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TECHNICAL MEMORANDUM WORK PLAN FOR SUPPLEMENTAL SAMPLING ACTIVITIES
AT UNEXPLODED ORDNANCE 7 (UXO 7) RANGES NSA CRANE IN
09/01/2011
TETRA TECH NUS INC

Comprehensive Long-term Environmental Action Navy

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Resource Conservation and Recovery Act

Technical Memorandum: Work Plan for Supplemental Sampling Activities at UXO 7 Ranges

Naval Support Activity Crane
Crane, Indiana

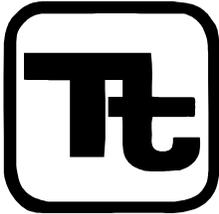
Contract Task Order F272

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Midwest

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TECHNICAL MEMORANDUM

DATE: August 28, 2011

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Project File – CTO F272

SUBJECT: NSA Crane UXO 7 – Small Arms Ranges - Supplemental Work Plan

1.0 Site Description

UXO 7 is located within Naval Support Activity (NSA) Crane (**Figure 1**) and formerly consisted of a 500-yard Firing Range, West Trap Range, East Trap Range, and South Pistol Range. **Figure 2** identifies the locations of the former UXO 7 small arms ranges. None of the aforementioned ranges are currently in use at UXO 7 and are closed. Samples were collected during 2007 as part of a Resource Conservation and Recovery Act (RCRA) Facility Investigations (RFI) to determine whether contamination was present.

2.0 RFI Results

Lead from bullets and shot, and polynuclear aromatic hydrocarbons (PAHs) from clay targets were the primary constituents of concern during the 2007 RFI at UXO 7. Soil samples collected during the RFI from areas within the former small arms ranges contained lead and/or PAHs at concentrations above specific screening levels for human health and/or ecological receptors. The RFI soil sampling program was performed to support human health and ecological risk assessments for typical small arms range

contaminants. The resulting RFI environmental data for UXO 7, although adequate for risk assessment purposes, was not sufficiently robust to fully delineate the nature and extent of lead and/or PAH contamination within the former range soil for focused removal actions.

The conceptual site model (CSM) for small arms ranges presumes that typically lead, whether from lead shot or lead bullets, and PAHs from clay pigeons will generally be restricted to the top one-foot of soil. However, if there has been any post-range use disturbance of this area, the extent of lead in soil could extend deeper than one foot below the ground surface. During the UXO 7 RFI, surface soil samples (typically from the surface to two feet below ground surface) were collected from various locations within the small arms ranges across the UXO 7 area. As indicated in **Figure 2**, the primary range infrastructure features from the former 500-yard rifle range (firing position berms, main target berm and barricade, etc.) are still present and visible at UXO 7. Only the dirt mound between the 300- and 400-yard firing positions is believed to be the result of operations that occurred after firing range activities at the site had permanently ended. Although the aboveground structures (i.e., trap houses and concrete firing positions) from the former trap ranges in the Central Zone of UXO 7 (depicted in **Figure 2**) have been removed from the range, minor amounts of trap house debris and electrical wiring observed at the site aided in locating the former range trap house sites. Furthermore, the distribution of lead and PAH contamination in range soil at the site is consistent with the areas of defined lead accumulation (range berms) and identified PAH accumulation (near the trap range centers about 75-175 feet from the firing line) which would be expected to occur on a typical small arms range. Therefore, based on these data, it appears that there has not been extensive disturbance of the soil in the former range areas of UXO 7.

The resulting analytical soil data were compared against human health and ecological screening levels in the RFI Report (Tetra Tech, July 2009). The results showed excess risk to humans (residential receptors) from PAHs and to ecological receptors (mammals and birds) from lead. During an initial review of the Draft-Final RFI Report for UXO 7, and in consultation with Naval Facilities Engineering Command (NAVFAC) and the United States Environmental Protection Agency (USEPA) Region V, an ecological screening concentration of 192 milligrams per kilogram (mg/kg) for lead in soil was identified for UXO 7. For the purpose of risk evaluation, the UXO 7 former small arms range area was divided into three zones consisting of a northern zone, a central zone, and a southern zone, based on the geographic position within the UXO 7 site (**Figure 2**).

3.0 Supplemental Delineation Sampling for Lead Concentrations in Soil

This proposed supplemental soil sampling program is focused on identified soil sampling locations from the RFI where the analytical data indicated elevated concentrations of lead present in the former small arms range soil. The collection of the supplemental soil sampling data will provide more detailed information on the nature and extent of the contaminated areas in the soil at the site (both laterally and vertically), and will guide the development of subsequent focused interim removal actions.

Table 1 provides a summary of the number of lead samples to be collected during the supplemental sampling field effort. The information below provides details on the sampling effort for the lead soil sample collection.

Real-time X-ray fluorescence (XRF) analyses will be used as a field-screening tool to determine the nature and extent of lead in soil. A screening value of 125 parts per million (ppm) lead will be used for the XRF screening of soil samples. This conservative XRF field screening value is nearly one-half the previously identified ecological lead screening concentration of 192 mg/kg and nearly one-quarter of the human health screening level of 400 mg/kg. The indicated XRF screening value for lead (125 mg/kg) was selected as a conservative concentration for field screening purposes and is not directly tied to a specific ecological or human health screening value. The more conservative XRF screening value provides the field team greater confidence in supporting field decisions for supplemental sampling locations (identifying the limits of soil lead contamination, supporting real-time decisions for additional step-out samples, or requiring the collection of soils samples from greater depths). It is the goal of this UXO 7 supplemental sampling to have the discrete areas of elevated soil lead concentrations fully bounded by field-screened XRF soil samples that display lead concentrations below the selected XRF screening value (both vertically and horizontally). These XRF data will be used to confirm that all areas of elevated soil lead have been clearly identified based on the XRF field screening process and serve as the basis for selecting a subset of collected soil samples for verification of lead concentrations by analysis at a fixed-base laboratory (FBL).

The XRF analyses will be performed on soil samples encircling the former RFI sample locations with elevated lead concentrations. These supplemental sampling locations will include samples initially collected from the surface to one-foot below ground surface (bgs). Should the 0 to 1-foot bgs soil sample exhibit an XRF lead concentration greater than the established XRF lead screening value of 125 ppm, then additional vertical samples will be collected at that location in one-foot increments until the lead XRF concentration is below the established XRF field screening concentration. Horizontal step-out samples

will also be collected in approximate 5-foot increments until the lead XRF concentration is below the established XRF field screening concentration. The collection of vertical samples from each location will be used to confirm the base of the lead contamination in the former range soil while the horizontal samples will ensure that the soil areas with elevated lead concentrations have been fully delineated. Representative soil samples evaluated by the field XRF approach will be submitted for FBL analysis to determine the overall accuracy of the XRF field screening process and to add greater credibility to the XRF data through a correlation analysis of the FBL and field data. This approach will guide the development of a subsequent soil removal action to address the lead-contaminated soil at UXO 7.

Three discrete areas within UXO 7 were identified where the soil lead concentrations were greater than the 192 mg/kg ecological screening level and all were located in the northern zone (**Figure 3**). Based on the XRF analysis of the soil samples collected from the northern zone, additional samples may be collected (using a step-out technique) to better delineate specific areas of elevated soil lead concentrations from those areas that do not exhibit elevated soil lead via XRF analysis.

The northernmost of the three soil lead areas occurs in a depression on the rifle range floor near a small drainage that passes through the range between the 400- and 500-yard firing positions (**Figure 3**). The soil lead contamination in this location has been partially bounded laterally. A sampling approach featuring 17 locations surrounding the three RFI sample locations with lead concentrations greater than 192 mg/kg will be initiated, with each sample location featuring two sampling depths (as previously described) (**Figure 4**). In the event that the XRF soil samples collected from the inner ring around the RFI soil lead hot spot exhibit field XRF lead concentrations greater than 125 ppm, then samples from the second ring of sampling locations will be added to the data set until the soil contamination is fully delineated vertically and laterally. Up to 24 secondary ring samples may be collected as necessary to clearly distinguish lead impacted soil (>192 mg/kg) from lower risk soil on the range.

A second area along the 400-yard firing location berm features a linear area with three RFI soil sample points containing elevated soil lead concentrations above 192 mg/kg (**Figure 3**). While the lead contamination in this area was partially bounded laterally during the RFI (i.e., there were several adjacent sampling locations below the 192 mg/kg ecological screening level for lead), the complete lateral extent of the contamination in the area has not been fully characterized (**Figure 5**). Furthermore, because this firing position was constructed to be elevated above the range floor, there is the potential that some lead contamination may be present at depth (past sampling only considered the surface to two-foot depth of soil). A series of 18 XRF sample locations are proposed to encircle the previously identified RFI soil lead hot spots with each sampling location featuring two sampling depths (as previously described and as

presented in **Table 1**) . These primary rings of six soil sample locations each will be used to delineate the lateral and vertical extent of the soil lead based on field XRF screening. In the event that the XRF soil samples collected from the inner rings around the RFI soil lead hot spots exhibit field XRF lead concentrations greater than 125 ppm, then additional soil samples from a series of secondary rings of sampling locations will be added to the data set until the soil lead contamination is fully delineated vertically and laterally. Up to 26 secondary ring samples may be collected as necessary to clearly distinguish lead impacted soils (>192 mg/kg) from lower risk soils on the range.

The third area in the northern zone consists of a dirt mound area between the 300- and 400-yard firing positions and had two discrete sampling points with elevated soil lead concentrations above 192 mg/kg (**Figure 3**). While the lead contamination in this area has been partially bounded laterally (i.e., there were several adjacent sampling locations below the 192 mg/kg ecological screening level for lead), the complete extent of the contamination in the area has not been fully characterized. Furthermore, because this location was elevated above the range floor, it is possible that there may be some lead contamination present in subsurface soils (past sampling only considered the surface to two-foot depth of soil). A series of 12 XRF sample locations (two sample depths per location as previously described, and as shown in **Table 1**) are proposed to initially surround the RFI soil lead hot spots in this area (**Figure 6**).

In the event that the XRF soil samples collected from the inner rings around the RFI soil lead hot spots exhibit field XRF lead concentrations greater than 125 ppm, then additional soil samples from secondary rings of sampling locations will be added to the data set until the soil lead contamination is fully delineated vertically and laterally. Up to 20 secondary ring samples may be collected as necessary to clearly distinguish lead impacted soils (>192 mg/kg) from lower risk soils on the range.

The goal of the supplemental sampling is to better delineate the lateral and vertical limits within each of the three areas where soil lead concentrations exceed the 192 mg/kg ecological screening level for lead.

Additional soil characterization testing in the form of a seven-point composite sample will be collected within the boundaries of each of the three lead-impacted soil areas for the purpose of supporting toxicity characteristic leaching procedure (TCLP) analyses. The TCLP testing will determine whether the specific soil to be excavated and removed from the site will require management as hazardous (based on leachable lead concentrations) or may be managed and transported as non-hazardous soil. The cost for the soil disposal is entirely dependent on its characterization as hazardous for lead or non-hazardous. An improved delineation of these elevated soil lead areas in the northern zone will aid in the development of

limited removal soil actions to address these areas and reduce the average post-removal lead concentrations at UXO 7 to more acceptable concentrations.

4.0 Supplemental Delineation Sampling for PAH Concentrations in Soil

This proposed supplemental soil sampling program is focused on identified soil sampling locations from the RFI where the analytical data indicated elevated concentrations of PAHs present in the former small arms range soil. The collection of the supplemental soil sampling data will provide more detailed information on the nature and extent of the contaminated areas in the surface soil at the site (both laterally and vertically), and will guide the development of subsequent focused interim removal actions.

During the 2007 RFI, surface soil samples were collected for analysis of PAH compounds which were commonly used as a binding agent (pitch tar) in the manufacture of clay trap and skeet targets. The soil PAH concentrations are typically higher on older trap and skeet ranges (when the clay targets contained pitch tars), especially in those areas of the range where the clay target shards impact and accumulate on the range floor below the aerial locations where the targets were successfully shot and broken in the air. In the UXO 7 central zone there were two separate trap ranges (East Trap Range and West Trap Range) that were sampled for soil PAHs (**Figure 7**).

As with the lead delineation sampling, a similar sampling approach is proposed for the PAH areas, except that there will be no field screening step. All proposed vertical and horizontal PAH soil samples as shown on **Figures 8 and 9**, and identified in **Table 1**, will be collected and shipped to the FBL. Due to the limited extraction time associated with PAH analysis, the FBL will be instructed to extract all samples, but to initially analyze only those samples collected from the 0- to 1-foot depth of the inner rings of the sample areas. Based on the analyses for these samples, the FBL will then be instructed by Tetra Tech to analyze only those particular samples from the outer rings and deeper depths associated with those samples exceeding the PAH screening levels which will delineate the extent of PAH contamination within that area.

As an example, if a inner ring soil sample (0- to 1-foot bgs) establishes that the PAH contamination is localized and does not extend beyond one-foot deep and does not continue laterally beyond the first ring of samples (approximately 15-feet from the known point of soil PAH contamination), then that area of elevated PAH compounds in soil has been fully delineated and a soil removal activity may be designed to address that PAH-contaminated soil area as it was delineated by the supplemental sampling. For the stated example, the outer ring of soil samples would not require analysis because the identified PAH

contamination had been fully encircled by “clean” (PAHs below screening level) samples and will have identified the limits of the soil above PAH screening levels. Consequently, if soil samples collected from the 0- to 1-foot and the 1- to 2-foot depth still exceed PAH screening levels, then additional soil samples would need to be collected from greater step-out distances and depths (performed in a subsequent sampling event) to fully bound the extent of PAH contamination in soil. The PAH project action limits (PALs) are presented and discussed later in this Supplemental Work Plan.

In the East Trap Range there were two soil samples with PAHs above screening levels and additional soil samples should be collected and analyzed to bound the extent of the PAH contamination in the surface soil (**Figure 8**). The current approach is to encircle each PAH hotspot with an inner ring of six samples collected at a distance of 15 feet from the subject hotspot (two sampling depths – 0 to 1- and 1 to 2-foot bgs). These samples will be shipped to the FBL for extraction and PAH analysis.

Two larger outer rings at a radius of 30 feet from each hotspot will have 10 sampling points (again with each point featuring two sampling depths – 0 to 1- and 1 to 2-foot bgs). This sampling approach produces 16 sampling points and 32 soil samples per hotspot. The outer rings of samples will also be shipped to the FBL to be extracted, but held for PAH analysis. The FBL will be instructed to perform the PAH analysis only on those outer ring samples where a respective inner circle location exceeded the PAH screening level; otherwise, the outer ring samples will be discarded.

A similar situation exists for the West Trap Range, except there were four soil samples with PAH concentrations above screening levels (**Figure 9**). The four areas of PAH-impacted soils (above relevant PAH compound-specific screening levels) occur in the central area of the former trap range and are sufficiently close to one another that portions of the proposed sampling rings around the PAH hot spots overlap to some extent. Consequently, as shown on **Figure 9**, there are 22 proposed sampling locations along the inner rings surrounding the PAH hot spots (with two sample depths per location). There are an additional 20 sampling locations with each point available to supplement the inner sample ring data, as necessary, to clearly distinguish PAH impacted soils (above screening levels) from lower risk soils on the trap range. The outer ring of samples will also be shipped to the FBL to be extracted and held for PAH analysis. The FBL will be instructed to perform the PAH analysis only on those outer ring samples where a respective inner ring location exceeded the PAH screening level; otherwise, the outer ring samples will be discarded.

As described in the RFI results section of this Technical Memorandum, there is minimal indication of small arms range soil disturbance following the end of the small arms range operations. For this reason,

deeper contamination (below a depth of 2-feet bgs) is not anticipated for UXO 7. The CSM for trap and skeet ranges indicate that skeet fragments and residues are likely restricted to the top few inches of soil. Those skeet materials were deposited onto the ground surface after falling to the ground from the point in the air where the skeet target was impacted by shot from the shotgun shell, The skeet residues that occur then fall through the air to the ground surface and would not be expected to occur at depth.

Similar to the supplemental soil lead sampling program for UXO 7, the end goal of the supplemental PAH soil sampling program is to better delineate the lateral and vertical extent of the soil PAHs that exceed human health or ecological screening levels for the individual PAH compounds in the former trap range soil. An improved delineation of these areas of elevated PAHs in central zone soil will support development of limited soil removal actions to address these areas and reduce the average post-removal PAH soil concentrations at UXO 7 to more acceptable concentrations.

4.1 PAH Project Action Levels

The table presented below summarizes the project action limits (PALs) for which the PAHs in the East and West Trap Range areas will be compared.

PAH Compound	Project Action Limit (PAL) (µg/kg)
2-METHYLNAPHTHALENE	31,000
ACENAPHTHENE	340,000
ACENAPHTHYLENE	340,000
ANTHRACENE	1,700,000
BENZO(A)ANTHRACENE	150
BENZO(A)PYRENE	15
BENZO(B)FLUORANTHENE	150
BENZO(G,H,I)PERYLENE	170,000
BENZO(K)FLUORANTHENE	1,500
CHRYSENE	15,000
DIBENZO(A,H)ANTHRACENE	15
FLUORANTHENE	230,000
FLUORENE	230,000
INDENO(1,2,3-CD)PYRENE	150
NAPHTHALENE	14,000
PHENANTHRENE	170,000
PYRENE	170,000
BAP EQUIVALENT-HALFND	15
BAP EQUIVALENT-POS	15

The United States Environmental Protection Agency (USEPA) has identified seven PAHs as potentially carcinogenic: benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(k)fluoranthene, chrysene, dibenzo(a,h)anthracene, and indeno-(1,2,3-cd)-pyrene (highlighted in the above table). Of these PAHs, benzo(a)pyrene has been subjected to the most toxicological study and the USEPA has used the toxicological data to establish quantitative toxicological parameters (cancer slope factors and inhalation unit risks) for benzo(a)pyrene. All seven of these PAHs have a similar chemical structure and similar chemical properties. Laboratory studies suggest that these chemicals act similarly from the perspective of carcinogenicity and that the carcinogenic potency of the individual PAHs can be evaluated with reference to the carcinogenic potency of benzo(a)pyrene. Therefore, the USEPA has developed a toxicity equivalency factor (TEF) for each potentially carcinogenic PAH that can be used to convert the concentration of that PAH to an equivalent concentration of benzo(a)pyrene. Since benzo(a)pyrene is often abbreviated BaP, this process is known as determining the BaP equivalent concentration.

Therefore, in addition to a direct comparison to individual PAH PALs, a total for calculated BaP equivalents will also be done. The calculated screening value for BaP is 15 micrograms per kilogram ($\mu\text{g}/\text{kg}$).

5.0 Quality Assurance and Quality Control

The environmental field work completed to date at UXO 7 has been performed in accordance with an EPA-approved QAPP (*Tetra Tech, 2007; RCRA Addendum No. 2 to the QAPP for SWMUs 8, 15, 18, 19, 20 and the Old Gun Tub Storage Lot, and for UXO 5 [Building 2044 Drop Tower/Test Rail Site] and UXO 7 [Ranges], August*) which supplemented earlier approved QAPPs for NSA Crane from 2006 and 2004. The proposed field effort will be conducted in accordance with the primary guiding principles presented in the approved QAPPs, as summarized in this Technical Memorandum, and as specified in the field Standard Operating Procedures (SOPs) included as Attachment A. The analytical quality assurance/quality control (QA/QC) requirements for the sampling and analysis activity at UXO 7 will be in accordance with the FBL SOPs included as Attachment B. Field duplicates are regarded as a typical approach to assess project field sampling quality and field sample duplicates are planned for collection as part of the UXO 7 supplemental sampling effort. Sample summaries and QC samples are identified in **Table 2**.

TABLE 1

**FIELD SAMPLE SUMMARY
 UXO 07 – SUPPLEMENTAL SOIL SAMPLING
 NSA CRANE
 CRANE, INDIANA**

Sample Location⁽¹⁾	Sample ID⁽²⁾	LEAD (XRF⁽³⁾)	LEAD (SW 846-6010C)	PAHs (SW 846-8270C)
Soil Samples				
X7-SB200	X7-SS200-0001	---	---	1
	X7-SS200-0102	---	---	1
X7-SB201	X7-SS201-0001	---	---	1
	X7-SS201-0102	---	---	1
X7-SB202	X7-SS202-0001	---	---	1
	X7-SS202-0102	---	---	1
X7-SB203	X7-SS203-0001	---	---	1
	X7-SS203-0102	---	---	1
X7-SB204	X7-SS204-0001	---	---	1
	X7-SS204-0102	---	---	1
X7-SB205	X7-SS205-0001	---	---	1
	X7-SS205-0102	---	---	1
X7-SB206	X7-SS206-0001	---	---	1
	X7-SS206-0102	---	---	1
X7-SB207	X7-SS207-0001	---	---	1
	X7-SS207-0102	---	---	1
X7-SB208	X7-SS208-0001	---	---	1
	X7-SS208-0102	---	---	1
X7-SB209	X7-SS209-0001	---	---	1
	X7-SS209-0102	---	---	1
X7-SB210	X7-SS210-0001	---	---	1
	X7-SS210-0102	---	---	1
X7-SB211	X7-SS211-0001	---	---	1
	X7-SS211-0102	---	---	1
X7-SB212	X7-SS212-0001	---	---	1

Sample Location⁽¹⁾	Sample ID⁽²⁾	LEAD (XRF⁽³⁾)	LEAD (SW 846-6010C)	PAHs (SW 846-8270C)
	X7-SS212-0102	---	---	1
X7-SB213	X7-SS213-0001	---	---	1
	X7-SS213-0102	---	---	1
X7-SB214	X7-SS214-0001	---	---	1
	X7-SS214-0102	---	---	1
X7-SB215	X7-SS215-0001	---	---	1
	X7-SS215-0102	---	---	1
X7-SB216	X7-SS216-0001	---	---	1
	X7-SS216-0102	---	---	1
X7-SB217	X7-SS217-0001	---	---	1
	X7-SS217-0102	---	---	1
X7-SB218	X7-SS218-0001	---	---	1
	X7-SS218-0102	---	---	1
X7-SB219	X7-SS219-0001	---	---	1
	X7-SS219-0102	---	---	1
X7-SB220	X7-SS220-0001	---	---	1
	X7-SS220-0102	---	---	1
X7-SB221	X7-SS221-0001	---	---	1
	X7-SS221-0102	---	---	1
X7-SB222	X7-SS222-0001	---	---	1
	X7-SS222-0102	---	---	1
X7-SB223	X7-SS223-0001	---	---	1
	X7-SS223-0102	---	---	1
X7-SB224	X7-SS224-0001	---	---	1
	X7-SS224-0102	---	---	1
X7-SB225	X7-SS225-0001	---	---	1
	X7-SS225-0102	---	---	1
X7-SB226	X7-SS226-0001	---	---	1
	X7-SS226-0102	---	---	1
X7-SB227	X7-SS227-0001	---	---	1
	X7-SS227-0102	---	---	1
X7-SB228	X7-SS228-0001	---	---	1
	X7-SS228-0102	---	---	1
X7-SB229	X7-SS229-0001	---	---	1

Sample Location⁽¹⁾	Sample ID⁽²⁾	LEAD (XRF⁽³⁾)	LEAD (SW 846-6010C)	PAHs (SW 846-8270C)
	X7-SS229-0102	---	---	1
X7-SB230	X7-SS230-0001	---	---	1
	X7-SS230-0102	---	---	1
X7-SB231	X7-SS231-0001	---	---	1
	X7-SS231-0102	---	---	1
X7-SB232	X7-SS232-0001	---	---	1
	X7-SS232-0102	---	---	1
X7-SB233	X7-SS233-0001	---	---	1
	X7-SS233-0102	---	---	1
X7-SB234	X7-SS234-0001	---	---	1
	X7-SS234-0102	---	---	1
X7-SB235	X7-SS235-0001	---	---	1
	X7-SS235-0102	---	---	1
X7-SB236	X7-SS236-0001	---	---	1
	X7-SS236-0102	---	---	1
X7-SB237	X7-SS237-0001	---	---	1
	X7-SS237-0102	---	---	1
X7-SB238	X7-SS238-0001	---	---	1
	X7-SS238-0102	---	---	1
X7-SB239	X7-SS239-0001	---	---	1
	X7-SS239-0102	---	---	1
X7-SB240	X7-SS240-0001	---	---	1
	X7-SS240-0102	---	---	1
X7-SB241	X7-SS241-0001	---	---	1
	X7-SS241-0102	---	---	1
X7-SB242	X7-SS242-0001	---	---	1
	X7-SS242-0102	---	---	1
X7-SB243	X7-SS243-0001	---	---	1
	X7-SS243-0102	---	---	1
X7-SB244	X7-SS244-0001	---	---	1
	X7-SS244-0102	---	---	1
X7-SB245	X7-SS245-0001	---	---	1
	X7-SS245-0102	---	---	1
X7-SB246	X7-SS246-0001	---	---	1

Sample Location⁽¹⁾	Sample ID⁽²⁾	LEAD (XRF⁽³⁾)	LEAD (SW 846-6010C)	PAHs (SW 846-8270C)
	X7-SS246-0102	---	---	1
X7-SB247	X7-SS247-0001	---	---	1
	X7-SS247-0102	---	---	1
X7-SB248	X7-SS248-0001	---	---	1
	X7-SS248-0102	---	---	1
X7-SB249	X7-SS249-0001	---	---	1
	X7-SS249-0102	---	---	1
X7-SB250	X7-SS250-0001	---	---	1
	X7-SS250-0102	---	---	1
X7-SB251	X7-SS251-0001	---	---	1
	X7-SS251-0102	---	---	1
X7-SB252	X7-SS252-0001	---	---	1
	X7-SS252-0102	---	---	1
X7-SB253	X7-SS253-0001	---	---	1
	X7-SS253-0102	---	---	1
X7-SB254	X7-SS254-0001	---	---	1
	X7-SS254-0102	---	---	1
X7-SB255	X7-SS255-0001	---	---	1
	X7-SS255-0102	---	---	1
X7-SB256	X7-SS256-0001	---	---	1
	X7-SS256-0102	---	---	1
X7-SB257	X7-SS257-0001	---	---	1
	X7-SS257-0102	---	---	1
X7-SB258	X7-SS258-0001	---	---	1
	X7-SS258-0102	---	---	1
X7-SB259	X7-SS259-0001	---	---	1
	X7-SS259-0102	---	---	1
X7-SB260	X7-SS260-0001	---	---	1
	X7-SS260-0102	---	---	1
X7-SB261	X7-SS261-0001	---	---	1
	X7-SS261-0102	---	---	1
X7-SB262	X7-SS262-0001	---	---	1
	X7-SS262-0102	---	---	1
X7-SB263	X7-SS263-0001	---	---	1

Sample Location ⁽¹⁾	Sample ID ⁽²⁾	LEAD (XRF ⁽³⁾)	LEAD (SW 846-6010C)	PAHs (SW 846-8270C)
	X7-SS263-0102	---	---	1
X7-SB264	X7-SS264-0001	---	---	1
	X7-SS264-0102	---	---	1
X7-SB265	X7-SS265-0001	---	---	1
	X7-SS265-0102	---	---	1
X7-SB266	X7-SS266-0001	---	---	1
	X7-SS266-0102	---	---	1
X7-SB267	X7-SS267-0001	---	---	1
	X7-SS267-0102	---	---	1
X7-SB268	X7-SS268-0001	---	---	1
	X7-SS268-0102	---	---	1
X7-SB269	X7-SS269-0001	---	---	1
	X7-SS269-0102	---	---	1
X7-SB270	X7-SS270-0001	---	---	1
	X7-SS270-0102	---	---	1
X7-SB271	X7-SS271-0001	---	---	1
	X7-SS271-0102	---	---	1
X7-SB272	X7-SS272-0001	---	---	1
	X7-SS272-0102	---	---	1
X7-SB273	X7-SS273-0001	---	---	1
	X7-SS273-0102	---	---	1
X7-SB274	X7-SS274-0001	1	TBD ⁽⁴⁾	---
	X7-SS274-0102	2	TBD ⁽⁴⁾	---
X7-SB275	X7-SS275-0001	1	TBD ⁽⁴⁾	---
	X7-SS275-0102	2	TBD ⁽⁴⁾	---
X7-SB276	X7-SS276-0001	1	TBD ⁽⁴⁾	---
	X7-SS276-0102	2	TBD ⁽⁴⁾	---
X7-SB277	X7-SS277-0001	1	TBD ⁽⁴⁾	---
	X7-SS277-0102	2	TBD ⁽⁴⁾	---
X7-SB278	X7-SS278-0001	1	TBD ⁽⁴⁾	---
	X7-SS278-0102	2	TBD ⁽⁴⁾	---
X7-SB279	X7-SS279-0001	1	TBD ⁽⁴⁾	---
	X7-SS279-0102	2	TBD ⁽⁴⁾	---
X7-SB280	X7-SS28-0001	1	TBD ⁽⁴⁾	---

Sample Location⁽¹⁾	Sample ID⁽²⁾	LEAD (XRF⁽³⁾)	LEAD (SW 846-6010C)	PAHs (SW 846-8270C)
	X7-SS280-0102	2	TBD ⁽⁴⁾	---
X7-SB281	X7-SS281-0001	1	TBD ⁽⁴⁾	---
	X7-SS281-0102	2	TBD ⁽⁴⁾	---
X7-SB282	X7-SS282-0001	1	TBD ⁽⁴⁾	---
	X7-SS282-0102	2	TBD ⁽⁴⁾	---
X7-SB283	X7-SS283-0001	1	TBD ⁽⁴⁾	---
	X7-SS283-0102	2	TBD ⁽⁴⁾	---
X7-SB284	X7-SS284-0001	1	TBD ⁽⁴⁾	---
	X7-SS284-0102	2	TBD ⁽⁴⁾	---
X7-SB285	X7-SS285-0001	1	TBD ⁽⁴⁾	---
	X7-SS285-0102	2	TBD ⁽⁴⁾	---
X7-SB286	X7-SS286-0001	1	TBD ⁽⁴⁾	---
	X7-SS286-0102	2	TBD ⁽⁴⁾	---
X7-SB287	X7-SS287-0001	1	TBD ⁽⁴⁾	---
	X7-SS287-0102	2	TBD ⁽⁴⁾	---
X7-SB288	X7-SS288-0001	1	TBD ⁽⁴⁾	---
	X7-SS288-0102	2	TBD ⁽⁴⁾	---
X7-SB289	X7-SS289-0001	1	TBD ⁽⁴⁾	---
	X7-SS289-0102	2	TBD ⁽⁴⁾	---
X7-SB290	X7-SS290-0001	1	TBD ⁽⁴⁾	---
	X7-SS290-0102	2	TBD ⁽⁴⁾	---
X7-SB291	X7-SS291-0001	1	TBD ⁽⁴⁾	---
	X7-SS291-0102	2	TBD ⁽⁴⁾	---
X7-SB292	X7-SS292-0001	1	TBD ⁽⁴⁾	---
	X7-SS292-0102	2	TBD ⁽⁴⁾	---
X7-SB293	X7-SS293-0001	1	TBD ⁽⁴⁾	---
	X7-SS293-0102	2	TBD ⁽⁴⁾	---
X7-SB294	X7-SS294-0001	1	TBD ⁽⁴⁾	---
	X7-SS294-0102	2	TBD ⁽⁴⁾	---
X7-SB295	X7-SS295-0001	1	TBD ⁽⁴⁾	---
	X7-SS295-0102	2	TBD ⁽⁴⁾	---
X7-SB296	X7-SS296-0001	1	TBD ⁽⁴⁾	---
	X7-SS296-0102	2	TBD ⁽⁴⁾	---
X7-SB297	X7-SS297-0001	1	TBD ⁽⁴⁾	---

Sample Location⁽¹⁾	Sample ID⁽²⁾	LEAD (XRF⁽³⁾)	LEAD (SW 846-6010C)	PAHs (SW 846-8270C)
	X7-SS297-0102	2	TBD ⁽⁴⁾	---
X7-SB298	X7-SS298-0001	1	TBD ⁽⁴⁾	---
	X7-SS298-0102	2	TBD ⁽⁴⁾	---
X7-SB299	X7-SS299-0001	1	TBD ⁽⁴⁾	---
	X7-SS299-0102	2	TBD ⁽⁴⁾	---
X7-SB300	X7-SS300-0001	1	TBD ⁽⁴⁾	---
	X7-SS300-0102	2	TBD ⁽⁴⁾	---
X7-SB301	X7-SS301-0001	1	TBD ⁽⁴⁾	---
	X7-SS301-0102	2	TBD ⁽⁴⁾	---
X7-SB302	X7-SS302-0001	1	TBD ⁽⁴⁾	---
	X7-SS302-0102	2	TBD ⁽⁴⁾	---
X7-SB303	X7-SS303-0001	1	TBD ⁽⁴⁾	---
	X7-SS303-0102	2	TBD ⁽⁴⁾	---
X7-SB304	X7-SS304-0001	1	TBD ⁽⁴⁾	---
	X7-SS304-0102	2	TBD ⁽⁴⁾	---
X7-SB305	X7-SS305-0001	1	TBD ⁽⁴⁾	---
	X7-SS305-0102	2	TBD ⁽⁴⁾	---
X7-SB306	X7-SS306-0001	1	TBD ⁽⁴⁾	---
	X7-SS306-0102	2	TBD ⁽⁴⁾	---
X7-SB307	X7-SS307-0001	1	TBD ⁽⁴⁾	---
	X7-SS307-0102	2	TBD ⁽⁴⁾	---
X7-SB308	X7-SS308-0001	1	TBD ⁽⁴⁾	---
	X7-SS308-0102	2	TBD ⁽⁴⁾	---
X7-SB309	X7-SS309-0001	1	TBD ⁽⁴⁾	---
	X7-SS309-0102	2	TBD ⁽⁴⁾	---
X7-SB310	X7-SS310-0001	1	TBD ⁽⁴⁾	---
	X7-SS310-0102	2	TBD ⁽⁴⁾	---
X7-SB311	X7-SS311-0001	1	TBD ⁽⁴⁾	---
	X7-SS311-0102	2	TBD ⁽⁴⁾	---
X7-SB312	X7-SS312-0001	1	TBD ⁽⁴⁾	---
	X7-SS312-0102	2	TBD ⁽⁴⁾	---
X7-SB313	X7-SS313-0001	1	TBD ⁽⁴⁾	---
	X7-SS313-0102	2	TBD ⁽⁴⁾	---
X7-SB314	X7-SS314-0001	1	TBD ⁽⁴⁾	---

Sample Location ⁽¹⁾	Sample ID ⁽²⁾	LEAD (XRF ⁽³⁾)	LEAD (SW 846-6010C)	PAHs (SW 846-8270C)
	X7-SS314-0102	2	TBD ⁽⁴⁾	---
X7-SB315	X7-SS315-0001	1	TBD ⁽⁴⁾	---
	X7-SS315-0102	2	TBD ⁽⁴⁾	---
X7-SB316	X7-SS316-0001	1	TBD ⁽⁴⁾	---
	X7-SS316-0102	2	TBD ⁽⁴⁾	---
X7-SB317	X7-SS317-0001	1	TBD ⁽⁴⁾	---
	X7-SS317-0102	2	TBD ⁽⁴⁾	---
X7-SB318	X7-SS318-0001	1	TBD ⁽⁴⁾	---
	X7-SS318-0102	2	TBD ⁽⁴⁾	---
X7-SB319	X7-SS319-0001	1	TBD ⁽⁴⁾	---
	X7-SS319-0102	2	TBD ⁽⁴⁾	---
X7-SB320	X7-SS320-0001	1	TBD ⁽⁴⁾	---
	X7-SS320-0102	2	TBD ⁽⁴⁾	---
X7-SB391	X7-SS391-0001	1	TBD ⁽⁴⁾	---
	X7-SS391-0102	2	TBD ⁽⁴⁾	---
X7-SB392	X7-SS392-0001	1	TBD ⁽⁴⁾	---
	X7-SS392-0102	2	TBD ⁽⁴⁾	---
X7-SB393	X7-SS393-0001	1	TBD ⁽⁴⁾	---
	X7-SS393-0102	2	TBD ⁽⁴⁾	---
X7-SB394	X7-SS394-0001	1	TBD ⁽⁴⁾	---
	X7-SS394-0102	2	TBD ⁽⁴⁾	---
X7-SB395	X7-SS395-0001	1	TBD ⁽⁴⁾	---
	X7-SS395-0102	2	TBD ⁽⁴⁾	---
X7-SB396	X7-SS396-0001	1	TBD ⁽⁴⁾	---
	X7-SS396-0102	2	TBD ⁽⁴⁾	---
Total Soil Samples		106	TBD⁽⁴⁾	148
Potential Step-Out Samples (collected as needed)				
X7-SB321 thru X7-SB390	X7-SS321-0001 thru X7-SS390-0001	69	TBD ⁽⁴⁾	---
	X7-SS321-0102 thru X7-SS390-0102	69	TBD ⁽⁴⁾	---
X7-SB397 thru X7-SB406	X7-SS397-0001 thru X7-SS406-0001	10	TBD ⁽⁴⁾	---
	X7-SS397-0102 thru X7-SS406-0102	10	TBD ⁽⁴⁾	---
Total Potential Step-Out Soil Samples		158⁽⁵⁾	TBD⁽⁴⁾	0

FBL = Fixed-base laboratory
PAH = Polynuclear aromatic hydrocarbons
ppm = parts per million
TBD = To be determined
XRF = X-ray fluorescence

- 1 X7 = UXO 7. SB = Soil boring.
- 2 SS = Surface soil. Last four digits of sample ID indicate depth below ground surface in feet.
- 3 1 = Sample analyzed in the field via XRF. 2 = Sample analyzed in the field via XRF if respective sample (0 to 1 foot bgs) exceeds field screening value of 125 ppm.
- 4 All soil samples used to define the interface between "contaminated" and "non-contaminated" soils will be selected for confirmatory lead analysis at the FBL.
- 5 Additional "step-out" lead soil samples may be collected in order to define the extent of lead contamination in the field based on XRF analysis.

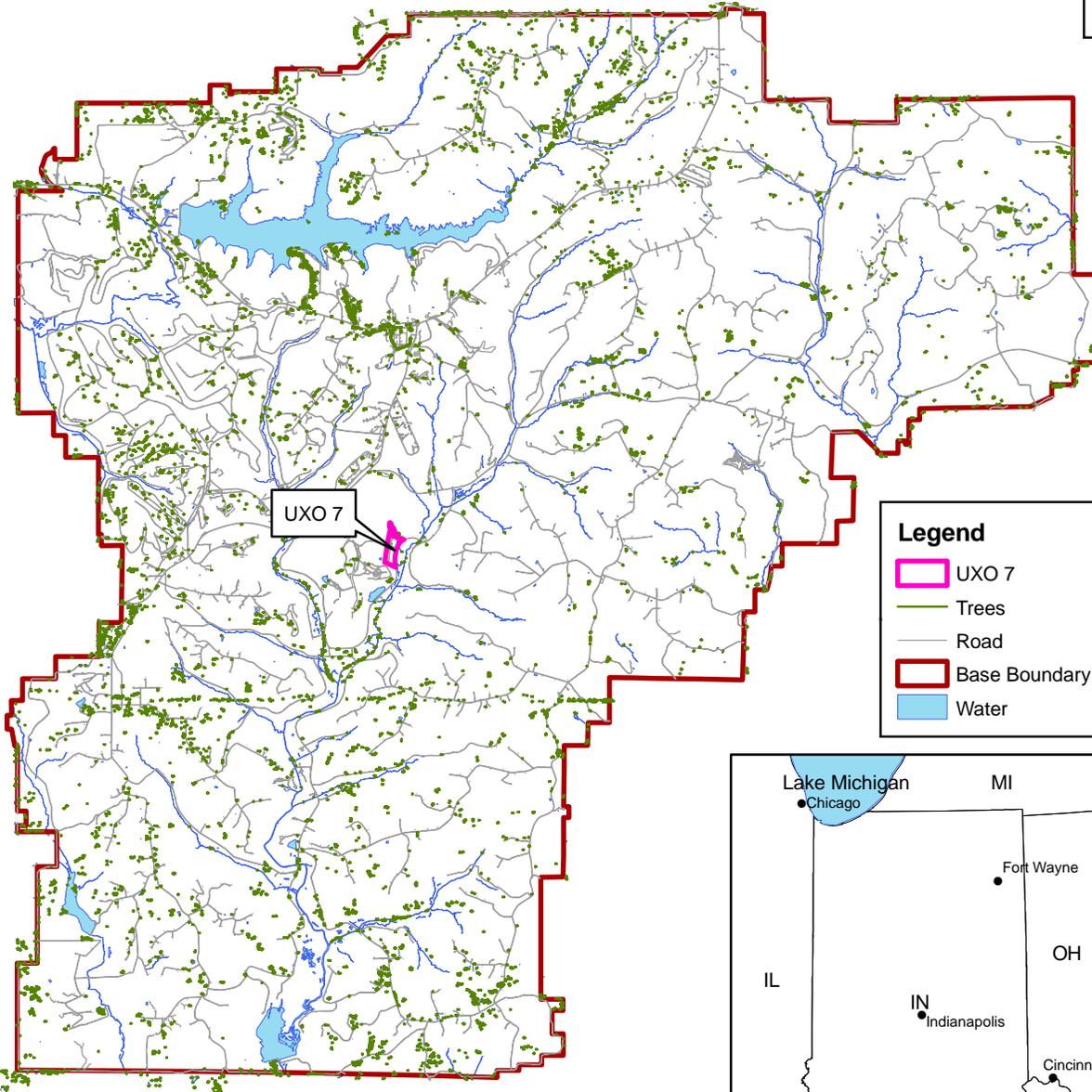
TABLE 2

**FIXED-BASE LABORATORY SAMPLE SUMMARY
UXO 07 – SUPPLEMENTAL SOIL SAMPLING
NSA CRANE
CRANE, INDIANA**

Matrix	Analytical Group	Concentration Level	Samples	Field Duplicates⁽¹⁾	MS/MSDs⁽¹⁾	Rinsate Blanks⁽²⁾	Total Samples to Lab
Solid	Lead SW 846-6010C ⁽³⁾	Low to Moderate	45 ⁽⁴⁾	3	3	1	52
Solid	PAHs SW 846-8270C ⁽³⁾	Low to Moderate	148	8	8	1	165

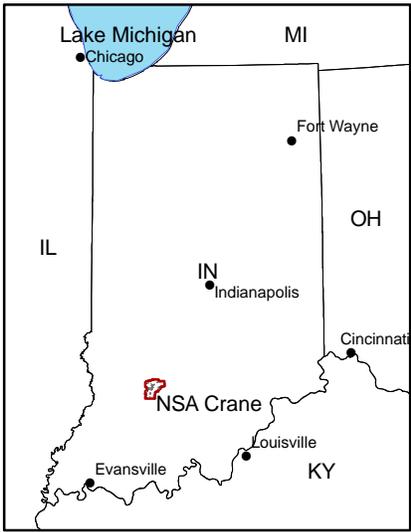
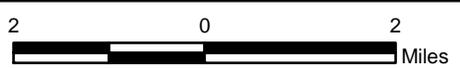
MS/MSD = Matrix spike/matrix spike duplicate

- 1 Field duplicates and MS/MSD samples will be collected at a frequency of 1 per 20 samples per analyte.
- 2 Rinsate blanks will be collected at a frequency of one per type of non-dedicated equipment.
- 3 Analysis to be performed by Empirical Laboratories.
- 4 Actual number of lead samples may vary slightly and are dependent on field XRF screening results.



Legend

- UXO 7
- Trees
- Road
- Base Boundary
- Water



DRAWN BY	DATE
T. WHEATON	05/12/10
CHECKED BY	DATE
J. GOERDT	07/08/11
REVISED BY	DATE
SCALE AS NOTED	



SITE LOCATION MAP
UXO 7 - SUPPLEMENTAL
SAMPLING WORK PLAN
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER CTO F272	
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO.	REV
FIGURE 1	0

Aerial photograph taken in 2009.



DRAWN BY J. ENGLISH CHECKED BY J. GOERDT REVISED BY DATE 07/07/11 DATE 07/08/11 DATE SCALE AS NOTED	 UXO 7 SMALL ARMS RANGES UXO 7 - SUPPLEMENTAL SAMPLING WORK PLAN NSA CRANE CRANE, INDIANA	CONTRACT NUMBER _____ CTO NUMBER F272 APPROVED BY _____ DATE _____ APPROVED BY _____ DATE _____ FIGURE NO. 2 REV 0
--	---	---

Aerial photograph taken in 2009.



