidity/ temperature extremes associated with outdoor work; wearing incorrect clothing for the weather (i.e. short sleeves when its cold); working in direct sunlight, working in wet clothing, lack of training on monitoring of the WBGT, and/or lack of training in monitoring of heart rates.	Workers will be cognizant of heat and cold stress symptoms and associated First Aid procedures. Electrolyte/fluids replacement will be readily available to workers. Appropriate clothing/PPE as defined in the SSHP will be donned. Work/Rest regimens will be established per the SSHP as necessary. The SSHO will have a WBGT meter on site for monitoring temperature according to manufacturer's instructions, and the SSHO will check and record the radial pulse of any worker showing signs of potential heat stress. The radial artery can be found close to the inside part of the wrist near the thumb. The SSHO will use the middle and index fingers to find the radial artery, and the SSHO will count the radial pulse for a full minute (60 seconds) to determine heart rate in beats per minute. In the field logbook, the SSHO will note the date/ time, the heart rate, relative strength of the beat, which wrist used, and anything anomalous. For individuals with normal cardiac performance, sustained (over several minutes) heart rate should remain below 180 beats per minute minus age (in years), recovery heart rate at one minute after a peak work effort should be below 110 bpm, and there should be no symptoms of sudden and severe fatigue, nausea, dizziness, or lightheadedness.	L
X	Load, Transport, and Recycle railroad ties	Continuing to work outside during thunderstorm; continuing to work in high wind conditions; working in especially cold/wet conditions	Work will cease when severe weather conditions prevail. Shelter will be sought in field vehicles, never under trees. The SSHO will monitor the weather and advise the workers with the approach of inclement weather. In the event of thunder/lightning, workers will stand down until at least 30 minutes after last lightning observed or thunder heard. Workers should be equipped with appropriate rain gear and dress in layers.	L
X	Load, Transport, and Recycle railroad ties	Walking through tall grass where ticks may be present; not being aware of bees, wasps, and/or hornets in work area; stepping on/running into nest that may be present in the work area; not being aware of potentially poisonous insects/ animals that may be encountered on site; being in area where worker should not be and disturbing insects/animals; not recognizing plants as poisonous and/or being in area where worker does not need to be where poisonous plants may exist	Workers will be briefed on the site specific biological hazards (as described in the SSHP) during the initial site orientation. Appropriate First Aid measures will be identified for these hazards; Workers will be advised to avoid contact with these hazards (i.e., walk around tall grass if possible, ensure long pants and long sleeves are worn) and to check themselves at end of the day for ticks. If tick are discovered on the skin, it must immediately be reported to the SSHO. Site personnel will be encouraged to wear light color clothing with cuffs and openings closed. If any worker is allergic to bee/wasp/hornet stings, the SSHO should be advised, and an epinephrine injector should be readily available for that individual(s). If any wild animals are encountered on site, workers will avoid the area until the animal moves and/or seek shelter in vehicles. The SSHP will be consulted so that workers can properly identify poisonous plants potentially encountered on site, and workers will avoid them. If these plants cannot be avoided, long pants, long sleeves, gloves, and/or barrier creams may be used.	L
X	Load, Transport, and Recycle railroad ties	Not wearing shirt with sleeves/long pants while being exposed to sunlight; not utilizing sunscreen; not utilizing shade where available; not shielding eyes from bright sun	Workers will be advised in the proper use of sun blocking agents and shaded areas will be sought out on site; Workers should avoid direct exposure to the sun for long periods of time. Pop up tents may be used to create shade, if available. Safety glasses may be worn with UVA/UVB protection.	L

ACTIVITY HAZARDS ANALYSIS

Add Items

	EQUIPMENT	TRAINING	INSPECTION
X	Truck	Proper use, inspection, and care of equipment prior to use	Daily Inspection
X	Hydraulic Excavator	Proper use, inspection, and care of equipment prior to use	Daily Inspection
X	Semi-tractors with end-dump trailers	Proper use, inspection, and care of equipment prior to use	Daily Inspection

Involved Personnel:

SSHO, Site Superintendent, Field Team, Operators

Relevant Competent Person(s): SSHO and Site Superintendent

PPE: Level D, modified to include safety-toe work boots, hard hats, safety vests, safety glasses with side shields (as needed), and leather work gloves (as needed)

Acceptance Authority (digital signature):

Digitally signed by Michael W. Barsa, CSP
Date: 2016.09.22 12:08:16 -04'00'