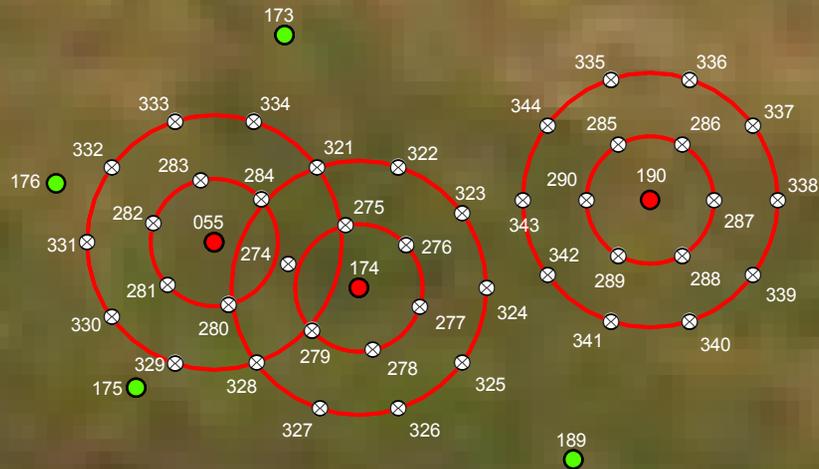
Legend	
●	192 mg/kg Lead Exceedance Detected
●	No Exceedance
—	Lead Sampling Boundary

DRAWN BY	DATE
J. ENGLISH	07/05/11
CHECKED BY	DATE
J. GOERDT	07/07/11
REVISED BY	DATE
SCALE	AS NOTED



SUPPLEMENTAL LEAD SOIL SAMPLING AREAS
NORTHERN ZONE
UXO 7 - SUPPLEMENTAL SAMPLING WORK PLAN
NSA CRANE
CRANE, INDIANA

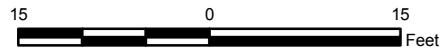
CONTRACT NUMBER	CTO NUMBER
	F272
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO.	REV
3	0



Legend

- ⊗ Proposed Lead Sampling Location
- Lead Sampling Boundary
- 192 mg/kg Lead Exceedance Detected
- No Exceedance

All location IDs begin with "X7-SB."



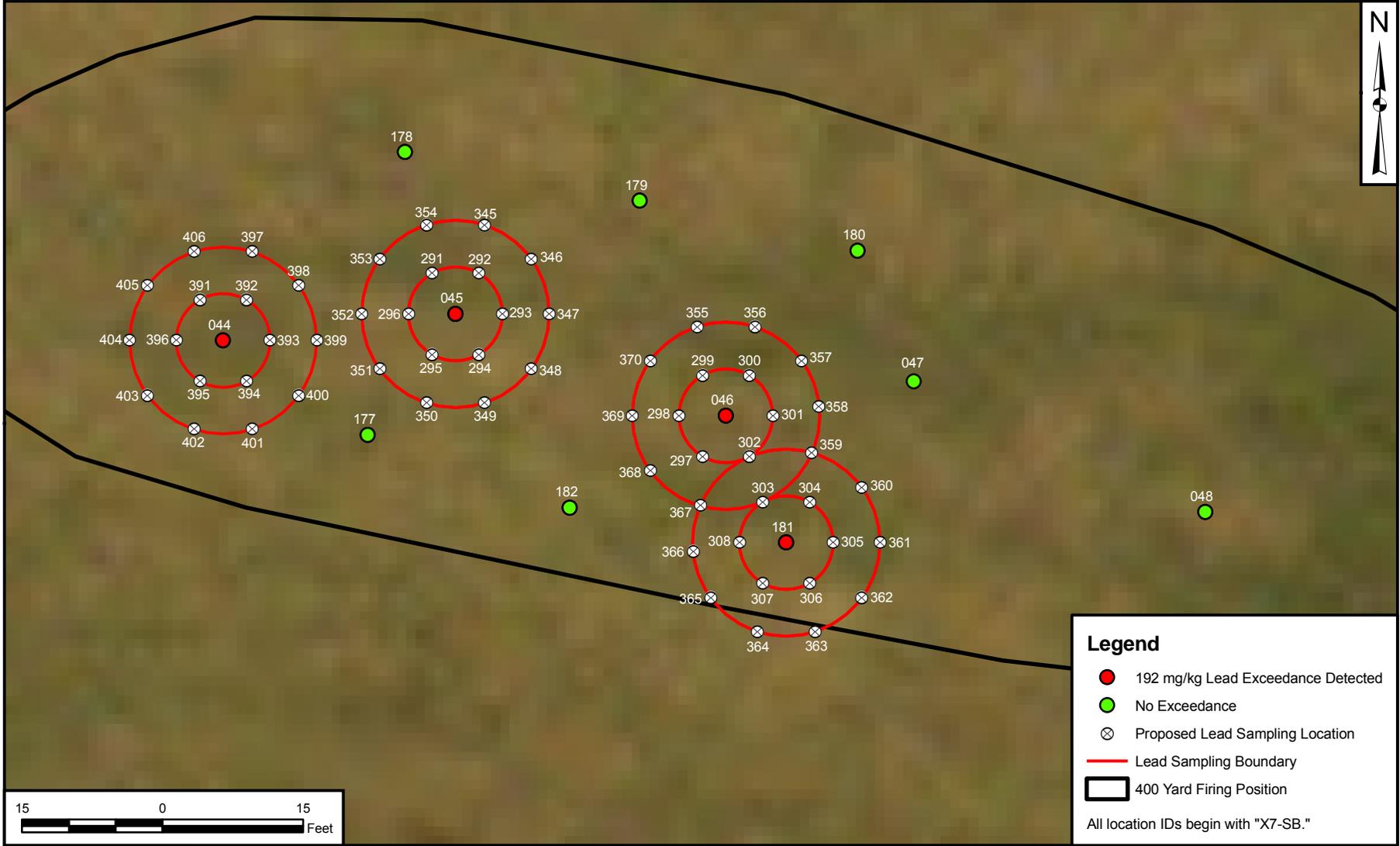
DRAWN BY	DATE
J. ENGLISH	07/05/11
CHECKED BY	DATE
J. GOERDT	07/08/11
REVISED BY	DATE
MK BOND	09/28/11



PROPOSED SUPPLEMENTAL LEAD SOIL SAMPLING
NORTHERNMOST AREA OF NORTHERN ZONE
UXO 7 - SUPPLEMENTAL SAMPLING WORK PLAN
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	CTO NUMBER
—	F272
APPROVED BY	DATE
—	—
APPROVED BY	DATE
—	—
FIGURE NO.	REV
4	0

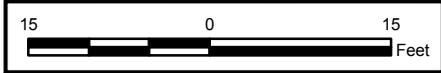
SCALE
AS NOTED



Legend

- 192 mg/kg Lead Exceedance Detected
- No Exceedance
- ⊗ Proposed Lead Sampling Location
- Lead Sampling Boundary
- 400 Yard Firing Position

All location IDs begin with "X7-SB."

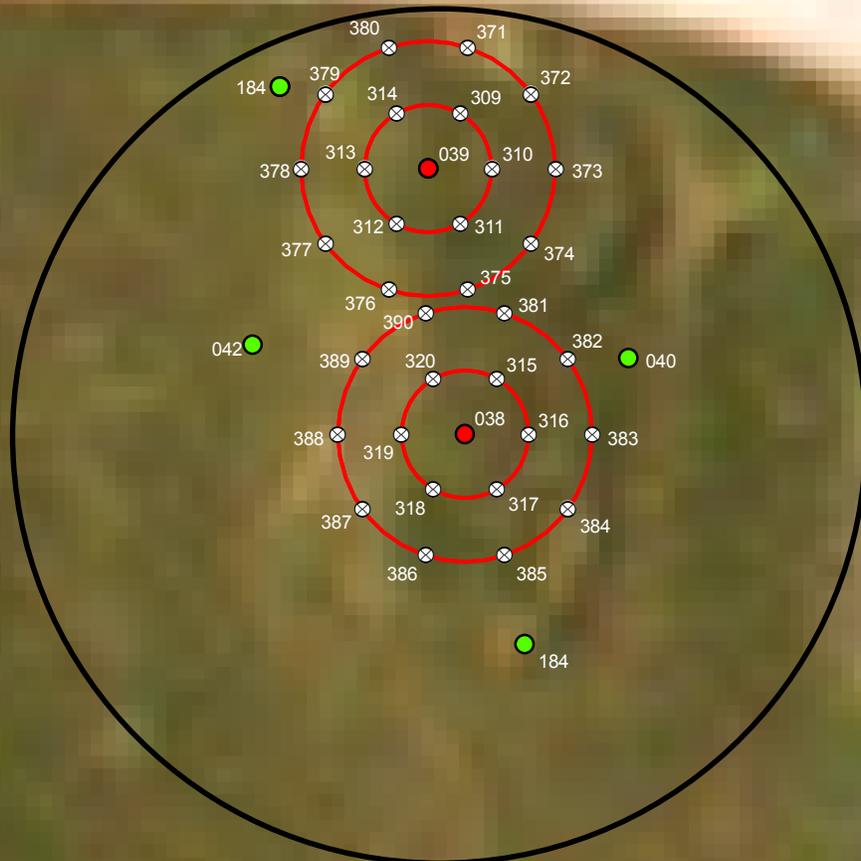


DRAWN BY	DATE
J. ENGLISH	07/05/11
CHECKED BY	DATE
J. GOERDT	07/28/11
REVISIED BY	DATE
MK BOND	07/28/11
SCALE AS NOTED	



PROPOSED SUPPLEMENTAL LEAD SOIL SAMPLING
CENTRAL AREA OF NORTHERN ZONE
UXO 7 - SUPPLEMENTAL SAMPLING WORK PLAN
NSA CRANE
CRANE, INDIANA

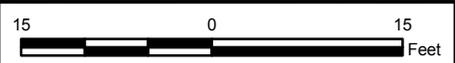
CONTRACT NUMBER	CTO NUMBER
---	F272
APPROVED BY	DATE
---	---
APPROVED BY	DATE
---	---
FIGURE NO.	REV
5	0



Legend

- ⊗ Proposed Lead Sampling Location
- 192 mg/kg Lead Exceedance Detected
- No Exceedance
- Lead Sampling Boundary
- Dirt Mound

All location IDs begin with "X7-SB."

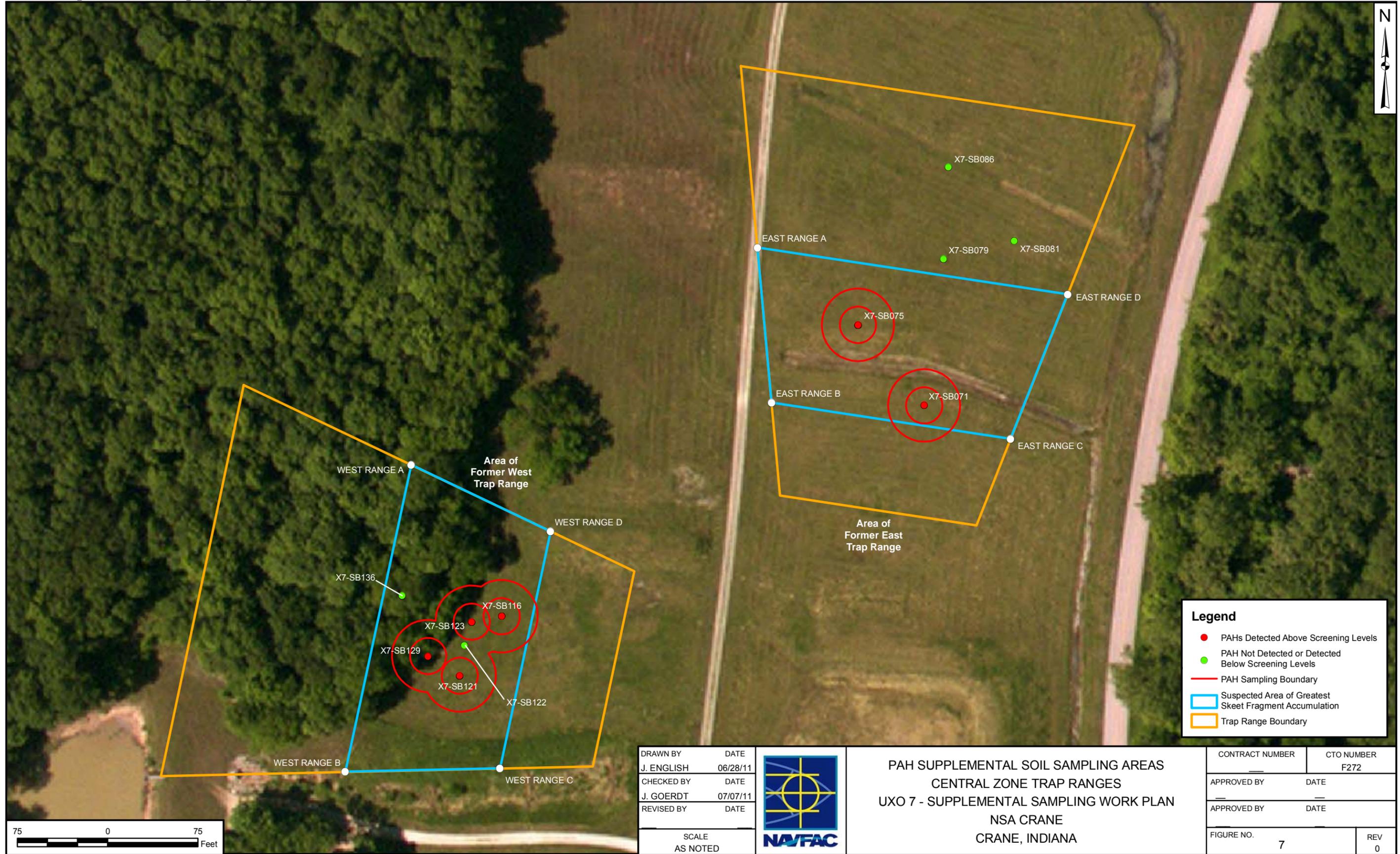


DRAWN BY	DATE
J. ENGLISH	07/05/11
CHECKED BY	DATE
J. GOERDT	09/28/11
REVISD BY	DATE
MK BOND	09/28/11
SCALE AS NOTED	



PROPOSED SUPPLEMENTAL LEAD SOIL SAMPLING
 SOUTHERNMOST AREA OF NORTHERN ZONE
 UXO 7 - SUPPLEMENTAL
 SAMPLING WORK PLAN
 NSA CRANE
 CRANE, INDIANA

CONTRACT NUMBER	CTO NUMBER
---	F272
APPROVED BY	DATE
---	---
APPROVED BY	DATE
---	---
FIGURE NO.	REV
6	0



Legend

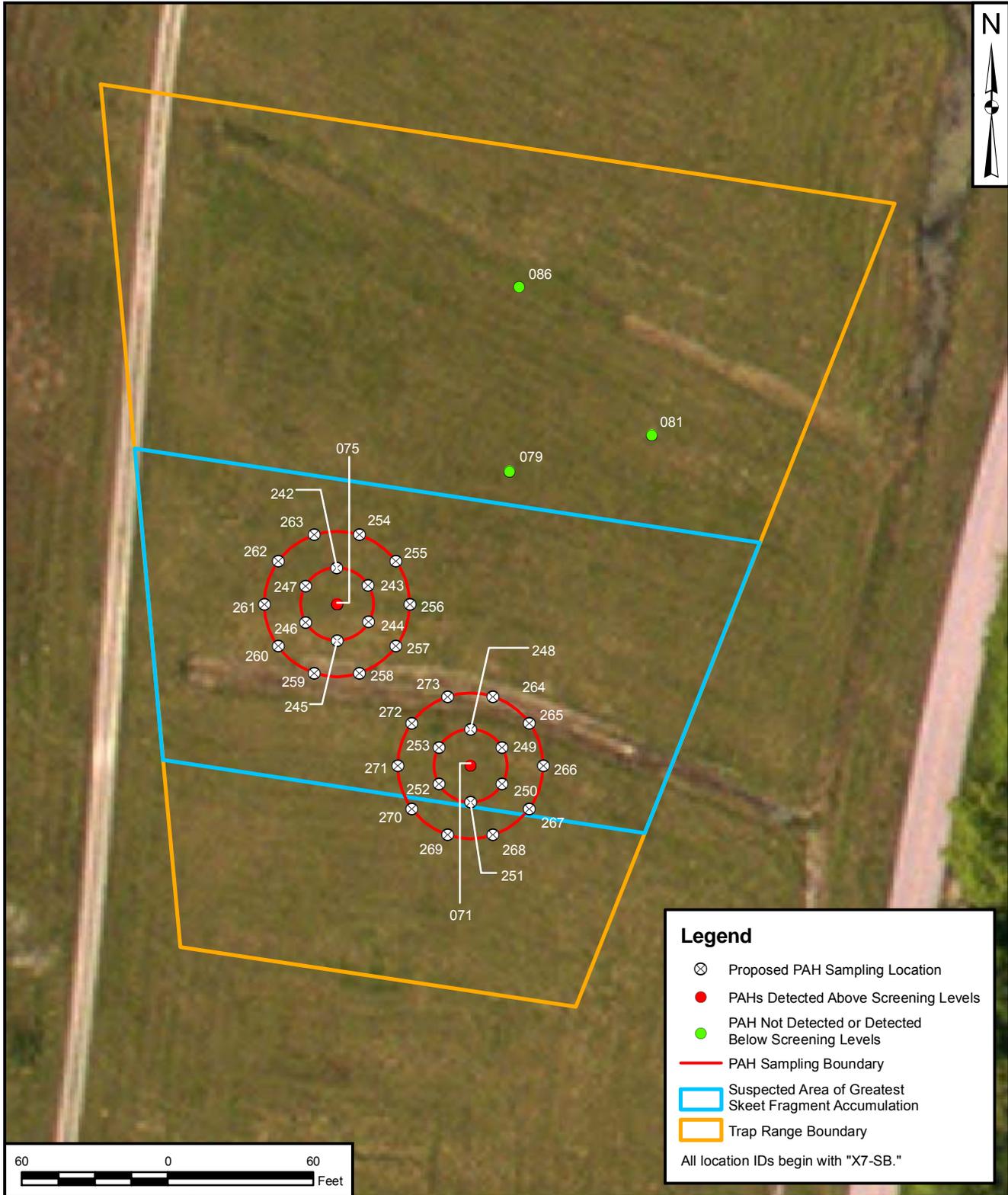
- PAHs Detected Above Screening Levels
- PAH Not Detected or Detected Below Screening Levels
- PAH Sampling Boundary
- Suspected Area of Greatest Sket Fragment Accumulation
- Trap Range Boundary

DRAWN BY	DATE
J. ENGLISH	06/28/11
CHECKED BY	DATE
J. GOERDT	07/07/11
REVISED BY	DATE
SCALE AS NOTED	



PAH SUPPLEMENTAL SOIL SAMPLING AREAS
CENTRAL ZONE TRAP RANGES
UXO 7 - SUPPLEMENTAL SAMPLING WORK PLAN
NSA CRANE
CRANE, INDIANA

CONTRACT NUMBER	CTO NUMBER
	F272
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO.	REV
7	0



Legend

- ⊗ Proposed PAH Sampling Location
- PAHs Detected Above Screening Levels
- PAH Not Detected or Detected Below Screening Levels
- PAH Sampling Boundary
- Suspected Area of Greatest Skeet Fragment Accumulation
- Trap Range Boundary

All location IDs begin with "X7-SB."

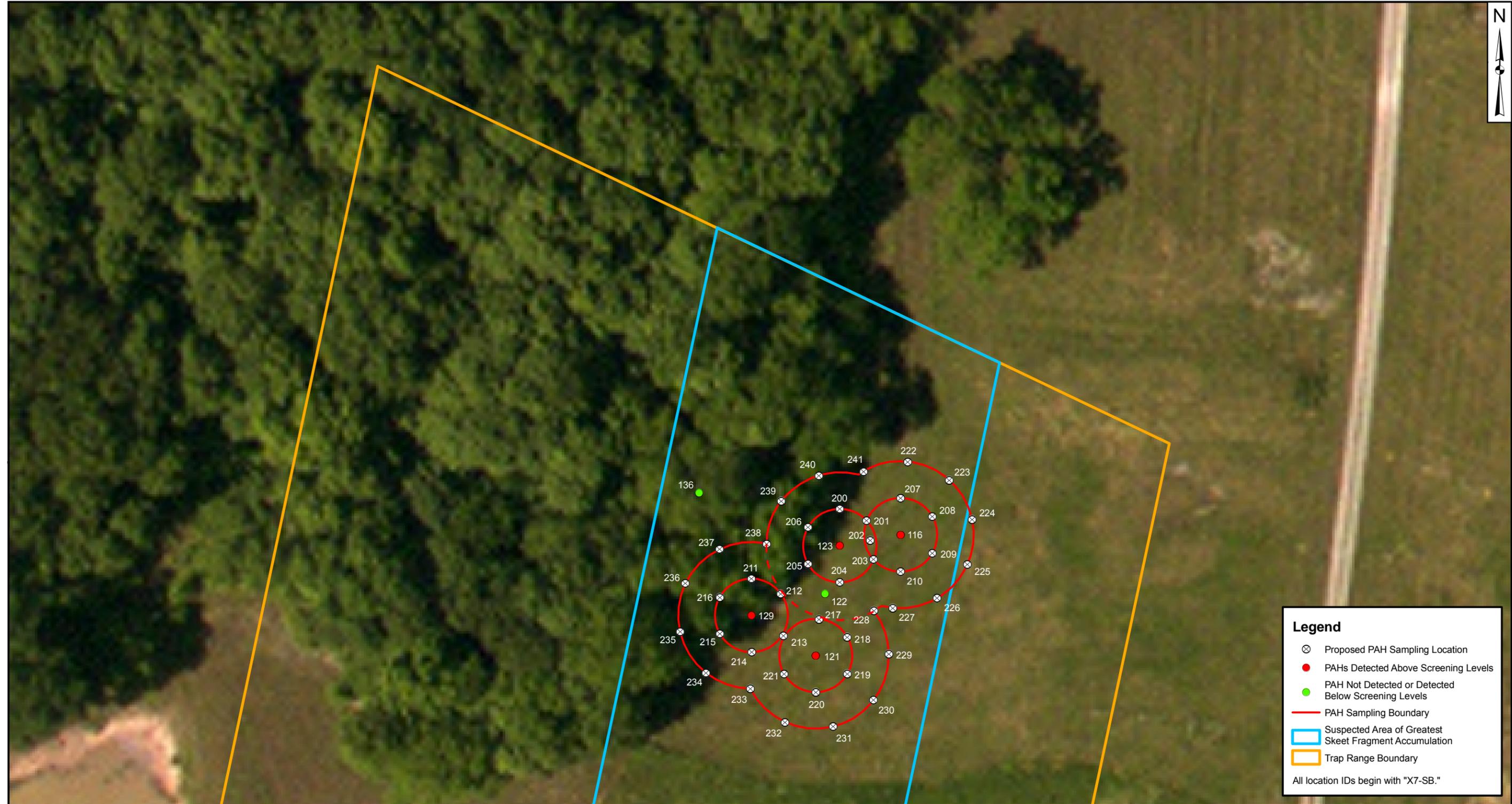


DRAWN BY	DATE
J. ENGLISH	06/28/11
CHECKED BY	DATE
J. GOERDT	07/07/11
REVISED BY	DATE
SCALE AS NOTED	



PROPOSED SUPPLEMENTAL PAH SOIL SAMPLING
EAST TRAP RANGE
UXO 7 - SUPPLEMENTAL
SAMPLING WORK PLAN
NSA CRANE
CRANE, INDIANA

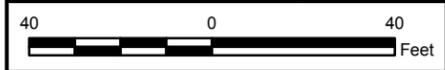
CONTRACT NUMBER	CTO NUMBER
---	F272
APPROVED BY	DATE
---	---
APPROVED BY	DATE
---	---
FIGURE NO.	REV
8	0



Legend

- ⊗ Proposed PAH Sampling Location
- PAHs Detected Above Screening Levels
- PAH Not Detected or Detected Below Screening Levels
- PAH Sampling Boundary
- ▭ Suspected Area of Greatest Skeet Fragment Accumulation
- ▭ Trap Range Boundary

All location IDs begin with "X7-SB."



DRAWN BY	DATE
J. ENGLISH	06/28/11
CHECKED BY	DATE
J. GOERDT	07/07/11
REVISED BY	DATE
SCALE AS NOTED	



PROPOSED SUPPLEMENTAL PAH SOIL SAMPLING
 WEST TRAP RANGE
 UXO 7 - SUPPLEMENTAL SAMPLING WORK PLAN
 NSA CRANE
 CRANE, INDIANA

CONTRACT NUMBER	CTO NUMBER
	F272
APPROVED BY	DATE
APPROVED BY	DATE
FIGURE NO.	REV
9	0

ATTACHMENT A
FIELD STANDARD OPERATING PROCEDURES
UXO 7 – SMALL ARMS RANGES
NSA CRANE
CRANE, INDIANA

Table of Contents

SOP-01	Sample Labeling
SOP-02	Sample Identification Nomenclature
SOP-03	Sample Custody and Documentation of Field Activities
SOP-04	Sample Preservation, Packaging, and Shipping
SOP-05	Soil Coring and Sampling Using Hand Auger Techniques
SOP-06	Soil Sample Logging
SOP-07	Decontamination of Field Sampling Equipment
SOP-08	Management of Investigation-Derived Waste
SOP-09	Global Positioning System
SOP-10	Field Portable X-Ray Fluorescence Analysis of Soil and Sediment Samples Using the INNOV-X Alpha Series Instrument

STANDARD OPERATING PROCEDURE

SOP-01

SAMPLE LABELING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures to be used for labeling sample containers. Sample labels are used to document the sample ID, date, time, analysis to be performed, preservative, matrix, sampler, and the analytical laboratory. A sample label will be attached to each sample container.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)

Disposable medical-grade gloves (e.g. latex, nitrile)

Sample log sheets

Required sample containers: All sample containers for analysis by fixed-base laboratories will be supplied and deemed certified clean by the laboratory.

Sample labels

Chain-of-custody records

Sealable polyethylene bags

Heavy-duty cooler

Ice

3.0 PROCEDURES

3.1 The following information will be electronically printed on each sample label prior to mobilizing for field activities. Additional “generic” labels will also be printed prior to mobilization to be used for field QC and backups.

- Project number (CTO F272)
- Sample location ID
- Contract Task Order number
- Sample ID
- Matrix

- Preservative
 - Analysis to be performed
 - Laboratory name
- 3.2 Select the container(s) that are appropriate for a given sample. Select the sample-specific ID label(s), complete date, time, and sampler name, and affix to the sample container(s).
- 3.3 Fill the appropriate containers with sample material. Securely close the container lids without overtightening.
- 3.4 Place the sample container in a bubble wrap sleeve and/or sealable polyethylene bag and place in a cooler containing ice.

Example of a sample label is attached at the end of this SOP.

4.0 ATTACHMENTS

1. Sample Label

ATTACHMENT 1 SAMPLE LABEL

Tetra Tech NUS, Inc. 661 Andersen Drive Pittsburgh, 15220 (412)921-7090		Project:
		Location:
		CTO:
Sample No:		Matrix:
Date:	Time:	Preserve:
Analysis:		
Sampled by:		Laboratory

STANDARD OPERATING PROCEDURE

SOP-02

SAMPLE IDENTIFICATION NOMENCLATURE

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to establish a consistent sample nomenclature system that will facilitate subsequent data management at the Naval Support Activity (NSA) Crane. The sample nomenclature system has been devised such that the following objectives can be attained.

- Sorting of data by site, location, or matrix
- Maintenance of consistency (field, laboratory, and database sample numbers)
- Accommodation of all project-specific requirements
- Accommodation of laboratory sample number length constraints
- Ease of sample identification

The NSA Crane Environmental Protection Department must approve any deviations from this procedure.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Pen with indelible ink

Sample tags

Sample container labels

3.0 SAMPLE IDENTIFICATION NOMENCLATURE

3.1 Confirmation Samples

All confirmation samples will be properly labeled with a sample label affixed to the sample container. Each sample will be assigned a unique sample tracking number.

3.1.1 Confirmation Sample numbering Scheme

The sample tracking number will consist of a four- or five-segment alpha-numeric code that identifies the sample's associated Unexploded Ordnance (UXO) site or Area of Concern (AOC), sample type, location, and sample depth. For soil samples, the final four tracking numbers will identify the depth in units of feet below ground surface (bgs) at which the sample was collected (rounded to the nearest foot). For sediment samples, the final four tracking numbers will identify the depth in units of inches bgs at which the sample was collected.

The alphanumeric coding to be used is explained in the following diagram and subsequent definitions:

AN	AA	NNNA	NNNN (Soils and Sediment only)
UXO or AOC Number	Matrix	Sample Location Number and Grab or Composite	Sequential depth interval from freshly exposed surface

Character Type:

A = Alpha
 N = Numeric

UXO Number (AN):

X7 = UXO 7

Matrix Code (AA):

SS = Surface Soil Sample
 SB = Subsurface Soil Sample

Location Number (NNNA):

Sequential number for each matrix, followed by a letter indicating grab (G) or composite (C) sample, if appropriate.

Depth Interval (NNNN):

This code section will be used for soil and sediment samples only. For soil samples, the final four tracking numbers will identify the depth in units of feet. Surface soil samples will be collected from 0- to 2-feet bgs. Subsurface soil samples will be collected at depths greater than 2-feet bgs. For sediment samples, the

final four tracking numbers will identify the depth in units of inches. Sediment samples will be collected from 0- to 6-inches below the sediment/water interface.

The depth code is used to note the depth bgs at which a soil or sediment sample is collected. The first two numbers of the four-number code specify the top interval, and the third and fourth specify the bottom interval of the sample depth. The depths will be noted in whole numbers only; further detail, if needed, will be recorded on the sample log sheet, boring log, logbook, etc. (If composite samples are collected: "location" refers to a particular sampling grid represented by a composite sample.

3.1.2 Examples of Confirmation Sample Nomenclature

A surface soil sample collected at UXO 7, sampling location 025, at a depth of 1-foot bgs would be labeled as "X7-SS025-0001".

3.3 Field Quality Assurance/Quality Control (QA/QC) Sample Nomenclature

Field QA/QC samples are described in the UXO 7 Technical Memorandum. They will be designated using a different coding system than the one used for regular field samples.

3.3.1 QC Sample Numbering

The QC code will consist of a four-segment alpha-numeric code that identifies the sample QC type, the date the sample was collected, and the number of this type of QC sample collected on that date.

AN	AA	NNNNNN	NN
UXO of AOC Number	QC Type	Date	Sequence Number (per day)

The QC types are identified as:

RB = Rinsate Blank

FD = Field Duplicate

The sampling time recorded on the Chain-of-Custody Form, labels, and tags for duplicate samples will be "0000" so that the samples are "blind" to the laboratory. Notes detailing the sample number, time, date, and type will be recorded on the sample log sheets and will document the location of the duplicate sample (sample log sheets are not provided to the laboratory).

3.3.2 Examples of Field QA/QC Sample Nomenclature

The first duplicate of the day at UXO 7 for a surface soil sample collected on October 26, 2011 would be designated as FD102611-01.

The third duplicate of the day taken at UXO 7 of a surface soil sample collected on September 3, 2011 would be designated as FD090311-03.

The first rinsate blank associated with samples collected on September 3, 2011 would be designated as RB090311-01.

STANDARD OPERATING PROCEDURE

SOP-03

SAMPLE CUSTODY AND DOCUMENTATION OF FIELD ACTIVITIES

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures for sample custody and documentation of field sampling and field analyses activities.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

The following logbooks, forms, labels, and equipment are required.

Writing utensil (preferably black pen with indelible ink)

Site logbook

Field logbook

Sample label

Chain-of-Custody Form

Custody seals

Equipment calibration log

Soil and Sediment Sample Log Sheet

3.0 PROCEDURES

This section describes custody and documentation procedures. All entries made into the logbooks, custody documents, logs, and log sheets described in this SOP must be made in indelible ink (black is preferred). No erasures are permitted. If an incorrect entry is made, the entry will be crossed out with a single strike mark, initialed, and dated.

3.1 Site Logbook

The site logbook is a hard-bound, paginated, controlled-distribution record book in which all major on-site activities are documented. At a minimum, the following activities and events will be recorded (daily) in the site logbook:

- All field personnel present
- Arrival/departure of site visitors
- Arrival/departure of equipment
- Start or completion of sampling activities
- Daily on-site activities performed each day
- Sample pickup information
- Health and safety issues
- Weather conditions

The site logbook is initiated at the start of the first on-site activity (e.g., site visit or initial reconnaissance survey). Entries are to be made for every day that on-site activities take place.

The following information must be recorded on the cover of each site logbook:

- Project name
- Project number
- Book number
- Start date
- End date

Information recorded daily in the site logbook need not be duplicated in other field notebooks but must summarize the contents of these other notebooks and refer to specific page locations in these notebooks for detailed information (where applicable). At the completion of each day's entries, the site logbook must be signed and dated by the Field Operations Leader (FOL).

3.2 Field Logbooks

The field logbook is a separate dedicated notebook used by field personnel to document his or her activities in the field. This notebook is typically hardbound and paginated.

3.3 Sample Labels

Adhesive sample container labels must be completed and applied to every sample container. Information on the label includes the project name, location, sample number, date, time, preservative, analysis, matrix, sampler's initials, and the name of the laboratory performing the analysis.

3.4 Chain-of-Custody Form

The Chain-of-Custody Form (COC) is a form that is initiated as samples are acquired and accompanies a sample (or group of samples) as it is transferred from person to person. Each COC is numbered. This form must accompany any samples collected for laboratory chemical analysis. A copy of a blank COC form is attached at the end of this SOP.

The FOL must include the name of the laboratory in the upper right hand corner section to ensure that the samples are forwarded to the correct location. If more than one COC is necessary for any cooler, the FOL will indicate "Page ___ of ___" on each COC. The original signed copy of the COC will be placed inside a sealable polyethylene bag and taped inside the lid of the shipping cooler. Once the samples are received at the laboratory, the sample custodian checks the contents of the cooler(s) against the enclosed COC(s). Any problems are noted on the enclosed COC Form (bottle breakage, discrepancies between the sample labels, COC form, etc.) and will be resolved through communication between the laboratory point-of-contact and the Project Manager (PM). The COC form is signed and retained by the laboratory and becomes part of the sample's corresponding analytical data package.

3.5 Custody Seal

The custody seal is an adhesive-backed label, and it is part of the chain-of-custody process and is used to prevent tampering with samples after they have been collected in the field and sealed in coolers for transit to the laboratory. The custody seals are signed and dated by the samplers and affixed across the opening edges of each cooler (two seals per cooler) containing environmental samples. The laboratory sample custodian will examine the custody seal for evidence of tampering and will notify the TtNUS PM if evidence of tampering is observed.

3.6 Equipment Calibration Log

The Equipment Calibration Log is used to document calibration of measuring equipment used in the field, if applicable. The Equipment Calibration Log documents that the manufacturer's instructions were followed for calibration of the equipment, including frequency and type of standard or calibration device. An Equipment Calibration Log must be maintained for each electronic measuring device requiring calibration. Entries must be made for each day the equipment is used.

3.7 Sample Log Sheets

The Soil and Sediment Sample Log Sheets are used to document the sampling of soil and sediment (see SOP-05).

4.0 ATTACHMENTS

1. Chain-of-Custody Record
2. Equipment Calibration Log
3. Soil and Sediment Sample Log

**ATTACHMENT 3
 SOIL AND SEDIMENT SAMPLE LOG SHEET**

SOIL & SEDIMENT SAMPLE LOG SHEET

Page ___ of ___

Project Site Name: _____		Sample ID No.: _____		
Project No.: _____		Sample Location: _____		
<input type="checkbox"/> Surface Soil <input type="checkbox"/> Subsurface Soil <input type="checkbox"/> Sediment <input type="checkbox"/> Other: _____ <input type="checkbox"/> QA Sample Type: _____		Sampled By: _____ C.O.C. No.: _____ Type of Sample: <input type="checkbox"/> Low Concentration <input type="checkbox"/> High Concentration		
GRAB SAMPLE DATA:				
Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)	
Time:				
Method:				
Monitor Reading (ppm):				
COMPOSITE SAMPLE DATA:				
Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				
OBSERVATIONS / NOTES:			MAP:	
Circle if Applicable:			Signature(s):	
MS/MSD	Duplicate ID No.:			

STANDARD OPERATING PROCEDURE

SOP-04

SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for sample preservation, packaging, and shipping to be used in handling soil and aqueous samples.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Shipping labels

Custody seals

Chain-of-custody (COC) form(s)

Sample containers with preservatives: All sample containers for analysis by fixed-base laboratories will be supplied, with preservatives added (if required) and deemed certified clean by the laboratory.

Sample shipping containers (coolers): All sample shipping containers are supplied by the laboratory.

Packaging material: Bubble wrap, sealable polyethylene bags, strapping tape, etc.

3.0 PROCEDURES FOR SAMPLE PRESERVATION, PACKAGING, AND SHIPPING

- 3.1 The laboratory provides sample containers with preservative already included (as required) for the analytical parameter for which the sample is to be analyzed. All samples will be held, stored, and shipped at or below 6°C, but above freezing. This will be accomplished through refrigeration (used to hold samples prior to shipment) and/or ice.
- 3.2 The sampler shall maintain custody of the samples until the samples are relinquished to another custodian or to the common carrier.
- 3.3 Check that each sample container is properly labeled, the container lid is securely fastened, and the container is sealed in a polyethylene bag.
- 3.4 If the container is glass, place the sample container into a bubble-out shipping bag and seal the bag using the self-sealing, pressure sensitive tape supplied with the bag.

- 3.5 Inspect the insulated shipping cooler. Check for any cracks, holes, broken handles, etc. If the cooler has a drain plug, make certain it is sealed shut, both inside and outside of the cooler. If the cooler is questionable for shipping, the cooler must be discarded.
- 3.6 Line the cooler with large plastic bag, and line the bottom of the cooler with a layer of bubble wrap. Place the sample containers into the shipping cooler in an upright position (containers will be upright, with the exception of any 40-ml vials). Continue filling the cooler with ice until the cooler is nearly full and the movement of the sample containers is limited.
- 3.7 Wrap the large plastic bag closed and secure with tape.
- 3.8 Place the signed copy of the COC form inside a sealable polyethylene bag. Tape the bag to the inside of the lid of the shipping cooler.
- 3.9 Close the cooler and seal the cooler with approximately four wraps of strapping tape at each end of the cooler. Prior to wrapping the last wrap of strapping tape, apply a signed and dated custody seal to each side of the cooler (one per side). Cover the custody seal with the last wrap of tape. This will provide a tamper evident custody seal system for the sample shipment.
- 3.10 Affix shipping labels to each of the coolers, ensuring all of the shipping information is filled in properly. Overnight (e.g., FedEx Priority Overnight) courier services will be used for all sample shipments.
- 3.11 All samples will be shipped to the laboratory no more than 72 hours after collection. Under no circumstances should sample hold times be exceeded.

STANDARD OPERATING PROCEDURE

SOP-05

SOIL CORING AND SAMPLING USING HAND AUGER TECHNIQUES

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the procedures for collecting surface and subsurface soil cores from unconsolidated overburden materials using hand augering techniques.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Disposable medical-grade gloves (e.g., latex, nitrile)

Writing utensil (preferably black pen with indelible ink)

Indelible marker

Stainless Steel Auger Buckets

Stainless Steel Extension Rods

Cross Handle

Required decontamination materials

Bentonite pellets

Sealable polyethylene bags

Sample labels

Shipping containers (containing ice)

Disposable plastic trowels or stainless steel trowels

Stainless steel mixing bowls

Sample containers: Sample containers are certified clean by the laboratory supplying the containers.

Soil Sample Log Forms

Daily Activity Logs

Chain-of-Custody Form

3.0 SOIL SAMPLING USING A HAND AUGER

Hand Augers may be employed to collect the soil cores. A hand augering system generally consists of a variety of all stainless steel bucket bits (i.e. cylinders 6-1/2" long and approximately 2-3/4", 3-1/4", or 4" in diameter), a series of extension rods (available in various lengths), a cross handle.

- 3.1 The hand auger can be used in a wide variety of soil conditions. It can be used to sample soil, both from the surface, or to depths in excess of 12 feet. However, the presence of rock layers and the collapse of the borehole normally contribute to its limiting factors.

Attach a properly decontaminated bucket bit into a clean extension rod and further attach the cross handle to the extension rod.

- 3.2 Clear the area to be sampled of any surface debris (vegetation, twigs, rocks, litter, etc.)
- 3.3 Turn the hand auger sampler into the ground to a depth of 6-inches. The 0- to 6-inch depth soil interval is considered to be the surface soil.
- 3.4 After reaching the desired depth, slowly and carefully withdraw the apparatus from the borehole.
- 3.4 Utilizing a properly decontaminated stainless steel trowel or disposable trowel, remove the sample material from the bucket bit and place into a sealable polyethylene bag. Note in a field notebook or on a standardized data sheet any changes in the color, texture or odor of the soil.
- 3.5 Thoroughly homogenize the sample material and write sample ID, date, and time on the bag with an indelible marker.
- 3.6 Complete required information on the Soil Sample Log Sheet (copy attached at the end of this SOP). Update the Chain-of-Custody (COC) Form.
- 3.7 Excess soil core materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole, and hydrated with potable water.
- 3.8 Decontaminate all soil sampling equipment in accordance with SOP-08 before collecting the next sample.
- 3.9 Soil samples shipped to a fixed-base laboratory for analysis will be in sample containers supplied by the laboratory. The sample labels will be completed and affixed to the sample container. The samples will then be packaged and shipped to the fixed-base laboratory in accordance with SOP-04.

4.0 ATTACHMENTS

1. Soil and Sediment Sample Log Sheet

ATTACHMENT 1
SOIL AND SEDIMENT SAMPLE LOG SHEET

SOIL & SEDIMENT SAMPLE LOG SHEET

Page ___ of ___

Project Site Name: _____		Sample ID No.: _____		
Project No.: _____		Sample Location: _____		
<input type="checkbox"/> Surface Soil <input type="checkbox"/> Subsurface Soil <input type="checkbox"/> Sediment <input type="checkbox"/> Other: _____ <input type="checkbox"/> QA Sample Type: _____		Sampled By: _____ C.O.C. No.: _____ Type of Sample: <input type="checkbox"/> Low Concentration <input type="checkbox"/> High Concentration		
GRAB SAMPLE DATA:				
Date:	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)	
Time:				
Method:				
Monitor Reading (ppm):				
COMPOSITE SAMPLE DATA:				
Date:	Time	Depth Interval	Color	Description (Sand, Silt, Clay, Moisture, etc.)
Method:				
Monitor Readings (Range in ppm):				
OBSERVATIONS / NOTES:			MAP:	
Circle if Applicable:			Signature(s):	
MS/MSD	Duplicate ID No.:			

STANDARD OPERATING PROCEDURE SOP-06

SOIL SAMPLE LOGGING

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes the standard procedures and technical guidance on the logging of soil samples.

2.0 FIELD FORMS AND EQUIPMENT

Knife

Ruler (marked in tenths and hundredths of feet)

Boring Log: An example of this form is attached.

Writing utensil (preferably black pen with indelible ink)

3.0 RESPONSIBILITIES

A field geologist or engineer is responsible for supervising all activities and assuring that each soil sample is properly and completely logged.

4.0 PROCEDURES FOR SAMPLE LOGGING

To maintain a consistent classification of soil, it is imperative that the field geologist understands and accurately uses the field classification system described in this SOP. This identification is based on visual examination and manual tests.

4.1 USCS Classification

Soils are to be classified according to the Unified Soil Classification System (USCS). This method of classification is detailed in Figure 1 (attached to this SOP).

This method of classification identifies soil types on the basis of grain size and cohesiveness.

Fine-grained soils, or fines, are smaller than the No. 200 sieve and are of two types: silt (M) and clay (C). Some classification systems define size ranges for these soil particles, but for field classification purposes, they are identified by their respective behaviors. Organic material (O) is a common component of soil but has no distinguishable size range; it is recognized by its composition. The careful study of the USCS will aid in developing the competence and consistency necessary for the classification of soils.

Coarse-grained soils will be divided into categories: rock fragments, sand, or gravel. The terms "sand" and "gravel" not only refer to the size of the soil particles but also to their depositional history. To insure accuracy in description, the term "rock fragments" will be used to indicate angular granular materials resulting from the breakup of rock. The sharp edges that are typically observed indicate little or no transport from their source area; and therefore, the term provides additional information in reconstructing the depositional environment of the soils encountered. When the term "rock fragments" is used, it will be followed by a size designation such as "(1/4 inch-1/2 inch)" or "coarse-sand size" either immediately after the entry or in the remarks column. The USCS classification would not be affected by this variation in terms.

4.2 Color

Soil colors will be described utilizing a single color descriptor preceded, when necessary, by a modifier to denote variations in shade or color mixtures. A soil could therefore be referred to as "gray" or "light gray" or "blue-gray." Because color can be utilized in correlating units between sampling locations, it is important for color descriptions to be consistent from one boring to another.

Colors must be described while the sample is still moist. Soil samples will be broken or split vertically to describe colors. Samplers tend to smear the sample surface, creating color variations between the sample interior and exterior.

The term "mottled" will be used to indicate soils irregularly marked with spots of different colors. Mottling in soils usually indicates poor aeration and lack of good drainage.

4.3 Relative Density and Consistency

To classify the relative density and/or consistency of a soil, the geologist is to first identify the soil type. Granular soils contain predominantly sands and gravels. They are non-cohesive (particles do not adhere well when compressed). Finer-grained soils (silts and clays) are cohesive (particles will adhere together when compressed).

Granular soils are given the USCS classifications GW, GP, GM, SW, SP, SM, GC, or SC (see Figure 1).

The consistency of cohesive soils is determined by performing field tests and identifying the consistency as shown in the following table.