ACTIVITY HAZARDS ANALYSIS

Overall Risk Assessment Code (RAC) (Use highest code): L

Date: 4 August 2016

Project: RVAAP, Atlas Scrap Yard AOC

Activity: Installation of Temporary Storage Facility

Activity Location: RVAAP, Atlas Scrap Yard AOC

Prepared By: Brandon Hack

Risk Assessment Code Matrix

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

Add Identified Hazards

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Transfer historical files and Administrative Record documents to the temperature-controlled temporary storage unit	Material Handling (manual) - muscle strain / back injury from manual lifting	Do not lift beyond your capabilities. Adhere to proper lifting techniques. Consider the following before engaging in a work activity that may cause back injury: • Know the weight to be lifted and postural requirements. • Check that the object will remain stable when moved. • Evaluate if a mechanical device should be used to move the object (e.g., hand truck, cart, dolly, pallet jack, etc.). • Determine number of people needed to move object safely. • Consider personnel medical limitations/restrictions. • Consider environmental conditions (e.g., slippery conditions, lighting, etc.). • Plan the lift and the travel path before starting activity. Implement one of the following controls for objects greater than 50 pounds: • Engineer the lift out of the task through work planning. • Use a mechanical device. • Use two or more people.	L
X	Location and setup a double-wide office trailer (i.e. 60' by 24') with shelving units between Buildings 1034 and 1036	Material Handling (manual) - injury from sharps (i.e., materials/equipment having corners, edges or projections capable of cutting or piercing the skin)	Wear cut-resistant work gloves when the possibility of lacerations or other injury may occur. Utilize mechanical device, if appropriate, to avoid directly handling sharps. Maintain all hand and power tools in a safe condition. Wear a face shield for protection from glass breakage.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Location and setup a double-wide office trailer	Heavy/Hauling Equipment Operation - general hazards	Adhere to USACE EM 385-1-1, Section 18 (Machinery and Mechanized Equipment). Use only properly trained and qualified personnel. Operate equipment in strict accordance with manufacturer's requirements and wear seat belts at all times. Perform daily inspections of equipment and take out-of-service any equipment determined to be unsafe or with a deficiency that effects the safe operation of the equipment. Maintain minimum 10 feet clearance between overhead power lines (rated 50 kV or below) and any part of the equipment. Use 3-point contact when accessing/egressing any equipment that requires personnel to climb. Clean tracks to provide safe walking/working surface and avoid walking on tracks whenever possible. Avoid walking on machine tracks whenever possible and clean tracks for safe walking/working surfaces. Isolate equipment swing and turn radius. Prohibit equipment operation when there is heavy fog with limited visibility. Restrict entry to the work area to authorized personnel. Utilize a dedicated spotter when necessary. Understand and review the posted hand signals. Wear hearing protection during operation of open-cab equipment for elevated noise levels. Equipment operators may not use a cell/mobile phone while the equipment is operating.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Location and setup a double-wide office trailer	Heavy/Hauling Equipment Operation - injury to ground personnel	Personnel shall not approach operating equipment (within 25 feet) without prior hand/eye/radio contact and permission from the operator of the equipment. Stop movement of equipment and lower booms/buckets when approached by ground personnel. Do not pass under overhead loads. Ensure dedicated spotters are readily and easily identifiable visually (e.g. orange hardhat or equivalent means). Approach operating equipment from front (in line of operator's vision) to get operator's attention while at a safe distance. Maintain a minimum 25 feet safe distance from operating equipment. Remain outside equipment swing and turn radius. Use caution when standing next to idle equipment. Avoid positioning between fixed objects and operating equipment and equipment pinch points. Maintain 25 feet upwind position during loading and dumping operations. Stay alert of equipment operation and listen for back-up alarms. Wear high visibility vest when required to work in close proximity to operating equipment. Wear high visibility vest with reflective material (stripe or panel) during work activities performed after daylight hours.	L
X	Location and setup a double-wide office trailer	Heavy/Hauling Equipment Operation - vehicle traffic	Look in the direction of travel before backing up. Arrange traffic flow to prevent foot traffic from crossing the routes of heavy/hauling equipment and moving loads. Adhere to posted speed limits. Use reflective warning vests when exposed to vehicle traffic. Reroute traffic as necessary to minimize potential for an accident. Utilize dedicated flaggers when necessary at site roadway crossings. Set-up signs, traffic cones and/or barricades to define and protect the project area from the non project-related vehicle traffic.	L