CONSISTENCY FOR COHESIVE SOILS

Consistency	Standard Penetration Resistance (Blows per Foot)	Unconfined Compressive Strength (Tons/Sq. Foot by pocket penetration)	Field Identification
Very soft	0 to 2	Less than 0.25	Easily penetrated several inches by fist.
Soft	2 to 4	0.25 to 0.50	Easily penetrated several inches by thumb.
Medium stiff	4 to 8	0.50 to 1.0	Can be penetrated several inches by thumb with moderate effort.
Stiff	8 to 15	1.0 to 2.0	Readily indented by thumb but penetrated only with great effort.
Very stiff	15 to 30	2.0 to 4.0	Readily indented by thumbnail.
Hard	Over 30	More than 4.0	Indented with difficulty by thumbnail.

Cohesive soils are given the USCS classifications ML, MH, CL, CH, OL, or OH (see Figure 1).

The consistency of cohesive soils is determined by hand by determining the resistance to penetration by the thumb. The thumb determination methods are conducted on a selected sample of the soil, preferably the lowest 0.5 foot of the sample. The sample will be broken in half and the thumb pushed into the end of the sample to determine the consistency. Do not determine consistency by attempting to penetrate a rock fragment. If the sample is decomposed rock, it is classified as a soft decomposed rock rather than a hard soil. One of the other methods will be used in conjunction with it. The designations used to describe the consistency of cohesive soils are shown in the above-listed table.

4.4 Weight Percentages

In nature, soils are consist of particles of varying size and shape and are combinations of the various grain types. The following terms are useful in the description of soil:

Terms of Identifying Proportion of the Component	Defining Range of Percentages by Weight
Trace	0 - 10 percent
Some	11 - 30 percent
Adjective form of the soil type (e.g., sandy)	31 - 50 percent

Examples:

- Silty fine sand: 50 to 69 percent fine sand, 31 to 50 percent silt.
- Medium to coarse sand, some silt: 70 to 80 percent medium to coarse sand, 11 to 30 percent silt.
- Fine sandy silt, trace clay: 50 to 68 percent silt, 31 to 49 percent fine sand, 1 to 10 percent clay.
- Clayey silt, some coarse sand: 70 to 89 percent clayey silt, 11 to 30 percent coarse sand.

4.5 Moisture

Moisture content is estimated in the field according to four categories: dry, moist, wet, and saturated. In dry soil, there appears to be little or no water. Saturated samples obviously have all the water they can hold. Moist and wet classifications are somewhat subjective and often are determined by the individual's judgment. A suggested parameter for this would be calling a soil wet if rolling it in the gloved hand or on a porous surface liberates water (i.e., dirties or muddies the surface). Whatever method is adopted for describing moisture, it is important that the method used by an individual remains consistent throughout an entire field activity.

4.6 Classification of Soil Grain Size for Chemical Analysis

To determine the gross grain size classification (e.g., clay, silt, and sand) from the USCS classification described above, the following table will be used.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
Clay	CL	inorganic clays of low to medium plasticity, gravelly clays, sandy clays, silty clays, lean clays.
	CH	inorganic clays of high plasticity, fat clays.
	OH	organic clays of medium to high plasticity, organic silts.
Silt	ML	inorganic silts and very fine sands, rock four, silty or clayey fine sands with slight plasticity.
	OL	organic silts and organic silty clays of low plasticity.
	MH	inorganic silts, micaceous or diatomaceous fine sand or silty soils.
Sand	SW	well graded sands, gravelly sands, little or no fines.

Gross Soil Grain Size Classification	USCS Abbreviation	Description
	SP	poorly graded sands, gravelly sands, little or no fines.
	SM	silty sands, sand-silt mixtures.
	SC	clayey sands, sand-clay mixtures.

4.7 Summary of Soil Classification

In summary, soils will be classified in a similar manner by each geologist/engineer at a project site. The hierarchy of classification is as follows:

- Density and/or consistency
- Color
- Plasticity (optional)
- Soil types
- Moisture content
- Other distinguishing features
- Grain size
- Depositional environment

5.0 ATTACHMENTS

1. Figure 1 - Unified Soil Classification System
2. Boring Log

ATTACHMENT 1
 FIGURE 1 - UNIFIED SOIL CLASSIFICATION SYSTEM

Unified Soil Classification System			
Coarse Grained Soils (more than half of soil > No. 200 sieve)	Gravels (More than half of coarse fraction > no. 4 sieve size)		GW Well graded gravels or gravel-sand mixtures, little or no fines
			GP Poorly graded gravels or gravel-sand mixtures, little or no fines
			GM Sandy gravels, gravel-sand-silt mixtures
			GC Clayey gravels, gravel-sand-silt mixtures
	Sands (More than half of coarse fraction < no. 4 sieve size)		SW Well graded sands or gravelly sands, little or no fines
			SP Poorly graded sands or gravelly sands, little or no fines
		SM Silty sands, sand-silt mixtures	
		SC Inorganic silts and very fine sands, rock flour, silty or clayey fine sands or clayey silts with slight plasticity	
Fine Grained Soils (more than half of soil < No. 200 sieve)	Sils and Clays LL = < 50		ML Inorganic silts and very fine sands, rock flour, silty fine sands or clayey silts with slight plasticity
			CL Inorganic clays of low to medium plasticity, gravelly clays, sandy clays, lean clays
			OL Organic silts and organic silty clays of low plasticity
	Sils and Clays LL = > 50		MH Inorganic silts, micaceous or diatomaceous fine sand or silty soils, elastic silts
			CH Inorganic silts of high plasticity, fat clays
Highly Organic Soils		OH Organic clays of high plasticity, organic silty clays, organic silts	
		Pt Peat and other highly organic soils	

Grain Size Chart

Classification	Range of Grain Sizes	
	U.S. Standard Sieve Size	Grain Size In Millimeters
Boulders	Above 12"	Above 305
Cobbles	12" to 3"	305 to 76.2
Gravel	3" to No. 4	76.2 to 7.76
	coarse 3" to 3/4"	76.2 to 4.76
fine 3/4" to No. 4	19.1 to 4.76	
Sand	No. 4 to No. 200	4.76 to 0.074
	coarse No. 4 to No. 10	4.76 to 2.00
	medium No. 10 to No. 40	2.00 to 0.420
fine No. 40 to No. 200	0.420 to 0.074	
Silt and Clay	Below No. 200	Below 0.074

Relative Density (SPT)

SANDS AND GRAVELS	BLOWS/FOOT
VERY LOOSE	0 - 4
LOOSE	4 - 10
MEDIUM DENSE	10 - 30
DENSE	32 - 50
VERY DENSE	OVER 50

Consistency (SPT)

SILTS AND CLAYS	BLOWS/FOOT
VERY SOFT	0 - 2
SOFT	2 - 4
MEDIUM STIFF	4 - 8
STIFF	8 - 16
VERY STIFF	16 - 22
HARD	OVER 32

STANDARD OPERATING PROCEDURE

SOP-07

DECONTAMINATION OF FIELD SAMPLING EQUIPMENT

1.0 PURPOSE

This Standard Operating Procedure (SOP) establishes the procedures to be followed when decontaminating non-dedicated field sampling equipment during the field investigations.

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Writing utensil (preferably black pen with indelible ink)

Non-latex rubber or plastic gloves

Cotton gloves

Field logbook

Potable water

Deionized water

Isopropanol (optional)

LiquiNox detergent

Brushes, spray bottles, paper towels, etc.

Container to collect and transport decontamination fluids

3.0 DECONTAMINATION PROCEDURES

3.1 Don non-latex and/or cotton gloves and decontaminate sampling equipment (in accordance with the following steps) prior to field sampling and between samples.

3.2 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.

3.3 Wash the equipment with a solution of LiquiNox detergent. Prepare the LiquiNox wash solution in accordance with the instructions on the LiquiNox container. Collect the LiquiNox wash solution into a container. Use brushes or sprays as appropriate for the equipment. If oily residue has accumulated on the sampling equipment, remove the residue with an isopropanol wash and repeat the LiquiNox wash.

- 3.4 Rinse the equipment with potable water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the potable water rinsate into a container.
- 3.5 Rinse the equipment with deionized water. Rinsing may be conducted by spraying with water from a spray bottle or by dipping. Collect the deionized water rinsate into a container.
- 3.6 Remove excess water by air drying, shaking, or by wiping with paper towels as necessary.
- 3.7 Document decontamination by recording it in the field logbook.
- 3.8 Containerized decontamination solutions will be managed in accordance with the procedures described in SOP-08.

STANDARD OPERATING PROCEDURE

SOP-08

MANAGEMENT OF INVESTIGATION-DERIVED WASTE

1.0 PURPOSE

This Standard Operating Procedure (SOP) describes how investigation-derived waste (IDW) will be collected, segregated, classified, and managed during the field investigations at NSA Crane. The following types of IDW will be generated during this investigation:

- Soil sampling residues
- Decontamination solutions
- Personal protective equipment and clothing (PPE)
- Miscellaneous trash and incidental items

2.0 REQUIRED FIELD FORMS AND EQUIPMENT

Health and safety equipment (with PPE)

Hand augers, plastic or stainless steel trowels

Bucket (with collected decon water)

Decontamination equipment

Field logbook

Writing utensil (preferably black pen with indelible ink)

Plastic sheeting and/or tarps

Plastic garbage bags

3.0 PROCEDURES

Management of IDW includes the collection, segregation, temporary storage, classification, final disposal, and documentation of the waste-handling activities if necessary.

3.1 Liquid Wastes

Liquid wastes that will be generated during the site activities include decontamination solutions from sampling equipment. These wastes will be collected and containerized in a central location at NSA Crane for proper disposal.

3.2 Solid Wastes

Solid wastes that may be generated during site activities include soil and sediment sampling residues. Excess soil core/sampling materials will be returned to the hole and tamped. If insufficient soil is available to fill the hole to the ground surface, then bentonite pellets mixed with the soil will be used to backfill the hole, and hydrated with potable water. Excess sediment sampling materials will be returned to the point of collection. The disposition of this materials will be carried out in a manner such as not to contribute further environmental degradation or pose a threat to public health or safety.

3.3 PPE and Incidental Trash

All PPE wastes and incidental trash materials (e.g., wrapping or packing materials from supply cartons, waste paper) will be decontaminated (if contaminated), double bagged, securely tied shut, and placed in a designated waste receptacle at NSA Crane.

STANDARD OPERATING PROCEDURE

SOP-09

GLOBAL POSITIONING SYSTEM

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide the Field Technicians with basic instructions for operating a handheld Global Positioning System (GPS) unit allowing them to set GPS parameters in the receiver, record GPS positions on the field device, and update existing Geographic Information System (GIS) data. This SOP is specific to GIS quality data collection for Trimble-specific hardware and software.

If possible, the Trimble GeoXM or GeoXH Operators Manual should be downloaded onto the operator's personal computer for reference before or while in the field. The manual can be downloaded at <http://trl.trimble.com/docushare/dsweb/Get/Document-311749/TerraSyncReferenceManual.pdf>

Unless the operator is proficient in the setup and operation of the GPS unit, the Project Manager (or designee) should have the GPS unit shipped to the project-specific contact listed below in the Pittsburgh, Pennsylvania office at least five working days prior to field mobilization so project-specific shape files, data points, background images, and correct coordinate systems can be uploaded into the unit.

Tetra Tech NUS, Inc.
Attn: Kevin Moore
661 Anderson Drive, Bldg #7
Pittsburgh, PA 15220

2.0 REQUIRED EQUIPMENT

The following hardware and software should be utilized for locating and establishing GPS points in the field:

2.1 Required GPS Hardware

- Hand-held GPS Unit capable of sub-meter accuracy (i.e. Trimble GeoXM or Trimble GeoXH). This includes the docking cradle, a/c adapter, stylus, and USB cable for data transfer.

Optional Accessories:

- External antenna
 - Range pole
 - Hardware clamp (for mounting Geo to range pole)
 - GeoBeacon
- Indelible marker
 - Non-metallic pin flags for temporary marking of positions

2.2 Required GPS Software

The following software is required to transfer data from the handheld GPS unit to a personal computer:

- Trimble TerraSync version 2.6 or later (pre-loaded onto GPS unit from vendor)
- Microsoft ActiveSync version 4.2 or later. Download to personal computer from:
http://www.microsoft.com/windowsmobile/en-us/downloads/eulas/eula_activesync45_1033.mspx?ProductID=76
- Trimble Data Transfer Utility (freeware version 2.1 or later). Download to personal computer from:
<http://www.trimble.com/datatransfer.shtml>

3.0 START-UP PROCEDURES

Prior to utilizing the GPS in the field, ensure the unit is fully charged. The unit may come charged from the vendor, but an overnight charge is recommended prior to fieldwork.

The Geo-series GPS units require a docking cradle for both charging and data transfer. The Geo-series GPS unit is docked in the cradle by first inserting the far domed end in the top of the cradled, then gently seating the contact end into the latch. The power charger is then connected to the cradle at the back end using the twist-lock connector. Attach a USB cable as needed between the cradle (B end) and the laptop/PC (A end).

It is recommended that the user also be familiar and check various Windows Mobile settings. One critical setting is the Power Options. The backlight should be set as needed to conserve power when not in use.

Start Up:

- 1) Power on the GPS unit by pushing the small green button located on the lower right front of the unit.
- 2) Utilizing the stylus that came with the GPS unit, launch **TerraSync** from the Windows Operating System by tapping on the start icon located in the upper left hand corner of the screen and then tap on **TerraSync** from the drop-down list.
- 3) If the unit does not default to the Setup screen, tap the Main Menu (uppermost left tab, just below the Windows icon) and select Setup.
- 4) If the unit was previously shipped to the Pittsburgh office for setup, you can skip directly to Section 4.0. However, to confirm or change settings, continue on to Section 3.1.

3.1 Confirm Setup Settings

Use the Setup section to confirm the TerraSync software settings. To open the Setup section, tap the Main Menu and select Setup.

- 1) Coordinate System
 - a. Tap on the Coordinate System.
 - b. Verify the project specs are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
Note: It is always best to utilize the Cancel tab rather than the OK tab if no changes are made since configurations are easily changed by mistake.
 - c. Tap on the Units.
 - d. Verify the user preferences are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
 - e. Tap Real-time Settings.
 - f. Verify the Real-time Settings are correct for your specific project by scrolling through the various settings. Edit as needed and then tap OK; otherwise, tap Cancel to return to Setup Menu.
 - g. The GPS unit is now configured correctly for your specific project.

4.0 ANTENNA CONNECTION

- 1) If a connection has been properly made with the internal antenna, a satellite icon along with the number of usable satellites will appear at the top of the screen next to the battery icon. If no connection is made (e.g.: no satellite icon), tap on the GPS tab to connect antenna.
- 2) At this point the GPS unit is ready to begin collecting data.

5.0 COLLECTING NEW DATA IN THE FIELD

- 1) From the Main Menu select Data.
- 2) From the Sub Menu (located below the Data tab) select New which will bring up the New Data File menu.
- 3) An auto-generated filename appears and should be edited for your specific project. If the integral keyboard does not appear, tap the small keyboard icon at the bottom of the screen.
- 4) After entering the file name, tap Create to create the new file.
- 5) Confirm antenna height if screen appears. Antenna height is the height that the GPS unit will be held from the ground surface (Typically 3 to 4 feet).
- 6) The Choose Feature screen appears.

5.1 Collecting Features

- 1) If not already open, the Collect Feature screen can be opened by tapping the Main Menu and selecting Data. The Sub Menu should default to Collect.
- 2) **Do not begin the data logging process until you are at the specific location for which you intend to log the data.**
- 3) A known reference or two should be shot at the beginning and at the end of each day in which the GPS unit is being used. This allows for greater accuracy during post-processing of the data.
- 4) Upon arriving at the specific location, tap on Point_generic as the Feature Name.
- 5) Tap Create to begin data logging.
- 6) In the Comment Box enter sample ID or location-specific information.
- 7) Data logging can be confirmed by viewing the writing pencil icon in the upper part of the screen. Also, the logging counter will begin. As a Rule of Thumb, accumulate a minimum of 20 readings on the counter, per point, as indicated by the logging counter before saving the GPS data.
- 8) Once the counter has reached a minimum number of counts (i.e. 20), tap on OK to save the data point to the GPS unit. Confirm the feature. All data points are automatically saved within the GPS unit.
- 9) Repeat steps 2 through 8, giving each data point a unique name or number.

Note: If the small satellite icon or the pencil icon is blinking, this is an indication the GPS unit is not collecting data. A possible problem may be too few satellites. While still in data collection mode, tap on Main Menu in upper left hand corner of the screen and select Status. Skyplot will display as the default showing the number of available satellites. To increase productivity (number of usable satellites) use the stylus to move the pointer on the productivity and precision line to the left. This will decrease precision, but increase productivity. The precision and productivity of the GPS unit can be adjusted as the number of usable satellites changes throughout the day. To determine if GPS is correctly recording data, see Section 5.2.

5.2 Viewing Data or Entering Additional Data Points to the Current File

- 1) To view the stored data points in the current file, tap on the Main Menu and select Map. Stored data points for that particular file will appear. Use the +/- and <-/-> icons in lower left hand corner of screen to zoom in/out and to manipulate current view.
- 2) To return to data collection, tap on the Main Menu and select Data. You are now ready to continue to collect additional data points.

5.3 Viewing Data or Entering Data Points from an Existing File

- 1) To view data points from a previous file, tap on Main Menu and select Data, then select File Manager from the Sub Menu.
- 4) Highlight the file you want to view and select Map from the Main Menu.
- 5) To add data points to this file, tap on Main Menu and select Data. Continue to collect additional data points.

6.0 NAVIGATION

This section provides instructions on navigating to saved data points in an existing file within the GPS unit.

- 1) From the Main Menu select Map.
- 2) Using the Select tool, pick the point on the map to where you want to navigate.
- 3) The location you select will have a box placed around the point.
- 4) From the Options menu, choose the Set Nav Target (aka set navigation target).
- 5) The location will now have double blue flags indicating this point is you navigation target.
- 6) From the Main Menu select Navigation.
- 7) The dial and data on this page will indicate what distance and direction you need to travel to reach the desired target.

- 8) Follow the navigation guide until you reach the point you select.
- 9) Repeat as needed for any map point by going back to Step 1.

7.0 PULLING IN A BACKGROUND FILE

This section provides instructions on pulling in a pre-loaded background file. These files are helpful in visualizing your current location.

- 1) From the Main Menu select Map, then tap on Layers, select the background file from drop down list.
- 2) Select the project-specific background file from the list of available files.
- 3) Once the selected background file appears, the operator can manipulate the screen utilizing the +/- and <-/-> functions at the bottom of the screen.
- 4) In operating mode, the operator's location will show up on the background file as a floating "x".

8.0 DATA TRANSFER

This section provides instructions on how to transfer stored data on the handheld GPS unit to a personal computer. Prior to transferring data from the GPS unit to a computer, Microsoft ActiveSync and Trimble Data Transfer Utility software must be downloaded to the computer from the links provided in Section 2.2 (Required GPS Software). If a leased computer is utilized in which the operator can not download files, see the Note at the end of Section 8.0.

- 1) See Attachment A at the end of this SOP for instructions on how to transfer data from the GPS to a personal computer.

Note: If you are unable to properly transfer data from the GPS unit to a personal computer, the unit should be shipped to the project-specific contact listed in Section 1.0 where the data will be transferred and the GPS unit then shipped back to the vendor.

9.0 SHUTTING DOWN

This section provides instruction for properly shutting down the GPS unit.

- 1) When shutting down the GPS unit for the day, first click on the "X" in the upper right hand corner.
- 2) You will be prompted to ensure you want to exit TerraSync. Select Yes.
- 3) Power off the GPS unit by pushing the small green button located on the bottom face of the unit.

- 4) Place the GPS unit in its cradle to recharge the battery overnight. Ensure the green charge light is visible on the charging cradle.

ATTACHMENT A

How to Transfer Trimble GPS Data between Data Collector and PC

original 11/21/06 (5/1/08 update)

Remember – Coordinate System, Datum, and Units are critical!!!

Trimble Data Collection Devices:

Standard rental systems include the Trimble ProXR/XRS backpack and the newer handheld GeoXT or GeoXH units. Some of the older backpack system may come with either a RECON “PDA-style” or a TSCe or TSC1 alpha-numeric style data collector.

The software on all of the above units should be Trimble TerraSync (v 2.53 or higher – current version is 3.20) and to the user should basically look and function similar. The newer units and software versions (which should always be requested when renting) include enhancements for data processing, real-time display functions, and other features.

Data Transfer:

Trimble provides a free transfer utility program to aid in the transfer of GIS and field data. The Data Transfer Utility is a standalone program that will run on a standard office PC or laptop.

To connect a field data collector such as a RECON, GeoXM, GeoXT, GeoXH, or ProXH, you must first have Microsoft ActiveSync installed to allow the PC and the data collector to talk to one another. A standard USB cable is also needed to connect the two devices.

A CD or USB drive is provided with the data collector for use in data transfer. If needed, these programs are also available without charge via the web at:

- **Trimble Data Transfer Utility** (v 1.38) program to download the RECON or GeoXH field data to your PC: <http://www.trimble.com/datatransfer.shtml>
- **ActiveSync** from Microsoft to connect the data collector to the PC. The latest version (v4.5) can be found at: <http://www.microsoft.com/windowsmobile/activesync/default.mspx>
(see page 2 for data transfer instructions)

To Transfer Data Collected in the Field:

- Install the Data Transfer and ActiveSync software installed on your PC
- Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
- ActiveSync should auto-detect the connection and recognize the data collector
- Make sure the data file desired is CLOSED in TerraSync prior to transfer
- Connect via ActiveSync as a guest (not a partnership)
- Run the Trimble Data Transfer Utility program on your PC
- Select "**GIS Datalogger on Windows CE**" or similar selection
- Hit the green connect icon to the right - the far right area should say "**Connected to**" if successful
- Select the "**Receive**" data tab (under device)
- Select "**Data**" from file types on the right
- Find the file(s) needed for data transfer. You can sort the data files by clicking on the date/time header
- Select or browse to a C-drive folder you can put this file for emailing
- When the file appears on the list, hit the "**Transfer All**"
- Go to your Outlook or other email, send a message to: kevin.moore@tetrattech.com (or GIS department)
- Attach the file(s) you downloaded from your C-drive. For each TerraSync data file created you should have a packet of multiple data files. All need to be sent as a group – make sure you attach all files (the number of files may vary – examples include: ssf, obx, obs, gix, giw, gis, gip, gic, dd, and car)

To Transfer GIS Data from PC to the Field Device (must be converted in Pathfinder Office):

- Obtain GIS file(s) desired from GIS Department and have converted to Trimble extension
- Contact Kevin Moore (kevin.moore@tetrattech.com) if needed for file conversion and upload support
- The GIS file(s) can be quickly converted if requested and sent back to the field user in the needed "Trimble xxx.imp" extension via email – then quickly downloaded from Outlook to your PC for transfer
- Install the Data Transfer and ActiveSync software installed on your PC
- Connect the RECON or GeoXH to your PC via an A/B USB cable (blade end and square end type "HP printer" style)
- ActiveSync should auto-detect the connection and recognize the data collector
- Connect via ActiveSync as a guest (not a partnership)
- Run the Trimble Data Transfer Utility program on your PC
- Select "**GIS Datalogger on Windows CE**" or similar selection
- Hit the green connect icon to the right - the far right area should say "**Connected to**" if successful
- Select the "**Send**" data tab (under device)
- Select "**Data**" from file types on the right (you can also send background files)
- Browse to the location of the data on your PC (obtain the file from Pathfinder Office or from the person who converted the data for field use)
- Select the options as appropriate for the name and location of the data file to go on the data collector (usually you can choose main memory or a data storage card)
- When the file(s) appears on the list, hit the "**Transfer All**"
- Run TerraSync on the field device and open the existing data files. Your transferred file should appear (make sure you have selected Main Memory, Default, or Storage Card as appropriate)

STANDARD OPERATING PROCEDURE

SOP-10

FIELD PORTABLE X-RAY FLUORESCENCE ANALYSIS OF SOIL AND SEDIMENT SAMPLES USING THE INNOV-X ALPHA SERIES INSTRUMENT

1.0 PURPOSE

This procedure is for the semiquantitative analysis of metallic lead particles and chemical compounds of lead in soil using a field portable x-ray fluorescence (FPXRF) spectrometer. This procedure is based on the United States Environmental Protection Agency (USEPA)-approved XRF field screening method for elemental analysis (Method 6200).

2.0 SCOPE, APPLICATION, AND LIMITATIONS

2.1 Scope of Procedure

Analysis of any other elements besides lead using FPXRF may require changes to this Standard Operating Procedure (SOP), and are therefore outside the scope of this SOP.

Although it is possible to use FPXRF to measure analytes in situ, this SOP requires removal of a soil sample from its native environment prior to analysis. By removing, drying, and homogenizing the sample prior to analysis, more precise and accurate results are obtained.

2.2 Analyst Training

Use of this method is restricted to personnel both trained and knowledgeable in the operation of the Innov-X alpha series XRF instrument or under the supervision of a trained and knowledgeable individual. Proper training for the safe operation of the instrument should be completed by the analyst prior to analysis. This training may be obtained directly from INNOV-X, an INNOV-X instrument distributor or lessor, or another trained Tetra Tech person.

3.0 ACRONYMS AND ABBREVIATIONS

FPXRF: Field portable x-ray fluorescence.

mg/kg: milligrams per kilogram.

MDL: Method detection limit.

PQL: Practical quantitation limit.

QC: Quality control.

RPD: Relative percent difference.

USGS: United States Geological Survey.

XRF: X-ray fluorescence.

4.0 RESPONSIBILITIES

Analyst/Chemist - Responsible for all aspects of sample preparation and analysis including equipment maintenance. Also responsible for maintaining chain-of-custody of samples after receipt from sampling personnel.

5.0 PROCEDURES

5.1 Safety

5.1.1 Radiation Safety

Radiation safety practices for the INNOV-X instrument can be found in the operator's manual (typically shipped with unit). Protective shielding should never be removed by the analyst or any personnel other than the manufacturer.

An additional hazard present with x-ray tubes is the danger of electric shock from the high voltage supply. The danger of electric shock is as substantial as the danger from radiation but is often overlooked because of its familiarity.

5.1.2 Protective Equipment

Analysts must wear disposable plastic gloves whenever sample aliquots are being transferred from one vessel to another. Consult the health and safety plan for other protection requirements.

5.2 Apparatus and Materials

Apparatus and materials consist of the following:

INNOV-X Alpha Series FPXRF spectrometer with data processing unit (iPAQ) pocket personnel computer):

INNOV-X Alpha Series XRF instrument manual to match the INNOV-X Alpha Series instrument.

Aluminum drying pans or aluminum foil: Sized suitably to hold as much as 50 grams of sample and fit into the drying oven.

Calibration verification check sample: A National Institute of Standards and Technology (NIST) or other Standard reference material (SRM) that contains lead in a concentration range that is compatible with the project objectives to verify the accuracy of the instrument. SRMs are shipped with the unit. Acceptable limits for SRM percent recoveries are usually provided with the SRMs. In their absence, a limit of ± 30 percent will be used as a guideline.

Instrument Blank: May be silicon dioxide, a Teflon block, a quartz block, "clean" sand, or lithium carbonate and is typically shipped with the unit.

Lead calibration check standard: Supplied by the FPXRF manufacturer.

Method blank material for performing method blank checks: May be lead-free silica sand or lithium carbonate that undergoes the same preparation procedure as the samples.

Battery charger.

Polyethylene sample cups: 31 millimeters (mm) to 40 mm in diameter with collar, or equivalent (appropriate for FPXRF instrument).

X-ray window film: Mylar™, Kapton™, Spectrolene™, polypropylene, or equivalent; 2.5 to 6.0 micrometers (μm) thick.

Sample containers: glass or plastic to store samples.

Sieves: 60-mesh Stainless steel, Nylon, or equivalent for preparing soil and sediment samples if necessary.

Trowels: for collecting soil samples.

Plastic bags: used for collection and homogenization of soil samples. May also be used as sample presentation device.

Drying oven: standard convection or toaster oven, for soil samples that require drying.

Rolling pin (optional): Wooden rolling pin for crushing dried samples.

5.3 Sample Collection, Preservation, and Handling

Samples shall be provided to the FPXRF analyst in plastic bags. The analyst is responsible for maintaining chain-of-custody of all samples until all analyses have been successfully completed. No sample preservation is necessary. All samples shall be handled in accordance with sample handling SOPs in effect for the field event.

5.4 Preventive Maintenance

Refer to the instrument manual for specific manufacturer's recommendations.

5.5 Instrument Start-Up

- 5.5.1 Ensure the pocket PC (iPAQ) is plugged into the FPXRF instrument body and install a fully charged battery into the instrument.
- 5.5.2 Press the ON/OFF button on the base of the pistol grip of the instrument. If the iPAQ does not automatically power up, press the Power button in the right corner of the iPAQ.
- 5.5.3 Tap the Microsoft icon at the upper left corner of the iPAQ.
- 5.5.4 Choose START.
- 5.5.5 Tap "Soil Mode" on the menu or choose Mode (bottom of screen) and then choose Soil Mode from the drop down menu.

5.5.6 Allow the instrument to warm up (approximately 3 minutes).

5.5.7 Release the manual trigger lock.

5.5.8 Standardize the instrument in accordance with Section 5.6.

5.6 Standardization/Calibration Check

It is not possible to start an analysis if the instrument has not been standardized. To verify proper calibration of the instrument it is necessary to periodically standardize it using the automated standardization procedure. This must be done anytime the instrument is restarted and every 4 hours of operation, although re-standardization may be done at any other time (e.g., when instrument drift is suspected).

5.6.1 Click the standardization piece (supplied with the instrument) on the front of the instrument, verifying that the solid portion of the standard completely covers the analysis window. If using a manufacturer supplied test stand, lay the standardization plate over the analysis window.

5.6.2 Select "Tap here to Standardize" or select *File* → *Standardize*. The red light on top of the instrument will blink indicating that the instrument is producing x-rays and the shutter is open. The amber light on the rear of the instrument will also be illuminated and a status bar will appear to display the progress of the standardization.

5.6.3 Upon successful standardization the message "Successful Standardization" will appear along with the instrument resolution. In this case tap "ok" to dismiss the completion message. If problems are encountered, either follow the prompts that appear and/or repeat the standardization. Contact the FOL if problems persist. Take note of any error messages that appear as they may be useful if the instrument manufacturer must be contacted. Additional assistance is also available in the manufacturer's instrument manual.

5.7 Quality Control

The quality control (QC) program includes analysis of blanks, calibration verification checks, duplicate analyses, and field duplicate samples. For all the above areas, any identified problems and corrective action must be documented in the instrument run log, analysis narrative report, and instrument

maintenance log or standards log (as applicable). Identical operating conditions will be used for each sample.

5.7.1 Laboratory Blanks

Two types of blank samples shall be analyzed for FPXRF analysis: instrument blanks and method blanks.

5.7.1.1 At the beginning of each day, at the end of each day, and after every 20th sample or when potential contamination of the instrument is suspected, analyze an instrument blank to verify that no contamination exists in the spectrometer or on the probe window.

If the lead concentration in the blank exceeds the method detection limit (MDL, see Section 5.9.3) check the probe window and other potentially contaminated instrument components for contamination. If contamination is not causing the elevated blank readings, “zero” the instrument according to manufacturer’s instructions.

5.7.1.2 After every 20th sample analyze a method blank. If the method blank lead concentration exceeds the practical quantitation limit (PQL, see Section 5.9.4), identify the cause of the elevated lead concentration and reanalyze all samples since the last acceptable method blank.

5.7.2 Calibration Verification Checks

5.7.2.1 After performing each blank check (Section 5.7.2), analyze a calibration verification check sample to check the accuracy of the instrument and to assess the stability and consistency of the analysis for the analytes of interest.

5.7.2.2 If the measured lead percent recovery (See Section 5.9.1) is less than 60 percent or greater than 135 percent, reanalyze the check sample. If the value continues to fall outside this acceptance range, the instrument should be recalibrated, or restandardized according to the manufacture instructions and the batch of samples analyzed before the unacceptable calibration verification check must be reanalyzed.

5.7.3 XRF Duplicate Samples

XRF duplicate samples are two portions of the same sample that have been prepared and homogenized together, and then split and analyzed in the same manner by the XRF analyst.

5.7.3.1 Analyze an XRF duplicate at a frequency of 1 per 20 or once per day, whichever is more frequent.

5.7.3.2 If the computed RPD (See Section 5.9.2) exceeds 50 percent reanalyze both samples. If the RPD again exceeds 50 percent RPD consider whether the high degree of imprecision is caused by sample heterogeneity or other causes. This assessment may be aided by repeating the analysis of a sample that was analyzed previously. If the observed imprecision is attributed to sample heterogeneity, increase the number of readings made per sample to try to limit the imprecision and repeat the analyses. If this does not correct the problem notify the FOL.

5.8 Sample Analysis

Note:

This section provides sample analysis instructions, assuming that appropriate instrument start-up and calibration checks have been completed. The longer the instrument count time, the lower the detection limits and the less uncertainty there is with a recorded result. Count time is user-selectable through the instrument's software. Because the XRF data will be used in a screening capacity to make preliminary decisions concerning the soil concentrations relative to 400 mg/kg, it is not necessary to obtain a high degree of accuracy or precision with the instrument. Therefore, count times should be limited to less than 180 seconds unless an usually high degree of precision is expected. To change the count time, select Options → Setup Testing and enter the same value (in seconds) to minimum and maximum count times.

Note:

Section 5.7 identifies the appropriate frequencies for conducting various QC sample analyses and the associated acceptance limits and corrective actions for potentially unsuitable conditions. The specified QC analysis frequencies are minimum frequencies. More frequent QC sample analyses are permitted, especially when diagnosing quality problems.

5.8.1 Ensure that calibration checks and blanks have been analyzed according to Sections 5.6, 5.7.1, and 5.7.2. Count times shall be at least 60 seconds but generally less than 180 seconds.

5.8.2 Acquire enough soil sample to fill an 8-ounce jar and separate from it all particles greater than the size of a pea.

5.8.3 Homogenize the remaining finer grained portion of the sample by simple mixing until it appears as uniform in texture and composition as practicable. Mixing may be done in a beaker or other suitable lead-free container. If the sample is moist and has high clay content, it may be kneaded

- in a plastic bag. Mixing shall continue for at least two minutes to ensure that the sample is well mixed. To aid mixing, the sample may be placed into a thick-walled (3 mil or thicker) gallon-sized freezer bag (e.g., ZipLoc[®]) and rolled flat with a rolling pin to break up large chunks of dirt.
- 5.8.4 Place approximately 20 to 50 grams (one U.S. nickel weighs about 5 grams) in a suitable container (e.g., aluminum drying pan) for drying.
 - 5.8.5 Dry the homogenized sample from Step 5.8.4 for approximately 20 to 30 minutes in the oven at a temperature not greater than 150°C (a setting of approximately 300°F). If the sample is not visibly dry after this initial drying time, place the sample back into the oven until the sample is dry.
 - 5.8.6 Re-homogenize the dried sample aliquot in a beaker or other suitable lead-free container to obtain a well mixed soil sample. Mixing shall continue for at least one minute.
 - 5.8.7 Place a portion (approximately 1.5 cubic inches) of the dried, homogenized sample aliquot into the instrument manufacturer's recommended sample cup (e.g., a 31.0-mm polyethylene sample cup (or equivalent) or place it in a thin-walled (1.0 mil or thinner) plastic sandwich bag (e.g., ZipLoc[®]).
 - 5.8.8 If using a disposable plastic sample cup, ensure the cup is at least three-quarters full and cover with mylar (or other) film per the manufacturer's recommendations.
 - 5.8.9 Present a portion of the sample to the instrument in Soil Mode.
 - 5.8.10 Perform a single pull of the trigger to start the count. The count time shall be the same as was used for the calibrations, calibration checks, and blank analyses. The message "Test in progress" will appear on the instrument and the red light on top of the instrument and will illuminate.
 - 5.8.11 When the predetermined count period has expired the message "Test complete" will appear on the instrument. A slight delay may also be incurred during which time the message "calculating" may appear to indicate that results are being computed.
 - 5.8.12 Record the displayed results for lead concentration in mg/kg on Figure 1.

CAUTION

Inconsistent positioning of samples in front of the probe window is a potential source of error because the x-ray signal decreases by the square of the distance from the radioactive source. This error is minimized by maintaining the same distance between the window and each sample. For the best results, the window of the probe should be in direct contact with the sample, which means that the sample surface should be flat and smooth to provide a good contact surface.

5.8.13 Rotate the sample cup approximately one-third of a turn, or if using a thin plastic baggie, read another 1/3 portion of the sample bag. Acquire another measurement by repeating Steps 5.8.10 through 5.8.12.

5.8.14 Repeat Steps 5.8.10 through 5.8.11 a third time, and calculate the average concentration.

5.8.15 Based on the degree of precision demonstrated by the three individual measurements, determine whether additional readings should be acquired on the sample. This determination shall be based on professional judgment of the FPXRF analyst and shall consider the degree of precision observed during calibration checks and previous sample analyses. The objective will be to ensure that the average reading reported for each sample is representative of the true sample concentration. If the analyst feels that non-representative readings are being obtained the analyst shall correct the analytical system or notify the FOL prior to continuing with analyses.

5.8.16 Ensure that measured results are reported to the following standards

- Results < 1000 mg/kg (or parts per million) are reported to two significant figures and results > 1000 mg/Kg are reported to three significant figures.
- All values < MDL shall be reported as the MDL and flagged with the letter “U”.
- All values > MDL and < PQL shall be reported as is and flagged with the letter “B”.

5.9 Calculations

5.9.1 Percent Recovery: The equation for determining percent recovery of calibration verification check standards and standard reference materials is:

$$\%R = \frac{\text{Experimental Concentration}}{\text{Certified or Known Concentration}} \times 100 \%$$

5.9.2 Relative Percent Difference: The equation for determining relative percent difference for laboratory and field duplicate samples is:

$$\text{RPD} = \frac{|\text{Amount in Sample 1} - \text{Amount in Sample 2}|}{0.5 (\text{Amount in Sample 1} + \text{Amount in Sample 2})} \times 100 \%$$

5.9.3 Method Detection Limit (MDL): Because the analyses governed by this SOP are semi-quantitative, the manufacturer-specified detection limit will be reported as the MDL unless the specified detection limit is less than 20 mg/kg. Care will be taken to ensure that the appropriate count time is consistent with the reported detection limit. However, no value less than 20 mg/kg will be reported as an MDL.

5.9.4 Practical Quantitation Limit (PQL): Multiply the MDL by 3 to obtain the PQL: $\text{PQL} = \text{MDL} \times 3$

6.0 REFERENCES

Innov-X Systems, Inc. Innov-X Systems X-Ray Fluorescence Spectrometers Instruction Manual. Woburn, MA. June 2002.

Stephen Shefsky, NITON Corporation. Comparing Field Portable X-Ray Fluorescence (XRF) To Laboratory Analysis of Heavy Metals In Soil. Presented at the International Symposium of Field Screening Methods for Hazardous Wastes and Toxic Chemicals. Las Vegas, Nevada. January 29-31, 1997.

USEPA (U.S. Environmental Protection Agency), Method 6200: Field Portable X-Ray Fluorescence Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment, Office of Solid Waste, Washington, D.C. January 1998.

USEPA, Region I, Northeast Waste Management Officials' Association (NEWMOA) Technology Review Committee Advisory Opinion. Innovative Technology: X-Ray Fluorescence Field Analysis. September 21, 1999.

ATTACHMENT B

ANALYTICAL STANDARD OPERATING PROCEDURES UXO 7 – SMALL ARMS RANGES NSA CRANE CRANE, INDIANA

Table of Contents

SOP-100	Metals Digestion/Preparation: Methods – USEPA SW 846 3005A, 3010A, 3030C, 3031, 3050B
SOP-105	Metals Analysis by Inductively Coupled Plasma-Atomic Emission Spectrometry (ICP-AES) Technique: Methods - 200.7, SW 846 (6010B, 6010C), Hardness Calculation (SM 19 th Edition 2340B), ILMO 4.1 (USEPA CLP)
SOP-201	GC/MS Low Level PAH's by SW-846 Method 8270C SIM
SOP-300	GC/MS Semi-Volatile BNA-Aqueous Matrix Extraction Using SW-846 Method 3510C for 8270C/625 Analysis
SOP-327	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC) Method 8330A and 8332
SOP-343	Soxhlet Extraction – BNA and Pest/PCB Using SW-846 Method 3541
QS10	Laboratory Sample Receiving, Log In and Storage Standard Operating Procedures
QS14	Analytical Laboratory Waste Disposal

Certifications:

Low-level PAH Analysis

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP100 REVISION #: 22 EFFECTIVE DATE: 20101117

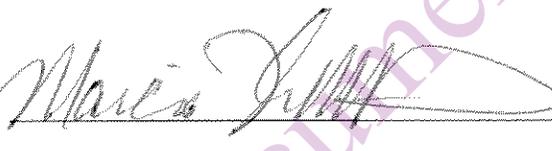
METALS DIGESTION/PREPARATION

References:

Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C 21st
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)

APPROVALS:

Lab Director:  Date: 11/19/10

Data Quality Manager:  Date: 11/19/10

Section Supervisor:  Date: 11/19/10

Changes Summary

Revision 22, 11/17/10

- The SOP is an update from Revision 21 dated 9/1/10
- Revised to add the need for matrix spike duplicates to be digested and analyzed for TCLP extracts.
- Requirement to hold samples 24 hours after in-house preservation was added to section III.

Revision 21, 9/1/10

- The SOP is an update from Revision 20 dated 04/27/10
- The SOP has been found to be up-to-date with Standard Methods 21st edition.
- Reference to adjusting filtrate volume for method 3030C has been removed.
- References to bound logbooks have been replaced with LIMS references.

Revision 20, 4/27/10

- The SOP is an update from Revision 19 dated 04/20/09.
- References to oil sample preparation have been removed.
- Extraction volumes for TCLP have been updated.

METALS DIGESTION/PREPARATION

References:

**Methods 3005A/USEPA CLPILM0 4.1 Aqueous, 3010A, 3030C, 3050B
USEPA CLPILM0 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C
See Addendum for USEPA CLPILM 05.2 (Aqueous & Soil/Sediment)**

I. SCOPE AND APPLICATION

A. AQUEOUS

1. Method 3005A and USEPA CLP ILM0 4.1, "Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by ICP Spectroscopy".
 - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
2. Method 200.7, "Determination of Metals and Trace Metals in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry"
 - a. This method is used to prepare surface water, ground water, drinking water and wastewater samples for analysis by inductively coupled argon plasma spectroscopy (ICP).
3. Method 3010A, "Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy".
 - a. This method is used to prepare aqueous samples, EP and mobility-procedure extracts, and wastes that contain suspended solids for analysis by ICP. The procedure is used to determine total metals.
4. Method 3030C (Standard methods), "Preliminary Treatment for Acid-Extractable Metals".
 - a. This method is used to prepare ground water samples from North Carolina for analysis by ICP.

B. SOLIDS

1. Method 3050B, "Acid Digestion of Sediments, Sludges and Soils".
 - a. This method is used to prepare sediments, sludges and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.
 - b. It should be noted that some metals could be biased high with the soil digestion when dilution is necessary. Take necessary measures to ensure that dilutions are made as accurately as possible.
2. USEPA CLP ILM0 4.1, "Acid Digestion of Soil/Sediment"
 - a. This method is used to prepare sediments and soil samples for analysis by ICP. Since certain matrices may result in poor recovery, the method of standard additions may be used when analyzed.

D. NOTES:

1. "Total Metals" includes all metals, inorganically and organically bound and both dissolved and particulate.
2. "Dissolved metals" includes all metals present in a sample after filtration through a 0.45 micron filter followed by digestion.

II. SUMMARY OF METHODS

A. A representative sample of water or soil is put into an acid medium and exposed to heat for a certain amount of time. This allows for reduction of interferences by organic matter and converts metals bound to particulates to form the free metal that can be determined by ICP-Atomic Emission Spectrometry.

NOTE: When a reporting limit is required for a project lower than is customary, a four times concentration or alternate soil digestion ratio must be used in order to reach that lower level. Care must be taken to matrix match this concentrated aliquot. A blank and laboratory control sample (at a reduced concentration) are required with this concentration. A matrix spike (not at reduced concentration) and duplicate or matrix spike and matrix spike duplicate is needed per 20 samples or per batch.

III. SAMPLE HANDLING AND PRESERVATION

A. AQUEOUS

1. Samples are taken in high density polyethylene, one liter bottles. Samples should be preserved with concentrated HNO₃ to a pH <2 immediately upon sampling. If dissolved metals are to be analyzed the sample should be filtered before the HNO₃ is added. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months. Note – samples received unpreserved and preserved in-house must be held 24 hours prior to preparation.

B. SOLIDS

1. Samples are taken in high density polyethylene (CLP only) or glass bottles. The samples should be maintained at 4°C until analysis. The holding time for metals samples is 180 days or approximately 6 months.

IV. INTERFERENCES

A. AQUEOUS

1. Solvents, reagents, glassware, and other sample processing hardware may 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.

B. SOLIDS

1. Sludge samples can contain diverse matrix types, each of which may present its own analytical challenge. Spiked samples and any relevant standard reference material should be processed to aid in determining whether this method is applicable to a given waste.

V. SAFETY

A. Normal accepted laboratory safety practices should be followed while performing this analysis.

B. Be certain the exhaust hood is functioning before you begin the digestion procedure.

C. Hot acids can be extremely corrosive. Avoid inhalation or contact with skin.

VI. EQUIPMENT/APPARATUS

A. Fume hood, Labconco or equivalent.

B. Hot plate, Thermolyne cimarec-3 or equivalent source for use at 95°C. The temperature of the hot plate must be monitored via the use of a temperature blank.