Add Items

	EQUIPMENT	TRAINING	INSPECTION
X	Truck	Proper use, inspection, and care of equipment prior to use	Daily Inspection
X	Trailer	Proper use, inspection, and care of equipment prior to use	Daily Inspection
X	Rack truck/support truck	Proper use, inspection, and care of equipment prior to use	Daily Inspection

ACTIVITY HAZARDS ANALYSIS

Involved Personnel:

SSHO, Site Superintendent, Field Team, Operators

Relevant Competent Person(s): SSHO and Site Superintendent

PPE: Level D, modified to include safety-toe work boots, safety glasses with side shields (as needed), and leather work gloves (as needed)

Acceptance Authority (digital signature): Digitally signed by Michael W. Barsa, CSP
Date: 2016.09.22 12:08:52 -04'00'

ACTIVITY HAZARDS ANALYSIS

Overall Risk Assessment Code (RAC)
(Use highest code)
L

Date: 12 September 2016

Project: RVAAP, Atlas Scrap Yard AOC

Activity: Waste Management Operations

Activity Location: RVAAP, Atlas Scrap Yard AOC

Prepared By: Michael Barsa, CSP

Risk Assessment Code Matrix

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

Add Identified Hazards

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Waste Characterization Sampling	Exposure to biological; Biological hazards such as stinging and biting insects, poisonous snakes, possible exposure to poison ivy, poison sumac may be present.	Work party members will be briefed on the site specific hazards during the initial site orientation. Appropriate First Aid measures will be identified for these hazards; Work party members will be advised to avoid contact with these hazards and to check themselves at end of the day for ticks. If tick are discovered on the skin, it must immediately be reported to the SSHO. Site personnel will be encouraged to wear light color clothing with cuffs and openings closed. If members of the work party are allergic to bee/wasp stings, the SSHO should be advised, and an epinephrine injector should be readily available for that individual(s).	L
X	Waste Characterization Sampling	Heat/Cold Stress	Workers will be trained and cognizant of heat and cold stress symptoms and associated First Aid procedures. Electrolyte/fluids replacement will be readily available to workers. Work/Rest regimens will be established per ACGIH/NIOSH guidelines.	L
X	Waste Characterization Sampling	Exposure to potential back strains during manual lifting operations	Workers will be advised on; Proper lifting techniques such as keeping the back straight; Lifting with the legs; Avoid twisting the back; Use mechanical help, such as a drum dolly, or two-man lifting for items that exceed 50 pounds; Ensure the path of travel is clear prior to lifting and transporting the load.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Waste Characterization Sampling	Exposure to Noise	High noise areas will be conspicuously identified; Hearing protection will be provided, as necessary, when sound levels exceed limits outlines in 29 CFR 1910.95(a). (85 dBA) Employees that routinely work in high noise areas will be enrolled in the corporate Hearing Control Program.	L
X	Waste Characterization Sampling	Slips, trips, and falls	The work area will be visually inspected continuously by the workers to mark, move, or barricade hazard areas to prevent mishaps. Work areas will be kept organized; work areas will be well lit.	L
X	Waste Characterization Sampling	Exposure to potential back strains during manual lifting operations	Workers will be advised on: Proper lifting techniques such as keeping the back straight; Lifting with the legs; Avoid twisting the back ; Use mechanical help or assistance from others whenever possible Break heavy loads down into smaller, lighter loads, if possible; Ensure the path of travel is clear prior to lifting and transporting the load. Wear gloves while moving equipment Move and handle containers preferably using a drum grappler. Movement by hand is to be offered as a last resort.	L
X	Waste Characterization Sampling	Hand or fingers caught between objects; lacerations and abrasions	Workers involved in activities that subject the hands to injury (for example cuts, abrasions, punctures) will wear cut resistant work gloves) .	L

Add Items

	EQUIPMENT	TRAINING	INSPECTION
X	Vehicles	Personnel will be trained in the equipment operation. Personnel in the area or assisting will be made aware of the hazard and will coordinate carefully during equipment handling operations. Guards will be kept in place during operation. A safe distance will be maintained from moving mechanical parts.	In accordance with manufacturers' manuals.
X	Sampling bottles and disposable equipment	Per UFP-QAPP	In accordance with manufacturers' manuals.