- C. Thermometer capable of reading 80 to 120 degrees C – ERTCO cat# 611-3-SC or equivalent.
 - D. Vacuum pump for filtering dissolved metals- Gast or equivalent.
 - E. Analytical balance capable of weighing to 0.01 gram. Mettler model BB300 or equivalent.
 - F. Beckman CS-6R centrifuge.
 - G. Various class A volumetric glassware and ribbed watchglasses, Pyrex or equivalent.
 - H. Whatman No. 41 filter paper or equivalent.
 - I. Whatman No. 42 filter paper or equivalent.
 - J. Whatman 0.45 micron filter paper or equivalent.
 - K. 250 mL beaker or other appropriate vessel such as polypropylene block digester tubes, watch glasses and caps.
 - L. Stirring device, e.g. magnetic stirrer, glass rod or equivalent.
 - M. Manual Sample Mill
 - N. Wiley Sample Mill
 - O. Clippers for cutting vegetation
- NOTE:** All glassware should be acid washed.

VII. REAGENTS AND STANDARD PREPARATION

A. REAGENTS

1. Metals grade Nitric acid (HNO₃). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
2. Metals grade Hydrochloric acid (HCl). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
3. 30% hydrogen peroxide reagent, ACS Grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
4. Metals grade Sulfuric acid (H₂SO₄). Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
5. Reagent water (Deionized water).
6. Potassium Permanganate - Ultra pure grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
7. Ammonium hydroxide, concentrated, reagent grade. Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.
8. Ammonium phosphate, reagent grade- Reagent should be analyzed to determine level of impurities. If method blank is <MDL, then the reagent can be used.

B. STANDARDS

1. Traceability

- a. A LIMS record shall be maintained on all reference materials. The record shall include date of receipt, source, purity, all compositional information, storage conditions and expiration date. These materials/solutions are to be identified by a unique number in the LIMS as well as on the container's label.
- b. All working standards made from reference materials shall be labeled with a unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the information is recorded in LIMS. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in LIMS. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded. There should be no container with

sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

c. The analyst must initial and date each entry made in LIMS.

2. PREPARATION

A. Laboratory control sample

1. Aqueous

- a. This solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO₃, 1 mL of CLP-CAL-1, Solution A, 1 mL of CLP-CAL-1 Solution B, 0.25 mL of CLP-CAL-2, and 0.25 mL of CLP-CAL-3 diluted to 1 L in a volumetric flask. Use 50 mL (100 mL for strict CLPILM0 4.1) for digestion. This solution is given a unique identifier and recorded in sample LIMS.
- b. For four times concentrated samples: The solution is prepared as follows: 50 mL concentrated HCl, 20 mL concentrated HNO₃, 1mL CLPP-SPK-4 (Inorganic Ventures) (This solution contains 10 mg/L Selenium, 100 mg/L Antimony, 50 mg/L Cadmium and Thallium, 40 mg/L Arsenic and 20 mg/L Lead) to 1 L in a volumetric flask. This solution is given a unique identifier. Use 12.5 mLs to 50 mLs and prepare two aliquots. Heat at 90 to 95°C to reduce the volume in each vessel to ten mLs and then combine each 10 mL aliquot into one vessel and take to a final volume of 25 mLs. Take care to matrix match acids so that the final 25 mL portion will contain 2% HNO₃ and 5% HCl. Use 0.125 mLs HNO₃ and 0.3125 mLs HCl to each 50 mL vessel.

2. Solids:

- a. 1.0 ±0.02 (or 2.0 ±0.02) gram aliquot of teflon chips is weighed and spiked using the same spiking solution used for matrix spikes. This sample is given a unique identifier according to the Lot# for the teflon chips used and when digested is given the descriptor. i.e. BS1 and then BS2 etc. plus the unique identifier number assigned. Alternatively a solid matrix standard reference material is obtained from the manufacturer. This sample is given a unique identifier and the weight is recorded in a bound logbook and transferred to LIMS.

B. Spiking solution

1. Sample is spiked using 0.1 mL of CLP-CAL-1, Solution A, 0.1 mL of CLP-CAL-1 Solution B, 0.025 mL of CLP-CAL-2 and 0.025 mL of CLP-CAL-3 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers. Record the amount spiked and the unique identifier of the standard.
2. CLP sample is spiked using 0.1 mL CLPP-SPK-1 and 0.1 mL CLPP-SPK-4 for a final volume of 100 mL. If only 50 mL is used, decrease amount used appropriately. These solutions are given unique identifiers.
3. For samples that require four times concentration, the sample is spiked using 0.0125 mLs of CLPP-SPK-4 to each of two vessels with 50 mLs of sample in each. The volume of each of the vessels is lowered to less than 10 mLs and combined and the final volume of this concentrated sample is 25mLs.

VIII. CALIBRATION

- A. The temperature of the samples must be maintained at 95°C and monitored via a temperature blank. Record in temperature logbook for later transfer into LIMS.

IX. PROCEDURE

- A. Glassware preparation for digestion or when the hot-block can not be used:
 1. Wash glassware with hot soapy water and rinse thoroughly. (Beakers must be washed as soon as possible after being used, dirty beakers must not be allowed to sit overnight.)
 2. Rinse glassware with reagent water that contains 5% HNO₃ and 5% HCl followed by a rinse with reagent water.
 3. Prior to use, all glassware must be confirmed clean via a glassware check. Otherwise, repeat step "2" until the glassware check passes.
- B. Aqueous sample filtration (for dissolved metals):
 1. Thoroughly clean a flask and funnel with hot soapy water. Next, rinse the flask and funnel with 1:5 HNO₃ followed by a thorough D.I. water rinsing. This step is very important because the filters contain some metals (namely Zn) which could contaminate the samples.
 2. Rinse a 0.45 micron filter with 1:5 HNO₃ thoroughly, followed by D.I. water.
 3. Filter the unpreserved sample. If dissolved Hg analysis is requested for the sample, filter at least 200 mL.
 4. Discard the first 50 to 100 mL.
 5. A preparation blank must be taken through the filtration step and analyzed with the sample.
 6. Preserve the sample with HNO₃ to pH<2.
 7. Soluble samples that are clean and clear do not have to be digested. Use 100 mL sample, add 5 mL of concentrated HCl and 2 mL of concentrated HNO₃. **Samples must be digested unless approval for analysis without digestion is received from the project manager.**
- C. Aqueous sample preparation
 1. Method 3005A and USEPA CLP ILM0 4.1, "**Acid digestion procedure for total recoverable or dissolved metals for analysis by ICP**".
 - a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a digestion vessel. For samples which require concentration pour 50 mLs of the well-mixed sample into two digestion vessels.
 - b. Add 0.50 mL (1 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO₃ to the sample. For samples which require concentration, add 0.125 mL (0.25 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HNO₃ to the sample.
 - c. Add 2.5 mL (5 mL of 1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample. For samples which require concentration, add 0.3125 mL (0.625 mL of (1+1) when strict CLP ILM0 4.1 is required) concentrated HCl to the sample.
 - d. Cover the sample with a ribbed watch glass or equivalent source.
 - e. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank will assure correct temperature. The temperature must be

- recorded in the temperature logbook. Take the volume down to between 5 to 10 mL, (12 to 25 mLs when strict CLP ILM0 4.1 is required) **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.** Remove the sample from the hot plate and cool
- f. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
 - g. Bring sample to its predigestion volume (or when samples require concentration, to a volume four times lower then what was started with) with DI water in the digestion vessel. The final volume must be recorded in the LIMS.
 - h. The sample is now ready for analysis.
 - i. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards plus identification #'s for standards used for spiking and the volume spiked into the sample.
2. Method 200.7, "**Acid digestion procedure for total recoverable metals**".
- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into the digestion vessel. If sample contains undissolved solids >1% refer to Section 11.3 of Method 200.7 for subsequent procedures.
 - b. Add 1.0 mL concentrated HNO₃ to the sample.
 - c. Add 2.50 mL concentrated HCl to the sample.
 - d. Cover the sample with a ribbed watch glass or equivalent source.
 - e. Transfer the digestion vessel to a pre-heated hot plate or equivalent source at 85°C. Take the volume down to between 10 to 15 mL, **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes.**
 - f. Leave sample on hot plate and gently reflux for 30 minutes. Remove from hot plate and cool.
 - g. Bring sample to its predigestion volume with DI water in the digestion vessel.
 - h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
 - i. The sample is now ready for analysis.
 - j. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
3. Method 3010A, "**Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by ICP Spectroscopy**".
- a. Shake sample thoroughly and pour 50 mL (5ml diluted to 50mL for TCLP, full 50ml volume for SPLP) of the well-mixed sample into the digestion vessel.
 - b. Add 1.5 mL concentrated HNO₃ to the sample.
 - c. Cover the sample with a ribbed watch glass.
 - d. Transfer the digestion vessel to a pre-heated hot plate or hot block at 90 to 95°C. A temperature blank must be used, with the temperature being recorded in the temperature logbook. Take the volume down to a low volume (~5 mL), **making certain that the sample does not boil. This is extremely important. Boiling may lead to vaporization of certain analytes. Also make certain that no portion of**

the bottom of the digestion vessel is allowed to go dry. This may lead to low recoveries. Remove the sample from the hot plate and cool.

- e. Add another 1.5 mL portion of concentrated HNO₃ to the sample.
 - f. Cover the sample with a ribbed watch glass.
 - g. Transfer the vessel to the hotblock or equivalent source. Increase the temperature so a gentle reflux occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing).
 - h. Uncover the vessel and evaporate to a low volume (~3 mL) **making certain that no portion of the bottom of the digestion vessel is allowed to go dry.** Remove and cool.
 - i. Add 2.5 ml of 1:1 HCl (10 mL/100 mL of final solution).
 - j. Cover the digestion vessel and reflux for an additional 15 minutes.
 - k. Bring sample to its predigestion volume in digestion vessel.
 - l. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
Note: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.
 - m. The sample is now ready for analysis.
 - n. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.
- 4 Method 3030C (Standard Methods), "**Preliminary treatment for Acid-Extractable Metals**"
- a. Shake sample thoroughly and pour 50 mL of the well-mixed sample into a 50 mL digestion vessel.
 - b. Add 2.5 mL 1:1 HCl to the sample.
 - c. Heat 15 minutes in a hot bath.
 - d. Filter through a membrane filter.
 - e. Transfer to ICP analyst.

D. Solid sample preparation

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering procedure should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*
- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container.

Grinding of Vegetation Samples

Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately.

1. USEPA CLP ILM0 4.1, "**Acid digestion of Soil/Sediment**"
 - a. Mix the sample thoroughly to achieve homogeneity. For each digestion procedure, weigh (to the nearest 0.01 g) a 1.0 to 1.5 g portion of sample and transfer to a digestion vessel.
 - b. Add 10 mL of 1:1 nitric acid (HNO_3), mix the slurry, and cover with a watch glass or equivalent source. Heat the sample to 92 to 95°C and reflux for 10 minutes without boiling. Allow the sample to cool, add 5.0 mL of concentrated HNO_3 , replace with watch glass or equivalent source, as appropriate, and reflux for 30 minutes. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the heating vessel.
 - c. After the second reflux step has been completed and the sample has cooled, add 2 mL of Type II water and 3.0 mL of 30% hydrogen peroxide (H_2O_2). Return the heating vessel to the hot plate or equivalent heating source for warming to start the peroxide reaction. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides, and cool the heating vessel.
 - d. Continue to add 30% H_2O_2 in 1 mL aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2 .)
 - e. If the sample is being prepared for ICP analysis of Al, As, Sb, Ba, Be, Ca, Cd, Cr, Co, Cu, Fe, Pb, Mg, Mn, Ni, K, Se, Ag, Na, Tl, V, and Zn, add 5 mL of 1:1 HCl and 10 mL of Type II water, return the covered heating vessel to the hot plate or equivalent heating source, and heat for an additional 10 minutes. After cooling, filter through Whatman No. 42 filter paper (or equivalent) and dilute to 50 mL with Type II water. NOTE: In place of filtering, the sample (after dilution and mixing) may be centrifuged or allowed to settle by gravity overnight to remove insoluble material. Dilute the digestate to 144 mL with DI water, add 5 mLs concentrated HCl and 1 mL of concentrated HNO_3 , mix well and place into the appropriate container. The diluted sample has an approximate acid concentration of 2.5% (v/v) HCl and 5% (v/v) HNO_3 . The sample is now ready for analysis.
 - f. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards and ID of matrix spikes and the amounts used for spiking.
2. Method 3050B, "**Acid digestion of Sediments, Sludges and Soils**"

- a. Mix the sample thoroughly for 5 minutes using a plastic spatula or Teflon coated spatula in a glass or plastic weigh boat to achieve homogeneity.
- b. Weigh approximately (to the nearest 0.01 g) a 1 to 1.5 g portion of the sample directly into a digestion vessel. For samples with low percent solids a larger sample size may be used as long as digestion is completed. Record the exact mass in the LIMS.

NOTE: To achieve the lowest reporting limit possible, use a 2.0 g portion of sample with an ending volume of 100 mLs.

- c. Add 5 mL D.I. water and 5 mL concentrated $\text{HNO}_3(1:1)$, mix the slurry and cover with a watch glass. Place the sample in a preheated hot block and reflux at 95°C for 10 to 15 minutes being certain that the sample does not boil. Record temperature in temperature logbook
- d. Allow the sample to cool. Add 5 mL concentrated HNO_3 , replace the watch glass and heat/reflux again for 30 minutes. If brown fumes are generated, indicating oxidation of the sample by HNO_3 , repeat this step (addition of 5 mL of concentrated HNO_3) over and over until no brown fumes are given off by the sample indicating the complete reaction with HNO_3 . Using a watch glass or equivalent allow the solution to evaporate to approximately 5 mL without boiling at $95^\circ\text{C} \pm 5^\circ\text{C}$ for approximately two hours. Maintain a covering of solution over the bottom of the vessel at all times. Do not allow the volume to be reduced to less than 5 mL while maintaining a covering of solution over the bottom of the beaker. If the volume does get low, add 2.5 mL of D.I. water to bring volume back up.
- e. Take the sample off the hot block and allow it to cool. Next, add 2 mL of D.I. water and 3 mL of 30% Hydrogen Peroxide. (The sample will bubble upon the addition of H_2O_2 if it is still warm.) Cover the vessel with a watch glass and return the sample to the hot block or equivalent source and heat until the bubbling subsides. Care must be taken to ensure that losses do not occur due to excessively vigorous effervescence. Heat until effervescence subsides and cool the beaker. Add two more 3 mL portions of H_2O_2 to the sample in the same manner as before. (NOTE: Do not add more than a total of 10 mL 30% H_2O_2 .)
- f. Cover the sample with a ribbed watch glass and continue heating the acid-peroxide digestate at $95^\circ\text{C} \pm 5^\circ\text{C}$ without boiling for approximately two hours until the volume has been reduced to approximately 2.5 mL. Maintain covering of solution over the bottom of the vessel at all times.
- g. Add 2.5 mL of DI water and 2.5 mL of concentrated HCl and 10 mL of DI water, cover the sample with a ribbed watch glass and continue refluxing for an additional 10 minutes without boiling
- h. When necessary, filter or centrifuge the sample to remove insoluble material that could clog the nebulizer. The filtering apparatus must be thoroughly cleaned with dilute nitric acid prior to filtration.
- i. Bring sample up to 50 mL with D.I. water in the vessel. Add 150 ml of DI water to a 250 ml sample bottle. Invert the 50 ml sample digestion vessel several times to mix the sample and pour sample into the 150 ml of the sample bottle. Pour some sample back into the 50 ml sample digestion vessel to rinse and pour back into the 250 ml sample bottle and cap and mix.

NOTE1: When preparing DoD project samples, if any sample in a digestion batch requires filtration, all samples (including QC samples) must be treated in the same manner.

NOTE2: To achieve the lowest reporting limit possible use 2.0 grams of sample with an ending volume of 100 mLs.

- j. The sample is now ready for analysis.
- k. The LIMS must contain the date, analyst, sample number, client, sample mass/volume, final volume of digestate, lot # of acids used and the preparation and ID of standards.

X. CALCULATIONS

- A. The analyst must be supplied with both beginning sample masses/volumes and final digestate volumes. This information must be recorded in the digestion log.

XI. QUALITY CONTROL

A. Digestion

1. Temperature blank

- a. The temperature of the hot plate/hot block must be monitored for temperature during the digestion process.
- b. The thermometer must be tagged with annual calibration information. Record the thermometer reading, correction factor and the corrected temperature in the digestion log.

2. Blanks

- a. Digest a blank with every batch of samples digested (20 sample maximum). The blank is prepared by adding all the same reagents added to the samples to a clean dry beaker and taking it through the same process as the samples.
- b. Also, there must be a blank for every different method of digestion that is set up that day, every 20 samples.
- c. There must also be a blank for every different matrix of samples that is to be digested, every 20 samples.
- d. Sample is given a unique identifier in the digestion log.

3. Laboratory Control Samples

- a. For water samples, one LCS is digested with every batch of samples digested (20 sample maximum).
- b. For water samples, a LCS is digested every day for each type of digestion, every 20 samples.
- c. For soil/sediment samples, a soil matrix standard reference material (SRM) must be digested per batch (20 samples maximum) or alternatively a spiked teflon chip sample.
- d. Sample is given a unique identifier in the digestion log.

4. Duplicates

- a. A duplicate is prepared every 20 samples. This usually takes the form of a matrix spike duplicate.
NOTE: Certain projects require a sample duplicate and a matrix spike duplicate with each set of twenty samples.

5. Blank Spike

- a. This is required for certain projects.

B. Sample Matrix

NOTE: Field blanks/duplicates, trip blanks, or equipment blanks are not to be used for sample matrix QC samples.

1. Matrix spike

- a. Digest a spike and spike duplicate every 20 samples where sample volume is adequate to do so. Choose a sample (if possible) that has a lot of metals requested to be analyzed.

NOTE: For some projects, a sample duplicate and sample spike may be required instead of a spike and spike duplicate. Your supervisor should make you aware of these projects.

- b. The following metals do not get digested spikes when using CLP spike.

Calcium

Magnesium

Sodium

Potassium

- c. For TCLP samples, a spike **and a spike duplicate** must be digested for every matrix. You should inspect the sample (original sample prior to extraction) or check the log book to determine matrix type. (Also the matrix spike aliquots must be added to the extracts after filtration but before preservation.)

- d. **The CLH project requires that a high and a low spike be prepared and analyzed. Spikes should be prepared at 40 mg/Kg and 400 mg/Kg for soil samples and 200 ug/L and 2000 ug/L for aqueous samples.**

XII. CORRECTIVE ACTIONS

A. Sample boils during digestion.

1. Redigest another sample aliquot.

B. Sample goes dry or portion of beaker bottom is exposed due to excess evaporation during digestion.

1. Redigest another sample aliquot.
2. Glass beaker dry for an extended period of time? Discard beaker.

XIII. SPECIAL NOTES

A. **Never** take for granted how a sample should be digested. If the sample looks strange or unusual, or if you are not sure what metals the sample gets, what detection limits are required, whether the sample is total or dissolved, or even what method of digestion should be used, always ask your supervisor or the person who is to analyze the sample. How metals need to be digested changes too often to take it for granted.

B. **Antimony (Sb) soils** should be analyzed within 48 hours of digestion whenever possible. When a soil requesting Antimony analysis is received, you must coordinate with the person who will be analyzing it to be sure that they can analyze it on the same day that it is digested.

C. Labels for the digested sample must be written in a neat and legible manner. The labels must include such information as sample number, client name, the date digested, and the volume or mass digested.

D. There are several precautions that must be taken to minimize the possibility of contamination.

1. All metals glassware must be kept separate from all other laboratory glassware.

2. Metals glassware must be washed as soon as possible after being used. **Dirty metals beakers must not be left overnight.**
 3. Acid to be used for metals digestions must be kept separate from all other laboratory acid.
- E. Samples must be digested in a timely manner to ensure ICP analysis remains on schedule for data generation. Samples received on or before Wednesday of week X must be prepared for ICP digestion by the end of week X. Your supervisor must be consulted if this schedule can not be met at a particular time.
- F. Please consult Waste Disposal SOP-QS14, for information concerning disposal of waste generated from this area. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

Addendum for USEPA CLPILM 05.2 AQUEOUS & SOIL/SEDIMENT

The following is a list of changes for sample preparation when the 5.2 statement of work is required:

1. Soluble samples are required to be digested unless the chain of custody specifically states that digestion is not required. An MDL study must be done on the unprepared MDL solution in order to provide MDL levels for samples that are not digested. When digestion is not required an LCSW and post digestion spike are not required.
2. Digestates must be stored until 365 days after delivery of a complete, reconciled data package.
3. Preparation codes are used on form 13's. They are found in the 5.2 statement of work page B-39 3.4.12.2.4.

DEFINITIONS – Refer to SOP-QS08 for common environmental laboratory definitions.

EMPIRICAL LABS, LLC.

Record of SOP Review and Implementation

Rev 22

TRAINING TOPIC SOP 100 - Metals Digestion/Preparation Methods 3005A, 3010A, 3020A, 3030, 3040A, 3050B USEPA CLPILMO 04.1

AQUEOUS & Soil/Sediment USEPA CLPILMO 05.2 Aqueous & Soil/Sediment, USEPA Method 200.7 (Standard Methods) 3030C

Group: Betty Deville

ATTENDEES:					
NAMES (print)	SIGNATURE	REMARK	DATE	TIME	INSTRUCTOR
1 Kendra Gentry	<i>Kendra Gentry</i>		1/17/11	14:54	BLD
2 Royer Buer	<i>Roy B</i>		1-17-2011	15:11	BLD
3 Fran Hula	<i>Fran Hula</i>		1/17/2011	15:17	KH
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**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

METALS: SOP 105

REVISION #: 17

EFFECTIVE DATE: 20110516

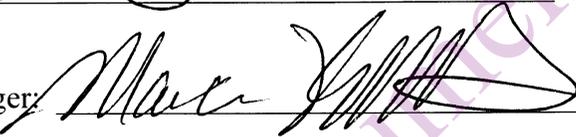
METALS

**BY INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION
SPECTROMETRY (ICP-AES) TECHNIQUE**

**SW846 6010B, SW846 6010C, EPA 200.7, SM 2340B (19th 20th and 21st Edition) for Hardness
Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June,
2003) and Addendum for USEPA CLPILM 05.2.**

APPROVALS:

Lab Director:  Date: 5/16/11

Data Quality Manager:  Date: 5/16/11

Section Supervisor: Betty Quillen Date: 5/16/11

Changes Summary

Revision 17, 20110516

- This is an update of SOP revision 16 dated 4/11/2010.
- Change all limit statements to include “after rounding to the nearest whole number”.
- Add procedure for recording digestates filtered prior to analysis within section 14.2.
- Training SOP reference updated to QS03 in section 14.6.
- References to DoD QSM 4.1 have been updated to DoD QSM 4.2.

Revision 16, 04/11/10

- The SOP is an update from Revision 15 dated 05/08/09
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory’s revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, SM 2340B (19th 20th and 21st Edition) for Hardness Calculation, (USEPA CLP) ILMO 4.1 (NJDEP does not accept CLPILM 04.1 after June, 2003) and Addendum for USEPA CLPILM 05.2.

2. Applicable Matrix or Matrices

This SOP is applicable to all matrices, including ground water, aqueous samples, TCLP, SPLP and EP extracts, industrial and organic wastes, soils, sludge samples, sediments, and other solid wastes, require digestion prior to analysis.

3. Detection Limit: Detection limits are found in **Table 1** of this SOP. Sensitivity and optimum ranges of the metals may be found in the ICP method file.

4. Scope of Application, Including components to be Analyzed

Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. This table also lists the associated Method Detection Limit and the Reporting Limit (also defined as the Limit of Quantitation).

5. Summary of the Test Method

- 5.1 Prior to analysis, samples must be solubilized or digested using appropriate. Sample Preparation Methods (e.g., Methods 3005-3050 and SOW ILM 04.1/05.2). When analyzing for dissolved constituents, acid digestion is not always necessary if the samples are filtered and acid preserved prior to analysis. If particulates form after filtration and preservation the sample must be digested prior to analysis.

NOTE: When selenium is required soluble samples must always be digested.

- 5.2 This method describes the simultaneous multi-elemental determination of elements by ICP. The method measures element-emitted light by optical spectrometry. Samples are nebulized and 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 decomposed to atoms and ions that become excited and emit characteristic light which is measured, giving a measurement of the concentration of each element type in the original sample. Background correction is required for trace element determination. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background-intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analytic wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. Control of the spectrometer is provided by PC based *iTEVA* software.

5.3 Inductively Coupled Argon Plasma (ICAP) primary advantage is that it allows simultaneous determination of any elements in a short time. The primary disadvantage of ICP is background radiation from other elements and the plasma gases. Although all ICP instruments utilize high-resolution optics and background correction to minimize these interferences, analysis for traces of metals in the presence of a large excess of a single metal is difficult. Examples would be traces of metals in an alloy or traces of metals in a limed (high calcium) waste. ICP and Flame AA have comparable detection limits (within a factor of 4) except that ICP exhibits greater sensitivity for refractories (Al, Ba, etc.). Furnace AA, in general, will exhibit lower detection limits than either ICP or FAA.

5.4 It is standard procedure to use an internal standard (scandium) with samples to increase the stability of the instrument as recommended by the manufacturer (Thermo Fisher). (When samples are suspected of containing scandium, internal standard cannot be used.)

6. Definitions

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

Additional definitions specific to this SOP are listed below:

- 6.1 **ICP or ICAP**- Inductively Coupled Plasma or Inductively Coupled Argon Plasma.
- 6.2 **Inter-element correction (IEC)**- Defined as a correction factor applied by the instrument when there is an overlap of the spectrum from the plasma gases or from another metal into the spectrum of another metal causing that metals concentration to either be inflated or deflated.

7. Interferences

7.1. Spectral interferences are caused by background contribution from continuum or recombination phenomena, stray light from the line emission of high-concentration elements, overlap of a spectral line from another element, or unresolved overlap of molecular band spectra.

7.1.1. Background emission and stray light can usually be compensated for by subtracting the background emission determined by measurements adjacent to the analyte wavelength peak. Spectral scans of samples or single element solutions in the analyte regions may indicate when alternate wavelengths are desirable because of severe spectral interference. These scans will also show whether the most appropriate estimate of the background emission is provided by an interpolation from measurements on both sides of the wavelength peak or by measured emission on only one side. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference (inter-element or molecular) or adequately corrected to reflect the same

change in background intensity as occurs at the wavelength peak. For multivariate methods using whole spectral regions, background scans should be included in the correction algorithm. Off-line interferences are handled by including spectra on interfering species in the algorithm.

- 7.1.2. To determine the appropriate location for off-line background correction, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. The location selected for background correction must be either free of off-line inter-element spectral interference or a computer routine must be used for automatic correction on all determinations. If a wavelength other than the recommended wavelength is used, the analyst must determine and document both the overlapping and nearby spectral interference effects from all method analytes and common elements and provide for their automatic correction on all analyses. Tests to determine spectral interference must be done using analyte concentrations that will adequately describe the interference. Normally, 100 mg/L single element solutions are sufficient; however, for analytes such as iron that may be found at high concentration, a more appropriate test would be to use a 200 mg/L or 500 mg/L concentration near the upper analytical range limit.
- 7.1.3. Spectral overlaps may be avoided by using an alternate wavelength or can be compensated by equations that correct for inter-element contributions. Instruments that use equations for inter-element correction require the interfering elements be analyzed at the same time as the element of interest. When operative and uncorrected, interferences will produce false positive determinations and be reported as analyte concentrations. More extensive information on interferant effects at various wavelengths and resolutions is available in reference wavelength tables and books. Users may apply inter-element correction equations determined on their instruments with tested concentration ranges to compensate (off line or on line) for the effects of interfering elements. Some potential spectral interferences observed for the recommended wavelength are listed in the method in table 2. For multivariate methods using whole spectral regions, spectral interferences are handled by including spectra of the interfering elements in the algorithm. The interferences listed are only those that occur between method analytes. Only interferences of a direct overlap nature are listed. These overlaps were observed with a single instrument having a working resolution of 0.035 nm.
- 7.1.4. When using inter-element correction equations, the interference may be expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interference element. For example, assume that Arsenic is to be determined (at 193.696 nm) in a sample containing approximately 10 mg/L of Aluminum. According to Table 2 from the method, 100 mg/L of Aluminum would yield a false signal for Arsenic equivalent to approximately 1.3 mg/L. Therefore, the presence of 10 mg/L of Aluminum would result in a false signal for Arsenic equivalent to approximately 0.13 mg/L. The user is cautioned that other instruments may exhibit somewhat different levels of interferences than that shown in Table 2 from the method. The interference effects must be evaluated for each individual instrument since the intensities will vary.

- 7.1.5. Inter-element corrections will vary for the same emission line among instruments because of differences in resolution, as determined by the grating, the entrance and exit slit widths, and by the order of dispersion. Inter-element corrections will also vary depending upon the choice of background correction points. Selecting a background correction point where an interfering emission line may appear should be avoided when practical. Inter-element corrections that constitute a major portion of an emission signal may not yield accurate data. Users should not forget that some samples may contain uncommon elements that could contribute spectral interferences.
- 7.1.6. The interference effects must be evaluated for each individual instrument. For each instrument, intensities will vary not only with optical resolution but also with operating conditions (such as power, viewing height and argon flow rate). When using the recommended wavelengths, the analyst is required to determine and document for each wavelength the effect from referenced interferences as well as any other suspected interferences that may be specific to the instrument or matrix. The instrument utilizes a computer routine for automatic correction on all analyses.
- 7.1.7. If the correction routine is operating properly, the determined, apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. The concentration range is calculated by multiplying the concentration of the interfering element by the value of the correction factor being tested and divided by 10. If after the subtraction of the calibration blank the apparent analyte concentration falls outside of this range in either a positive or negative direction, a change in the correction factor of more than 10% should be suspected. The cause of the change should be determined and corrected and the correction factor updated. The interference check solutions should be analyzed more than once to confirm a change has occurred. Adequate rinse time between solutions and before analysis of the calibration blank will assist in the confirmation.
- 7.1.8. When inter-element corrections are applied, their accuracy should be verified, daily, by analyzing spectral interference check solutions (IFA/IFB). If the correction factors or multivariate correction matrices tested on a daily basis are found to be within 20% criteria for 5 consecutive days, the required verification frequency of those factors in compliance may be extended to a weekly basis. Also, if the nature of the samples analyzed is such they do not contain concentrations of the interfering elements at \pm one reporting limit from zero, daily verification is not required. All inter-element spectral correction factors or multivariate correction matrices must be verified and updated every six months or when an instrumentation-change, such as in the torch, nebulizer, injector, or plasma conditions occurs. Standard solution should be inspected to ensure that there is no contamination that may be perceived as a spectral interference.

7.2. Physical interferences are effects associated with the sample nebulization and transport processes. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If

physical interferences are present, they must be reduced by diluting the sample or by using a peristaltic pump, by using an internal standard or by using a high solids nebulizer. Another problem that can occur with high dissolved solids is salt buildup at the tip of the nebulizer, affecting aerosol flow rate and causing instrumental drift. The problem can be controlled by wetting the argon prior to nebulization, using a tip washer, using a high solids nebulizer or diluting the sample. Also it has been reported that better control of the argon flow rate, especially to the nebulizer, improves instrument performance: this may be accomplished with the use of mass flow controllers.

- 7.3. Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. Memory effects can result from sample deposition on the uptake tubing to the nebulizer and from the build-up of sample material in the plasma torch and spray chamber. The site where these effects occur is dependent on the elements and can be minimized by flushing the system with a rinse blank between samples. The possibility of memory interferences should be recognized.
- 7.4. Users are advised that high salt concentrations can cause analyte signal suppressions and confuse interference tests. When the instrument displays negative values, dilution of the samples may be necessary.

8. Safety

Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed lab-wide.

- 8.1 Normal accepted laboratory safety practices should be followed while performing this analysis.
 - 8.1.1. Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of appropriate safety gloves and lab coats is highly recommended.
 - 8.1.2 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples that need special consideration have applicable notes on the sample logs.
 - 8.1.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves in the Quality Assurance Officers office.

9. Equipment & Supplies

- 9.1. Inductively coupled argon plasma emission spectrometer: Thermo Scientific 6500 DUO.
- 9.2. Computer-controlled emission spectrometer with background correction: Thermo Scientific 6500 DUO or equivalent.
- 9.3. Radio frequency generator compliant with FCC regulations: Thermo Fisher or equivalent.
- 9.4. Auto-sampler: Thermo Fisher or equivalent.

- 9.5. Printer capable of printing results every 4 minutes.
- 9.6. Cooling Water recycler.
- 9.7. Iteva software.
- 9.8. Argon gas supply – Liquid Argon
- 9.9. Class A volumetric flasks
- 9.10. Analytical balance - capable of accurate measurement to a minimum of three significant figures (0.001gm).
- 9.11. Variable Eppendorf Pipettes 1000 μ L; 5000 μ L
- 9.12. Disposable beakers 10, 20 and 50 mL size.
- 9.13. Hood system capable of venting the heat from the system off of the instrument during analysis.

10. Reagents and Standards

The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:

- 10.1. Reagent Water. All references to water in the method refer to reagent grade water unless otherwise specified. Reagent water will be interference free.
- 10.2. 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. If the purity of a reagent is in question analyze for contamination. If the concentration is less than the MDL then the reagent is acceptable.
- 10.3. Hydrochloric acid (concentrated), HCl. A method blank is digested and analyzed before a new lot number of HCl is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.
- 10.4. Nitric acid (concentrated), HNO₃. A method blank is digested and analyzed before a new lot number of HNO₃ is put into use, to ascertain purity. The lot # is logged into Element and the data kept on file.
- 10.5. Calibration standards

- 10.5.1. All standards have an acid matrix of 2% HNO₃ and 5% HCl and should be prepared using class A volumetric flasks and calibrated Eppendorfs).
- 10.5.2. CAL1 is the calibration blank: Reagent grade water matrix matched as in
Note: when this standard is analyzed the intensities should be compared to a previous run to make sure that no contamination has occurred. Prepare this solution fresh daily.
- 10.5.3. Stock QC21 solution: (100 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals - Sb, As, Be, Cd, Ca, Cr, Co, Cu, Fe, Pb, Li, Mg, Mn, Mo, Ni, Se, Sr, Tl, Ti, V, and Zn.
- 10.5.4. Stock QC7 solution: Order from the manufacturer already prepared. This solution is given a unique identifier within Element and includes the following metals- (50 ug/mL)- silver; (100 ug/mL)- aluminum, boron, barium and sodium; (1000 ug/mL)- potassium; (500 ug/mL or 100 ug/mL note we use two sources of this standard and each have different concentrations for Si) –Silica.
- 10.5.5. Boron solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.6. Stock Tin solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.7. Stock Silver solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.8. Stock Aluminum solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.9. Stock Calcium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier. Note: Two sources are needed.
- 10.5.10. Stock Magnesium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.11. Stock Iron solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.

- 10.5.12. Stock Potassium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.13. Stock Barium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.14. Stock Sodium solution: (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element. Note: Two sources are needed.
- 10.5.15. Stock Arsenic solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.16. Stock Cobalt solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.17. Stock Chromium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.18. Stock Copper solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.19. Stock Manganese solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.20. Stock Nickel solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.21. Stock Lead solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.22. Stock Selenium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.23. Stock Thallium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.24. Stock Beryllium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.25. Stock Cadmium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.26. Stock Antimony solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

- 10.5.27. Stock Molybdenum solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.28. Stock Strontium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.29. Stock Titanium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.30. Stock Vanadium solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.31. Stock Zinc solution: (1000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.
- 10.5.32. Stock Scandium solution (10000 ug/mL). Order from the manufacturer already prepared. This solution is given a unique identifier within Element.

10.6. Calibration and Calibration Verification standards

- 10.6.1. The calibration standards and calibration verification standards preparations are recorded in Element. Please find method of preparation in Appendix I.
- 10.6.2. The CRL solution is analyzed to check the accuracy of the instrument at the reporting limit. The stock standard solutions A and B are prepared from single element standards listed in 10.5 above. Please find method of preparation in Appendix I. This solution is stable for 6 months. The working solutions are made up as needed or every 3 months as follows: Prepared by adding 1.0 ml of RL Stock solution A and 1.0 ml of RL Stock Solution B to de-ionized water with 2% HNO₃ and 5% HCL matrix and diluting to 100 mLs , mix well. This solution is stable for 3 months.
- 10.6.3. The interference check standard solutions (IFA and IFB) are prepared to provide an adequate test of the IECs. A purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe is diluted 10x to prepare the IFA. The IFB is prepared by diluting 100x a purchased solution containing 10 ug/mL of As and Tl; 20 ug/mL Ag; 50 ug/mL Ba, Be, Cr, Co, Cu, Mn, and V; 100 ug/mL Cd, Ni and Zn; 5 ug/mL Pb and Se; and 60 ug/L Sb. Add to this a purchased solution containing 500 ug/mL Al, Ca, Mg and 200 ug/mL Fe diluted 10x. These solutions are prepared as needed or monthly and assigned an Element # for traceability.

10.7 Digestion standards

10.7.1 The Blank Spike (BS) is prepared from High Purity solutions CLP-CAL-1 solution A and B; CLP-CAL-2 and CLP-CAL-3. 0.50 mL of CLP-CAL-1 A and B; and 0.50 mLs of the 1000 ug/mL single element standards for Molybdenum, Boron, Titanium and Strontium is diluted to 500 mL with 0.125 mL of CLP-CAL-2 and CLP-CAL-3 and 0.050 mLs of 10000 ug/mL Tin. 25 mL of HCl and 10 mL of HNO₃ are added for preservation. This solution is stored in a Teflon bottle. A portion is reserved in case of a problem with digestion. When there is a problem with the analysis of the BS the solution is checked first before action is taken to make sure that it was made properly and has not deteriorated since it was made up. This solution is given a unique identifier within Element. The BS is prepared from a source independent from that used in the calibration standards. This solution is prepared daily or as needed. 50 mLs of this solution is used for digestion for normal level water samples and the sample is brought back to 50 mLs after digestion. Low level water samples start with two 50 mLs vials with only 1.0 mL of the stock blank spike solution in each taken to 50 mLs. The samples are cooked down to below 25 mLs and combined and then cooked down to below 25 mLs again and then brought back to 25 mLs. This low level BS is given a unique identifier in Element.

10.7.2. The solid BS used with soil samples is prepared by weighing up 1.0 gram of Teflon chips for regular level and 2.0 grams of Teflon chips for low level and spiking using the same spiking solutions used to spike the sample matrix. This standard is given a unique identifier i.e. Batch #-BS1. Note: Amount of spiking solution used varies according to whether the samples are being digested for normal level or low level soils. See spiking solutions in 10.7.3.1 for how to prepare the BS for a solid sample, it is prepared the same way that a soil spike is prepared only the known amounts of metals are added to laboratory water.

10.7.3. The spiking solutions are prepared as follows:

10.7.3.1. Stock Multi-element Spiking Solutions: High Purity CLP-CAL-1 solution A: 2000 ug/mL Al and Ba; 50 ug/mL Be; 200 ug/mL Cr; 500 ug/mL Co, Mn, Ni, V and Zn; 250 ug/mL Cu; 1000 ug/mL Fe; 5000 ug/mL Ca, Mg, K and Na; solution B: 250 ug/mL Ag; CLP-CAL-2: 1000 ug/L Sb; CLP-CAL-3: 1000 ug/mL As, Pb, Se, Tl; 500 ug/mL Cd. Order from the manufacturer already prepared. These solutions are given a unique identifier within Element. Add 0.050 mL for water samples and 0.20 mL for normal level soil samples and 0.10 for low level soil samples of CLP-CAL-1 solutions A and B, and 0.0125 mL for water samples and 0.05 mLs for normal level soil samples and 0.025 mLs for low level soil samples of CLP-CAL-2 and 3 to 50 mL of sample for water samples and 1 gram of sample for normal level soils and 2 grams of sample for low level soils for the following spike values: 2000 ug/L Al and Ba; 50 ug/L Be; 200 ug/L Cr; 500 ug/L Co, Mn, Ni, V and Zn; 250 ug/L Cu; 1000 ug/L Fe; 5.0 mg/L Ca, Mg, K and Na, 250 ug/L Ag, Sb, As, Pb, Se and Tl; 125 ug/L Cd. A blank

spike should be prepared at the time the samples are spiked to check the actual spike value and accuracy.

10.7.3.2. TCLP Spiking Solution: Use 0.50 mL diluted to 50 mL for digestion:

2.5 mL 10000 mg/L Ba stock standard diluted to 100 mL; 2.5 mL Cr, Pb and As 1000 mg/L stock standard diluted to 100 mL; 0.50 mL Cd and Se diluted to 100 mL. Store in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 2500 ug/L Ba; 250 ug/L Cr, Pb and As; and 50 ug/L of Cd and Se. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed.

10.7.3.3. TCLP Silver Spiking Solution: Use 5.0 mL diluted to 50 mL for digestion:

0.40 mL of 1000 mg/L stock Ag solution diluted to 200 mL. Store this solution in a Teflon bottle. A blank spike should always be prepared at the same time a sample is being spiked. This solution should produce a spike value of 200 ug/L. Note: Since the samples are diluted 10x when digested the spike value will appear to be 10x greater when analyzed. Also this solution is not very stable and may require fresh preparation at least weekly.

11. Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.

11.1. Preliminary treatment of most matrices is necessary because of the complexity and variability of sample matrices. Water samples which have been pre-filtered and acidified will not need acid digestion as long as the samples and standards are matrix matched and particulates do not form after the filtration and preservation take place. Solubilization and digestion procedures are presented in Sample Preparation Methods (Methods 3005A-3050A).

11.2. Sample digestates are stored at room temperature for at least 2 months unless a longer time is requested by the client. The samples contain an acid matrix of 3:1. All metal samples are neutralized before disposal in the receiving section of the laboratory.

11.3. **The appropriate SOPs should be consulted regarding sample preparation.** The following is a brief summary of the methods we use for metals preparation.

11.3.1. Method 3005A prepares groundwater and surface water samples for total recoverable and dissolved metals determination by ICP. The unfiltered or filtered sample is heated with dilute HCl and HNO₃ prior to metal determination.

11.3.2. Method 3010A prepares waste samples for total metal determination by ICP. The samples are vigorously digested with a mixture of nitric acid and hydrochloric acid followed by dilution with laboratory water. The method is applicable to aqueous samples, TCLP and mobility-procedure extracts.

11.3.3. Standard Methods Method 3030C prepares ground-waters and surface water samples for acid extractable metals: (lead and chromium.) This preparation has a holding time of 72 hours. The samples are preserved at collection with 5mL/L of HNO₃, in the laboratory 5 mL/100mL of 1+1 HCl is added and the sample is heated for 15 minutes in a block digester. The sample is filtered through a membrane filter and the filtrate is carefully transferred to a volumetric flask and brought back to 100 mLs.

11.3.4. Method 3050B prepares wastes samples for total metals determination by ICP. The samples are vigorously digested in nitric acid and hydrogen peroxide followed by dilution with either laboratory water or hydrochloric acid and laboratory water. The method is applicable to soils, sludges, and solid waste samples.

12. Quality Control

Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.

12.1. Daily run and batch QC

12.1.1. Calibration is required daily. Either a blank and a high standard or a client specific three standard concentration points and a blank calibration is required daily.

12.1.2. IEC correction standards for aluminum and iron are required daily.

12.1.3. ICV within $\pm 5\%$ for 200.7 and within $\pm 10\%$ for all other methods.

12.1.4. ICB/CCB less than two times \pm MDL or less than \pm LOD for DOD. The ICB/CCB must immediately follow the ICV/CCV.

12.1.5. RL standard run against the curve within $\pm 20\%$ initially and client specific requirement of $\pm 30\%$ at the end of the analysis.

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times \pm MDL or less than \pm LOD unless verified standard contamination for DOD. The IFB must recover within $\pm 20\%$ for all analytes in the IFB standard solution. If the IFA/IFB solution is not within the required limits- if possible reanalyze all associated samples, if not possible to reanalyze all associated samples must be flagged with an “Q” on the final report for DOD.

12.1.7. CCV must be analyzed every ten samples or at the end of the analysis within $\pm 10\%$ or the samples are reanalyzed if possible. If samples cannot be reanalyzed, all samples are flagged with a "Q" for DOD.

12.1.8. CCB must be analyzed every ten samples immediately following the CCV or at the end of the analysis less than two times $\pm \text{MDL}$ or $\leq \text{LOD}$ for DOD. If the CCB is out of the allowable range the samples are flagged with "B".

12.1.9. *The following should be analyzed with each preparation batch containing a matrix spike.*

- Serial dilution: If the analyte concentration is sufficiently high (minimally, a factor of 50 above the instrumental detection limit after dilution), an analysis of a 1:4 dilution (volumetric glassware must be used) should agree within $\pm 10\%$ of the original determination. If not, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.
- Post digestion spike addition: An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75% to 125% of the known value for SW6010B and 80 to 120% for SW6010C and is required especially if the pre-digestion matrix spike is outside of control limits. The spike addition should produce a minimum level of 10 times and a maximum of 100 times the instrumental detection limit. If the spike is not recovered within the specified limits, a matrix effect should be suspected. Run all associated samples in the preparatory batch by method of standard additions (MSA) or apply "J" flag. The analyst and or section manager must note this situation on the final analytical report. Apply "J" flag if the post spike is outside the range of 75% to 125% for 6010B or 80% to 120% for 6010C.