Involved Personnel:

SSHO, Site Superintendent, Field Team, Operators

Relevant Competent Person(s): SSHO and Site Superintendent

PPE: Level D, modified to include safety-toe work boots, safety glasses with side shields (as needed), and leather work gloves (as needed)

ACTIVITY HAZARDS ANALYSIS

Acceptance Authority (digital signature): Digitally signed by Michael W. Barsa, CSP
Date: 2016.09.22 12:09:28 -04'00'

ACTIVITY HAZARDS ANALYSIS

Overall Risk Assessment Code (RAC)
(Use highest code)
L

Date: 12 September 2016

Project: RVAAP, Atlas Scrap Yard AOC

Activity: Restoration

Activity Location: RVAAP, Atlas Scrap Yard AOC

Prepared By: Michael Barsa, CSP

Risk Assessment Code Matrix

E = Extremely High Risk
H = High Risk
M = Moderate Risk
L = Low Risk

		Probability				
		Frequent	Likely	Occasional	Seldom	Unlikely
Severity	Catastrophic	E	E	H	H	M
	Critical	E	H	H	M	L
	Marginal	H	M	M	L	L
	Negligible	M	L	L	L	L

Add Identified Hazards

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Re-grade site	Vehicle Accidents/Collisions	All equipment will be properly secured during transport. Use ground guide and back-up alarm during backing. Vehicle and equipment operators should look in the direction of travel; look before backing up. Arrange traffic flow to prevent foot traffic from crossing the routes of heavy equipment and moving loads. Ensure the operator acknowledges your presence before walking near equipment in operation. Use of cell phones by the driver/operator is prohibited while the vehicle/equipment is in motion.	L
X	Re-grade site	Heavy/Hauling Equipment Operation - vehicle traffic	Look in the direction of travel before backing up. Arrange traffic flow to prevent foot traffic from crossing the routes of heavy/hauling equipment and moving loads. Adhere to posted speed limits. Use reflective warning vests when exposed to vehicle traffic. Reroute traffic as necessary to minimize potential for an accident. Utilize dedicated flaggers when necessary at site roadway crossings. Set-up signs, traffic cones and/or barricades to define and protect the project area from the non project-related vehicle traffic.	L

ACTIVITY HAZARDS ANALYSIS

	JOB STEPS	HAZARDS	ACTIONS TO ELIMINATE OR MINIMIZE HAZARDS	RAC
X	Re-grade site	Material Handling (manual) - muscle strain / back injury from manual lifting	Do not lift beyond your capabilities. Adhere to proper lifting techniques. Consider the following before engaging in a work activity that may cause back injury: • Know the weight to be lifted and postural requirements. • Check that the object will remain stable when moved. • Evaluate if a mechanical device should be used to move the object (e. g., hand truck, cart, dolly, pallet jack, etc.). • Determine number of people needed to move object safely. • Consider personnel medical limitations/restrictions. • Consider environmental conditions (e.g., slippery conditions, lighting, etc.). • Plan the lift and the travel path before starting activity. Implement one of the following controls for objects greater than 50 pounds: • Engineer the lift out of the task through work planning. • Use a mechanical device. • Use two or more people.	L
X	Re-grade site	Material Handling (manual) - injury from sharps (i.e., materials/equipment having corners, edges or projections capable of cutting or piercing the skin)	Wear cut-resistant work gloves when the possibility of lacerations or other injury may occur. Utilize mechanical device, if appropriate, to avoid directly handling sharps. Maintain all hand and power tools in a safe condition. Wear a face shield for protection from glass breakage.	L
X	Re-grade site	Biological Hazards - biting/stinging insects (e.g., mosquitoes, bees and wasps) and animals (e.g., stray dogs/cats, rodents and snakes)	Notify supervision if allergic to biting/sting insects. Avoid insect nest areas and likely habitats of vermin. Avoid all contact with wild animals. Apply insect repellant (≤ 30% DEET) according to manufacturer's instructions. Wear appropriate clothing and cover exposed skin to protect against biting/stinging insects. Tape pants legs at the ankles. Be alert and watch for rodents, snakes, etc. Avoid rodent urine, droppings or nesting materials. Avoid areas with significant pigeon droppings. Check skin/clothing for ticks at breaks and end of shift.	L
X	Re-grade site	Slip/Trip/Fall	Use caution when working on uneven or wet ground. Practice good housekeeping Use caution when working on uneven or wet ground. Eliminate or clearly mark protrusions and obstructions.	L

ACTIVITY HAZARDS ANALYSIS

EQUIPMENT	TRAINING	INSPECTION
Add Items		

	EQUIPMENT	TRAINING	INSPECTION
X	Dozer and potentially excavator and tandem haul trucks	Daily Inspection	Proper use, inspection, and care of equipment prior to use.

Involved Personnel:

SSHO, Site Superintendent, Field Team

Relevant Competent Person(s): SSHO and Site Superintendent

PPE: Level D, modified to include safety-toe work boots, safety glasses with side shields (as needed), and leather work gloves (as needed)

Acceptance Authority (digital signature): Digitally signed by Michael W. Barsa, CSP
Date: 2016.09.22 12:10:11 -04'00'

ATTACHMENT 4

Camp Ravenna First Responder Reporting Form

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FIRST RESPONDER REPORTING FORM
(Print all information)

Collect as much of the information on the top half of this form as possible before making initial notification. Complete the top and bottom of the form before turning in to Camp Ravenna.

Name of individual reporting spill: ____

When did the spill occur (Date and Time)? ____

Spill Location (Building or area name / number, indoors or out; if vehicle involved, type and bumper number):
____

What was spilled? _______________________________________ **How much was spilled?** ____________________________________

Rate at which material is currently spilling. ____

Extent of spill travel? ____

Did the spill reach water (ditch, creek, stream, pond, well head) ____

Number of injured personnel and type injuries, if applicable. ____

Do you need the Fire Department to respond to protect life, property, and environment? _______________________________________

Unit: ____ **State:** _____________ **Report Date & Time:** _____________________________

On Scene Coordinator Name and Grade: ____ **Phone:** _________________________

How did the spill occur (be specific). ____

____

What remedial action was taken? ____

____

Was soil and absorbent material generated? ____________ **How much?** ____

What is the location of the soil and absorbents? ____

Was the Environmental Office contacted (yes or No, date and time)? ____

Who did you talk to in the Environmental Office? ____

Was the site cleared by the Env. Office (Yes or No, date and time)? ____

Who cleared the site (name and grade, date and time)? ____

Initial information is critical. Get as much information as you can, but don't hesitate to make the initial notification if a spill is moving or worsening rapidly!

This form must be completed for all releases and turned-in to Camp Ravenna Range Control within 24 hours.

FIRST RESPONDER SPILL/RELEASE RESPONSE ACTIONS

Units or contractors performing training or other operations at Camp Ravenna shall be responsible for adhering to the provisions identified in the Camp Ravenna Integrated Contingency Plans (ICP). A copy of the ICP may be obtained from the Camp Ravenna Environmental Supervisor. Following discovery of a spill (any size), the procedures outlined below shall be executed where applicable:

1. **If necessary, initiate evacuation of the immediate area.**

2. **Notify Camp Ravenna Range Control via two-way radio or by calling (614) 336-6041, and report information contained on the "First Responder Reporting Form" if it is known or can reasonably be determined. This form has been copied on the opposite side of this page. If Range Control cannot be reached, contact a Camp Ravenna OSC (listed below).**

3. **Stop spill flow when possible without undue risk of personal injury.**

4. **If trained, contain the spill using available spill response equipment or techniques.**

5. **Make spill scene OFF LIMITS to unauthorized personnel.**

6. **Restrict all sources of ignition when flammable substances are involved.**

7. **Report to the OSC upon his/her arrival to the scene.**

8. **Turn in a completed copy of the Camp Ravenna First Responder Form to Camp Ravenna Range Control for ALL releases, even ones cleaned up by the reporter.**

TELEPHONE NUMBER

When Camp Ravenna Range Control is not available, the Camp Ravenna OSC must to be contacted by the discoverer/first responder following a release if it is in water, at or above a reportable quantity (25 gallons or more of POL), a hazardous or extremely hazardous substance, a hazardous waste, or involves fire, explosion, or is otherwise a major incident.

NAME	JOB TITLE	OFFICE	24 HOUR
Camp Ravenna Range Control	Operations and Training	(614)336-6041	(614) 202-5783
Tim Morgan (Primary OSC)	Environmental Supervisor	(614)336-6568	(330)322-7098
Katie Tait	Environmental Specialist	(614)336-6136	Contact Alternate
CPT Mike Yates	Range Operations	(614)336-6193	(330) 819-5038
MAJ Richard Saphore	Logistics Officer	(614)336-6790	(614) 593-1654
LTC Ed Meade	Garrison Commander	(614)336-6560	(614)307-0493
Joint Forces Command (Alternate POC)	OHARNG Emergency Center	(888)637-9053	(888)637-9053

Off-site (from Camp Ravenna area code 614 phones)

Portage County Fire Department (Portage Dispatch) ....……………………………………..…. 9-1-330 296-6486
Portage County Sheriff ………………………………………………………………………… 9-1-330-296-5100
Trumbull County Fire Department and Sheriff (Trumbull Dispatch) ..……………………… 9-1-330-675-2730