12.2 Quarterly and/or every six months

12.2.1. Linear range standards must be analyzed at a frequency no less than once every six months. The linear range standard is required for verification that samples are actually linear to the degree claimed. The analyst is responsible for completing this task in a timely manner. The linear range standard must be within $\pm 10\%$ of true value. This standard can be analyzed as the linear dynamic range.

12.2.2. The inter-element correction factors (IEC) should be verified at the time the linear range standards are analyzed or whenever there is any question about whether an IEC is correcting correctly.

12.2.3. IDL's, linear range and IEC checks must be performed quarterly if straight CLP work is required.

12.3. Digested Batch QC

- 12.3.1. All quality control data should be maintained and available for easy reference or inspection.
- 12.3.2. Employ a minimum of one method blank per sample batch to determine if contamination or any memory effects are occurring. A method blank (BLK), sometimes referred to as the preparation blank is a volume of reagent water acidified with the same amounts of acids as were the standards and samples. These blanks are taken through the same digestion/preparation steps as the sample being tested. The result for the method blank should not indicate contamination greater than $\pm \frac{1}{2}$ RL for DOD or \pm RL/CRDL for other or CLP. If exceeded, the impact upon the data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The extracted blank associated with TCLP batches must be less than 100 X the regulatory limit for barium.
- 12.3.3. Employ a minimum of one blank spike (BS) for aqueous samples or one Teflon chip spiked sample per sample batch to verify the digestion procedure. These blank spikes are taken through the same digestion/preparation steps as the sample being tested. The control limits are $\pm 15\%$ method 200.7 - aqueous and soil samples or $\pm 20\%$ for all other methods aqueous and soil samples. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be re-digested. Consult your supervisor for further action. Qualifying the associated data may not be permissible for some clients.

12.4. Sample

- 12.4.1. Analyze one replicate sample for every twenty samples or per analytical batch, whichever is more frequent. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. It is acceptable to substitute a matrix spike duplicate for the sample replicate. Project specific requirements will take precedence in these situations. NJDEP demands that this requirement be met with a client specific duplicate rather than a spike duplicate. The control limits are less than or equal to 20% RPD (if both are $>5x$ RL) or \pm the RL (if either are $<5x$ RL). Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. Apply "J" flag for DOD if acceptance criteria are not met. Apply "*" flag for CLP and other work if acceptance criteria are not met.
- 12.4.2. Analyze a minimum of one spiked sample and/or spiked sample duplicate for every twenty samples or per analytical batch, whichever is more frequent. Project specific requirements will take precedence in determining whether a matrix spike duplicate is employed in these situations. If the analyte level in the sample is not greater than 4X the spiking level, the spike recoveries should be within $\pm 20\%$ of the true value. If not, and sufficient sample volume exist, a post digestion spike should be analyzed. Apply "J" flag for DOD if acceptance

criteria are not met. Apply “N” flag or CLP and other work if acceptance criteria are not met.

13. Calibration and Standardization

Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.

- 13.1. Set up the instrument with proper operating parameters. The instrument must be allowed to become thermally stable before beginning (usually requiring at least 30 minutes of operation prior to calibration).
- 13.2. Operating conditions - **The instrument settings can be found in method file within the iTEVA software.** For operation with organic solvents, use of the auxiliary argon inlet is recommended, as are solvent-resistant tubing, increased plasma (coolant) argon flow, decreased nebulizer flow, and increased RF power to obtain stable operation and precise measurements. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be established for each individual analyte line on that particular instrument. The analyst must (1) verify that the instrument configuration and operating conditions satisfy the analytical requirements and (2) maintain quality control data confirming instrument performance and analytical results.
- 13.3. Auto-peak when some change has been made to the introductory system and calibrate the instrument according to the instrument manufacturers recommended procedures, using the specified calibration standard solutions. Flush the system with 2% HNO₃ / 5% HCl between each standard or as the manufacturer recommends. (Use the average intensity of multiple exposures for both standardization and sample analysis to reduce random error.) The calibration curve consists of a blank and three standards ($r \geq 0.998$). If a three point calibration curve is not required for the client samples being analyzed by Empirical Laboratories may use a blank and one standard as referenced in USEPA - CLP protocols.
- 13.4. Before beginning the sample run, analyze single element Iron and Aluminum standards at their linear range to check for IEC drifts. Analyze these standards first as QC samples with an IEC check table and action taken should be to calculate IECs using the iTEVA software. Make sure to rinse thoroughly after running these linear range standards, they can cause carry over into the initial QC samples which are analyzed next. The analysis order follows as: ICV ($\pm 10\%$) for 200.7 ($\pm 5\%$) and ICB ($< \pm 2 \times \text{MDL}$, $< \pm \text{LOD-DOD}$ or $\pm \text{RL/CRDL}$ for others or CLP, first, then analyze a reporting limit standard (a standard at the concentration of the reporting limit). This standard should be within $\pm 20\%$ for DOD projects and $\pm 30\%$ for samples analyzed for 6010C. Then reanalyze the highest mixed calibration standard(s) as if it were a sample. Concentration values obtained should not deviate from the actual values by more than 5%. If they do, follow the recommendations of the instrument manufacturer to correct for this condition. Note: Supervisor must be notified if the control limit is

not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

- 13.5. For **CLP projects**, verify the validity of the curve in the region of 2x the contract required detection limit (CRDL) before and after each batch of 20 samples in the specific order of CRI, ICSA, ICSAB, CCV and CCB (CCB criteria: $< \pm\text{MDL}$ or $\pm\text{RL}/\text{CRDL}$ for others or CLP, or twice during every 8-hour work shift, whichever is more frequent. Results should be within $\pm 20\%$. Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation. (For Internal QC)
- 13.6. Verify the inter-element and background correction factors at the beginning of the sequence in the specific order of IFA, IFB, CCV and CCB (IFA criteria: non-spiked analytes $< \pm 2\text{xMDL}$ or $< \text{LOD}$ for DOD beginning of sequence. Do this by analyzing the interference check solution IFA and IFB. Absolute value of concentration for all non-spiked analytes in the IFA must be $< \text{LOD}$ (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within $\pm 20\%$ of the true value for IFB. If corrective action fails, apply Q-flag to all results for specific analyte(s) in all samples associated with the ICS. (CRI, ICSA and ICSAB required at the end for CLP projects only).

Note: Supervisor must be notified if the control limit is not met. Supervisor will dictate corrective action if required. The final analytical report must document this situation.

13.7. The instrument must be calibrated once every 24 hours.

13.8. Instrument Autosampler Report example:

Calibration Rack (used by instrument software to insert QC)

- 1) Cal Std 1 (blank)
- 2) Cal Std 2 (Low Cal)
- 3) Cal Std 3 (Mid Cal)
- 4) Cal Std 4 (Ba @ 5000 ppb)
- 5) Cal Std 5 (QC5)
- 6) Cal Std 6 (QC 21)
- 7) Cal Std 7 (NAK 100)
- 8) Cal Std 8 (QC3)
- 9) Cal Std 9 (Ag)
- 10) Al IEC-(correction using ITEVA software)
- 11) Fe IEC-(correction using ITEVA software)

Sample Sequence RACK 1

- 1) SEQ-ICV

- 2) SEQ-ICB
- 3) SEQ-CRL1-reporting limit standard 1
- 4) SEQ-CRL2-reporting limit standard 2
- 5) Ba@ 5000 ppb (readback)
- 6) QC5
- 7) NAK High-(readback)
- 8) QC 21 High-(readback)
- 9) Salt Cal at 500 ppm (readback)
- 10) Rinse
- 11) SEQ-IFA1
- 12) SEQ-IFB1
- 13) Rinse
- 14) SEQ-CCV
- 15) SEQ-CCB
- 16) Method Blank (*Batch # -BLK1*)
- 17) Blank Spike (*Batch # -BS1*)
- 18) Sample 1
- 19) Sample 2
- 20) Sample 3
- 21) Sample 4
- 22) Sample 5
- 23) Sample 6
- 24) Sample 7
- 25) Sample 8
- 26) Sample 9
- 27) Sample 10
- 28) SEQ-CCV
- 29) SEQ-CCB
- 30) Sample 11
- 31) Sample 12
- 32) Sample 13
- 33) Sample 14
- 34) Sample 15
- 35) Sample 16
- 36) Sample 17
- 37) Sample 18
- 38) Sample 19
- 39) Sample 20
- 40) Sample matrix spike (*batch#- MS1*)
- 41) Sample matrix spike duplicate (*batch# -MSD1*)
- 42) Sample post digestion spike (*batch# -PS1*)
- 43) Sample serial dilution (*batch# -DUP1*)
- 44) SEQ-CCV
- 45) SEQ-CCB
- 46) Preparation Blank (*batch# -BLK1*)
- 47) Blank Spike (*batch# -BS1*)
- 48) Sample 1
- 49) Sample 2

- 50) Sample 3
- 51) Sample 4
- 52) Sample 5
- 53) Sample 6
- 54) Sample 7
- 55) Sample 8
- 56) Sample 9
- 57) Sample 10
- 58) SEQ-CCV
- 59) SEQ-CCB
- 60) Sample 11

RACK 2

- 1) Sample 12
- 2) Sample 13
- Etcetera...

Each rack holds 60 samples and there are 4 racks that are used for samples, CCVs and CCBs and run QC.

14. Procedure

- 14.1. Once the instrument has been calibrated, begin the analysis of samples.
- 14.2. If particulates are visible in the digestate, the sample must be filtered prior to analysis. If filtration is required, a filter blank must be prepared by filtering reagent grade water which has been properly acidified. The sample requiring filtration must be recorded on the bench sheet and added to the bench sheet comments in the LIMS. **In the event USACE samples are filtered, all USACE samples and the QC samples in that QC batch must be filtered. All USACE solid samples and their associated batch QC samples must be filtered prior to analysis.**
- 14.3. Flush the system with 2% HNO₃ / 5% HCl for at least 1 minute before the analysis of each sample.
- 14.4. Dilute and reanalyze samples that are more concentrated than the linear calibration limit or, for 200.7, $\pm 10\%$ of the linear range standard. **In the case of USACE samples, the criterion changes and requires dilution and reanalysis of all samples which produce a concentration that exceeds the highest calibration standard. Sample results detected between the MDL and LOQ are flagged as estimated with a "J" flag.**
- 14.5. Verify calibration every 10 samples or every 2 hours, whichever is more frequent and at the end of the analytical run, using a continuing calibration verification (CCV) sample and a continuing calibration blank (CCB) sample.

- 14.5.1. The results of the CCV are to agree within $\pm 10\%$ for 6010 (5% for 200.7) on initial verification of the expected value, with relative standard deviation (RSD) $< 5\%$ from 3 replicates (minimum of three integrations). If not, terminate the analysis, correct the problem, and reanalyze the previous ten samples. The analyst may continue the analytical run, and after conferring with the section manager it may be necessary to reanalyze a group of samples. The analyst must notify the section manager within 24 hours.
- 14.5.2. The results of the calibration blank (this is not the method/preparation blank) are to be $< 2x \pm MDL$, for CLP $< RL$, for **DOD no analytes detected** $> \pm LOD$. If the calibration blank is not in control, evaluate the impact upon the previous 10 samples. Reanalysis may be required after an evaluation of the data. If the blank $< 1/10$ the concentration of the action level of interest and no sample is within 10% of the action limit, samples need not be reanalyzed. One must also evaluate the reporting limit (RL) as it relates to 3X the IDL/MDL. If the RL is significantly above 3X IDL or MDL then reanalysis may not be required (Na, K, Mg and Ca are good examples of this situation).
- 14.6. Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS03 for guidance.

15. Data Analysis and Calculations

Quality Systems SOP QS09 “General and commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

- 15.1. Total hardness is reported from HNO₃ preserved sample. The final concentration is calculated from the calcium and magnesium results as follows: Ca mg/L x 2.5 + Mg mg/L x 4.1 = total Hardness in mg/L as CaCO₃.
- 15.2. The instrument will generate data results in mg/L or $\mu\text{g/L}$ (labeled appropriately). Each result represents an average of three individual readings per metal channel.
- 15.3. For aqueous samples, if a post/pre-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution.
- 15.4. For solid samples, if a post-digestion dilution is performed, the result must be multiplied by this factor or the dilution factor must be entered into the instrument data table in which case the instrument will generate data corrected for the dilution. Also, the result must be converted to reporting units which are usually mg/kg.

$$SR (\text{ug/g or mg/kg}) = IR * DF * FED / SM$$

- SR = Sample result
 IR = Instrument result ($\mu\text{g/L}$)
 DF = Dilution factor (post digestion)

FED = Final volume of digestate (L)
SM = Sample mass digested (g)

16. Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality Manual is completed by each analyst and then provided to the supervisor for further processing and approval.

DOC LCS Preparation: See BS preparation under 10.7.1 through 10.7.3 above.

DOC Accuracy and Precision Criteria: The LOD is analyzed at 2 times the MDL and must result in a concentration 3 times the noise. The LOQ is analyzed at the RL or 2 times the RL and must be recovered within $\pm 50\%$.

17. Pollution Prevention:

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

18. Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. Table 2 of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19. Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. Table 2 within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

CORRECTIVE ACTIONS

19.1. INSTRUMENT RELATED

- 19.1.1. ICV not within $\pm 10\%$ or $\pm 5\%$ for 200.7
 - a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
 - b. Is the problem with the calibration?
 - i. Recalibrate through analysis of appropriate standards and recheck ICV.

19.1.2. ICB not \pm MDL or within \pm 3X IDL or CRDL for CLP, **DOD no analytes detected >LOD**

- a. Is the problem with the solution?
 - i. Re-prepare
- b. Is the problem with the calibration?
 - i. Recalibrate with the blank solution or the low level standard. Restart analysis with the ICV.

19.1.3. Check standards not within \pm 5%

- a. Is the problem with the solution?
 - i. Re-pour, re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.4. CLP only-CRI not within \pm 20% (Internal QC, only required for CLP work).

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.5. IFA metals not present are not less than the detection limit for that metal, **for IFA DOD, absolute value of concentration for all non-spiked analytes $<\pm$ LOD.**

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.6. IFB not within \pm 20%

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. Recalibrate thru analysis of appropriate standards. Restart analysis with the ICV.

19.1.7. CCV not within \pm 10%

- a. Is the problem with the solution?
 - i. Re-prepare or obtain new stock.
- b. Is the problem with the calibration?
 - i. If appropriate, continue the analysis. Discuss effect of the out of control situation with your supervisor. The samples will be reanalyzed or the data will be qualified.

19.1.8. CCB not \pm 2xMDL or CRDL for CLP, DOD no analytes detected $>\pm$ LOD.

- a. Is the problem with the solution?

- i. Re-prepare
- b. Is the problem with the calibration?
 - i. Re-calibrate and reanalyze.

19.2. DIGESTION RELATED

- 19.2.1. Preparation blank (BLK) not within $\pm \frac{1}{2}$ RL and \pm RL for common contaminants DOD or RL/CRDL for other or CLP
 - a. Is the problem with the instrument?
 - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
 - b. Is the problem with the digestion?
 - i. If associated samples are less than 10X the level of the preparation blank but above the RL, the sample must be re-digested or the data must be qualified on the final report.
- 19.2.2. BS not within control limits
 - a. Is the problem with the instrument?
 - i. Evaluate with respect to instrumental bias or reanalyze when instrument is in control.
 - b. Is the problem with the digestion?
 - i. If biased low, associated samples must be re-digested.
 - ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-digested or the data will be qualified on the final report.

19.3. SAMPLE MATRIX RELATED

- 19.3.1. Replicate analysis RPD not within $\pm 20\%$ (if both are $>5X$ CRDL) or \pm the CRDL (if either are $<5X$ CRDL).
 - a. The associated sample data must be qualified on the final report.
- 19.3.2. Spike analysis recovery not within $\pm 20\%$.
 - a. Is the analyte level in the sample greater than 4X the spiking level?
 - i. If yes, the spike recovery is not evaluated.
 - ii. If no, a post digestion spike must be analyzed and the associated sample data must be qualified on the final report.
- 19.3.3. When required, post digestion spike analysis recovery not within $\pm 25\%$ for SW6010B, DOD or $\pm 20\%$ SW6010C.
 - a. The associated sample data must be qualified on the final report.
 - b. For USACE analysis by MSA is required.
- 19.3.4. Serial dilution analysis percent difference not within $\pm 10\%$
 - a. Is the analyte concentration a factor of 50 above the instrumental detection limit after dilution?
 - i. If no, the serial dilution data can not be evaluated.

- ii. If yes, a chemical or physical interference effect should be suspected. The analyst and or section manager must note this situation on the final analytical report.

20. Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21. References

21.1. *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 6010B and Method 6010C.*

21.2. *USEPA Code of Federal Regulations, 40, CH 1, PT 136; Method 200.7; APX-B.*

21.3. *USEPA Contract Laboratory Program (CLP) for Inorganics ILM04.1; ILM05.2*

21.4. DOD Quality Systems Manual for Environmental Laboratories Version 4.2. (Based on NELAC Voted Revision June 5, 2003. 10/25/2010)

22. Tables, Diagrams, Flowcharts and Validation Data

Table 1 contains all applicable parameters with the applicable RL/LOQ, LOD and Detection Limit.

Table 1A, contains a list of the wavelengths used for each analyte.

Table 2, for all technical methods, contains the QA/QC summary table.

Table 3, Technical Completeness / Accuracy Checklist

Table 4, Data Reviewers Checklist

Table 1 Water				
Analyte	MDL	LOD	MRL	Units
Aluminum	50.0	100	200	ug/L
Antimony	5.00	8.00	15.0	ug/L
Arsenic	3.00	6.00	10.0	ug/L
Barium	5.00	10.0	40.0	ug/L
Beryllium	1.00	2.00	5.00	ug/L
Boron	10.0	20.0	30.0	ug/L
Cadmium	1.00	2.00	5.00	ug/L
Calcium	1000	2000	5000	ug/L
Chromium	2.00	4.00	10.0	ug/L
Cobalt	5.00	10.0	12.5	ug/L
Copper	4.00	8.00	10.0	ug/L
Iron	30.0	60.0	100	ug/L
Lead	1.50	3.00	3.00	ug/L
Magnesium	1000	3000	5000	ug/L
Manganese	3.00	6.00	15.0	ug/L
Molybdenum	5.00	10.0	15.0	ug/L
Nickel	3.00	6.00	10.0	ug/L
Potassium	1000	3000	5000	ug/L
Selenium	3.00	5.00	6.00	ug/L
Silver	1.00	2.00	10.0	ug/L
Sodium	1000	3000	5000	ug/L
Thallium	3.00	4.00	8.00	ug/L
Tin	10.0	20.0	30.0	ug/L
Titanium	5.00	10.0	15.0	ug/L
Vanadium	5.00	10.0	12.5	ug/L
Zinc	5.00	10.0	20.0	ug/L

Table 1 TCLP				
Analyte	MDL	LOD	MRL	Units
Antimony	0.00500	0.00800	0.0150	mg/L
Arsenic	0.00300	0.00600	0.0100	mg/L
Barium	0.00500	0.0100	0.0400	mg/L
Cadmium	0.00100	0.00200	0.00500	mg/L
Chromium	0.00200	0.00400	0.0100	mg/L
Copper	0.00400	0.00800	0.0100	mg/L
Lead	0.00150	0.00300	0.00300	mg/L
Selenium	0.00300	0.00500	0.00600	mg/L
Silver	0.00100	0.00200	0.0100	mg/L

Table 1 Soil				
Analyte	MDL	LOD	MRL	Units
Aluminum	10.0	20.0	40.0	mg/Kg
Antimony	1.00	1.60	3.00	mg/Kg
Arsenic	0.600	1.20	2.00	mg/Kg
Barium	1.00	2.00	8.00	mg/Kg
Beryllium	0.200	0.400	1.00	mg/Kg
Boron	2.00	4.00	6.00	mg/Kg
Cadmium	0.200	0.400	1.00	mg/Kg
Calcium	200	400	1000	mg/Kg
Chromium	0.400	0.800	2.00	mg/Kg
Cobalt	1.00	2.00	2.50	mg/Kg
Copper	0.800	1.60	2.00	mg/Kg
Iron	6.00	12.0	20.0	mg/Kg
Lead	0.300	0.600	0.600	mg/Kg
Magnesium	200	600	1000	mg/Kg
Manganese	0.600	1.20	3.00	mg/Kg
Molybdenum	1.00	2.00	3.00	mg/Kg
Nickel	0.600	1.20	2.00	mg/Kg
Potassium	200	600	1000	mg/Kg
Selenium	0.600	1.00	1.20	mg/Kg
Silver	0.200	0.400	2.00	mg/Kg
Sodium	200	600	1000	mg/Kg
Thallium	0.600	0.800	1.60	mg/Kg
Tin	2.00	4.00	6.00	mg/Kg
Titanium	1.00	2.00	3.00	mg/Kg
Vanadium	1.00	2.00	2.50	mg/Kg
Zinc	1.00	2.00	4.00	mg/Kg

TABLE 1A

METAL	WAVELENGTH
Aluminum	396.1
Antimony	206.8
Arsenic	189.0
Barium	233.5
Beryllium	313.0
Boron	249.7
Cadmium	228.8
Calcium	317.9
Chromium	267.7
Cobalt	228.6
Copper	324.7
Iron	261.1
Lead	220.3
Magnesium	279.0
Manganese	257.6
Molybdenum	202.0
Nickel	231.6
Potassium	766.4
Selenium	196.0
Silver	328.0
Sodium	589.5
Strontium	421.5
Thallium	190.8
Tin	189.9
Titanium	334.9
Vanadium	292.4
Zinc	206.2

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Interference Check	<ul style="list-style-type: none"> once per calibration 	<ul style="list-style-type: none"> IFA less than LOD if not verified contamination of standard. IFB must be within $\pm 20\%$. 	<ul style="list-style-type: none"> Check IEC corrections for metals in the IFA.
Calibration Curve	<ul style="list-style-type: none"> Prior to analyzing any samples A minimum of a blank and 3-points for linear fits client specific requirement or a blank and high standard. Low standard at the RL level run against the curve within 20% initially and within 30% for subsequent analysis (6010C). 	<ul style="list-style-type: none"> Linear calibration Corr. of 0.998 Must follow curve processing requirements from SOP QS08 	<ul style="list-style-type: none"> Re-evaluate curve mix and makeup Re-run curve Check instrument for maintenance needs Re-prepare the curve standards <p>Samples cannot be analyzed until there is a passing calibration</p>
ICB	At the beginning of every sequence	Must meet the $\leq \pm \text{LOD}$ for DOD or $< 2 \times \text{MDL}$	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	<ul style="list-style-type: none"> Must be in the range 90 to 110% for 6010B&C, or 95 to 115% for 200.7. 	<ul style="list-style-type: none"> Re-analyze an ICV from a different source Re-prepare and re-analyze the ICV Re-calibrate and verify standard preps and sources
CCV	<ul style="list-style-type: none"> At the beginning of every sequence For every 10-client samples 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
Closing CCV	<ul style="list-style-type: none"> At the end of every sequence 	<ul style="list-style-type: none"> Must be in the range 90 to 110% 	<ul style="list-style-type: none"> Samples must be reanalyzed if possible, if not samples are flagged with a "Q".
BLK	One per prep batch	<ul style="list-style-type: none"> Must be less than $\frac{1}{2} \pm \text{RL}$. 	<ul style="list-style-type: none"> Re-analysis to confirm the positive value Ascertain if there are any samples within the batch that meet the MB criteria and provide the information for the decision makers If results are between the LOD or RL/LOQ, then assess the data and notify the PM for further action Re-prepare of samples associated with the MB NCR will be required for data reported Final Report data flagging will be required

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
BS	One per prep batch	Must be in the range of 80 to 120% for 6010B, DOD; or 85 to 115% for 200.7.	<ul style="list-style-type: none"> • Rerun to confirm problem. • All samples associated with the LCS must be re-digested, reanalyzed if possible. • NCR will be required for data reported • If samples cannot be re-digested or re-analyzed Final Report data flagging will be required
MS	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
MSD	One per prep batch	Must be in the range of 80 to 120%	Final Report data flagging will be required
Sample Duplicate	One per prep batch	20%	Flag samples
Post Digestion Spike	One per batch	±25% for DOD/6010B, ±20% 6010C	If possible MSA required, Flag samples
DOC Study	<ul style="list-style-type: none"> • Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples 	<ul style="list-style-type: none"> • Must meet the criteria of the BS for average accuracy 	<ul style="list-style-type: none"> • Re-prep and / or • Re-analysis
MDL Study	Once per year		
LOD Verification	Every quarter		
LOQ Verification	Every quarter		
Linear Dynamic Range Study (LDR)	Every six months		

Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were dilution factors applied correctly
5. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
6. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
7. Were proper data qualifiers applied to the data in LIMS
8. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST Sample Number(s):				
Batch Number(s):				
Method: 6010B or 6010C (ICP)				

QA/QC Item	Yes	No	NA	Second Level Review
1. Were samples analyzed within USACE holding times?	_____	_____	_____	_____
2. Was initial calibration curve QC criteria met?	_____	_____	_____	_____
3. Was all continuing calibration criteria in control?	_____	_____	_____	_____
4. Did any sample exceed the highest calibration standard? (If yes, were appropriate dilutions made to generate samples concentration within calibration range?)	_____	_____	_____	_____
5. Did BS or blank spike meet control limits?	_____	_____	_____	_____
6. Did MS/MSD meet control limits?	_____	_____	_____	_____
7. Was the preparation (Method) Blank (BLK) below the project required detection limits?	_____	_____	_____	_____
8. Did you return samples back to cold storage immediately after use?	_____	_____	_____	_____
9. Was hot plate temperature monitored/documented and did you apply the thermometer correction factor?	_____	_____	_____	_____
10. Sample preparation information is correct and complete.	_____	_____	_____	_____
11. Analytical results are correct and complete.	_____	_____	_____	_____
12. The appropriate SOP's have been used and followed.	_____	_____	_____	_____
14. "Raw data" including all manual integration's have been correctly interpreted.	_____	_____	_____	_____
15. "Special" sample preparation and analytical requirements have been met.	_____	_____	_____	_____
16. Documentation complete (e.g., all anomalies in the analytical sequence have been documented, corrective action forms are complete.	_____	_____	_____	_____

Comments on any "No" response:

Analyst: _____ Date: _____

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 201 REVISION #: 21 EFFECTIVE DATE: 20110516

**GC/MS SEMIVOLATILES and LOW-CONCENTRATION PAHs
BY EPA METHOD 625 AND SW846 METHOD 8270C AND 8270D
INCLUDING ADDITIONAL APPENDIX IX COMPOUNDS**

APPROVALS:

Lab Director:  Date: 5/16/11

Data Quality Manager:  Date: 5/16/11

Section Supervisor:  Date: 05/16/11

Changes Summary

Revision 21, 05/16/2011

- The SOP is an update from Revision 20 dated 4/13/2010
- A requirement to record the amount and ID of the internal standard used was added to section 14.3.

Revision 20, 4/13/10

- The SOP is an update from Revision 19 dated 4/11/2010
- The SOP is formatted to simplify the text and place all method/program specifications in the SOP tables.

Revision 19, 4/11/10

- The SOP is an update from Revision 18 dated 9/16/08
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DOD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DOD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1.0 Identification of the Test Method

This SOP is based primarily on SW-846 Methods 8000B/8000C/8270C/8270D. Methods *Federal Register* Method 625 and CLP Method for Semi-volatiles have also been used in the development of this SOP.

2.0 Applicable Matrix or Matrices

This SOP is used for the analysis of semi-volatile organic compounds (including low concentration PAHs) in a variety of matrices (soils, sediments, waters, etc.).

3.0 Detection Limits – Reporting Limits

See Table 1

4.0 Scope of Application, Including Components to Be Analyzed

4.1 Each parameter that is routinely analyzed and reported under the scope of this SOP is listed in the Appendix of this SOP. This table also lists the associated Detection Limit, Limit of Detection and Reporting Limit (also defined as the Limit of Quantitation).

4.2 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

5.1 After sample preparation using the appropriate extraction technique, the sample is introduced into the GC/MS using direct injection. The analytes are separated in the gas chromatograph by a combination of the temperature program, the pressure program and the capillary column. The analytes are then detected by the mass spectrometer. Analytes are identified by comparing the mass spectra of known standards with the mass spectra from the sample. Analytes are quantitated relative to known standards using the internal standard method.

6.0 Definitions –

Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7.0 Interferences

7.1 All raw data (samples & QC) must be evaluated for interferences. If contamination occurs, determine whether the source of interference is in the preparation or clean-up of the samples and take corrective action to eliminate the problem.

7.2 Contamination by carryover can occur when samples of high-concentration and low-concentration are analyzed sequentially. To reduce carryover, the sample syringe must be rinsed with solvent between injections. If an unusually high sample is detected, a solvent blank should be analyzed for cross contamination or the subsequent sample should be evaluated for cross-contamination.

8.0 Safety

8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed lab-wide.

- 8.2 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.
- 8.3 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- 8.4 MSDS sheets are available for all reagents and standards which have been purchased. These are located on the bookshelf outside the office supply storage room.

9.0 Equipment & Supplies

- a HP 5890/6890/7890GC complete with electronic pressure control and temperature programmable gas chromatograph suitable for split-less injection.
- b Column: RTX-5MS (or equivalent) 30 m x 0.25 mm I.D. x 0.25 μ m film thickness fused silica capillary column.
- c HP 5971/5973/5975 mass spectrometer capable of scanning from 35 to 500 amu every second or less, using 70 volts electron energy in electron impact ionization mode. The mass spectrometer is capable of producing a mass spectrum for decafluorotriphenylphosphine, DFTPP, which meets all the tuning criteria of the EPA methods.
- d HP 7673/7683 autosampler capable of reproducibility from one injection to another proven by meeting QC and calibration criteria.
- e HP GC/MS interface that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.
- f Acquisition Software: HP Chemstation system is interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.
- g Data Processing Software: Target DB on Windows NT server data system is interfaced to the HP Chemstation. The system accepts and stores acquired data. It plots by extracted ion current profile (EICP). The system is also capable of integrating the abundances in any EICP between specified times or scan-number limits.
- h Micro syringes – gas tight 5 μ L and larger.
- i Liners – 2mm or 4mm single goose-neck.
- j Septa 11mm.
- k Seals- dual vespel stainless steel or gold plated 0.8mm.
- l Vials- 2ml and larger amber.
- m Volumetric flasks- 10ml and larger class A with glass stopper.

10.0 Reagents and Standards –

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory.
- 10.2 Reagent grade chemicals shall be used in all tests unless otherwise specified. 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.

- 10.3 Methylene chloride (Please read SOP-336 before handling this solvent in our laboratory.) – Trace analysis grade.
- 10.4 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label and recorded on the certificate of analysis sheet. The date they are opened is noted on the label and recorded in LIMS. Each standards label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. All stocks and standards are stored in the freezer at a temperature of $-15^{\circ}\text{C} \pm 5^{\circ}\text{C}$ from the date they are received/prepared. Standards are brought to room temperature before being used to make standards. Sonication is used if precipitation is observed after bringing to room temperature. The refrigerator and freezer temperature are monitored daily with an annually calibrated thermometer and recorded with calibration correction in the Extraction temperature/calibration logbook.
- 10.5 Individual standard makeup is recorded in LIMS with specific details concerning the standard being used, concentration, amount, solvent and expiration date.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and table 3-1 of the Empirical Laboratories' Quality Assurance Manual include details concerning sample preservation, containers and handling of semi-volatile samples and extracts. All water and soil samples are stored in the appropriate walk-in coolers at a temperature of 4°C . All extracts are stored in the Hobart in the Extraction lab at a temperature of 4°C . Water samples have a holding time of 7 days from date of sampling while soil samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). Extracts have 40 days from date of extraction to be analyzed.

12.0 Quality Control

- 12.1 Internals - All samples and QC are spiked with internal standards prior to analysis.
- 12.2 Surrogates - All samples and QC are spiked with surrogates prior to extraction. See **Table 2** for criteria and corrective action.
- 12.3 LCS Sample - The LCS is extracted 1 per extraction batch of up to 20 samples to provide accuracy results. It is spiked using an alternate source or lot number than the calibration standards. See **Table 2** for criteria and corrective action.
- 12.4 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2** for criteria and corrective action.
- 12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2** for criteria and corrective action. Some factors that may affect MS/MSD results are:
 - 12.5.1 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.
 - 12.5.2 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was four or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.

- 12.5.3 MS vs. MSD - If a spiked compound has a similar problem in both the MS and MSD and is not traced to a method problem, no further action may be necessary other than the generation of a non-conformance report to document the problem.
- 12.5.4 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.
- 12.6 Demonstration of Capability (DOC) – Each new analyst must complete a demonstration of capability by analyzing four LCSs with acceptable precision and accuracy. This also must be done when a new instrument is installed or a significant change to the method has been made.

13.0 Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.2 Initial Calibration - An initial multi-point calibration curve must be analyzed and shown to meet the initial calibration criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. The lowest standard must be less than or equal to the reported quantitation limit and the highest standard must not exceed the linear range of the detector. Generally, levels for the curve range from 1.0ug/mL to 100ug/mL for regular SVOCs and 0.1µg/mL to 50µg/mL for low-concentration PAHs.. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.
- 13.3 Initial Calibration Verification (ICV) - A second source standard at the continuing calibration verification (CCV) level must be analyzed and calculated against the initial calibration curve, then shown to meet the ICV criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.
- 13.4 Continuing Calibration Verification (CCV) - Every 12 hours, a CCV must be analyzed and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analyses may be performed. See **Table 2** for criteria and corrective action. For ICV standard preparation, refer to LIMS. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, reason indicated and dated with the quantitation report and chromatogram. All integrations are second-checked for acceptability by a senior analyst. Refer to SOP-QS07 for guidance.

14.0 Procedure

Prior to analysis the samples are prepared for chromatography using the appropriate sample preparation and clean up methods (generally SW-846 methods 3510, 3520, 3541, 3546 3550, 3580, EPA method 625 or CLP).

- 14.1 Chromatographic conditions: Refer to corresponding instrument maintenance log for current gas chromatograph and mass spectrometer conditions.
- 14.2 Tuning - Prior to any calibration or analysis, DFTPP tuning criteria must be met for a 50 ng injection of the tuning standard. The injection port performance compounds (pentachlorophenol, benzidine and 4,4'-DDT) are also injected to verify the performance of the injection port. See **Table 2** for criteria and corrective action.
- 14.3 Extracts - Prior to analysis, 1.0 mL extracts are prepared by verifying volume and spiking with 20uL of the internal standard solution. Record the amount and ID of internal standard spiked on the sequence log printed from the LIMS.

- 14.5 Instrument sequence-The instrument sequence log is filled out prior to sample analyses. An example of a typical instrument sequence log follows:

1-SEQ-TUN1 (12:00 am)
2-SEQ-CCV1
3-SEQ-BS1
4-SEQ-BLK1
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-SEQ-MS1
15-SEQ-MSD1
16-SEQ-TUN2 (12:00pm - 12 hours since last DFTPP/CCV)
17-SEQ-CCV2
18-Sample
19-Sample
20-Sample

- 14.6 Data Reduction/Evaluation - Each sample analysis sequence is documented using the computer run log generated on the Chemstation. This run log is signed, dated and paginated then placed in a 3 ring binder for that instrument. After the sample has been analyzed, the data is processed through Target DB on the Windows NT data system. The following must be checked to determine if the sample will need reanalysis or dilution. Criteria and corrective action are found in Table 2. Formal data evaluation is detailed in SOP QS05 and documented using the Analyst Data Review Checklist (see Appendix). Manual integration guidance is found in SOP QS07.

14.6.1 Internal Standard Area Counts and Retention Times

- 14.6.2 Surrogate Recoveries and Retention Times
- 14.6.3 Analyte concentration.
- 14.6.4 Analyte identification based on spectrum and retention time.
- 14.6.5 Analyte quantitation verification.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 “General and Commonly used Laboratory Calculations” provides details on general calculations used throughout the laboratory.

15.2 The RF is calculated as follows:

$$RF = \frac{A_s \times C_{is}}{A_{is} \times C_s}$$

where:

A_s = Peak area (or height) of the analyte or surrogate.

A_{is} = Peak area (or height) of the internal standard.

C_s = Concentration of the analyte or surrogate.

C_{is} = Concentration of the internal standard.

15.2 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where:

Calculated concentration is determined from the initial calibration.

Theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV RF} - \text{Average RF}) * 100}{\text{Average RF}}$$

where:

CCV RF is the response factor from the analysis of the verification standard

Average RF is the average of the response factors from the initial calibration.

15.3 Concentration in water samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/mL, which is equivalent to $\mu\text{g/L}$.]

$$\text{Concentration } (\mu\text{g/L}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(RF)(V_s)(1000)}$$

where:

A_s = Area (or height) of the peak for the analyte in the sample.

A_{is} = Area (or height) of the peak for the internal standard.

C_{is} = Concentration of the internal standard in the volume extracted in $\mu\text{g/L}$.

D = Dilution factor, if the sample was diluted prior to analysis. If no dilution was made, D = 1. The dilution factor is always dimensionless.

V_i = Volume of the extract injected (μL). The nominal injection volume for samples and calibration standards must be the same.

$\overline{\text{RF}}$ = Mean response factor from the initial calibration.

V_s = Volume of the aqueous sample extracted (mL). If units of liters are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

- 15.4 Concentration in non-aqueous samples is calculated as follows: [Note: Using the units specified here for these terms will result in a concentration in units of ng/g, which is equivalent to $\mu\text{g}/\text{kg}$.]

$$\text{Concentration } (\mu\text{g}/\text{kg}) = \frac{(A_s)(C_{is})(D)(V_i)}{(A_{is})(\overline{\text{RF}})(W_s)(1000)}$$

where: A_s ,

A_{is} , C_{is} , D , and $\overline{\text{RF}}$ are the same as for aqueous samples, and

W_s = Weight of sample extracted (g). Either a dry weight or wet weight may be used, depending upon the specific application of the data. If units of kilograms are used for this term, multiply the results by 1000.

The 1000 in the denominator represents the number of μL in 1 mL. If the injection (V_i) is expressed in mL, then the 1000 may be omitted.

- 15.3 Any questions left unanswered by this SOP should be clarified by reading the referenced method. If questions still remain unanswered, check with the Section Manager, Technical Director and/or Data Quality Manager.

16.0 Method Performance

See SOP QS08 and Table 2 for criteria and corrective actions associated to the following method performance items:

- 16.1 Method Detection Limit Study or Detection Limit Determination
- 16.2 Limit of Detection Verification
- 16.3 Limit of Quantitation or Reporting Limit Verification
- 16.4 Demonstration of Capability (DOC)
- 16.5 PT Studies

17.0 Pollution Prevention

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

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2** within this SOP also lists corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

40 CFR, Part 136; Appendix A

Test Methods for Evaluating Solid Waste, SW-846

National Environmental Laboratory Accreditation Conference; CH. 5, 2003

USACE, EM 200-1-3; Appendix 1; Shell, 2/2001

DOD Quality Systems Manual for Environmental Laboratories,

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters with the applicable DL(MDL)/LOD/LOQ(MRL).