SEE REVERSE FOR FIRST RESPONDER REPORTING FORM

Appendix C
Grading, Drainage, and Erosion Control Plan

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FINAL
GRADING, DRAINAGE, AND EROSION CONTROL PLAN
FOR
FY16 RECYCLING OF MATERIALS AT RVAAP-50 ATLAS SCRAP YARD AREA OF CONCERN AND SETUP OF TEMPORARY STORAGE FACILITY

Former Ravenna Army Ammunition Plant
Portage and Trumbull Counties, Ohio

Contract No.: W912QR-12-D-0011
Delivery Order 0017

Prepared for:

United Stated Army Corps of Engineers
Louisville District
600 Martin Luther King, Jr. Place
Louisville, Kentucky 40202

Prepared by:

ERT, Inc.
14401 Sweitzer Lane, Suite 300
Laurel, Maryland 20707
(301) 361-0620

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Final
Grading, Drainage, and Erosion Control Plan
for
FY16 Recycling of Materials at RVAAP-50 Atlas Scrap Yard Area of Concern and Setup of Temporary Storage Facility

Former Ravenna Army Ammunition Plant
Portage and Trumbull Counties, Ohio

Prepared for:

U.S. Army Corps of Engineers
Louisville District

Contract W912QR-12-D-0011
Delivery Order 0017

Prepared by:

ERT, Inc.
14401 Sweitzer Lane, Suite 300
Laurel, Maryland 20707
(301) 361-0620

Approvers:

20 December 2016

Sean Carney, Project Management Professional (PMP) Date
Project Manager

26 September 2016

Jennifer Harlan, PMP Date
Division Manager

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LIST OF ATTACHMENTS

LIST OF ACRONYMS AND ABBREVIATIONS

AOC	Area of Concern
CELRL	U.S. Army Corp of Engineers, Louisville District
DO	Delivery Order
ERT	ERT, Inc.
GD&E	Grading, Drainage, and Erosion Control Plan
PWS	Performance Work Statement
RVAAP	Former Ravenna Army Ammunition Plant
RVAAP-50	Atlas Scrap Yard
USACE	U.S. Army Corp of Engineers

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1.0 INTRODUCTION

ERT, Inc., (ERT) has been contracted to perform recycling/disposal of stockpiled material at the Atlas Scrap Yard (RVAAP-50) Area of Concern (AOC), and setup a temporary storage facility for the restoration document repository at the Former Ravenna Army Ammunition Plant (RVAAP), now known as the Camp Ravenna Joint Military Training Center (Camp Ravenna), Portage and Trumbull Counties, Ohio. The activities will be performed under a Firm Fixed Price Delivery Order (DO), as outlined in the Performance Work Statement (PWS), under United States Army Corps of Engineers (USACE), Louisville District (CELRL) contract W912QR-12-D-0011, DO 0017.

1.1 General

The ERT team has prepared this Grading, Drainage, and Erosion Control (GD&E) Plan to present the objectives of the PWS regarding the grading, drainage, and erosion control at this site and to identify the management practices that ERT will use to achieve those objectives.

1.2 Objectives

The GD&E Plan objectives are:

1. Ensure that a Filtrexx is installed and maintained to filter storm water run-off to protect down-gradient areas;
2. Prevent the erosion of surface soil during the removal of stockpiled material at RVAAP-50;
3. Minimize and control the dispersal of soils onto the facility roadways; and,
4. Return the site to a final condition that facilitates future maintenance operations (e.g., mowing).

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2.0 GRADING AND DRAINAGE

Grading of disturbed areas is necessary as part of the restoration of the site after the removal of stockpiled materials. The intent of the grading activity is to return the entire disturbed areas to a gently sloped parcel that promotes proper drainage and renders the area amenable to future maintenance operations (e.g., mowing). The ERT team will evaluate the need for fill if the volume of available material is insufficient to achieve the desired drainage pattern. However, based on the current conditions of the site, additional fill is unlikely to be required. ERT estimates that approximately 0.25 acres total will be disturbed during the removal activities; therefore there is no need for a National Pollutant Discharge Elimination System permit or Erosion, Sediment, and Pollution Control Plan. However, best management practices will be utilized during the execution of the project.

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3.0 EROSION CONTROL

3.1 Temporary Erosion Control Best Management Practices

Before removal activities begin, temporary erosion and sediment control best management practices (BMPs) will be established to prevent impacts to down-gradient areas. **Figure 3-1** illustrates the proposed layout of these erosion control measures at the site. Based on the type of field activities that will be implemented during the construction phases at RVAAP-50 (removal and load out of stockpiled materials, and re-grading of disturbed areas), the proposed best management practices for erosion and sediment control will include the following items:

1. Filtrex will be installed on the down-gradient side of the load out areas to prevent sediment from migrating off from the disturbed areas. Filtrexx will remain in place until proper vegetation has been established that will prevent erosion in the future.
2. A gravel construction entrance (i.e., rock apron) will be placed as a controlled site entrance to reduce the amount of sediment transported by construction vehicles onto facility and public roads.
3. Surface stabilization will include compaction via tracking equipment over the disturbed areas and permanent seeding to stabilize disturbed areas.
4. Plant the any disturbed areas with 50% Kentucky Bluegrass (Blueridge, Argyl, BlueStar, Ken Blue, Limousine or Baron varieties), 40% Perennial Ryegrasses (Peak, Sonata, Wind Dance, Integra, Morning Star or Shining Star varieties), and 10% Creeping Red Fescue (Lustrous or Boreal varieties) to promote the establishment of the vegetative cover.
5. Temporary erosion control BMPs will be left in place until the vegetative cover is 75% established, at which point they will be removed upon inspection and approval by CELRL, ARNG, and OHARNG.

3.2 Inspections

The Site Superintendent will inspect the erosion and sediment control measures daily during construction activities, as well as after each runoff producing rainfall event, using the Erosion Control Inspection Form presented as **Attachment 1**. Upon completion of the construction restoration activities, temporary erosion and sediment control measures will be removed. Each inspection event will be captured in the field notes generated for the field activities. Below is a list of items that will be checked during each inspection.

1. The Filtrexx will be checked for undermining or deterioration of the fabric. Sediment will be removed when the level of sediment causes bulging of the fence or reaches one-half of the fabric height.
2. The gravel construction entrance will be maintained to minimize tracking of sediment onto facility or public roads. Where track-out has occurred, sweeping will be performed by the end of the work week.

All erosion and sediment control devices will be inspected and maintained until the USACE has formally accepted the completion of field activities. Erosion control structures will be adequate to sustain weather damage and degradation, and will be maintained by ERT. Once the area has been restored, stabilized, and accepted by the USACE, ERT will remove temporary erosion and sediment control structures such as the Filtrexx.

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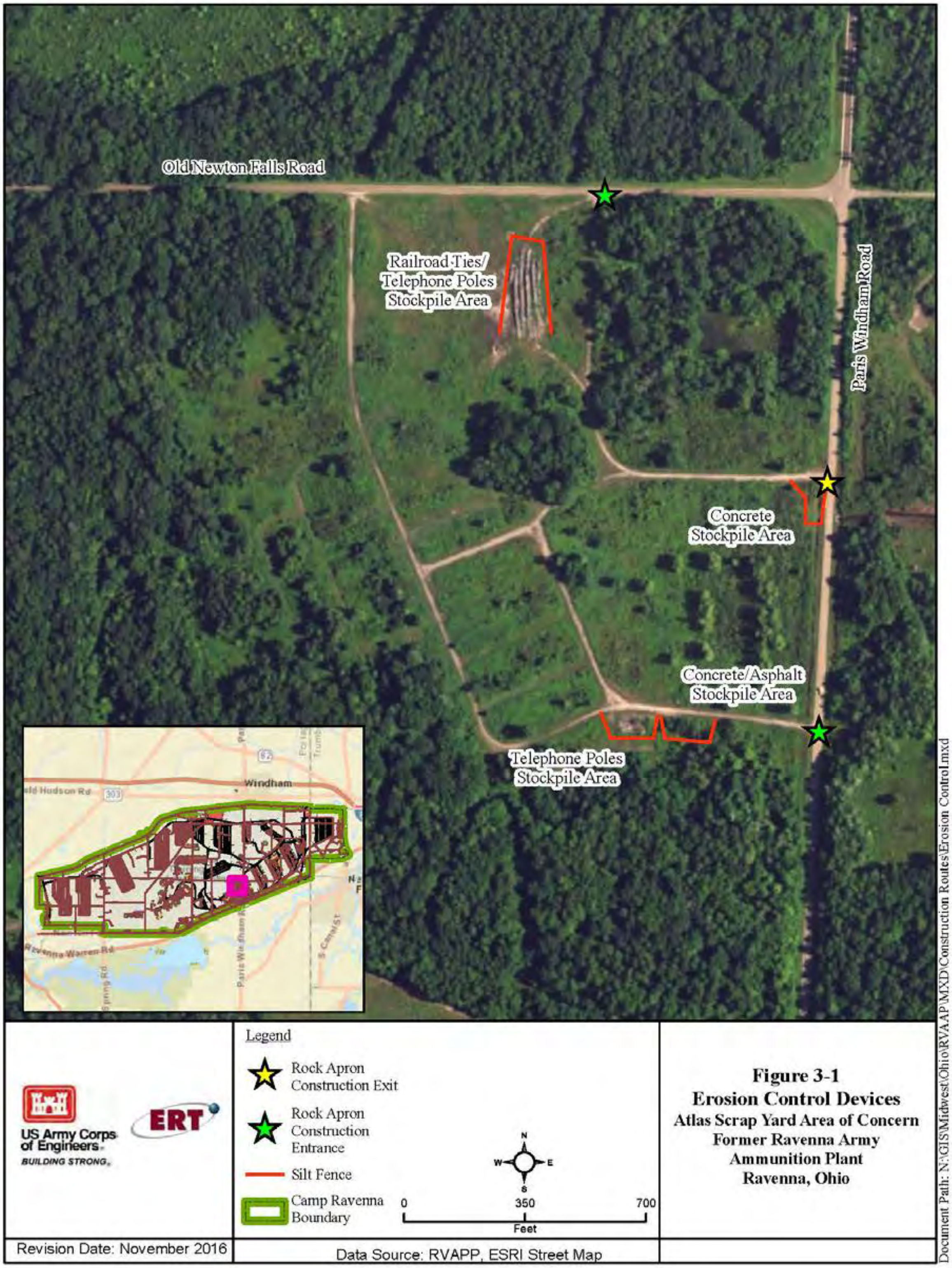