22.2 Table 2, QA/QC summary table

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist(s)

22.5 Table 5, 625 QC Limits

22.6 Table 6, Standards Used

22.7 Table 7, INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis

22.8 Table 8, LOW CONCENTRATION PAH INTERNAL STANDARD/SURROGATE SPECIFICATIONS

22.9 Figure 1, Tailing Factor Calculation

22.10 Table 9, DFTPP Tuning Criteria

TABLE 1

Analyte (Water)	DL	LOD	MRL/LOQ	Units
1,1'-Biphenyl	1.25	2.50	5.00	ug/L
1,2,4,5-Tetrachlorobenzene	1.25	2.50	5.00	ug/L
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/L
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/L
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/L
2,3,4,6-Tetrachlorophenol	1.25	2.50	5.00	ug/L
2,4,5-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4,6-Trichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dichlorophenol	1.25	2.50	5.00	ug/L
2,4-Dimethylphenol	5.00	10.0	20.0	ug/L
2,4-Dinitrophenol	12.5	25.0	50.0	ug/L
2,4-Dinitrotoluene	1.25	2.50	5.00	ug/L
2,6-Dinitrotoluene	1.25	2.50	5.00	ug/L
2-Chloronaphthalene	1.25	2.50	5.00	ug/L
2-Chlorophenol	1.25	2.50	5.00	ug/L
2-Methylnaphthalene	1.25	2.50	5.00	ug/L
2-Methylphenol	1.25	2.50	5.00	ug/L
2-Nitroaniline	5.00	10.0	20.0	ug/L
2-Nitrophenol	1.25	2.50	5.00	ug/L
3,3'-Dichlorobenzidine	1.25	2.50	5.00	ug/L
3-Nitroaniline	5.00	10.0	20.0	ug/L
4,6-Dinitro-2-methylphenol	5.00	10.0	20.0	ug/L
4-Bromophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Chloro-3-methylphenol	1.25	2.50	5.00	ug/L
4-Chloroaniline	1.25	2.50	5.00	ug/L
4-Chlorophenyl phenyl ether	1.25	2.50	5.00	ug/L
4-Methylphenol	1.25	2.50	5.00	ug/L
4-Nitroaniline	5.00	10.0	20.0	ug/L
4-Nitrophenol	5.00	10.0	20.0	ug/L
Acenaphthene	1.25	2.50	5.00	ug/L
Acenaphthylene	1.25	2.50	5.00	ug/L
Acetophenone	1.25	2.50	5.00	ug/L
Anthracene	1.25	2.50	5.00	ug/L
Atrazine	1.25	2.50	5.00	ug/L
Benzaldehyde	1.25	2.50	5.00	ug/L
Benzo (a) anthracene	1.25	2.50	5.00	ug/L
Benzo (a) pyrene	1.25	2.50	5.00	ug/L
Benzo (b) fluoranthene	1.25	2.50	5.00	ug/L
Benzo (g,h,i) perylene	1.25	2.50	5.00	ug/L
Benzo (k) fluoranthene	1.25	2.50	5.00	ug/L
Bis(2-chloroethoxy)methane	1.25	2.50	5.00	ug/L
Bis(2-chloroethyl)ether	1.25	2.50	5.00	ug/L
Bis(2-chloroisopropyl)ether	1.25	2.50	5.00	ug/L
Bis(2-ethylhexyl)phthalate	1.25	2.50	5.00	ug/L
Butyl benzyl phthalate	1.25	2.50	5.00	ug/L
Caprolactam	1.25	2.50	5.00	ug/L
Carbazole	1.25	2.50	5.00	ug/L
Chrysene	1.25	2.50	5.00	ug/L
Dibenz (a,h) anthracene	1.25	2.50	5.00	ug/L
Dibenzofuran	1.25	2.50	5.00	ug/L
Diethyl phthalate	1.25	2.50	5.00	ug/L
Dimethylphthalate	1.25	2.50	5.00	ug/L
Di-n-butyl phthalate	1.25	2.50	5.00	ug/L

Table 1 (Continued)

Analyte (Water)	DL	LOD	MRL/LOQ	Units
Di-n-octyl phthalate	1.25	2.50	5.00	ug/L
Fluoranthene	1.25	2.50	5.00	ug/L
Fluorene	1.25	2.50	5.00	ug/L
Hexachlorobenzene	1.25	2.50	5.00	ug/L
Hexachlorobutadiene	1.25	2.50	5.00	ug/L
Hexachlorocyclopentadiene	1.25	2.50	5.00	ug/L
Hexachloroethane	1.25	2.50	5.00	ug/L
Indeno (1,2,3-cd) pyrene	1.25	2.50	5.00	ug/L
Isophorone	1.25	2.50	5.00	ug/L
Naphthalene	1.25	2.50	5.00	ug/L
Nitrobenzene	1.25	2.50	5.00	ug/L
N-Nitrosodi-n-propylamine	1.25	2.50	5.00	ug/L
N-Nitrosodiphenylamine	1.25	2.50	5.00	ug/L
Pentachlorophenol	5.00	10.0	20.0	ug/L
Phenanthrene	1.25	2.50	5.00	ug/L
Phenol	1.25	2.50	5.00	ug/L
Pyrene	1.25	2.50	5.00	ug/L
Analyte (Soil)	DL	LOD	MRL/LOQ	Units
1,1'-Biphenyl	83.3	167	333	ug/Kg
1,2,4,5-Tetrachlorobenzene	83.3	167	333	ug/Kg
1,2,4-Trichlorobenzene	83.3	167	333	ug/Kg
1,2-Dichlorobenzene	83.3	167	333	ug/Kg
1,3-Dichlorobenzene	83.3	167	333	ug/Kg
1,4-Dichlorobenzene	83.3	167	333	ug/Kg
2,3,4,6-Tetrachlorophenol	83.3	167	333	ug/Kg
2,4,5-Trichlorophenol	83.3	167	333	ug/Kg
2,4,6-Trichlorophenol	83.3	167	333	ug/Kg
2,4-Dichlorophenol	83.3	167	333	ug/Kg
2,4-Dimethylphenol	333	667	1330	ug/Kg
2,4-Dinitrophenol	833	1670	3330	ug/Kg
2,4-Dinitrotoluene	83.3	167	333	ug/Kg
2,6-Dinitrotoluene	83.3	167	333	ug/Kg
2-Chloronaphthalene	83.3	167	333	ug/Kg
2-Chlorophenol	83.3	167	333	ug/Kg
2-Methylnaphthalene	83.3	167	333	ug/Kg
2-Methylphenol	83.3	167	333	ug/Kg
2-Nitroaniline	333	667	1330	ug/Kg
2-Nitrophenol	83.3	167	333	ug/Kg
3,3'-Dichlorobenzidine	83.3	167	333	ug/Kg
3-Nitroaniline	333	667	1330	ug/Kg
4,6-Dinitro-2-methylphenol	833	1670	3330	ug/Kg
4-Bromophenyl phenyl ether	83.3	167	333	ug/Kg
4-Chloro-3-methylphenol	83.3	167	333	ug/Kg
4-Chloroaniline	83.3	167	333	ug/Kg
4-Chlorophenyl phenyl ether	83.3	167	333	ug/Kg
4-Methylphenol	83.3	167	333	ug/Kg
4-Nitroaniline	333	667	1330	ug/Kg
4-Nitrophenol	333	667	1330	ug/Kg
Acenaphthene	83.3	167	333	ug/Kg
Acenaphthylene	83.3	167	333	ug/Kg
Acetophenone	83.3	167	333	ug/Kg
Anthracene	83.3	167	333	ug/Kg
Atrazine	83.3	167	333	ug/Kg
Benzaldehyde	83.3	167	333	ug/Kg
Benzo (a) anthracene	83.3	167	333	ug/Kg

Table 1 (Continued)

Analyte (Soil)	DL	LOD	MRL/LOQ	Units
Benzo (a) pyrene	83.3	167	333	ug/Kg
Benzo (b) fluoranthene	83.3	167	333	ug/Kg
Benzo (g,h,i) perylene	83.3	167	333	ug/Kg
Benzo (k) fluoranthene	83.3	167	333	ug/Kg
Bis(2-chloroethoxy)methane	83.3	167	333	ug/Kg
Bis(2-chloroethyl)ether	83.3	167	333	ug/Kg
Bis(2-chloroisopropyl)ether	83.3	167	333	ug/Kg
Bis(2-ethylhexyl)phthalate	83.3	167	333	ug/Kg
Butyl benzyl phthalate	83.3	167	333	ug/Kg
Caprolactam	83.3	167	333	ug/Kg
Carbazole	83.3	167	333	ug/Kg
Chrysene	83.3	167	333	ug/Kg
Dibenz (a,h) anthracene	83.3	167	333	ug/Kg
Dibenzofuran	83.3	167	333	ug/Kg
Diethyl phthalate	83.3	167	333	ug/Kg
Dimethylphthalate	83.3	167	333	ug/Kg
Di-n-butyl phthalate	83.3	167	333	ug/Kg
Di-n-octyl phthalate	83.3	167	333	ug/Kg
Fluoranthene	83.3	167	333	ug/Kg
Fluorene	83.3	167	333	ug/Kg
Hexachlorobenzene	83.3	167	333	ug/Kg
Hexachlorobutadiene	83.3	167	333	ug/Kg
Hexachlorocyclopentadiene	83.3	167	333	ug/Kg
Hexachloroethane	83.3	167	333	ug/Kg
Indeno (1,2,3-cd) pyrene	83.3	167	333	ug/Kg
Isophorone	83.3	167	333	ug/Kg
Naphthalene	83.3	167	333	ug/Kg
Nitrobenzene	83.3	167	333	ug/Kg
N-Nitrosodi-n-propylamine	83.3	167	333	ug/Kg
N-Nitrosodiphenylamine	83.3	167	333	ug/Kg
Pentachlorophenol	333	667	1330	ug/Kg
Phenanthrene	83.3	167	333	ug/Kg
Phenol	83.3	167	333	ug/Kg
Pyrene	83.3	167	333	ug/Kg
Analyte Low PAH (Water)	DL	LOD	MRL/LOQ	Units
1-Methylnaphthalene	0.0500	0.100	0.200	ug/L
2-Methylnaphthalene	0.0500	0.100	0.200	ug/L
Acenaphthene	0.0500	0.100	0.200	ug/L
Acenaphthylene	0.0500	0.100	0.200	ug/L
Anthracene	0.0500	0.100	0.200	ug/L
Benzo (a) anthracene	0.0500	0.100	0.200	ug/L
Benzo (a) pyrene	0.0500	0.100	0.200	ug/L
Benzo (b) fluoranthene	0.0500	0.100	0.200	ug/L
Benzo (g,h,i) perylene	0.0500	0.100	0.200	ug/L
Benzo (k) fluoranthene	0.0500	0.100	0.200	ug/L
Chrysene	0.0500	0.100	0.200	ug/L
Dibenz (a,h) anthracene	0.0500	0.100	0.200	ug/L
Fluoranthene	0.0500	0.100	0.200	ug/L
Fluorene	0.0500	0.100	0.200	ug/L
Indeno (1,2,3-cd) pyrene	0.0500	0.100	0.200	ug/L
Naphthalene	0.0500	0.100	0.200	ug/L
Phenanthrene	0.0500	0.100	0.200	ug/L
Pyrene	0.0500	0.100	0.200	ug/L
Analyte Low PAH (Soil)	DL	LOD	MRL/LOQ	Units
1-Methylnaphthalene	1.67	3.33	6.67	ug/Kg

Table 1 (Continued)

Analyte Low PAH (Soil)	DL	LOD	MRL/LOQ	Units
2-Methylnaphthalene	1.67	3.33	6.67	ug/Kg
Acenaphthene	1.67	3.33	6.67	ug/Kg
Acenaphthylene	1.67	3.33	6.67	ug/Kg
Anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) anthracene	1.67	3.33	6.67	ug/Kg
Benzo (a) pyrene	1.67	3.33	6.67	ug/Kg
Benzo (b) fluoranthene	1.67	3.33	6.67	ug/Kg
Benzo (g,h,i) perylene	1.67	3.33	6.67	ug/Kg
Benzo (k) fluoranthene	1.67	3.33	6.67	ug/Kg
Chrysene	1.67	3.33	6.67	ug/Kg
Dibenz (a,h) anthracene	1.67	3.33	6.67	ug/Kg
Fluoranthene	1.67	3.33	6.67	ug/Kg
Fluorene	1.67	3.33	6.67	ug/Kg
Indeno (1,2,3-cd) pyrene	1.67	3.33	6.67	ug/Kg
Naphthalene	1.67	3.33	6.67	ug/Kg
Phenanthrene	1.67	3.33	6.67	ug/Kg
Pyrene	1.67	3.33	6.67	ug/Kg
Analyte (TCLP)	DL	LOD	MRL/LOQ	Units
1,4-Dichlorobenzene	0.00125	0.00250	0.00500	mg/L
2,4,5-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4,6-Trichlorophenol	0.00125	0.00250	0.00500	mg/L
2,4-Dinitrotoluene	0.00125	0.00250	0.00500	mg/L
2-Methylphenol	0.00125	0.00250	0.00500	mg/L
3-Methylphenol	0.00125	0.00250	0.00500	mg/L
4-Methylphenol	0.00125	0.00250	0.00500	mg/L
Hexachlorobenzene	0.00125	0.00250	0.00500	mg/L
Hexachlorobutadiene	0.00125	0.00250	0.00500	mg/L
Hexachloroethane	0.00125	0.00250	0.00500	mg/L
Nitrobenzene	0.00125	0.00250	0.00500	mg/L
Pentachlorophenol	0.0050	0.0100	0.0200	mg/L
Pyridine	0.00125	0.00250	0.00500	mg/L

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specific criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	NA.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C of DoD QSM 4.1. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Tuning	Prior to ICAL and at the beginning of each 12-hour period.	Refer to table 8 of this SOP.	Retune instrument and verify. Rerun affected samples.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be accepted without a valid tune.
Breakdown check (DDT Method 8270 only)	At the beginning of each 12-hour period, prior to analysis of samples.	Degradation \leq 20% for DDT. Benzidine and pentachlorophenol should be present at their normal responses, and should not exceed a tailing factor of 2. [Method 625 – benzidine and pentachlorophenol tailing limits are 3 and 5, respectively, when benzidine or acids are target analytes. Benzidine tailing is specific to benzidine analysis and pentachlorophenol tailing is specific to acid analyte analyses according to 625.]	Correct problem then repeat breakdown checks.	Flagging criteria are not appropriate.	No samples shall be run until degradation \leq 20%. Not applied when low concentration PAHs are the only target analytes.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	<p>1. Average response factor (RF) for SPCCs: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] Note 1: See table 4 of 8270D SPCC analytes and limits. Note 2: ≥ 0.050 for all low-level PAHs</p> <p>2. RSD for RFs for CCCs: SVOCs $\leq 30\%$ and one option below: Option 1: RSD for each analyte $\leq 15\%$; [$\leq 20\%$ for non-DoD 8270D; or, $\leq 35\%$ for non-DoD 625] Option 2: linear least squares regression $r \geq 0.995$ or $r^2 \geq 0.990$; [$r \geq 0.990$ for non-DoD analyses] Option 3: non-linear regression—coefficient of determination (COD) $r^2 \geq 0.990$ (6 points shall be used for second order, 7 points shall be used for third order).</p>	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD projects.
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within $\pm 20\%$ of true value [$\pm 25\%$ for non-DoD 8270C; or, $\pm 30\%$ for non-DoD 8270D]	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Retention time window position establishment for each analyte and surrogate	Once per ICAL.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the sequence CCV is used.	NA.	NA.	

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Evaluation of relative retention times (RRT)	With each sample.	RRT of each target analyte within ± 0.06 RRT units. Note - retention times may be updated based on the CCV to account for minor performance fluctuations or after routine system maintenance (such as column clipping).	Correct problem, then rerun ICAL.	Flagging criteria are not appropriate.	With each sample, the RRT shall be compared with the most recently updated RRT. If the RRT has changed by more than ± 0.06 RRT units since the last update, this indicates a significant change in system performance and the laboratory must take appropriate corrective actions as required by the method and rerun the ICAL to reestablish the retention times.
Continuing calibration verification (CCV)	Daily before sample analysis and every 12 hours of analysis time.	1. Average RF for SPCCs: SVOCs ≥ 0.050 [2,4-dinitrophenol, hexachlorocyclopentadiene, N-Nitrosodi-n-propylamine, 4-nitrophenol] Note 1: See table 4 of 8270D SPCC analytes and limits. Note 2: ≥ 0.050 for all low-level PAHs 2. %Difference/Drift for all target compounds and surrogates: SVOCs $\leq 20\%D$ (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [$\pm 20\%$ for CCCs only non-DoD 8270C]	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV.	If reanalysis cannot be performed, data should be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since last acceptable CCV. [For non-DoD 8270C, if CCCs exceed, evaluate all analytes for 20%D and qualify as above]	Problem should be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed, holding time has been exceeded or client has approved reporting.
Internal standards verification	Every field sample, standard, and QC sample.	Retention time ± 30 seconds from retention time of the midpoint standard in the ICAL or daily CCV; EICP area within -50% to +100% of ICAL midpoint standard or daily CCV.	Inspect mass spectrometer and GC for malfunctions. Reanalysis of samples analyzed while system was malfunctioning is mandatory.	If corrective action fails in field samples, apply qualifier to analytes associated with the non-compliant IS. Flagging criteria are not appropriate for failed standards.	Sample results are not acceptable without a valid IS verification.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Method blank	One per preparatory batch.	No analytes detected > ½ RL/LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results. For common laboratory contaminants, no analytes detected > RL/LOQ.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
LCS containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by client or DoD (appendix G), if available. AFCEE 4.0.02 limits are applied for low concentration PAHs as they are not addressed by DoD. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery. Low concentration PAH limits	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix	Use LCS criteria, above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: $RPD \leq 30\%$ or client specified limit (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 625/8270) (continued)

QC Check	Minimum Frequency	Acceptance Criteria			Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	Surrogate	Water	Solid	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply qualifier to all associated analytes if acceptance criteria are not met. For acid surrogate, qualify acid analytes, for base/neutral surrogates, qualify base/neutral analytes.	
		Nitrobenzene-d5	40-110	35-100			
		2-Fluorobiphenyl	50-110	45-105			
		Terphenyl-d14	50-135	30-125			
		Phenol-d6	10-115	40-100			
		2-Fluorophenol	20-110	35-105			
		2,4,6-Tribromophenol	40-125	35-125			
		QC acceptance criteria specified by DoD (above) or Client. Low PAH surrogate limits are 14%-129% soil and 34%-167% water. Otherwise, in-house control limits may be used. No limits specified for Method 625.					
Results reported between DL and LOQ	NA.	NA.			NA.	Apply J-flag to all results between DL and LOQ.	

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Table 3, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 of the SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory or senior-scientist approval for manual integrations on standards and batch QC samples
7. Was the data uploaded into LIMS via direct upload (i.e. datatool) – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 4, Data Reviewers Checklist (Prior to approving data)

1. Does the hard copy raw data (or electronic raw data) package look complete and include all data points
2. Were QA objectives met and for failures were the appropriate actions taken
3. For direct uploads to LIMS, did a subset cross check match the raw data
4. Did all the manual entries into LIMS match the raw data
5. Were there appropriate signatures and documentation on the raw data
6. Were appropriate LIMS flags used
7. Were manual integrations signed
8. Were manual integrations for calibration and QC samples approved by supervisor
9. Were manual calculations verified

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8260B/624/8270C/8270D/625 (Circle One)

QA/QC Item	Yes	No	NA	Second Review	Level
1. Is the BFB/DFTPP tune performed every 12 hours and is the tuning criteria met?					
Are the RRFs and % RSDs within QC limits for appropriate analytes for the initial calibration? Check the retention times for compounds with the same spectra. Check compounds with different conc.(e.g. m/p-xylene, ketones, etc.).					
3. Was the initial calibration curve verified by a second source calibration standard (ICV) and have criteria been met?					
4. Does the Continuing Calibration Standard (CCV) meet the criteria for the CCCs, SPCCs and/or 20%D for all analytes.					
5. Is the Method Blank run at the desired frequency and is its concentration for target analytes less than the MDLs?					
6. Are the LCS, MS, MSD within control limits and run at the desired frequency?					
7. Are all sample holding times met, analytes within calibration range, IS areas and surrogate recoveries within QC limits?					
8. Were the Method Blank, LCS, MS, MSD and samples uploaded to the LIMS and verified (at least one calculation per batch uploaded)?					

Comments on any "No" response:

Primary-Level Review: _____ Date: _____

Second-Level Review: _____ Date: _____

Table 5 - 625 QC limits

COMPOUND	SPIKE ADDED (ug/L)	SAMPLE CONCENTRATION (ug/L)	LCS CONCENTRATION (ug/L)	LCS % REC #	QC. LIMITS REC.
Acenaphthene	100.00	0.0000	100.00	100	47-145
Acenaphthylene	100.00	0.0000	100.00	100	33-145
Anthracene	100.00	0.0000	100.00	100	27-133
Benzidine	100.00	0.0000	100.00	100	D-110
Benzo(a)anthracene	100.00	0.0000	100.00	100	33-143
Benzo(b)fluoranthene	100.00	0.0000	100.00	100	24-159
Benzo(k)fluoranthene	100.00	0.0000	100.00	100	11-162
Benzo(g,h,i)perylene	100.00	0.0000	100.00	100	D-219
Benzo(a)pyrene	100.00	0.0000	100.00	100	17-163
bis(2-Chloroethoxy)meth	100.00	0.0000	100.00	100	33-184
bis(2-Chloroethyl)ether	100.00	0.0000	100.00	100	12-158
bis(2-Chloroisopropyl)e	100.00	0.0000	100.00	100	36-166
Bis(2-ethylhexyl)phthal	100.00	0.0000	100.00	100	8-158
4-Bromophenyl-phenyleth	100.00	0.0000	100.00	100	53-127
Butylbenzylphthalate	100.00	0.0000	100.00	100	D-152
4-Chloro-3-methylphenol	100.00	0.0000	100.00	100	22-147
2-Chloronaphthalene	100.00	0.0000	100.00	100	60-118
2-Chlorophenol	100.00	0.0000	100.00	100	23-134
4-Chlorophenyl-phenylet	100.00	0.0000	100.00	100	25-158
Chrysene	100.00	0.0000	100.00	100	17-168
Dibenz(a,h)anthracene	100.00	0.0000	100.00	100	D-227
1,2-Dichlorobenzene	100.00	0.0000	100.00	100	32-129
1,3-Dichlorobenzene	100.00	0.0000	100.00	100	D-172
1,4-Dichlorobenzene	100.00	0.0000	100.00	100	20-124
3,3'-Dichlorobenzidine	100.00	0.0000	100.00	100	D-262
2,4-Dichlorophenol	100.00	0.0000	100.00	100	39-135
Diethylphthalate	100.00	0.0000	100.00	100	D-114
2,4-Dimethylphenol	100.00	0.0000	100.00	100	32-119
Dimethylphthalate	100.00	0.0000	100.00	100	D-112
Di-n-butylphthalate	100.00	0.0000	100.00	100	1-118
4,6-Dinitro-2-methylphe	100.00	0.0000	100.00	100	D-181
2,4-Dinitrophenol	100.00	0.0000	100.00	100	D-191
2,4-Dinitrotoluene	100.00	0.0000	100.00	100	39-139
2,6-Dinitrotoluene	100.00	0.0000	100.00	100	50-158
Di-n-octylphthalate	100.00	0.0000	100.00	100	4-146
Fluoranthene	100.00	0.0000	100.00	100	26-137
Fluorene	100.00	0.0000	100.00	100	59-121
Hexachlorobenzene	100.00	0.0000	100.00	100	D-152
Hexachlorobutadiene	100.00	0.0000	100.00	100	24-116
Hexachlorocyclopentadie	100.00	0.0000	100.00	100	15- 70
Hexachloroethane	100.00	0.0000	100.00	100	40-113
Indeno(1,2,3-cd)pyrene	100.00	0.0000	100.00	100	D-171
Isophorone	100.00	0.0000	100.00	100	21-196
Naphthalene	100.00	0.0000	100.00	100	21-133
Nitrobenzene	100.00	0.0000	100.00	100	35-180
2-Nitrophenol	100.00	0.0000	100.00	100	29-182
4-Nitrophenol	100.00	0.0000	100.00	100	D-132
N-Nitroso-di-methylamin	100.00	0.0000	100.00	100	29- 66
N-Nitrosodiphenylamine	100.00	0.0000	100.00	100	23-100
N-Nitroso-di-n-propylam	100.00	0.0000	100.00	100	D-230
Pentachlorophenol	100.00	0.0000	100.00	100	14-176
Phenanthrene	100.00	0.0000	100.00	100	54-120
Phenol	100.00	0.0000	100.00	100	5-112
Pyrene	100.00	0.0000	100.00	100	52-115
1,2,4-Trichlorobenzene	100.00	0.0000	100.00	100	44-142
2,4,6-Trichlorophenol	100.00	0.0000	100.00	100	37-144

Table 6 - BNA STANDARDS USED

<u>base/neutral mix (2000ppm)</u>	<u>acids mix (2000ppm)</u>
bis(2-Chloroethyl)ether	2,4-Dinitrophenol
bis(2-Chloroisopropyl)ether	2-Methylphenol
1,3-Dichlorobenzene	4-Methylphenol
1,2-Dichlorobenzene	Benzoic acid
1,4-Dichlorobenzene	4,6-Dinitro-2-methylphenol
Hexachloroethane	4-Nitrophenol
N-Nitroso-di-methylamine	2,4,5-Trichlorophenol
N-Nitroso-di-n-propylamine	2,4,6-Trichlorophenol
2,4-Dinitrotoluene	Phenol
2,6-Dinitrotoluene	Pentachlorophenol
Fluorene	2-Nitrophenol
Dimethylphthalate	4-Chloro-3-methylphenol
Hexachlorocyclopentadiene	2,4-Dichlorophenol
Anthracene	2,4-Dimethylphenol
4-Bromophenyl-phenylether	Benzoic acid
Di-n-butylphthalate	
bis(2-Chloroethoxy)methane	
1,2-Diphenylhydrazine	<u>semivoa misc. mix(2000ppm)</u>
Fluoranthene	Aniline
Hexachlorobenzene	Benzyl alcohol
N-Nitrosodiphenylamine	Carbazole
Phenanthrene	4-Chloroaniline
Hexachlorobutadiene	Dibenzofuran
Isophorone	2-Methylnaphthalene
Naphthalene	2-Nitroaniline
Nitrobenzene	3-Nitroaniline
1,2,4-Trichlorobenzene	4-Nitroaniline
Acenaphthene	Pyridine
Acenaphthylene	
2-Chloronaphthalene	<u>Benzidine mix (2000ppm)</u>
4-Chlorophenyl-phenylether	Benzidine
Diethylphthalate	3,3'-Dichlorobenzidine
Benzo(a)anthracene	
Bis(2-ethylhexyl)phthalate	
Butylbenzylphthalate	
Chrysene	<u>Individual or misc. mixes (2000/5000/20,000ppm)</u>
p-(Dimethylamino)azobenzene	Caprolactam
Pyrene	Benzaldehyde
Benzo(b)fluoranthene	Atrazine
Benzo(k)fluoranthene	1,1'-Biphenyl
Benzo(g,h,i)perylene	1,4-Dioxane
Benzo(a)pyrene	1-methylnaphthalene
Dibenz(a,h)anthracene	2,6-dichlorophenol
Di-n-octylphthalate	2,3,4,6-tetrachlorophenol
Indeno(1,2,3-cd)pyrene	

<u>BNA internals (2000ppm)</u>	<u>Acid surrogate (7500ppm)</u>
1,4-Dichlorobenzene-d4 (I.S)(1)	2-Fluorophenol (S)
Naphthalene-d8 (I.S)(35)	Phenol-d6 (S)
Acenaphthene-d10 (I.S) (59)	2,4,6-Tribromophenol (S)
Phenanthrene-d10 (I.S) (79)	2,-Chlorophenol-d4 (S)
Chrysene-d12 (I.S) (92))	<u>BN surrogate (5000ppm)</u>
Perylene-d12 (I.S) (101)	Nitrobenzene-d5 (S)
	Terphenyl-d14 (S)
	2-Fluorobiphenyl (S)
	1,2-Dichlorobenzene-d4 (S)

Table 7 INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis					
COMPOUND	*I.S	Q.M	COMPOUND	*I.S	Q.M
1,4-Dichlorobenzene-d4 (I.S)(1)		152	Dimethylphthalate	59	163
Acetophenone	1	105	Hexachlorocyclopentadiene	59	237
Aniline	1	93	2,4-Dinitrophenol	59	184
Benzaldehyde	1	106	2,4-Dinitrotoluene	59	165
Benzyl alcohol	1	108	2,6-Dinitrotoluene	59	165
bis(2-Chloroethyl)ether	1	93	Fluorene	59	166
bis(2-Chloroisopropyl)ether	1	45	2-Nitroaniline	59	65
1,3-Dichlorobenzene	1	146	3-Nitroaniline	59	138
1,2-Dichlorobenzene	1	146	4-Nitroaniline	59	138
1,4-Dichlorobenzene	1	146	4-Nitrophenol	59	65
2-Methylphenol	1	108	2,4,5-Trichlorophenol	59	196
4-Methylphenol	1	108	2,4,6-Trichlorophenol	59	196
3-Methylphenol	1	108	2-Fluorobiphenyl (S)	59	172
Phenol	1	94	Phenanthrene-d10 (I.S) (79)		188
Pyridine	1	79	Anthracene	79	178
Hexachloroethane	1	117	Atrazine	79	200
N-Nitroso-di-methylamine	1	42	4-Bromophenyl-phenylether	79	248
N-Nitroso-di-n-propylamine	1	70	Carbazole	79	167
2-Fluorophenol (S)	1	112	Di-n-butylphthalate	79	149
Phenol-d6 (S)	1	99	4,6-Dinitro-2-methylphenol	79	198
Naphthalene-d8 (I.S)(35)		136	1,2-Diphenylhydrazine	79	77
Benzoic acid	35	105	Fluoranthene	79	202
bis(2-Chloroethoxy)methane	35	93	Hexachlorobenzene	79	284
Caprolactam	35	113	N-Nitrosodiphenylamine	79	169
4-Chloroaniline	35	127	Pentachlorophenol	79	266
4-Chloro-3-methylphenol	35	107	Phenanthrene	79	178
2,4-Dichlorophenol	35	162	2,4,6-Tribromophenol (S)	79	330
2,4-Dimethylphenol	35	107	Chrysene-d12 (I.S) (92)		240
Hexachlorobutadiene	35	225	Benzidine	92	184
Isophorone	35	82	Benzo(a)anthracene	92	228
2-Methylnaphthalene	35	141	Bis(2-ethylhexyl)phthalate	92	149
Naphthalene	35	128	Butylbenzylphthalate	92	149
Nitrobenzene	35	77	Chrysene	92	228
2-Nitrophenol	35	139	3,3'-Dichlorobenzidine	92	252
1,2,4-Trichlorobenzene	35	180	p-(Dimethylamino)azobenzene	92	225
Catechol	35	110	Pyrene	92	202
Nitrobenzene-d5 (S)	35	82	Terphenyl-d14 (S)	92	244
Acenaphthene-d10 (I.S) (59)		164	Perylene-d12 (I.S) (101)		264
Acenaphthene	59	153	Benzo(b)fluoranthene	101	252
Acenaphthylene	59	152	Benzo(k)fluoranthene	101	252
1,1'-Biphenyl	59	154	Benzo(g,h,i)perylene	101	276
2-Chloronaphthalene	59	162	Benzo(a)pyrene	101	252
4-Chlorophenyl-phenylether	59	204	Dibenz(a,h)anthracene	101	278
Dibenzofuran	59	168	Di-n-octylphthalate	101	149
Diethylphthalate	59	149	Indeno(1,2,3-cd)pyrene	101	276

I.S=internal standard, Q.M=quant mass, S=surrogate

Table 7 INTERNAL STANDARD ASSOCIATION / QUANT MASS – Standard SVOC analysis (contd)					
COMPOUND	*I.S	Q.M	COMPOUND	*I.S	Q.M
1,4-Dichlorobenzene-d4 (I.S)(1)		152	Diphenylamine	59	169
Pentachloroethane	1	167	Thionazin	59	107
2-Picoline	1	93		59	
N-Nitrosomethylethylamine	1	88		59	
Methyl methanesulfonate	1	80		59	
N-Nitrosodiethylamine	1	102		59	
Ethyl methanesulfonate	1	79		59	
N-Nitrosopyrrolodine	1	100		59	
N-Nitrosomorpholine	1	56		59	
O-Toluidine	1	106		59	
	1		Phenanthrene-d10 (I.S) (79)		188
	1		4-Nitroquinoline-1-oxide	79	190
	1		Phenacetin	79	108
	1		4-Aminobiphenyl	79	169
	1		Pentachloronitrobenzene	79	237
	1		Sulfotepp	79	97
	1		Phorate	79	75
Naphthalene-d8 (I.S)(35)		136	Diallate	79	86
1- Methylnaphthalene	35	141	Dimethoate	79	87
N-Nitrosopiperidine	35	114	Pronamide	79	173
a,a-Dimethylphenethylamine	35	58	Disulfoton	79	88
O,O,O-Triethylphosphorothioate	35	97	Dinoseb	79	211
Hexachloropropene	35	213		79	
2,6-Dichlorophenol	35	162		79	
p-Phenylenediamine	35	108	Chrysene-d12 (I.S) (92)		240
N-Nitrosodi-n-butylamine	35	84	Methapyrilene	92	97
Safrole	35	162	p-(Dimethylamino)azobenzene	92	225
1,2,4,5-Tetrachlorobenzene	35	216	Chlorobenzilate	92	251
	35		3,3'- Dimethylbenzidine	92	212
	35		2- Acetylaminofluorene	92	181
	35		7,12-Dimethylbenz[a]anthracene	92	256
	35		Aramite	92	185
	35		Methyl parathion	92	109
	35		Parathion	92	109
Acenaphthene-d10 (I.S) (59)		164	Isodrin	92	193
Isosafrole	59	162	Kepone	92	272
1,4-Naphthoquinone	59	158	Famphur	92	218
Pentachlorobenzene	59	250	Perylene-d12 (I.S) (101)	101	
2-Naphthylamine	59	143	3-Methylcholanthrene	101	268
1-Naphthylamine	59	143	Hexachlorophene	101	196
2,3,4,6-Tetrachlorophenol	59	232		101	
5-Nitro-o-toluidine	59	152		101	

I.S=internal standard, Q.M=quant mass, S=surrogate

Table 8: LOW CONCENTRATION PAH INTERNAL STANDARD/SURROGATE SPECIFICATIONS

INTERNAL STD ASSOCIATION

Phenanthrene-d10 (IS)

Naphthalene
2-Methylnaphthalene
1-Methylnaphthalene

2-Fluorobiphenyl(SUR)

Acenaphthylene
Acenaphthene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene

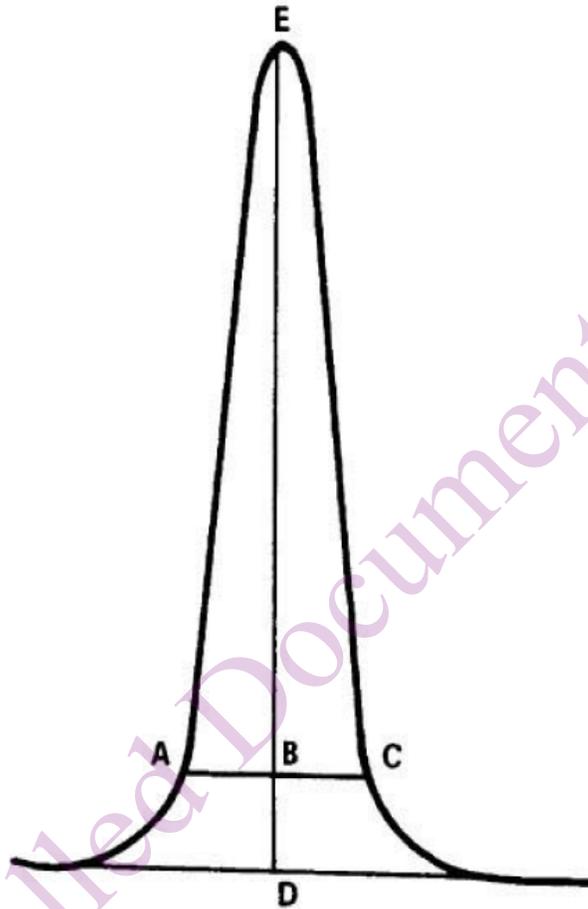
Perylene-d12 (IS)

Terphenyl-d14(SUR)

Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(g,h,i)perylene

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FIGURE 1
TAILING FACTOR CALCULATION



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

$$\text{Therefore: Tailing Factor} = \frac{12}{11} = 1.1$$

Table 9, DFTPP Tuning Criteria

Mass	Ion Abundance Criteria
51	30-60% of mass 198
68	<2% of mass 69
70	<2% of mass 69
127	40-60% of mass 198
197	<1% of mass 198
198	Base peak, 100% relative
199	5-9% of mass 198
275	10-30% of mass 198
365	>1% of mass 198
441	Present, but less than mass 443
442	>40% of mass 198
443	17-23% of mass 442

Note: While 8270D table 3 indicates different criteria, section 11.3.1.2 allows the use of alternate criteria.

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EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 300

REVISION #: 19

EFFECTIVE DATE: 20101117

GC/MS SEMI-VOLATILE
BNA-AQUEOUS MATRIX
EXTRACTION USING
SW-846 METHOD 3510C
FOR 8270/625 ANALYSIS

APPROVALS:

Lab Director:



Date:

11/19/10

Data Quality Manager:



Date:

11/19/10

Section Supervisor:



Date:

11/19/10

Changes Summary

Revision 19, 11/17/2010

- SOP has been updated to reflect current procedure used to prepare the “Baked Sodium Sulfate” and 10N NaOH solution.
- Redundant statement concerning holding time under sec. 14.0 has been removed.
- Volume correction for MeCl₂ added.
- Size and labeling of containers used as well as drying procedure prior to concentration of extracts updated.
- The requirement for a matrix spike duplicate for TCLP extracts has been added.

Revision 18, 04/26/10

- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory’s revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

1.0 Identification of the Test Method

1.1 This SOP is compliant with SW-846 Method 3510C and Method 625.

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to aqueous samples.

3.0 Detection Limit

Not Applicable to this SOP

4.0 Scope of Application, including components to be analyzed

Not Applicable to this SOP

5.0 Summary of the Test Method

5.1 Aqueous samples are extracted with methylene chloride. The extracts are dried through sodium sulfate and concentrated to an appropriate final volume.

6.0 Definitions

6.1 Laboratory Quality System SOP QS08 "Technical/Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" provides information on the commonly used definitions.

7.0 Interferences

- 7.1 Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
- 7.2 Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
- 7.3 Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

8.0 Safety

8.1 Laboratory SOP QS13 "Safety Program & Chemical Hygiene Plan" discusses the safety program that is to be followed labwide.

9.0 Equipment and Supplies

- 9.1 Separatory Funnel – 2L with Teflon stopcock
- 9.2 Beaker – 250mL or 400mL
- 9.3 Drying/Chromatographic column – 20mm I.D. x 300mm
- 9.4 Filter funnel
- 9.5 Turbo-Vap evaporation tube – 200mL tube made by Zymark or equivalent
- 9.6 Metal rack – capable of holding six glass evaporation tubes
- 9.7 Turbo-Vap Evaporator – heated and capable of temperature control (+5°C); the bath should be vented into a hood
- 9.8 Vials, 2.0 mL glass with Teflon-lined screw cap
- 9.9 pH indicator paper – wide range (1.0-12.0)
- 9.10 Syringe – 1mL

- 9.11 Graduated cylinder – 1000mL, 500mL, and 100mL, glass, Class A
- 9.12 Pasteur pipette – length 9”
- 9.13 Pasteur pipette bulb
- 9.14 Labels – DYMO
- 9.15 Teflon Bottles – 500mL
- 9.16 Volumetric Flasks – 500mL, 100mL, 50mL, and 10mL, glass, Class A
- 9.17 Ring Stand – 3-prong
- 9.18 Burette clamp – double
- 9.19 Aluminum foil – heavy duty
- 9.20 Nitrogen tank – equipped with pressure regulator
- 9.21 Boiling chips – Teflon
- 9.22 Glass Wool – Roving, 9989 purchased from Fisher #11-388 or equivalent

10.0 Reagents and Standards

- 10.1 Reagent Water - Reagent water is gathered in a carboy from source in the instrument lab as needed.
- 10.2 Sodium Hydroxide Solution - (10N). Weigh 800g NaOH (purchased in a fiber drum from Tennessee Reagents # 2-31825-25lb or equivalent) into a glass or plastic container. Add approximately 1000mL of reagent water to a 2000mL volumetric flask, add a stir bar, place on stir plate, and stir. Add pellets slowly and swirl until pellets are mostly dissolved. This mixture will get very hot. Continue to add reagent water while mixture is being stirred to keep volume at approximately 1000mL. Let stand until cool. Bring to final volume. Transfer to 1000mL Teflon containers.
- 10.3 Sodium Sulfate – Granular, anhydrous, trace pure 10-60 mesh (purchased in 200lb bulk fiber drum from Fisher #S415-200lb or equivalent). For low level tests, place an aliquot in a 1500mL heavy duty Pyrex beaker and bake in muffle furnace at 400°C for a minimum of 8 hours. Remove and cool in open air and place in designated “Baked Sodium Sulfate” container at room temperature
- 10.4 Glass Wool – Roving , 9989 Glass (purchased from Fisher #11-388 or equivalent).
- 10.5 Sulfuric Acid Solution - (1:1), slowly add 500mL of H₂SO₄ (Fisher, suitable for trace metal analysis #A300C-212 or equivalent) to 500mL of reagent water in a 1000mL Teflon container. This mixture will get very warm. Allow to cool before use.
- 10.6 Extraction Solvent - Methylene Chloride (purchased from Fisher #D151-4 or equivalent) Please read SOP-336 before handling this solvent in our laboratory.
- 10.7 The extraction analyst makes up surrogates and spikes. Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes.
 - 10.7.1 **BNA Surrogate** – The base neutral and acid surrogate are mixed together in one solution (purchased from NSI #WL-371-C at concentrations of 100-200ug/mL). The expiration for this standard is 6 months from the date opened. Use 0.5mL of this solution per 1000mL of aqueous sample.
 - 10.7.2 **BNA Spiking Solution** – The base neutral and acid spiking solutions are mixed together in one solution called BNA LCS#1 (This spiking solution contains all the compounds that are normally calibrated by GC/MS). This solution, with a final concentration of 100ug/mL, is prepared in Methanol by

making a dilution of stock purchased from reputable vendors (BNA LCS #1 spike kit #K-943 and 1-methylnaphthalene #1288-01-08 are purchased from NSI, 2,6 Dichlorophenol #95591 is purchased from Absolute Standards and 1,4 Dioxane #30287 is purchased from Restek). Use 0.5mL of this solution per 1000mL of aqueous sample. Another spiking solution is also used, called BNA LCS#2. This solution contains short or matrix spike list base extractable compounds. This solution, with a final concentration of 100ug/mL, is prepared in Methanol by making a dilution of stock purchased from NSI #Q-6104-0. Use 0.5mL of this solution in combination with BNA LCS#1 for all full list BNA requirements. BNA LCS #2 may be omitted from samples requiring PAH analysis. (For low level PAHs, use 1.0mL of a 1.0ug/mL solution made from BNA LCS #1, called "LLPAH spiking solution.") All standards expire 6 months from the date they are made.

10.7.3 BNA TCLP Spike – 0.5mL of BNA LCS#1 and BNA LCS#2 is added per 100mL volume. This volume is provided by Wet Chemistry in a 1L glass amber bottle. 100mL is removed from this container and measured using a graduated cylinder.

11.0 Sample Collection, Preservation, Shipment, and Storage

- 11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.
- 11.2 Aqueous samples have a hold time of 7 days from the date of sampling.

12.0 Quality Control

- 12.1 Quality Systems SOP QS08 "Technical/ Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures" outlines details related to laboratory wide protocols on quality control.

13.0 Calibration and Standardization

Not Applicable to this SOP

14.0 Procedure

- 14.1 Determine the samples necessary to extract from the following sources (Note: never extract samples of unknown origin without discussion with supervisor):
 - 14.1.1 Each day the extractions group leader will generate a sample backlog using LIMS.
 - 14.1.2 This backlog is used to determine extraction priorities based on hold times and due dates.
 - 14.1.3 Samples requiring RUSH turn around time may be logged in throughout the day, which will require immediate attention. Sample receiving personnel will generally communicate this need.
 - 14.1.4 Samples are placed in LIMS "batches" based on parameter and extracted accordingly.
- 14.2 Wearing lab coat, gloves and safety glasses, get samples from refrigerator. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether

they are in glass amber jar and have a Teflon lid. Find out if any special dilutions need to be made for this client. Routine procedures for difficult matrices are listed below:

14.2.1 SLUDGE - use only 100mL and dilute to 1000mL with reagent water.

14.2.2 TCLP EXTRACT - use only 100mL and dilute to 1000mL with reagent water. A separate matrix spike and matrix spike duplicate of 100mLs should be set up at the same time. Dilute to 1000mL with reagent water.

14.2.3 BAD MATRIX – for example a liquid that is partially sediment, see your supervisor to find out what dilution, if any should be made. SPLP extract- use 1 liter.

14.2.4 NPDES client - a special list of compounds is required including benzidine. Method 625 requires that there be a spike every ten samples. The sample must be extracted and concentrated in the same day. A GC/MS screen is recommended; therefore this extraction should be coordinated with the GC/MS operator. 1mL of the BNA spiking solution is added to the LCS and the matrix spike.

14.2.5 ACID EXTRACT WITH BAD MATRIX - a cleanup step is added. Samples are taken to a high pH, extracted with 60mL methylene chloride one time as explained below in the BASE NEUTRAL EXTRACTION section. This extract is discarded. The samples are then taken to a low pH and extracted as an acid extraction. Acid extractions may be concentrated in the TurboVap.

14.3 LOW LEVEL POLYAROMATIC HYDROCARBONS (PAHs) – Samples require a BNA extraction. Use the surrogate and spiking solution specified.

14.4 Mark the amber glass container of each sample at the water meniscus with "white out" for later determination of sample volume. Check the pH by inverting the sample and touching the wide range pH paper to the portion that remains on the lid. Record this pH on the LIMS bench sheet and, later, in LIMS.

14.5 Get out enough separatory funnels and 250mL beakers to extract the number of samples you have plus any additional spikes and a method blank. A method blank and an LCS must be processed with each set of samples. If the sample is a TCLP, blank fluid may be provided along with the extracted TCLP sample(s). Use only 100mL and dilute to 1000mL with reagent water. Process a matrix spike and matrix spike duplicate on aqueous samples if requested by client. If not, a LCSD must be processed. Rinse separatory funnels with methanol. Place label from sample bottle onto separatory funnel as samples are poured into funnels to ensure proper identification. Use Avery labels to properly identify method blank, LCS, and LCSD as well as each beaker for each sample.

14.6 Using the 1000mL glass graduated cylinder marked NANO PURE WATER ONLY, measure 1000mL of reagent water from the carboy and transfer it to a separatory funnel for the method blank and LCS. Transfer sample to separatory funnel that corresponds to the lab # on the sample bottle.

14.7 Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature.

- 14.8 Generally 0.5mL of BNA surrogate is added to each sample, spike, and blank with a syringe designated for BNA surrogate. Someone must verify that the surrogate has been added by initialing LIMS bench sheet.
- 14.8.1 NOTE: Be sure to invert syringe and eliminate air bubble when obtaining surrogate solution and spiking solution.
- 14.9 For the sample in each analytical batch selected for spiking, use the 0.5mL glass syringe designated for BNA spike, to add 0.5mL of BNA spiking solution. **For low level PAHs use 1.0mL of the 1.0ppm LLP AHs spiking solution.** Someone must verify that the spike has been added by initialing the LIMS bench sheet. For DOD QSM projects, all target compounds will be spiked into the LCS and MS/MSD.
- 14.10 Enter the ID# of the surrogate/spike used on the LIMS bench sheet and, later, in LIMS.
- 14.11 ACID EXTRACTION: Adjust the pH to between 1.0 and 2.0, using 2mL of 1:1 H₂SO₄. Add to each sample, spike and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more H₂SO₄ solution in small increments, as required to attain the proper pH.
- 14.12 Add 60mL of Methylene Chloride to each empty sample bottle and to the LCS, method blank and MS/MSD funnels. Swirl the 60mL of methylene chloride that you added to the empty sample bottle and transfer to the corresponding separatory funnel.
- 14.13 Seal and shake the separatory funnel vigorously for 2 minutes with periodic venting to release excess pressure. Alternatively, Teflon funnels may be used and placed in the shaker apparatus with the stopcocks slightly open. When this apparatus is used, the shake should be for 3 minutes.
- 14.13.1 NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, initial venting should be done immediately after the separatory funnel has been sealed and shaken once.
- 14.14 Allow the sample to sit for 10 minutes, if necessary, after it has been shaken. It will separate into two layers with the solvent layer on the bottom. If it forms an emulsion (thick, cloudy, viscous, mixture that you cannot see through), drain what you believe to be 60mL into a labeled (w/Avery label) 250mL centrifuge bottle. If the layers are clearly separated, drain the solvent layer into a labeled (w/Avery label) 250mL glass beaker.
- 14.15 Following Steps 14.12 through 14.14, extract two more times with 40mL of methylene chloride. Combine the two solvent extracts into the same 250mL beaker.
- 14.16 BASE NEUTRAL EXTRACTION: Adjust the pH to 11 or slightly greater, using 10N NaOH. Start by adding 7.0mL to each sample, spike, and method blank. Stopper and shake to insure that pH throughout the sample is changed. Check the drop of liquid hanging from the lid with short-range pH paper. Compare the color to the chart on the pH paper. If the color is not within range add more 10N NaOH in small increments, as required to attain the proper pH. BNA extraction is necessary when doing low level PAHs.
- 14.16.1 NOTE: This step is critical to the extraction procedure. Too much NaOH solution could cause you to lose certain Base Neutral compounds. Be careful on this step.