Figure 3-1. Erosion Control Devices

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4.0 ADDITIONAL CONSTRUCTION PRACTICES TO PREVENT STORM WATER POLLUTION

Municipal waste materials will be collected and stored by site personnel in appropriate waste containers bins. Good housekeeping and spill-control practices will be followed during field activities to minimize storm water contamination from petroleum products and vehicle maintenance. Good housekeeping practices include the following:

1. Vehicles on-site will be monitored for leaks and receive regular preventive maintenance to reduce the chance of leakage or spills;
2. Petroleum products will be stored in tightly sealed containers which are clearly labeled;
3. Spill kits and/or absorbent material will be located at fueling sources and maintenance activity sites;
4. Safety Data Sheets, a material inventory, and appropriate emergency contact numbers shall be maintained and stored on-site;
5. Portable toilet(s) will be located away from concentrated flow paths and traffic flow;
6. Sanitary waste will be collected from portable units regularly to avoid overfilling;
7. Project personnel will be instructed on the correct procedure for waste disposal; and
8. Dust-generating activities will be kept to the minimum required for accomplishment of excavation activities. Water for dust suppression will be applied on an as-needed basis.

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ATTACHMENT 1

EROSION CONTROL INSPECTION FORM

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Erosion and Sediment Control Inspection Report Form

Project Name and Location

Weather: ______________________________

Rain in last 24 hrs (inches): ______________________________

Owner / Permittee: ______________________________

Pollution Control Measures (BMP) Checklist:

________ Inlet Barrier (ie: gravel bags)
________ Sediment Barriers (ie: ditch checks)
________ Erosion Blankets, Hydromulch / Seed, etc
________ Stabilized Construction Entrance
________ Stream Crossings
________ Seed / Sod Areas
________ Sediment Basins & Discharge Locations
________ Borrow Areas
________ General Site Condition (trash, etc)

A. Current Construction / Active Areas:

B. Problem Areas / Special Observations(*Note problem areas ONLY below*):

BMP	Location	Observations, Effectiveness, & Corrective Actions Ordered

C. Listing of Areas where construction operations have permanently or temporarily stopped; stabilization measures initiated.

D. Have items noted on last inspection been corrected? Yes No (if No, Explain:)

Note: Inspection comments above indicate deficiencies only. Deficiencies must be corrected within 7 days, unless otherwise noted. All other BMP's on site are considered to be in good working condition.

______________________ ______________________

Date of Inspection **Inspector Signature**

6 Goals • No Sediment Leaves the Site • Lines of Defense Everywhere & Always • Cover Quickly
• Protect the Swale, Ditch ,and Channel • Keep Clean Water Clean • Inspect, Clean & Fix