- 14.17 FOR 8270 extraction: Extract one more time with 20mL of methylene chloride following Step 14.16. Do not combine BN and Acid extracts in a same 250mL beaker. However, you may filter BN and Acid extracts through the same sodium sulfate filter and combine into the same turbo in order to concentrate BN and acid extracts for one final extract.
- 14.17.1 NOTE: It has been demonstrated that two acid and one BN extraction can be used for normal 8270 samples. This procedure cannot be used for DOD or 625 samples.
- 14.18 For 625 extractions: extract 2 more times with 40 mL methylene chloride following steps 14.12 through 14.14. Combine BN extracts in the empty 250mL sample beaker as the acid portion concentrates in the turbo vap. Following step 14.24, concentrate the acid extract to ~5mL and then filter the BN extract into the same turbo.
- 14.19 Prepare to dry the sample by either of the following methods:
- 14.19.1 Set up a ring stand with funnels. Place a small amount of glass wool in the bottom of the funnels, add ~2" sodium sulfate to the funnel and rinse with 20-30 mL methylene chloride. Discard this rinse into the Chlorinated Waste container in the hood.
- 14.20 If the extract was drained into a centrifuge bottle, at this point you will need to take it to the centrifuge. Push the "ON" button to turn the centrifuge on. Be sure that the large holders are available for the 250-mL centrifuge bottles. The sample must always be balanced. If necessary use a dummy bottle making it similar weight using reagent water. Set the rpm at 2500 and the temperature at 0°C. Close the lid and be sure to press it down until you hear it click. Move the lever at the front of the lid to the "LOCK" position. Turn the time to approximately 15 minutes and bring it back to 10 minutes. As the rotor begins to move, you will be able to see the rpm's in the digital readout. Stay with the centrifuge until it has come up to the rpm's set to insure that it does not become unbalanced. This looks like 8888 on the digital readout. Should this occur, refer to the manual. When the cycle is complete, the digital readout will read 0000. Push the "OPEN" button and the lid will pop up. Move the lever at the front of the lid to the "UNLOCK" position. Open lid and remove sample. The sample will usually be in two layers with the extract on the bottom.
- 14.21 Remove any water layer from the extract in the beaker or centrifuge bottle, by one of two methods. Remove with a Pasteur pipette by carefully pulling up the water layer, on top, and not the solvent. Discard this layer in the sink. Use the smallest amount possible of Na₂SO₄ by sprinkling the top layer with Na₂SO₄ until it hardens, separates, and drops to the bottom.
- 14.22 Determine the original sample volume by refilling the sample bottle to the mark made with "white out." Transfer the liquid to a plastic 1000-mL graduated cylinder and record the sample volume on the LIMS bench sheet to the nearest 10-mL and record, later, in LIMS.
- 14.23 Prepare sample vial tray using labels printed off from LIMS that identify sample numbers, initial/final volumes, client, parameter, and date extracted.
- 14.24 TURBO-VAP CONCENTRATION
- 14.24.1 Rinse a Turbo-Vap tube with methylene chloride and arrange it underneath a rinsed, funnel. Pour the extract through the funnel so that it will collect in the

tube. Rinse the 400-mL beaker, which contained the solvent extract twice with 10 to 15 mL of methylene chloride and add each rinse to the funnel to complete the quantitative transfer. After all the extract has passed through the funnel, rinse the funnel with 10 to 15 mL of methylene chloride. Total volume in the glass evaporator tube should not exceed 200 mLs to avoid splattering on the lid of the Turbo-Vap.

- 14.24.2 Record the numbers of the Turbo-Vap tube on the LIMS bench sheet and place the tube in a metal holder.
- 14.24.3 Turbo-Vap Operation: Adjust the pressure of nitrogen gas tank to >30 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 40°C -50°C.
- 14.24.4 Place the glass evaporator tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be in the sensor and need removal. (See Turbo-Vap manual). Note which TV position was used for each sample on bench sheet.
- 14.24.5 When the beep sounds indicating the end of concentration, the extract will be at approximately one half mL (half way up tip of tube). Remove the tube from the bath. Use a 9" Pasteur pipette to draw up the sample and transfer it to the 2-mL vial. **THIS IS THE MOST CRITICAL PART OF THE ENTIRE OPERATION!!!** A single drop represents about 10 percent of the total sample. Before you move the tip of the pipette from the tube to the vial, be sure that a drop will not form on the end and fall off.
- 14.24.6 Draw ~0.25 mL of methylene chloride into a 9" Pasteur pipette and add this aliquot to the turbo-vap. Draw the methylene chloride into a pipette and rinse the sides of the tube several times. Transfer this rinse to the appropriately labeled 2-mL vial. Add methylene chloride from the designated clean pipette and repeat the rinsing process until you have ~ 1 mL in the sample extract vial. Compare this volume to a 2-mL dummy vial containing 1 mL of solvent to insure that you have not exceeded 1 mL. The methylene chloride rinse volume must be adjusted to achieve this final volume. Cover the extract with a Teflon-sealed screw cap.
- 14.25 The extract is now ready to be analyzed. Refrigerate at 4°C or carry directly to the instrument operator. Samples must be signed into the Sample Extract refrigerator. On log provided, enter the sample numbers, the analyst initials, and the date and time the samples were placed into the refrigerator.
- 14.26 Transfer handwritten extraction details from bench sheet to LIMS and archive bench sheet for future reference.

15.0 Data Analysis and Calculations

Not Applicable to this SOP

16.0 Method Performance

16.1 Demonstration of Capability (DOC): Each analyst must perform a DOC prior to independently extracting samples and yearly thereafter. The analyst must prepare 4 LCS samples. The data is calculated for accuracy and precision requirements.

17.0 Pollution Prevention

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

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Not Applicable to this SOP

19.0 Contingencies for Handling out of control or unacceptable data

Not Applicable to this SOP

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

21.1 Test Methods for Evaluating Solid Waste, SW-846, Third Edition

21.2 40 CFR, Method 625.

22.0 Tables, Diagrams, Flowcharts, and Validation Data

Not Applicable to this SOP

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 327

REVISION #: 20

EFFECTIVE DATE: 20110514

**NITROAROMATICS AND NITRAMINES BY
HIGH PERFORMANCE
LIQUID CHROMATOGRAPHY (HPLC)
METHOD 8330, 8330A, 8330B and 8332**

APPROVALS:

Lab Director:  Date: 5/16/11

Data Quality Manager:  Date: 5/16/11

Section Supervisor:  Date: 05/14/11

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Changes Summary

Revision 20, 5/14/2011

- 8330B constant drying requirement updated from +/- 1% to +/- 4% in section 14.6.2 based on Standard Methods SM2540.
- Removed reference to chilled sonication bath and updated reference to chilled shaker to indicate validation by SW846 team as included in method 8330B.
- Added a requirement to monitor the temperature of the chilled shaker in section 14.8.2.
- Added a reference to the CRM for 8330B in section 10.6 and 14.7.6
- Added 30mesh sieve to list of equipment in section 9.4.10.
- Removed Nitroguanidine from Table 1.
- Updated references to Table 2 to reflect Table 2 or 3 where appropriate.
- Removed reference to section 8.6 calculations within section 15.

Revision 19, 10/04/2010

- 8330B drying equipment has been added
- 8330B drying process has been updated
- Standard expiration dates have been clarified
- 8330B DL/LOD/LOQ have been added to Table 1.

Revision 18, 9/7/2010

- The SOP is an update from Revision 17 dated 02/12/10
- The SOP is formatted to include all 22-elements required per the NELAC standards
- The laboratory's revision of all technical SOPs now includes a Table of Contents that provides the map of the technical information contained within the SOP.
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.
- **8330B requirements updated with addition of Table 3 and Table 6.**

Table of Contents

1. Identification of the Test Method
2. Applicable Matrix or Matrices
3. Detection Limit
4. Scope of Application, Including components to be Analyzed
5. Summary of the Test Method
6. Definitions
7. Interferences
8. Safety
9. Equipment & Supplies
10. Reagents and Standards
11. Sample Collection, Preservation, Shipment, and Storage
12. Quality Control
13. Calibration and Standardization
14. Procedure
15. Data Analysis and Calculations
16. Method Performance
17. Pollution Prevention
18. Data Assessment and Acceptance Criteria for Quality Control Measures
19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
22. Tables, Diagrams, Flowcharts and Validation Data

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1.0 Identification of the Test Method

This SOP, based on SW-846 Method 8330, 8330A, 8330B and 8332, is used for the trace analysis of explosives residues by high performance liquid chromatography (HPLC) using a UV-VIS detector in water, solid, or sediment matrices..

2.0 Applicable Matrix or Matrices

This SOP is used for the trace analysis of explosives residues by high performance liquid chromatography (HPLC) using a UV-VIS detector in water, solid, or sediment matrices.

3.0 Detection Limit

See **Table 1**.

4.0 Scope of Application, Including Components to Be Analyzed

- 4.1 Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. When applicable, surrogates are listed and indicated as such within this table.
- 4.2 Extreme care should be taken when working with pure standard and stock standard solutions of these compounds and all handling of standards should be done in a hood. These compounds have been classified as known or suspected human or mammalian carcinogens.

5.0 Summary of the Test Method

- 5.1 Samples are analyzed after appropriate sample preparation using HPLC with identification at 210nm on a C-18 reverse phase column and confirmation at 210nm on a Biphenyl column. The preparation is performed using a solid phase extraction method, SW846 method 3535 for low concentrations of explosives residues in water. Dilution and filtration prepare high concentration water samples for direct injection. Extraction with acetonitrile in an ultrasonic bath or shaker followed by filtration prepares soil and sediment samples.

6.0 Definitions

- 6.1 Laboratory Quality System SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” provides information on the commonly used definitions.

7.0 Interferences

- 7.1 Solvents, reagents, glassware and other sample processing hardware may yield discrete artifacts and/or elevated baselines, causing misinterpretation of the chromatograms. All of these materials must be demonstrated to be free from interferences.
- 7.2 2-Am-DNT and 4-Am-DNT elute at similar retention times (retention time difference of 0.2 minutes). A large concentration of one isomer may mask the response of the other isomer. If it is not apparent that both isomers are present (or are not detected), an isomeric mixture should be reported.
- 7.3 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. Degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak, peak heights rather than peak areas should be used when tetryl is present in concentrations that are significant relative to the concentration of 2,4,6-TNT.

8.0 Safety

- 8.1 Laboratory SOP QS13 “Safety Program & Chemical Hygiene Plan” discusses the safety program that is to be followed labwide.

- 8.2 Standard precautionary measures used for handling other organic compounds should be sufficient for the safe handling of the analytes targeted by Method 8330A. The only extra caution that should be taken is when handling the analytical standard neat material for the explosives themselves and in rare cases where oil or waste samples are highly contaminated with the explosives. The HMX, RDX, Tetryl, and 2,4,6-TNT are explosives and the neat material should be handled carefully. Drying at ambient temperature requires several days. Do not dry at heated temperatures!
- 8.3 Care should be used in handling all samples. Safety glasses must be worn in the lab at all times. The use of blue nitrile gloves and lab coats is highly recommended.
- 8.4 Research into expected sample content and concentration should be done in order to be prepared for additional safety considerations. Generally, any samples which need special consideration have applicable notes on the sample logs.
- 8.5 MSDS sheets are available for all reagents and standards which have been purchased. These are located in the office next to the technical director.

9.0 Equipment & Supplies

- 9.1 Instrumentation
- 9.1.1 Agilent Series 1100 HPLC System
 - 9.1.2 Agilent G1311A Quaternary Pump
 - 9.1.3 Agilent G1379A Degasser
 - 9.1.4 Agilent 1313A 100 Position Autosampler
 - 9.1.5 Agilent G1316A Column Compartment
 - 9.1.6 Agilent G1314A Variable Wavelength Detector
 - 9.1.7 Agilent HPLC Chemstation
- 9.2 Hewlett Packard Series 1050 HPLC System
- 9.2.1 Hewlett Packard Model 79852 1050 Quaternary HPLC Pump
 - 9.2.2 Hewlett Packard Model 79853 Variable Wavelength UV-VIS Detector
 - 9.2.3 Hewlett Packard 1050 79855A 21 Position Autosampler
 - 9.2.4 Hewlett Packard 1050 100 Position Autosampler Upgrade
 - 9.2.5 Hewlett Packard Model 79856A 1050 Solvent Module
 - 9.2.6 Hewlett Packard Model G1303A Vacuum Degassing Module
 - 9.2.7 Dell OptiPlex 933 GX150 Pentium III Computer
 - 9.2.8 Hewlett Packard PC Communication for HP Chemstation
 - 9.2.9 Hewlett Packard HPLC Chemstation
 - 9.2.10 SideWinder Temperature Control Module
- 9.3 Solid-phase extraction system consisting of:
- 9.3.1 Manifold Station, *J.T. Baker spe-12G*, or equivalent
 - 9.3.2 Tubing and connectors
 - 9.3.3 SFE extraction cartridges, *Porapak®RDX Cartridges* or equivalent
 - 9.3.4 Vacuum system capable of maintaining 18 inches of mercury
 - 9.3.5 Balance ± 0.01 g.
 - 9.3.6 Vortex mixer.
- 9.4 Other Components:
- 9.4.1 Chilled Shaker (validation by SW846 team as included in method 8330B.)
 - 9.4.2 Disposable cartridge filters - 0.45 mm PTFE filter.
 - 9.4.3 Scintillation Vials - 20 mL, glass.
 - 9.4.4 Vials - 15 mL, glass, Teflon-lined cap.

- 9.4.5 Vials - 40 mL, glass, Teflon-lined cap.
- 9.4.6 Disposable syringes - Plastipak, 3 mL and 10 mL or equivalent.
- 9.4.7 Volumetric flask with ground glass stopper - 100 mL and 1000 mL.
- 9.4.8 Vacuum desiccator - Glass.
- 9.4.9 Mortar and pestle - Steel.
- 9.4.10 Sieve – 10 mesh, 30 mesh & 200 mesh
- 9.4.11 Graduated cylinders - 10 mL, 25 mL, 250 mL, 1000 mL.
- 9.4.12 Pasteur pipet - length 9 ".
- 9.4.13 Manual Sample Mill.
- 9.4.14 Wiley Sample Mill.
- 9.4.15 ESSA model LM-2P pulverizing mill (specified in 8330B)
- 9.4.16 Clippers for cutting vegetation
- 9.4.17 Drying cabinet & shelving inside

10.0 Reagents and Standards

- 10.1 The laboratory's LIMS system allows for complete documentation and for the traceability of reagents and standards used within the laboratory. The following information relates to the specific reagents and standards used for the performance of the method:
- 10.2 HPLC grade chemicals shall be used in all tests. 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. Certified stock standards are purchased from Restek, Protocol, Ultra and other vendors depending on their availability. The date they are received is noted on the label or container they are received in and in the LIMS system. The date the standards are opened they are recorded and given a sequential number in the LIMS system. All stock standards are stored at 4°C.
- 10.3 List of Reagents
 - Acetonitrile, CH₃CN - HPLC grade.
 - Methanol, CH₃OH - HPLC grade.
 - Calcium chloride, CaCl₂ - Reagent grade. Prepare an aqueous solution of 5g/L. This is for use with soil/sediment samples.
 - Organic-free reagent water. Obtained from the Nano Pure Water System in the GC lab.
 - Sodium Chloride solution - 325 g NaCl per 1000 mL reagent water.
- 10.4 Stock standards are purchased in mixtures from reputable vendors. The date they are received and opened is recorded in LIMs along with their lot number and vendor. Each standard that is prepared is recorded in the LIMs and given a sequential number. Each standards label is completed with the standard number, name, concentration, expiration date, and analyst initials. All standards are stored in the refrigerator in the dark at a temperature of 4°C ± 2°C or less from the date they are received/prepared. The refrigerator temperature is monitored daily with an annually calibrated thermometer and recorded with calibration correction in the GC refrigerator/freezer logbook or the Extraction temperature/calibration logbook. Makeup of some common standards is detailed below. The makeup of other standards can be found in the LIMs. In general, stock standards expire 6 months from the date opened. If the manufacturer expiration date is earlier then the earlier date is used. If the manufacturer expiration date lists month/year, the last day of the month is used.
- 10.5 Calibration Solutions - The 8330 calibration standards are prepared as follows using solutions purchased from Restek or an equivalent vendor.

- 10.5.1 Explosive (Mix#1,#2 and Surrogate) Calibration Stock Solutions: Using a 100 μ L syringe, 100 μ L of Mix # 1, Mix # 2, and Surrogate (Restek at 1000 μ g/mL) are injected into a 10mL volumetric flask containing approximately 9.5mL 1:1 acetonitrile and water and diluted to volume with same to make a 10 μ g/mL standard. After capping and inverting several times, the solution is transferred into a labeled, 12ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. These standards are used to make the calibration curve standards in 1:1 acetonitrile and water at concentrations of 10, 5.0, 1.0, 0.50, 0.10 and 0.025 μ g/mL. The lowest calibration standard can be extended down to 0.010 μ g/mL, if required.
- 10.5.2 Mix # 3 Calibration Stock Solution: Using a 500 μ L syringe, 500 μ L of Mix # 3 (Restek PETN & Nitroglycerin, NG, at 1000 μ g/mL) is injected into a 10mL volumetric flask containing approximately 9.0mL 1:1 acetonitrile and water and diluted to volume with same to make a 10 μ g/mL standard. After capping and inverting several times, the solutions are transferred into a labeled, 12ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. These standards are used to make the calibration curve standards in 1:1 acetonitrile and water at concentrations of 50, 25, 5, 2.5, 0.50 and 0.25 μ g/mL. The working calibration standards must be prepared fresh the day of use.
- 10.5.3 Matrix/LCS Spike Standard - The 8330 spiking solution is prepared by adding 0.5mL of a solution purchased from Ultra (Combined Stock Solution) in 50mL of acetonitrile for a final concentration of 10 μ g/mL. Samples are spiked with 1 mL of this solution.
- 10.5.4 Second Source Calibration Solution - The 8330 second source standard is prepared as follows using a solution purchased from Ultra(Combined Stock Solution) and Accustandard Inc.(Mix #3) or an equivalent vendor.
- 10.5.5 Second Source Stock Solutions: Using a 100 μ L syringe, 100 μ L of Combined Stock Solution (Ultra at 1000 μ g/mL) is injected into a 10mL volumetric flask containing approximately 9.5mL 1:1 acetonitrile and water and diluted to volume with same to make a 10 μ g/mL standard. After capping and inverting several times, the solution is transferred into a labeled, 12ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. This standard is used to make a second source check standard 1:1 acetonitrile and water at 1.0 μ g/mL. Using a 500 μ L syringe, 500 μ L of Mix #3(Accustandard at 1000 μ g/mL) is injected into a 10mL volumetric flask containing approximately 9.0mL 1:1 acetonitrile and water and diluted to volume with same to make a 10 μ g/mL standard. After capping and inverting several times, the solution is transferred into a labeled, 12ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. This standard is used to make a second source check standard 1:1 acetonitrile and water at 5.0 μ g/mL.
- 10.5.6 Surrogate Spike Solution- The 1,2-dinitrobenzene solution (1-Chloro-3-Nitrobenzene may be used as an alternate) is prepared as follows using a standard purchased from Restek or an equivalent vendor.
- 10.5.7 Surrogate Spiking Solution: Using a 250 μ L syringe, 200 μ L of 1,2-dinitrobenzene, (or 1-Chloro-3-Nitrobenzene), (Restek at 1000 μ g/mL) is injected into a 100mL volumetric flask containing approximately 95mL acetonitrile and diluted to volume with same to make a 2.0 μ g/mL standard. After capping and inverting several times, the solution is transferred into several labeled, 40ml, teflon-lined, screw-capped vial and stored in the refrigerator in the dark at 4°C or less for up to 30 days. Samples are spiked with 1 mL of this solution.
- 10.6 Certified Reference Material – 8330B reference materials are purchased from reputable vendors such as Wibby. The date they are received and opened is recorded in LIMs along with their lot number, vendor and a scanned copy of their COA. Alternatively, Soil PT study samples may be

purchased and used once the results are returned with the true values reported (report used as COA in LIMS).

11.0 Sample Collection, Preservation, Shipment, and Storage

- 11.1 Quality Systems SOP QS10 related to Sample Receipt, Handling, & Processing provides details for collection, preservation, shipment, and storage.
- 11.2 Samples and sample extracts are stored in the dark at 4°C. Samples are stored in the sample storage walk-in coolers. Extracts are stored in the Hobart in the Semivolatiles laboratory. The holding time for samples is 7 days for waters and 14 days for soils. The holding time for extracts is 40 days.
- 11.3 RT Windows
Retention time (RT) windows are determined for each compound through the analysis of 3 standards over a 72 hour period. The standard deviation of the standard retention times is calculated and the RT windows are determined to be $\pm 3x$ this standard deviation. New in-house retention time windows are established after every major change to the system (new column or flow) and after a new initial calibration using the mid-point standard. Retention times of each analyte in each CCV are compared to the established retention time window. Each analyte must fall within its respective RT window. If this criterion is not met, the system must be adjusted to allow another CCV to meet criteria, or a new initial calibration performed and new retention time windows established.

12.0 Quality Control

- 12.1 Quality Systems SOP QS08 “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” outlines details related to laboratory wide protocols on quality control.
- 12.2 An initial demonstration must be performed by each analyst performing this method. See **Table 2 or 3** for acceptance criteria.
- 12.3 Surrogate - All samples and QC are spiked with the surrogate. See **Table 2 or 3** for acceptance criteria and corrective action.
- 12.4 LCS Sample - The LCS is analyzed at the frequency required by the regulatory agency or client (every batch or 20 samples). To prepare the LCS, a blank is spiked with the LCS mix standards. See **Table 2 or 3** for acceptance criteria and corrective action..
- 12.5 Method Blanks - The Method Blank is extracted 1 per extraction batch of up to 20 samples. See **Table 2 or 3** for acceptance criteria and corrective action.
- 12.6 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for a MS/MSD, if sample is available. If no sample is available, an LCSD must be extracted to provide precision results. See **Table 2 or 3** for acceptance criteria and corrective action. MS data evaluation must include the consideration of the following factors.
- 12.7 Sample matrix - If the sample is a soil, grab sample or sequentially collected water sample it may affect the %R and RPD of the MS/MSD. Corrective action must be taken in the form of reanalysis if a method problem is indicated.
- 12.8 Original sample concentration - If a spiked compound has a problem and the concentration of that compound in the original sample was two or more times the concentration of the spike, no further corrective action may be necessary other than the generation of a corrective action report to document the problem.
- 12.9 MS vs. MSD - If a spiked compound has a similar problem in the MS and the MSD which is not traceable to the execution of the method, no further corrective action may be necessary other than the generation of a corrective action report to document the problem as matrix effect.

- 12.10 Non-target Interference - The presence of significant non-target interference should be brought to the immediate attention of your supervisor who should discuss the problem with the client/project manager to determine the action to be taken.

13.0 Calibration and Standardization

- 13.1 Quality Systems **SOP QS08** “Technical / Operational Definitions, Minimum Essential Quality Control Elements, and Laboratory Calibration Procedures” related to Calibration Procedures provides laboratory wide protocols for calibration and standardization.
- 13.2 Calibration of HPLC
- 13.2.1 Upon initial startup of the pump, flow is increased to 5.0mL/min after opening the purge valve to bleed air from the solvent/water lines. When all air bubbles have been removed, the flow is reduced to normal run conditions and the purge valve is closed. The instrument is then pumped with 100% solvent for 45 minutes (or less) and then pumped with the appropriate solvent/water mixture for an additional 45 minutes (or longer).
- 13.2.2 Initial Calibration: Injections of each calibration standard over the concentration range of interest are sequentially injected into the HPLC. Peak areas or heights are obtained for each analyte. (Peak height may be used instead of peak area for 2,4,6-TNT because degradation products of tetryl appear as a shoulder on the 2,4,6-TNT peak.) The calibration curve should be linear. However, some target analytes may be difficult to optimize without application of quadratic or higher order mathematical functions. Linearity may be determined using linear regression analysis for each target analyte by calculating the correlation coefficient r . Another term used to describe the goodness of fit of the line is coefficient of determination r^2 (the square correlation coefficient). The resulting line would normally not be forced through the origin or use the origin as a calibration point unless it is demonstrated that the intercept of the regression line is not statistically different from zero at 95% level of confidence. See **Table 2 or 3** for acceptance criteria and corrective action.
- 13.2.3 Due to the lack of resolution between 2-Am-DNT and 4-AM-DNT, calibration of these compounds can be based on “isomeric pairs”. Improved resolution may be obtained using a Supelco C-18 column with eluant of 57%/43% (v/v) methanol and water at 1.0 mL/min.
- 13.2.4 A visual inspection of the calibration curve should also be used as a diagnostic tool when nonlinear behavior is observed to verify if there is a large percentage error in any particular portion of the calibration curve. If the visual inspection indicates problems, or if one criteria is not met, then evaluate the following items for implementation based on an understanding of the detector response/contaminant concentration relationship.
- 13.2.5 Check the instrument operating conditions or the initial calibration standards used and make adjustments to achieve a linear calibration curve.
- 13.2.6 Narrow the calibration range using the same number of standards. Generally the highest standard is lowered first. The consequences of all actions taken must also be evaluated, i.e., reduction of the calibration range, raising of the Method Quantitation Limit, MQL, etc.
- 13.2.7 Evaluate the use of a nonlinear curve, when applicable. When nonlinear calibration models are used, the resultant line should not be forced through the origin and the origin should not be used as a calibration point. No higher than a third-order (cubic) calibration model shall be used. When a nonlinear calibration model is employed, more data points are needed to maintain at least three degrees of freedom. For example, use of a quadratic function requires at least a six-point initial calibration curve. The resulting r^2 should be greater than or equal to 0.99 for this to be considered acceptable.

- 13.2.8 Use of alternative techniques (e.g., relative standard error (RSE) outlined in the USEPA Memorandum, "Clarification Regarding Use of SW-846 Methods" (EPA/SW-846).
- 13.2.9 The standards used to make the calibration curve are verified to be accurate using a standard obtained from a second source, initial calibration verification (ICV). See Table 2 for acceptance criteria.
- 13.2.10 Daily Calibration: Continuing calibration verification (CCV) standards must be analyzed, at a minimum, at the beginning of the day, after every 20 samples and at the end of the sequence. See [Table 2 or 3](#) for acceptance criteria and corrective action.

14.0 Procedure

- 14.1 All waters have a 7-day holding time and soils have a 14-day holding time. Determine the samples necessary to extract as follows:
 - 14.1.1 Each day a backlog report will be provided indicating sample numbers with the respective analysis required. Line through all the extractions that have been completed and plan to do the remaining analysis within the required holding time.
 - 14.1.2 Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
 - 14.1.3 Check with log-in throughout the day and examine the COC (chain of custody) forms that arrive with each set of samples. If an analysis is ongoing, extra QC may be avoided by picking up those extractions on the same day.
 - 14.1.4 Wearing lab coat, gloves and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials and the date and time removed on the log provided. Inspect as to whether they are in glass(soil)/glass amber jar(water) and have a Teflon lid. Find out if any special dilutions or screens need to be made for this client.
- 14.2 Before extraction, all glassware must be prepared as instructed in SOP-306. Before weighing, the balance must be calibrated with ASTM Class I weights which bracket the amount to be weighed and recorded in the Extraction temperature/calibration logbook. If a heavy container is to be used for weighing, place a representative container on the balance, tare the balance and then calibrate the balance with the chosen weights. During extraction, all pertinent information (glassware, amounts, reagent lots, standards, etc.) is recorded in the HPLC 8330 extraction logbook so as to allow reconstruction of the extraction in the future.
- 14.3 High Level Aqueous Extraction Method
 - 14.3.1 Before extraction begins get out enough scintillation vials for each sample, method blank, laboratory control sample, matrix spike and matrix spike duplicate. Place an Avery label on each vial containing the following information: Lab #, Client name, Type of Analysis, Initial Volume - Final Volume, and the Lab Prep Batch Code.
 - 14.3.2 Place a 5 mL aliquot of each water sample in an appropriately labeled scintillation vial and add 4 mL of acetonitrile (3 mL for MS/MSD samples). Add 1.0 mL of the surrogate standard (2.0 µg/mL) using a 1.0 mL syringe to each sample, method blank and QC sample. Add 1.0 ml of 8330 standard spiking solution to each appropriate QC sample (LCS, MS&MSD). Shake samples thoroughly, and filter through a 0.45mm PTFE filter using a disposable syringe. Discard the first 3 mL of filtrate, and retain the remainder in a Teflon-capped vial for HPLC analysis. HMX quantitation can be improved with the use of methanol rather than acetonitrile for dilution before filtration. For screening purposes, 1 ml of sample is placed in a 4 ml vial along with 0.5 ml of acetonitrile and 0.5 ml of water.

14.4 Solid-Phase Extraction

This extraction method may not be appropriate for aqueous samples with greater than 1% suspended solids. Consult SW-846 Method 3535 for additional information.

- 14.4.1 Mark the outside of the sample container at the sample meniscus with "white-out". This mark will be used to determine the initial sample volume after processing the contents. Add 1.0 mL of the surrogate standard (2.0 µg/mL) using a 1.0 mL syringe to each sample, method blank and QC sample. Add 1.0 ml of standard spiking solution to each appropriate QC sample (LCS, MS&MSD).
 - 14.4.2 Assemble the manifold for multiple extractions with SPE cartridges.
 - 14.4.3 Wash the cartridges with 6 mL acetonitrile 3 times and 6 mL reagent water 6 times with gravity flow, do not let cartridge go dry. If it goes dry, you must start over.
 - 14.4.4 Add sample to the cartridge and attach connectors and tubing.
 - 14.4.5 Turn on the vacuum pump and begin drawing sample through the cartridge, while adjusting the flow to 10mL/min.
 - 14.4.6 Empty the water trap as needed.
 - 14.4.7 After the sample extraction is complete draw air through the cartridge for 15 minutes to dry.
 - 14.4.8 Add 4 mL of acetonitrile to the cartridge and allow it to pass through with gravity flow collecting it in a 12 mL vial. Note: the volume of acetonitrile may be reduced to 3ml to lower detection limits. Place an Avery label on each tube containing the following information: Lab #, Client name, Type of Analysis, Initial Volume - Final Volume, and the Lab Prep Batch Code. Bring extract up to 4 ml with acetonitrile. Sample extracts are diluted 2x with DI water prior to analysis. Record this volume in the HPLC extraction logbook. The extract is ready for analysis, proceed to Section 8.0.
 - 14.4.9 Determine the original sample volume by refilling the sample bottle to the mark made with "white out". Transfer the liquid to a plastic 1000-mL graduated cylinder and record the sample volume in the LIMS bench sheet to the nearest 10-mL.
- 14.5 Soil, Sediment and Nonaqueous Samples
- 14.5.1 Dry representative soil samples at room temperature, normally overnight, being careful not to expose the samples to direct sunlight. Grind and homogenize the dried sample thoroughly in an acetonitrile rinsed mortar so it will pass through a 30 mesh sieve. In other words, grind to a fine-dust like particle size. If one grinds the sample down to this small of a partical size, then the sieve would not be required.
 - 14.5.2 NOTE : Soil samples may be screened by a commercially available test kit prior to grinding in a mortar and pestle. Visually observe the sample for lumps of material that have a chemical appearance. These lumps should be suspect and not ground. Explosives are generally a very finely ground grayish-white material. Soil samples as high as 2% 2,4,6-TNT have been safely ground. Samples containing higher concentrations should not be ground in the mortar and pestle. 2,4,6-TNT is the analyte most often detected in high concentration in soil samples.
- 14.6 Sample Drying (8330B)
- 14.6.1 Samples should be thoroughly mixed and placed in the large drying shelves that are located in the 8330B drying cabinet. The shelves must be lined with aluminum foil before samples are placed in them. This technique applies only to a sample of at least 1kg. Make sure that the cabinet exhaust fan has been turned on before you place the samples in the cabinet.
 - 14.6.2 Dry the entire sample at room temperature overnight, to a maximum of two (2) days, being careful to avoid direct sunlight. When sample has dried and come to a constant weight (+/-4%), weigh the entire sample and record in LIMS then sieve the entire sample with a 10 mesh sieve. Break-up pieces of soil (especially clay) with gloved hands. Do

- not include vegetation in the portion of the sample that passes through the sieve unless this has been identified for inclusion as a project specific requirement.
- 14.6.3 Collect and weigh any portion unable to pass through the sieve. Record on bench sheet and in LIMS.
- 14.6.4 Record date, time, and ambient temperature in the drying area on a daily basis when drying samples.
- 14.7 Sample Grinding (8330B)
- 14.7.1 Sample grinding for soil samples from ammunition plants, depots, and firing ranges.
- 14.7.2 Initial demonstration: The lab must initially show that the mechanical grinder is capable of reducing the particle size to < 75 microns by passing representative portions of ground sample through a 200 mesh sieve. The data for this demonstration will be kept on file with the DQM.
- 14.7.3 A grinder blank (between each sample), using Ottawa sand, must be prepped (ground and sub-sampled) and analyzed in the exact same manner as a field sample. Each analytical batch will have a grinder blank sample. See **Table 3** for acceptance criteria and corrective action.
- 14.7.4 Soil Sub-sampling Procedure: Each ground sample is mixed and spread out on a large flat surface like a baking tray or pie pan, and 30 or more randomly located increments are removed from the entire depth to obtain the sum of at least a 10g sample.
- 14.7.5 Soil Sample Triplicate Determination: At the subsampling step, one sample per batch (cannot be a blank) will undergo triplicate analysis. Three 10g subsamples are taken from a sample expected to contain the highest level of explosives. If this information is not available, a random sample will be picked. See **Table 3** for acceptance criteria and corrective action.
- 14.7.6 Certified Reference Material: A solid CRM must be extracted with every batch of 8330B soil samples. See **Table 3** for acceptance criteria and corrective action.
- 14.8 Grinding of Vegetation Samples: Remove sample from shipping container and brush off dirt particles. Chop sample into about half inch pieces with clippers or other cutting tool. Place the sample in an aluminum pan and air-dry in an exhaust hood to the appropriate dryness for grinding. It should be dry enough where it won't stick to the inside of the mill. Grind the dried sample to fineness in either the manual sample mill or the Wiley mill or both if needed. Place the ground sample in a container and label immediately. Use 6g for extraction.
- 14.9 Sample Extraction: Get out enough 40 mL vials for each sample, method blank and QC sample to be extracted. Place an Avery label on each vial containing the following information: Lab #, Client name, Type of Analysis, Initial Weight - Final Volume, and the Lab Prep Batch Code. Weigh-out a 2.0 – 2.3 g subsample of each soil (use a blank matrix soil for each method blank and LCS) into the appropriately labeled 40 mL vial. To each QC sample, (LCS, MS&MSD), add 8.0 mL of acetonitrile. Then add 1.0 mL of surrogate (2.0µg/mL) standard and 1.0 ml of 8330 standard spiking solution. To each sample and method blank add 9.0 mL of acetonitrile and 1.0 mL of surrogate (2.0 µg/mL) standard using a 1.0 mL syringe. Cap each vial with a Teflon-lined cap and place in chilled shaker for 16-18 hours. Record the temperature of the chilled shaker daily while shaker is in use.
- 14.9.1 After shaking, allow the sample to settle (10-15 minutes should be adequate). Add 10mL of calcium chloride and centrifuge samples for 10 minutes. (The calcium chloride solution is added to the samples to coagulate suspended particles and remove them from the supernatant.) Make sure the sample is labeled correctly.
- 14.10 Sample Analysis

- 14.10.1 Samples will be prepared, analyzed and reported in batches and will be traceable to their respective batches. Quality control, QC, samples are required with each batch. A method blank, matrix spike/matrix spike duplicate and laboratory control sample is required for each sample matrix batch (normally sets of 20 samples).
- 14.10.2 Analyze the samples using the same conditions as the standards. Compounds identified on the C-18 column must be confirmed by injection on the Biphenyl column with an RPD limit of 40%. If the RPD exceeds 40%, results should be evaluated to determine if coelution or matrix is causing the exceedance and the reason noted. In cases of coelution or obvious matrix interference, the lower concentration may need to be reported. If no anomalies are noted, review the chromatographic conditions. If there is no evidence of chromatographic problems, report the higher result.
- 14.10.3 Identification of a compound is made if a peak is found within the RT window on the C-18 column and then confirmed on the Biphenyl column. Column temperature control is employed so retention time shifts should not be a problem.

15.0 Data Analysis and Calculations

15.1 Quality Systems SOP QS09 "General and Commonly used Laboratory Calculations" provides details on general calculations used throughout the laboratory.

15.2 Data Reduction/Evaluation

15.2.1 Each sample analysis sequence is documented in the HPLC run log. After the samples have been analyzed, the data is reduced using Target. The following must be checked to determine if the sample will need any reanalysis or dilution:

15.2.2 The initial CCV must be within $\pm 15\%$ difference of the calibration curve. See **Table 2** for acceptance criteria and corrective action.

15.2.3 Analyte concentration must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed.

15.2.4 Surrogate recovery should be within the limits established by the laboratory of 40-145% for water and 55-140% for solids/project sample matrix. See **Table 2** for acceptance criteria and corrective action.

15.2.5 After the data has been reduced and determined to be acceptable, it is uploaded into the LIMS and reviewed. Any manual integrations are documented by inclusion of the integrated signals (before and after manual integration) initialed, reason and dated with the quantitation report and chromatogram. Refer to QS07 for guidance.

15.3 Calculations

15.3.1 Calculate the calibration factor for each analyte at each concentration as:

$$CF = \frac{\text{Peak Area (or Height) of the Compound in the Standard}}{\text{Mass of the Compound Injected (in nanograms)}}$$

15.3.2 The mean CF is calculated as follows:

$$\overline{CF} = \frac{\sum_{i=1}^n CF_i}{n}$$

13.3.3 The standard deviation (SD) and the relative standard deviation (RSD) of the calibration factors for each analyte are calculated as follows:

$$SD = \sqrt{\frac{\sum_{i=1}^n (CF_i - \overline{CF})^2}{n - 1}}$$

$$RSD = \frac{SD}{CF} \times 100$$

15.3.4 Calibration verification involves the calculation of the percent drift (linear or quadratic) or the percent difference (average) of the instrument response between the initial calibration and each subsequent analysis of the verification standard. Use the equations below to calculate % Drift or % Difference, depending on the calibration procedure used.

$$\% \text{ Drift} = \frac{(\text{Calculated concentration} - \text{Theoretical concentration}) * 100}{\text{Theoretical Concentration}}$$

where the calculated concentration is determined from the initial calibration and the theoretical concentration is the concentration at which the standard was prepared.

$$\% \text{ Difference} = \frac{(\text{CCV CF} - \text{Average CF}) * 100}{\text{Average CF}}$$

15.3.5 External standard calibration - The concentration of each analyte in the sample may be determined by calculating the amount of standard injected, from the peak response, using the calibration curve. The concentration of a specific analyte is calculated as follows:

A. Aqueous Samples:

$$\text{Concentration } (\mu\text{g/L}) = \frac{[(A_s) (V_t) (D)]}{[(CF) (V_i) \overline{(D)}]}$$

where:

A_s = Response for the analyte in the sample, units may be in area counts or peak height.

V_t = Total volume of the concentrated extract..

D = Dilution factor, if dilution was made on the sample prior to analysis. If no dilution was made, $D = 1$.

\overline{CF} = Mean calibration factor from initial calibration (area/ng)

V_i = Volume of extract injected, μL .

V_s = Volume of aqueous sample extracted, mL.

Using the units specified here for these terms will result in concentration units of ng/mL, which is $\mu\text{g/L}$.

B. Nonaqueous Samples:

$$\text{Concentration } (\mu\text{g/kg}) = \frac{[(A_s) (V_t) (D)]}{[(CF) (V_i) (W_s)]}$$

where:

W_s = Weight of sample extracted, g. The wet weight or dry weight may be used, depending upon the specific applications of the data.

A_s , V_t , D , CF and V_i have the same definition as for aqueous samples.

16.0 Method Performance

Demonstration of Capability (DOC): Each analyst must perform a DOC prior to reporting data. The analyst must prepare (for prep technicians) and analyze (analysts reviewing and reporting data) 4-LCS samples. The data is calculated for accuracy and precision requirements. See **Table 2** for acceptance criteria and corrective action.

17.0 Pollution Prevention

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

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on data assessment and acceptance criteria for Quality Control Measures. **Table 2** of this SOP provides information on QC samples, frequency, and the associated criteria specific to the performance of this method.

19.0 Contingencies for Handling out-of-control or unacceptable data

Quality Control SOP QS05, "Data Deviations / Interpretations / Exceptions: Laboratory Non-Conformance / Corrective Action Procedures, Decision Making Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results", provides details on handling out of control data. **Table 2 and 3** within this SOP also list corrective actions associated with the failure of the various QC samples employed for the performance of this method.

20.0 Waste Management

20.1 Laboratory SOP QS14 on Waste Handling discusses general guidelines for the appropriate handling of wastes and the laboratory program on waste management.

21.0 References

21.1 40 CFR, Part 136; Appendix A

21.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition

- 21.3 National Environmental Laboratory Accreditation Conference; Chap. 5, 2003
- 21.4 DOD Quality Systems Manual for Environmental Laboratories, Ver. 3, Jan. 2006.
- 21.5 DOD Quality Systems Manual for Environmental Laboratories, Ver. 4.1, April, 2009.

22.0 Tables, Diagrams, Flowcharts and Validation Data

- 22.1 Table 1, all applicable parameters, including the surrogates and internals with the applicable RL and lowest calibration standard.
- 22.2 Table 2, 8330/8330A QA/QC summary table.
- 22.3 Table 3, 8330B QA/QC summary table.
- 22.4 Table 4, Technical Completeness / Accuracy Checklist
- 22.5 Table 5, Data Reviewers Checklist 8330/8330A
- 22.6 Table 6, Data Reviewers Checklist 8330B

Uncontrolled Document if Printed

TABLE 1 (DL/LOD/LOQ).

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,3,5-Trinitrobenzene 8330A	0.100	0.200	0.400	mg/Kg
1,3-Dinitrobenzene 8330A	0.100	0.200	0.400	mg/Kg
2,4,6-Trinitrophenylmethylnitramine (Tetryl) 8330A	0.100	0.200	0.400	mg/Kg
2,4,6-Trinitrotoluene (TNT) 8330A	0.100	0.200	0.400	mg/Kg
2,4-Dinitrotoluene (DNT) 8330A	0.100	0.200	0.400	mg/Kg
2,6-Dinitrotoluene 8330A	0.100	0.200	0.400	mg/Kg
2-Amino-4,6-dinitrotoluene 8330A	0.100	0.200	0.400	mg/Kg
2-Nitrotoluene (ONT) 8330A	0.100	0.200	0.400	mg/Kg
3,5-Dinitroaniline 8330A	0.100	0.200	0.400	mg/Kg
3-Nitrotoluene 8330A	0.100	0.200	0.400	mg/Kg
4-Amino-2,6-dinitrotoluene 8330A	0.100	0.200	0.400	mg/Kg
4-Nitrotoluene (PNT) 8330A	0.100	0.200	0.400	mg/Kg
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) 8330A	0.100	0.200	0.400	mg/Kg
Nitrobenzene 8330A	0.100	0.200	0.400	mg/Kg
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) 8330A	0.250	0.500	1.00	mg/Kg
Nitroglycerin 8330A	0.100	0.200	0.400	mg/Kg
PETN 8330A	0.250	0.500	1.00	mg/Kg
1,3,5-Trinitrobenzene 8330A/B	0.0800	0.160	0.320	ug/L
1,3-Dinitrobenzene 8330A/B	0.0800	0.160	0.320	ug/L
2,4,6-Trinitrophenylmethylnitramine (Tetryl) 8330A/B	0.0800	0.160	0.320	ug/L
2,4,6-Trinitrotoluene (TNT) 8330A/B	0.0800	0.160	0.320	ug/L
2,4-Dinitrotoluene (DNT) 8330A/B	0.0800	0.160	0.320	ug/L
2,6-Dinitrotoluene 8330A/B	0.0800	0.160	0.320	ug/L
2-Amino-4,6-dinitrotoluene 8330A/B	0.0800	0.160	0.320	ug/L
2-Nitrotoluene (ONT) 8330A/B	0.0800	0.160	0.320	ug/L
3,5-Dinitroaniline 8330A/B	0.0800	0.160	0.320	ug/L
3-Nitrotoluene 8330A/B	0.0800	0.160	0.320	ug/L
4-Amino-2,6-dinitrotoluene 8330A/B	0.0800	0.160	0.320	ug/L
4-Nitrotoluene (PNT) 8330A/B	0.0800	0.160	0.320	ug/L
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) 8330A/B	0.0800	0.160	0.320	ug/L
Nitrobenzene 8330A/B	0.0800	0.160	0.320	ug/L
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) 8330A/B	0.200	0.400	0.800	ug/L
Nitroglycerin 8330A/B	0.0800	0.160	0.320	ug/L
PETN 8330A/B	0.200	0.400	0.800	ug/L
1,3,5-Trinitrobenzene 8330B	0.080	0.040	0.020	mg/Kg
1,3-Dinitrobenzene 8330B	0.080	0.040	0.020	mg/Kg
2,4,6-Trinitrophenylmethylnitramine (Tetryl) 8330B	0.080	0.040	0.020	mg/Kg
2,4,6-Trinitrotoluene (TNT) 8330B	0.080	0.040	0.020	mg/Kg
2,4-Dinitrotoluene (DNT) 8330B	0.080	0.040	0.020	mg/Kg
2,6-Dinitrotoluene 8330B	0.080	0.040	0.020	mg/Kg
2-Amino-4,6-dinitrotoluene 8330B	0.080	0.040	0.020	mg/Kg
2-Nitrotoluene (ONT) 8330B	0.080	0.040	0.020	mg/Kg
3,5-Dinitroaniline 8330B	0.080	0.040	0.020	mg/Kg
3-Nitrotoluene 8330B	0.080	0.040	0.020	mg/Kg
4-Amino-2,6-dinitrotoluene 8330B	0.080	0.040	0.020	mg/Kg
4-Nitrotoluene (PNT) 8330B	0.080	0.040	0.020	mg/Kg
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX) 8330B	0.080	0.040	0.020	mg/Kg
Nitrobenzene 8330B	0.080	0.040	0.020	mg/Kg
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX) 8330B	0.080	0.040	0.020	mg/Kg
Nitroglycerin 8330B	0.400	0.200	0.100	mg/Kg
PETN 8330B	0.400	0.200	0.100	mg/Kg

Table 2. Organic Analysis by High-Performance Liquid Chromatography (Method 8330 and 8330A)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	Not Applicable (NA).	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
MDL determination	Initial method demonstration required for some states – not required for DoD	Refer to SOP QS09.			
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Retention time (RT) window width calculated for each analyte and surrogate	At method set-up and after major maintenance (e.g., column change).	RT width is ± 3 times standard deviation for each analyte RT from a 72-hour study.	NA.	NA.	
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	One of the options below: Option 1: RSD for each analyte $\leq 20\%$ Option 2: linear least squares regression: $r \geq 0.995$ Option 3: non-linear regression: coefficient of determination (COD) $r^2 \geq 0.99$ (6 points shall be used for second order, 7 points shall be used for third order).	Correct problem then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. Calibration may not be forced through the origin for DoD projects.
Retention time window position establishment for each analyte and surrogate	Once per ICAL and at the beginning of the analytical shift.	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.	NA.	NA.	

Table 2. Organic Analysis by High-Performance Liquid Chromatography (Method 8330 and 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Following ICAL, prior to sample analysis.	All project analytes within established retention time windows. <u>HPLC methods:</u> All project analytes within $\pm 15\%$ of expected value from the ICAL.	Correct problem, rerun ICV. If that fails, repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples (maximum of 20 for non-DoD projects), and at the end of the analysis sequence.	All project analytes within established retention time windows. <u>HPLC methods:</u> All project analytes within $\pm 15\%$ of expected value from the ICAL.	DoD project level approval must be obtained for each of the failed analytes or corrective action must be taken. Correct problem, then rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Retention time windows are updated per the method.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and $> 1/10$ the amount measured in any sample or $1/10$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Laboratory control sample (LCS) containing all analytes to be reported, including surrogates	One per preparatory batch.	QC acceptance criteria specified by client or DoD (appendix G), if available. Otherwise, use in-house control limits. In-house control limits may not be greater than ± 3 times the standard deviation of the mean LCS recovery.	Correct problem. Reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed. In general, the LCS is analyzed on the primary column only.
Matrix spike (MS)	One per preparatory batch per matrix.	For matrix evaluation, use LCS acceptance criteria above.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error. In general, the MS is analyzed on the primary column only.

Table 2. Organic Analysis by High-Performance Liquid Chromatography (Method 8330 and 8330A)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: RPD \leq 30% (between MS and MSD or sample and sample duplicate).	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference. In general, the MSD is analyzed on the primary column only.
Surrogate spike	All field and QC samples.	QC acceptance criteria specified by DoD, if available. Otherwise, use in-house control limits.	For QC and field samples, correct problem then reprep and reanalyze all failed samples for failed surrogates in the associated preparatory batch, if sufficient sample material is available. If obvious chromatographic interference with surrogate is present, reanalysis may not be necessary.	Apply qualifier to all associated analytes if acceptance criteria are not met.	Alternative surrogates are recommended when there is obvious chromatographic interference.
Confirmation of positive results (second column or second detector)	All positive results must be confirmed.	Calibration and QC criteria same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	NA.	Apply P-flag if RPD > 40%. Discuss in the case narrative.	Use project-specific reporting requirements if available; otherwise, use method reporting requirements; otherwise, report the result from the primary column. In general, all spiked batch QC is analyzed on the primary column only.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Demonstrate acceptable analytical capability	Prior to using any test method and at any time there is a significant change in instrument type, personnel, test method, or sample matrix.	QC acceptance criteria published by DoD, if available; otherwise, method-specified criteria.	Recalculate results; locate and fix problem, then rerun demonstration for those analytes that did not meet criteria (see Section C.1.f of DoD QSM 4.1).	Flagging criteria are not appropriate.	This is a demonstration of analytical ability to generate acceptable precision and bias per the procedure in Appendix C. No analysis shall be allowed by analyst until successful demonstration of capability is complete.
LOD determination and verification	Prior to initial analysis then quarterly verification.	See Box D-13 of DoD QSM 4.1			
LOQ establishment and verification	Prior to initial analysis then quarterly verification.	See Box D-14 of DoD QSM 4.1			
Soil drying procedure	Each sample and batch LCS.	Laboratory must have a procedure to determine when the sample is dry to constant weight. Record date, time, and ambient temperature on a daily basis while drying samples.	NA.	Flagging criteria are not appropriate.	
Soil sieving procedure	Each sample and batch LCS.	Weigh entire sample. Sieve entire sample with a 10 mesh sieve. Breakup pieces of soil (especially clay) with gloved hands. Do not intentionally include vegetation in the portion of the sample that passes through the sieve unless this is a project specific requirement. Collect and weigh any portion unable to pass through the sieve.	NA.	Flagging criteria are not appropriate.	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Soil grinding procedure	Initial demonstration.	The laboratory must initially demonstrate that the grinding procedure is capable of reducing the particle size to < 75 µm by passing representative portions of ground sample through a 200 mesh sieve (ASTM E11).	NA.	Flagging criteria are not appropriate.	
Soil grinding blank	Between each sample.	A grinding blank using clean solid matrix (such as Ottawa sand) must be prepared (e.g., ground and subsampled) and analyzed in the same manner as a field sample. Grinding blanks can be analyzed individually or composited. No target analytes detected greater than 1/2 Reporting Limit (RL).	All blank results must be reported and the affected samples must be flagged accordingly if blank criteria is not met.	If the composite grinding blank exceeds the acceptance criteria, apply B-flag to all samples associated with the grinding composite. If any individual grinding blank is found to exceed the acceptance criteria, apply B-flag to the sample following that blank.	
Soil subsampling process	Each sample, duplicate, and batch LCS.	Entire ground sample is mixed, spread out on a large flat surface (e.g., baking tray), and 30 or more randomly located increments are removed from the entire depth to sum a ~10 g subsample.	NA.	Flagging criteria are not appropriate.	
Soil sample triplicate	At the subsampling step, one sample per batch. Cannot be performed on any type of blank sample.	Three 10 g subsamples are taken from a sample expected to contain the highest levels of explosives within the Quantitation Range of the method. The RSD for results above the RL must not exceed 20%.	Corrective action must be taken if this criterion is not met (e.g., the grinding process should be investigated to ensure that the samples are being reduced to a sufficiently small particle size).	Apply J-flag if corrective action does not solve problem and no sample available.	

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Aqueous sample preparation	Each sample.	Solid phase extraction (SPE) using resin-based solid phase disks or cartridges is required.	NA.	Flagging criteria are not appropriate.	
Initial calibration (ICAL)	Minimum of 5 calibration standards with the lowest standard concentration at or below the RL. Once calibration curve or line is generated, the lowest calibration standard must be re-analyzed.	The apparent signal-to-noise ratio at the RL must be at least 5:1. If linear regression is used, $r \geq 0.995$. If using Internal Standardization, $RSD \leq 15\%$.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	No samples can be run without a valid ICAL. Analysis by HPLC UV, LC/MS, or LC/MS/MS is allowed.
Second source calibration verification (ICV)	Following ICAL, prior to sample analysis.	All analyte(s) and surrogates within $\pm 20\%$ of true value.	Correct problem and verify second source standard. Rerun ICV. If that fails, correct problem and repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples should be run until calibration has been verified.
Continuing calibration verification (CCV)	Prior to sample analysis, after every 10 field samples, and at the end of the analysis sequence.	All target analytes and surrogates within $\pm 20\%$ of the expected value from the ICAL.	Correct problem, rerun calibration verification. If that fails, then repeat ICAL. Reanalyze all samples since the last successful calibration verification.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier to all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results should not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Method blank	One per preparatory batch.	No analytes detected $> \frac{1}{2}$ RL and greater than $\frac{1}{10}$ the amount measured in any sample or $\frac{1}{10}$ the regulatory limit (whichever is greater). Blank result must not otherwise affect sample results.	Correct problem. If required, reprep and reanalyze method blank and all samples processed with the contaminated blank.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply B-flag to all results for the specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid method blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
LCS containing all analytes to be reported	One per preparatory batch.	A solid reference material containing all reported analytes must be prepared (e.g., ground and subsampled) and analyzed in exactly the same manner as a field sample. Recoveries for the LCS must demonstrate the laboratory's ability to meet the project's MQOs.	Correct problem, then reprep and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply Q-flag to specific analyte(s) in all samples in the associated preparatory batch.	Problem must be corrected. Results may not be reported without a valid LCS. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Matrix Spike (MS)	One per preparatory batch per matrix.	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. Percent recovery should be evaluated against LCS limits.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	For matrix evaluation only. If MS results are outside the LCS limits, the data shall be evaluated to determine the source of difference and to determine if there is a matrix effect or analytical error.
Matrix spike duplicate (MSD) or sample duplicate	One per preparatory batch per matrix.	For matrix evaluation only, therefore is taken post grinding from same ground sample as parent subsample is taken. Percent recovery should be evaluated against LCS limits and relative percent difference (RPD) < 20%.	Examine the project-specific DQOs. Contact the client as to additional measures to be taken.	For the specific analyte(s) in the parent sample, apply qualifier if acceptance criteria are not met.	The data shall be evaluated to determine the source of difference.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Confirmation analysis	When target analytes are detected on the primary column using the UV Detector (HPLC) at concentrations exceeding the Limit of Detection (LOD).	Calibration and QC criteria are the same as for initial or primary column analysis. Results between primary and second column RPD \leq 40%.	Report from both columns.	If there is a > 40% RPD between the two column results, data must be P-flagged accordingly.	Confirmation analysis is not needed if LC/MS or LC/MS/MS was used for the primary analysis. Secondary column – Must be capable of resolving (separating) all of the analytes of interest and must have a different retention time order relative to the primary column. Any HPLC column used for confirmation analysis must be able to resolve and quantify all project analytes. Detection by HPLC UV, LC/MS or LC/MS/MS. Calibration and calibration verification acceptance criteria is the same as for the primary analysis.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

Table 4, Technical Completeness / Accuracy Checklist

1. Were all the QC check elements analyzed – refer to Table 2 or 3 of this SOP
2. Were the QC criteria met
3. In cases of failures, was there an NCR written
4. Were all manual integrations signed
5. Were dilution factors applied correctly
6. Was there supervisory approval for manual integrations on standards and QC samples
7. Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
8. If the data was entered into LIMS manually, was a check of all entered values performed
9. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
10. Were proper data qualifiers applied to the data in LIMS
11. Was the hard copy package checked for completeness to include all data for the sequence such that the data reviewer could reconstruct sample analyses and validate / approve the data

Table 5, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8330 and 8330A

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Does the curve consist of at least five Calibration Standards?	_____	_____	_____	_____
2. Is the low RL standard in the calibration curve?	_____	_____	_____	_____
3. Are the % RSDs within QC limits for all analytes?	_____	_____	_____	_____
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have QC criteria been met?	_____	_____	_____	_____
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples and at the end of the sequence?	_____	_____	_____	_____
2. Are the % differences within QC limits for all analytes?	_____	_____	_____	_____
D. Sample Analysis				
1. Are all sample holding times met?	_____	_____	_____	_____
2. Was pH checked and recorded for all water samples?	_____	_____	_____	_____
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?	_____	_____	_____	_____
4. Are all compounds identified on the primary column confirmed on the secondary column?	_____	_____	_____	_____
5. Are Surrogate recoveries within QC limits?	_____	_____	_____	_____

**ANALYST DATA REVIEW CHECKLIST
8330 and 8330A (Explosives)**

QA/QC Item	Yes	No	NA	Second Level Review
E. QC Samples				
1. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than 1/2 the LOQ?	_____	_____	_____	_____
2. Is the Laboratory Control Sample and its percent recovery within QC limits?	_____	_____	_____	_____
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits?	_____	_____	_____	_____
F. Others				
1. Are all nonconformances included and noted?	_____	_____	_____	_____
2. Are all calculations checked at the minimum frequency?	_____	_____	_____	_____
3. Did analyst initial/date the appropriate printouts and report sheets?	_____	_____	_____	_____
4. Are all sample ID and units checked for transcription errors?	_____	_____	_____	_____
5. Are all manual integrations checked by a second reviewer to verify why they were performed?	_____	_____	_____	_____

Comments on any "No" response:

Analyst: _____

Second Level Review: _____

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Table 6, ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: 8330B

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Does the curve consist of at least five Calibration Standards?	_____	_____	_____	_____
2. Is the low RL standard in the calibration curve?	_____	_____	_____	_____
3. Are the % RSDs within QC limits for all analytes?	_____	_____	_____	_____
B. Second Source Verification				
1. Was the initial calibration curve verified by a second source calibration standard (ICV) and have QC criteria been met?	_____	_____	_____	_____
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 20 samples and at the end of the sequence?	_____	_____	_____	_____
2. Are the % differences within QC limits for all analytes?	_____	_____	_____	_____
D. Sample Analysis				
1. Are all sample holding times met?	_____	_____	_____	_____
2. Was pH checked and recorded for all water samples?	_____	_____	_____	_____
3. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?	_____	_____	_____	_____
4. Are all compounds identified on the primary column confirmed on the secondary column?	_____	_____	_____	_____
5. Are Surrogate recoveries within QC limits?	_____	_____	_____	_____

**ANALYST DATA REVIEW CHECKLIST
8330B (Explosives)**

QA/QC Item	Yes	No	NA	Second Level Review
E. QC Samples				
1. Is the Grinding Blank extracted at the desired frequency and is its concentration for target analytes less than ½ the LOQ?	_____	_____	_____	_____
2. Is the Method Blank extracted at the desired frequency and is its concentration for target analytes less than ½ the LOQ?	_____	_____	_____	_____
3. Is the Laboratory Control Sample and its percent recovery within QC limits?	_____	_____	_____	_____
4. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and is the percent recovery/RPD within QC limits?	_____	_____	_____	_____
5. Are the soils triplicates (DUP1/DUP2), if applicable, extracted at the desired frequency and is the RSD within QC limits?	_____	_____	_____	_____
F. Others				
1. Are all nonconformances included and noted?	_____	_____	_____	_____
2. Are all calculations checked at the minimum frequency?	_____	_____	_____	_____
3. Did analyst initial/date the appropriate printouts and report sheets?	_____	_____	_____	_____
4. Are all sample ID and units checked for transcription errors?	_____	_____	_____	_____
5. Are all manual integrations checked by a second reviewer to verify why they were performed?	_____	_____	_____	_____

Comments on any "No" response:

Analyst: _____

Second Level Review: _____

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

ORGANICS: SOP 343 REVISION #: 02 EFFECTIVE DATE: 20101117

**BNA & Pesticide/PCBs & TPH NON-AQUEOUS MATRIX
(MICROWAVE EXTRACTION) USING SW-846 METHOD 3546**

APPROVALS:

Lab Director:  Date: 11/30/10

Data Quality Manager:  Date: 11/30/10

Section Supervisor:  Date: 11/30/10

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Changes Summary

Revision 02, 11/17/2010

- SOP has been updated to reflect added volume correction for BNA surrogate
- clarified addition of sodium sulfate to microwave tubes
- Corrected # of samples associated to each of the specified microwave methods and added the name of the method identified on the instrument.

Revision 01, 09/09/2010

- SOP has been updated to reflect the correct QS SOPs and include missing solvent/spike information.

Revision 00, 08/01/09

- Review of SOP indicated no changes were necessary
- Additional requirements, based upon the DoD QSM 4.1, have been integrated into the routine sample flow; however, if the requirement is different from routine sample flow, then the requirement is outlined and documented as such to be followed only when DoD samples are analyzed.

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BNA & Pesticide/PCB & TPH NON-AQUEOUS MATRIX
(Microwave Extraction)
Using SW846 METHOD 3546

1. SCOPE AND APPLICATION

- a. This SOP describes the extraction of BNAs, pesticides/PCBs, and TPHs from soil, sediment, sludges and waste solids by an automated method (3546).

2. SUMMARY

- a. Soil and solid samples are mixed with sodium sulfate and extracted with solvent in a Microwave extractor for BNAs, Pesticides/PCBs, or TPHs. The extracts are then concentrated by a Turbo Vap concentrator.

3. INTERFERENCES

- a. Solvents, reagents, glassware, and other sample processing apparatus can add interferences to sample analysis. Method blanks must be extracted under the same conditions as samples to demonstrate freedom from interferences.
- b. Phthalate esters commonly found in plastics can interfere with the analysis. Plastics should be avoided.
- c. Soap residue can degrade certain analytes such as aldrin and heptachlor. Glassware should be solvent rinsed to avoid this problem.

4. APPARATUS AND MATERIALS

- d. Stainless Steel spatula
- e. Microwave extractor unit with 40 position carousel, electronic components, and ample ventilation
- f. Microwave extraction Teflon tubes, capacity approximately 75mL
- g. Suitable Teflon cap and screw-top lid
- h. Drying column (Chromatographic column) – 20mm I.D. x 300mm
- i. Vial – 2mL clear with Teflon-lined screw cap
- j. Vial – 12mL clear with Teflon-lined screw cap
- k. Syringe – 1mL, 500uL
- l. Pasteur pipet – 9” length
- m. Pasteur pipet bulb
- n. Labels – Dymo
- o. Aluminum foil – heavy duty
- p. Nitrogen tank – equipped with pressure regulator
- q. TurboVap Concentrator with 200mL concentrator tubes
- r. Teflon funnels for pouring off
- s. Balance – capable of weighing to 0.1grams
- t. Aluminum pie pans for mixing samples
- u. Filter paper – 185mm

5. REAGENTS

- a. Sodium Sulfate (Na_2SO_4) – Granular, anhydrous, trace pure 10-60 mesh (purchased in bulk containers from Fisher #S415-10S or equivalent)
- b. Methylene Chloride (Please read SOP – 336 before handling this solvent in our laboratory) (Dichloromethane) – suitable for spectrophotometry and gas chromatography (Fisher #D151-4 or equivalent)
- c. Hexane – suitable for spectrophotometry and gas chromatography (Fisher #H303-4)
- d. Surrogate/Spike Solutions – Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes or if the initial concentration of stock is different than that listed below:
 - i. **BNA Surrogate (100ug/mL)** – The base neutral and acid surrogates are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor. Use 0.5mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 0.5mL of 100ppm BN Surrogate spiking solution.)**
 - ii. **BNA Spiking Solution #1 & #2 (100 ug/mL)** – The base neutral and acid spiking solutions are mixed together in one solution. This solution is prepared in methanol by making a dilution of stock purchased from a reputable vendor with same compounds as for calibration. Use 0.5 mL of this solution per 15g of non-aqueous sample. **(For low-level PAHs use 1.0mL of 1.0 ug/mL LL PAH spiking solution.) The BNA Spiking solutions contain all targets that are calibrated for GC/MS. DOD QSM requires all targets to be spiked in the LCS and MS/MSD.**
 - iii. **TCMX/DCB(2,4,5,6-Tetrachloro-meta-xylene/Decachlorobiphenyl) Surrogate solution** is prepared in acetone by making a cut on stock purchased from a reputable vendor. 0.5mL at 0.5 ug/mL of this solution is added per 15g of non-aqueous sample.
 - iv. **PCB Spiking Solution** – Arochlor 1016/1260 or the PCB of choice (1242, 1248, 1254, or 1260 are the most common) is prepared in acetone at a concentration of 5.0ug/mL. PCB stock is usually purchased from RESTEK or equivalent. The PCB to use may be determined by viewing historical data or asking the GC operator. Use 0.5mL per 15.0g of non-aqueous sample.
 - v. **Pesticide Spiking Solution** – A spiking solution is prepared at 1.0 ug/mL. Use 0.5mL per 15g of non-aqueous sample.
 - vi. **TPH Surrogate** – Surrogate solution is prepared in acetone by diluting stock ortho-terphenyl standard to a final concentration of 20 ug/mL. Use 1mL per 15 grams of sample.
 - vii. **TPH Spike** – A spiking solution is prepared by extractions analyst that has a concentration of 1000 ug/mL in acetone.

6. SAMPLE COLLECTION, PRESERVATION, AND HOLDING TIMES

- a. Samples are collected in an appropriate size wide-mouth glass jar (4oz. or 8 oz.) with a Teflon-lined cap.
- b. Samples are preserved by cooling to 4°C.
- c. Holding time is 14 days from collection date to extraction.

7. PROCEDURE

- a. All soils have a 14-day holding time counted from the day they are sampled. Determine the samples necessary to extract using the following information. (DO NOT extract samples for which you have no information.):
 - i. Each day a backlog is generated in the LIMS providing all relevant sample information, including samples numbers and respective analysis required.
 - ii. Samples requiring RUSH turn around time may be logged in throughout the day which will require your immediate attention. Log-in personnel will generally communicate this need.
 - iii. Check the backlog throughout the day to re-evaluate priority if needed.
- b. Wearing lab coat, gloves, and safety glasses, get samples from cooler. Samples must be signed out of the walk-in refrigerator. Enter the sample numbers, your initials, and the date and time removed on the log provided. Inspect as to whether they are in glass and have a Teflon lid. Find out if any special dilutions need to be made for this client. If the sample has a particularly bad matrix or a strange matrix, see your supervisor to find out if a microwave extraction is truly necessary.
- c. Get twice the number of aluminum pie pans to prepare the number of samples you have plus any additional spikes of LCSs and a method blank. A method blank and LCS must be processed with each set of samples. A matrix spike, a duplicate or a matrix spike duplicate and a LCS must be processed for each analytical batch (up to a maximum of 20 samples). Using the LIMS, create a batch of samples and print off sample labels. The LIMS will create a unique batch sequence number.
- d. Decant and discard any water layer on a sediment sample by carefully pouring this off into a trashcan.
- e. Dump the entire sample into an aluminum pie pan and mix sample thoroughly with a spatula until mixture is homogenous. Discard any foreign objects such as sticks, leaves, and rocks.

It is extremely important that waste (when appropriate), soil and sediment samples be mixed thoroughly to ensure that the sample is as representative as possible of the sample media. The most common method of mixing is referred to as quartering. The quartering process should be performed as follows:

- *The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.*

- *Two quarters should then be mixed to form halves.*
- *The two halves should be mixed to form a homogenous matrix.*

This procedure should be repeated several times until the sample is adequately mixed.

NOTE: Samples that are clay type materials should be handled in a different manner. Due to these type sample matrices having an affinity to stick to most anything that touches it, another approach must be followed. Obtain a representative sub-sample aliquot from the center or middle section of the sample container

Place an aluminum pie pan on the balance and zero it. Calibrate balance with ASTM class-1 Troemner weights or equivalent, bracketing desired weight (50g, 20g, 10g, 5g, 1g). Record calibration in the Extraction calibration/temperature logbook. Using a spatula, transfer the appropriate weight, {10-20 grams depending upon client or project specific Detection Limits (DL) and/or Reporting Limits (RL)}, of a representative sample to the nearest 0.1 gram. Normally 10 or 15g sample weights are used. Record this amount on your label. Put your label on the side of the 400-mL beaker. For spiking purposes, weigh 3 aliquots of the appropriate sample. Pick a sample with a good matrix, one that mixes well, non-oily, etc.

- Add ~ 15 grams of sodium sulfate to the aluminum pie pan. Using a spatula and/or a glass rod, mix the sample thoroughly with the sodium sulfate until it becomes a sandy texture. If necessary, add additional sodium sulfate. When removing the spatula or glass rod from the mixed sample, leave behind all the sample possible. Cover the aluminum pie pan with foil and continue to weigh up the remaining samples. For the method blank and LCS, pour up approximately 15g of sodium sulfate in microwave tube. The matrix used for the method blank and LCS must be free of the analytes of interest and processed through the same analytical steps as the samples.
- Carefully transfer samples to microwave tubes. Make sure samples are loaded in the rack in the order of the bench sheet.
- Verify the amount of surrogate/spike to add to the sample prior to addition. It can change if a different detection limit is required or the volume of sample being analyzed changes. Set out the surrogate/spike at least ten minutes before use to allow it to warm to room temperature. Someone must verify that the surrogate/spike has been added by watching and signing off on bench sheet.
- Surrogate: **BNA/PAH** - using the 1-mL glass syringe designated for BNA surrogate, add 0.5 mL of BNA surrogate to each sample, spike, and blank. **Pest/PCBs** - using the 1.0-mL glass syringe marked TCMX/DCB surrogate, add 0.5 mL of TCMX/DCB surrogate to each sample, blank and spike. TPH – use the appropriate 1.0-mL glass syringe to add 1.0 mL of the appropriate surrogate to each sample, blank and spike.
- Spiking: For the BNA sample in each analytical batch selected for spiking, use the 0.5-mL glass syringe marked Base Neutral Acid Spiking to add 0.5 mL of the Base Neutral Acid Spiking solution. **(For low level PAHs use 1.0 ml of the 1.0µg/mL PAH spiking solution.)**
For Pest/PCB samples, determine if the sample will require a Pesticide

Spike and/or a PCB Spike. Proceed as follows:

Pesticide and PCB - set up two LCS's – one for Pesticide getting an AB MIX spike and one for PCB, which should be spiked with PCB 1660. In addition to the LCSs, a matrix spike/matrix spike duplicate is necessary for the pesticide. Prepare a PCB matrix spike/ matrix spike duplicate if requested by the client.

Pesticide only – To the sample in each analytical batch selected for spiking, add 0.5 mL of Pesticide Spike (Mix A&B) with a glass syringe dedicated for Pesticide Spike.

PCB only - To the sample in each analytical batch selected for spiking, add 0.5 mL of PCB 1016/1260 (unless otherwise specified, 1248 for BB&L) using a 1.0 mL glass syringe dedicated to that PCB.

For TPH - To the sample in each analytical batch selected for spiking, add 1mL of the appropriate spiking solution (i.e. DRO or TNEPH or MAEPH) using a 1.0 mL glass syringe dedicated to that spike.

- k. **Solvent:** Add 30mL methylene chloride for BNA/PAH/TPH extractions or 30ml hexane for Pest/PCB extractions.
- l. Place a Teflon cap and Teflon screw top on the Teflon microwave tube. Using the cap tightener station, tighten the caps and invert sample to insure proper mixing and check for leaks in cap.
- m. Place microwave tubes in microwave carousel making sure they are in order and spaced evenly throughout the carousel to insure proper heating while in microwave. Note position on bench sheet.
- n. Place microwave carousel in microwave making sure the carousel is properly lined up with the turning mechanism.
- o. Choose saved program option based on total number of samples to extract and begin process by pressing the start button. The program is set to EPA method 3546 specifications. **Note the method used on the bench sheet.**

For 1-16 samples (800W):

Method: 3546 800W 100% 16-Express

Max power: 800W

Ramp time: 15:00

Control temperature (in Celsius): 110

Hold time: 10:00

Cool down: 5:00

For 17-40 samples (1600W):

Method: 3546 1600W 100% 40-Express

Max power: 1600W

Ramp time: 15:00

Control temperature (in Celsius): 110

Hold time: 10:00

Cool down: 5:00

- p. Allow samples to cool in the carousel for an additional 30 minutes before attempting to handle the extracts.
- q. Transfer the extract to a pre-rinsed turbo vap tube by first passing through a funnel with P4 filter paper sodium sulfate. All tubes and funnels should be pre-rinsed with Methylene Chloride. After pouring the extract into the turbo, rinse the microwave tube 3 times with the extraction solvent and transfer the rinsate to the turbo. Finally, rinse the funnel with an adequate amount of the extraction solvent using a Teflon squirt bottle. This ensures optimum transfer of all compounds of interest.
- r. Now concentrate the extract to 1.0mL using the turbovap concentrator.
 - i. **Turbo-Vap Operation:** Adjust the pressure of nitrogen gas tank to 50 psi. Make sure the tank has 200 psi or more on the main valve. The temperature of the bath should be approximately 45°C. The pressure target range should be about 20-25 psi.
 - ii. Place the turbo vap tube in the Turbo-Vap. Be sure to push tube down so the tip slides into the sensor well. Close the lid to start concentration. Check that each position with a tube has an orange light showing. If the orange light is not steady, bubbles may be detected by the sensor and need removal. (See Turbo-Vap manual).
 - iii. When the beep sounds indicating the end of concentration, the extract will be at approximately 1 mL. Remove the tube from the bath.
- s. BNA and TPH samples need to be concentrated to ~1.0mL while Pesticides and PCB should be concentrated to ~5.0mL in turbo vap. Using clean solvent, rinse turbo with Pasteur pipet and bring sample to volume in sample vial.

8. DOCUMENTATION OF CAPABILITY (DOC)

- a. Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS08 for guidance.

9. WASTE MANAGEMENT AND POLLUTION PREVENTION

- a. Please see Waste Disposal SOP QS14 for the proper disposal of waste generated from this area.
- b. Quantity of chemicals purchased should be based on expected usage during its shelf-life and the disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

10. METHOD PERFORMANCE

- a. Refer to SOP-201, SOP-211 and SOP-219 for method performance.

11. REFERENCES

- a. EPA Methods SW-846, Method 3546

12. DEFINITIONS

- a. Refer to SOP QS08 for definitions.

13. HEALTH AND SAFETY

- a. Wear appropriate personal protection equipment when working with chemicals or samples.
- b. Use the lab hoods when working with solvents.
- c. Use caution when mixing strong acids or bases. Solutions will become extremely hot when mixing with water. Avoid splashing these solutions so they won't come in contact with the skin or eyes. If this happens, flush with lots of water. Contact your supervisor if serious and medical attention is needed.

Uncontrolled Document if Printed

**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

QUALITY SYSTEMS: QS10

REVISION #: 18

EFFECTIVE DATE: 20110916

LABORATORY SAMPLE RECEIVING, LOG IN AND STORAGE

APPROVALS:

Lab Director:  Date: 9-16-11

Data Quality Manager:  Date: 9-16-11

Section Supervisor:  Date: 9/16/11

Changes Summary

Revision 18, 09/16/11

- The cooler receipt form was revised.
- Added requirement that samples must be refrigerated within 2 hours of opening cooler.
- Added cooler receipt form completion requirement upon opening cooler.
- Updated procedure for pH measurement.
- Changed default time to 00:01 in the case a time is not listed on COC.
- Removed reference to ATSD in SOP 187.

Revision 17, 05/16/11

- The list of employees has been removed from section 8.

Revision 16, 11/17/10

- Added requirement “Notes to sample analytical comments indicating date/time/initials preserved.” for metals samples preserved within house.

Revision 15, 10/13/10

- Updated CRF on page 23, also added at the beginning of page 6 statement about recording the final temperature value.

Revision 14, 09/07/10

- The SOP combines SOPs 404, 406, 410, 415 and 432 into one SOP with updated naming.

Table of Contents

1. Sample Acceptance Criteria
2. Sample Receiving
3. Sample Log In
4. Sample Storage
5. Laboratory Information Management System (LIMS)
6. Daily Follow Up for Sample Receiving/Log In
7. Miscellaneous
8. Sample Storage, Secure Areas, and Sample Custody
9. Sample Custodian's Duties and Responsibilities
10. Procedure for Treatment of Soil Samples from Quarantined Areas
11. Subcontracting Laboratory Samples
12. Attachments

1.0 Sample Acceptance Criteria

This SOP lists in as much detail as possible our daily procedures for sample receiving, log in and storage of laboratory samples. Keep in mind that there may be project specific requirements that are more strict or different than our routine procedures. In these instances, the project specific requirements must be met and followed. Although a few project specific requirements are detailed in this SOP, i.e. DoD certification issues, not every situation can be addressed. If there is ever any uncertainty on what procedures must be followed, please see the Project Manager or your section manager immediately. If ever in doubt, always go with the more stringent requirements. This document will constantly be reviewed and revised as necessary.

- 1.1 A sample may be rejected for compliance purposes if it does not meet the following criteria. Analyses may only proceed after notification and approval to proceed from the client or from the laboratory manager.
 - 1.1.1 Sample must be properly preserved and in the proper container for the requested analysis.
 - 1.1.2 Sample integrity must be maintained. The container shall be intact without cracks, leaks, or broken seals.
 - 1.1.3 Adequate sample volume must be received for the requested analysis, including volume for any requested QA/QC (MS/MSD).
 - 1.1.4 The sample ID on the bottle label must match the sample ID listed on the chain of custody.
 - 1.1.5 The sample container label and the chain of custody must be completed with indelible ink. The sample label must be intact and list all necessary information; to include: sample date, sample time, sampler, and sample ID/location. The chain of custody shall also indicate sample date and time, requested analyses, and all necessary client information.
 - 1.1.6 Sample temperature must be less than 6°C or received on ice.
 - 1.1.7 Sample must be within holding time for the requested analysis.
 - 1.1.8 Samples must be refrigerated within 2 hours of being unloaded.

These issues are discussed in more detail below under the “Sample Receiving” section of this document.

2.0 Sample Receiving

- 2.1 Samples are received at the Empirical Laboratories on 621 Mainstream Drive, Suite 270 Nashville, TN 37228.
 - 2.1.1 The majority of samples are shipped in coolers by couriers such as Federal Express and UPS. All couriers are generally received in the Empirical Laboratories Sample Receiving (SR) area loading dock in back of the laboratory. The laboratory is located close to the Federal Express (FedEx) distribution station, therefore we do pick up our coolers at the FedEx location and transport them back directly to the laboratory. Some coolers and/or samples are delivered directly to the SR area by the sampler and/or client.
 - 2.1.2 Some coolers and/or samples may be received directly by Empirical Laboratories Sample Receiving personnel. If samples are hand delivered by the client make sure that necessary paperwork is included and that you sign and date the chain of custody, as well as record the final value temperature of the samples on the chain of custody as well. If the *Empirical Laboratories Chain of Custody [Attachment II]* is used the white and yellow copy of the chain of custody is retained and the pink copy must be given to the client.

- 2.2 Visually inspect all coolers for tampering, custody seals, (intact if applicable) leakage, etc. If a cooler has been damaged beyond repair, unpack the samples and discard the cooler as to not reuse it. If you suspect a cooler may be damaged or is extremely dirty this cooler must not be reused. If coolers were sent by Federal Express, examine the Federal Express air bills for the number of packages in the shipment and make sure that all the packages (coolers, boxes etc.) in a group have been received. If there are any problems the Project Manager must be contacted immediately. If anything looks unusual, take the time to check it out and document the situation and findings.
- 2.3 Each cooler must be opened and a cooler receipt form must be filled in placed in cooler. Open each cooler in order to quickly inspect the contents and to locate the chain of custody. Sample receiving personnel should wear the following personal protection equipment: gloves, safety glasses and a laboratory coat. Your signature and the date and time the samples were received must be placed onto the chain of custody. The time received must reflect the actual time the samples were received even though they may be logged into the system at a later time. Samples received late in the day during the week may be processed the next morning. All cooler(s) must be opened, examined (for leakage, breakage etc.), the temperature measured and the chain of custody signed and dated to reflect the actual date and time which they were received. The samples must be delivered to the appropriate analytical department or put in cold storage as soon as possible 2 hours after unloading.
- 2.3.1 Attach any shipping receipts, work orders, documentation, etc. to the chain of custody.
- 2.3.2 If a chain of custody or other paperwork is not sent, the client must be contacted and the samples temporarily placed on hold in cold storage. The required information may be found on the sample containers or it may be necessary to call the client to get the missing information (i.e. sample ID, collection date and time, etc.). All attempts to encourage our customers to complete a chain of custody or submit written information for samples must be made.
- 2.3.3 Project specific paperwork may be required. For all projects, a *Cooler Receipt Form [Attachment IV]* must be completed for each cooler received. Sample receiving personnel must begin completing this form as soon as a cooler is received and complete this form as samples go through the log in process.
- 2.4 The temperature of each cooler or set of samples must be measured as quickly as possible using a thermometer with 0.1°C increments. This thermometer must be calibrated against a NIST certified thermometer monthly and this information recorded in a bound notebook. The Certificate of Calibration for the NIST thermometer is kept on file at the QAO's desk. The thermometer must be tagged with the unique identification number of SR#1 and serial #; (Sample Receiving #1), the date calibrated and the correction factor. This information must also be recorded in a bound notebook. Only this thermometer can be used for recording the temperature of sample coolers upon receipt.
- 2.4.1 To measure the temperature, point the IR thermometer at the cooler temperature blank (if supplied) or a direct sample and wait a few seconds for the temperature to stabilize. The IR gun should be held 6 inches away (from the temperature blank or sample) for an accurate reading. Read the temperature to the nearest 0.1 °C. The final value temperature value must also be recorded on the chain of custody. (This value will also be recorded into the LIMS at a later point.). This is calculated by measuring the initial value temperature and adding the correction value temperature (IR temperature gun calibration factor) together, to obtain the final temperature value. All regulatory compliance samples received from North Carolina that does not meet the temperature requirement will be segregated and

the client will be notified of the non-compliance. The samples will not be analyzed until we receive client notification to proceed with analyses.

- 2.4.2 If the temperature exceeds 6°C for any sample, the project manager must contact the client immediately. There may be tighter temperature control limits for specific project requirements. The customer must make the decision to either continue with the analyses or resample. Make sure the client is aware that if the samples are analyzed, the following qualifier is normally included on the final report: "The shipping cooler temperature exceeded 6°C upon receipt to Empirical Laboratories. This may have an impact on the analytical results. The concentration may be considered as estimated." Not all samples for the project will be flagged, just those samples received above 6°C.

Many times we are not able to get in touch with the client quickly and the best judgment on how to handle the samples must be made after discussion with the Project Manager and/or Laboratory Director. The samples may still need to go through the log in process although it may be eventually determined that the samples will not be analyzed or the samples may temporarily be placed on hold and not logged in, with the exception of short hold and rush samples. Above all do not allow the samples to set out at room temperature for an extended period of time while waiting for a decision.

The only exceptions to the 6°C rule are:

- 2.4.2.1 Water samples for all Metals, (except Chrome 6+ and mercury) that have been preserved with HNO₃ to a pH of ≤ 2. *Keep in mind that non-aqueous sample for Metals must be cooled.*
- 2.4.2.2 Samples for Fluoride, Chloride and Bromide.
- 2.4.2.3 Waste/Product samples for all parameters.
- 2.4.2.4 Samples generated in the Aquatic Toxicology laboratories and brought directly to Sample Receiving after they are collected. Sample receiving personnel should place these in cold storage as soon as possible.
- 2.4.2.5 Samples collected locally by Empirical Laboratories personnel or local customers that hand deliver their samples. In some instances these samples may not have had time to cool down; however, these samples should have been placed on ice in an attempt to cool them to the proper temperature. This exception is only applicable if the samples were collected the same day as the laboratory receives them. It should be noted if samples are "Received on Ice" (ROI).
- 2.5 If several coolers are received at once, they must be inspected to determine the order in which the samples should be unpacked and logged in. The following priorities should be given:
- 2.5.1 Any analyses which have a 24-72 hour holding time. It is the log-in person's responsibility to notify the department manager or section group leader of such samples via e-mail and verbally.
- 2.5.2 Any sample which has almost exceeded its' holding time. (Especially watch for this with waters organic extractions, Solids and Sulfides, all of which have only 7 days). A list of parameters and holding times is posted in sample receiving.
- 2.5.2.1 If a sample is received already out of holding time, this must be documented and the project manager must be contacted. The sample can be analyzed at the client's request, but it will be qualified on the final

report as being analyzed out of holding time. The project manager must inform you of the client's need.

- 2.5.2.2 If a sample is received with limited holding time remaining for any parameter it may be necessary to contact the project manager so that he/she can contact the client. If the sample has to be analyzed on a rush basis to meet the holding time a rush charge may apply. Also it may not be possible to analyze the sample within the holding time due to sample load, etc.
- 2.5.3 Samples requiring rush turnaround.
 - 2.5.3.1 If sample(s) require 24 or 48-hour turnaround they will take first priority. Other rush requests also have high priority.
 - 2.5.3.2 The project manager and/or section manager must be contacted for approval concerning any unscheduled rush requests.
- 2.6 Unpack all samples from the cooler. If there are any known or suspected hazards this must be done under a hood. It may be necessary to rinse off the outside of the containers in the sink and/or wipe them off with a paper towel.
 - 2.6.1 Visually inspect them for tampering and custody seals (if applicable). Sort and inventory the samples against the chain of custody by arranging them in the same order as they are listed on the chain of custody. Normally samples are assigned log numbers in the same order as they are listed on the chain of custody but for certain projects or situations it is acceptable to arrange them in a manner which will make them easiest to log in.
 - 2.6.2 Check for leakage and sample container breakage as this could compromise the sample integrity. If any spillage occurred in the cooler make sure this is noted. Also list all the other samples in the cooler as cross contamination could occur. The Project Manager and/or the customer may need to be notified in these situations. It may be necessary to resample.
- 2.7 Check the chain of custody information against the information recorded on the containers. If these do not agree, this must be documented and the Project Manager must be notified.
 - 2.7.1 If major changes are made on the chain of custody received from an engineering job, then the PE should submit written confirmation of these changes or make the corrections and initial them directly on the chain of custody.
 - 2.7.2 Any error found on the chain of custody must be marked through with one line, initialed, dated and the correction written in.
- 2.8 Note any unusual requests, methodology, hazards (known or suspected) to the project manager and/or laboratory section manager or analysts before the samples are actually logged in. Make notes of any problems (improper containers, preservatives, temperature, or descriptions, etc.).

3.0 Sample Log In

- 3.1 After samples have been unpacked, sorted and reviewed, they are then ready to be assigned log numbers and continue through the log in process. Make sure that the parameters for the samples are clearly marked on the chain of custody. Contact the project manager if there are any questions, problems, etc.
- 3.2 Assign a work-order and sample number to each individual sample and record it on each sample container and the chain of custody
 - 3.2.1 All containers with the same description must have the same sample number even if they have different preservatives and require different tests. However, each

different fraction (bottle type and/or preservative) should be designated with a letter (A, B, C, etc.).

- 3.2.2 Grab and composite samples from the same sample location must be considered as separate samples. It may be necessary to use "grab" or "composite" as part of the sample description to distinguish between the samples. Only assign different log numbers to them if the parameters are clearly marked as grab and as composite. Do not assume that VOC must be analyzed from grab samples so therefore the client must have taken a grab sample.
 - 3.2.3 Sample numbers must begin with 001 at the beginning of each year (e.g. 0101001).
- 3.3 Check the following items and record this information on the cooler receipt form to further ensure sample integrity. If any of the following requirements are not met it may be necessary to contact the client. We can perform the analyses in most cases and will do so with the client's approval, however the results may be qualified in some manner on the final report.

Preserving sample integrity throughout the log in procedure must be one of our section's top priorities. This includes not only ensuring that the proper chemical preservatives have been added but also that the samples are received and maintained at the proper temperature. Samples should not set out at room temperature if there is a delay. The samples must temporarily be placed in cold storage until you are able to complete the log in procedure.

[Make sure the VOC containers are not temporarily stored in a non designated VOC only storage area.]

- 3.3.1 Determine if the samples were received at the proper temperature.
- 3.3.2 The sample descriptions on the bottle should match those on the chain of custody.
- 3.3.3 Check to determine if the proper chemical preservatives were added to adjust the sample to the correct pH. All regulatory compliance samples received from North Carolina that does not meet the preservation requirement will be segregated and the client will be notified of non-compliance. The samples will not be analyzed until notification to proceed with analyses is received from the client. A list of parameters and the required chemical preservatives is posted in the log-in room. The verification of this preservation will be recorded on the Cooler Receipt Form for all projects. If Empirical Laboratories prepared and shipped out the sample containers they will have been pre-preserved unless instructed otherwise by the client. Complete traceability of the preservatives used to pre-preserve the sample containers and to preserve samples in the log-in area is required. A bound notebook must be used to trace this information and must include the following: Lot #, Type of preservative, Date Prepped, Amount and Analyst Name. This information must also be labeled on each container, re-pipetter, etc. that the preservative is stored in. Each lot of HNO₃ used for Metals preservation must be tested prior to using them for preservation. These analyses are kept on file.
 - 3.3.3.1 The pH of each container (except VOA vials) which requires pH preservation must be checked. Do not open and check the pH of VOA vials in sample receiving/log-in. This information is then documented on the project cooler receipt form.
 - 3.3.3.2 The pH of preserved samples is checked and confirmed using pH narrow range indicator paper. When the client request pH analysis on samples and they must be reported and measured for pH using the narrow range paper

rather than a pH meter, the accuracy of each batch of indicator paper must be calibrated to the nearest tenth versus certified pH buffer and recorded into a bound logbook in accordance with SW846 method 9041A pH Paper method.

- 3.3.3.3 When taking the pH reading, **DO NOT PUT THE pH PAPER DIRECTLY INTO THE SAMPLE CONTAINER.** Use capillary tube to measure pH. For some samples (wastes) the indicator paper may not be accurate due to interferences. The observation of the appropriate color change is a strong indication that no interferences have occurred. If it appears as if there is interference, the pH must be measured using the pH meter. [See SOP - 187 pH, Electrometric.]
- 3.3.4 The following guidelines must be followed to check pH preservation:
 - 3.3.4.1 Water samples for Cyanide analyses must be preserved to a pH of >12.0 with NaOH upon collection. If the pH of these samples is <12.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to >12.0.
 - 3.3.4.2 Water samples for Metals analyses must be preserved to a pH of <2.0 with HNO₃ upon collection. If the pH of these samples is >2.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to <2.0.
 - 3.3.4.3 Samples requiring analyses which are preserved with H₂SO₄ (i.e., Nitrogen compounds, Total Phenolics, Oil and Grease, Total Phosphorus, etc.) should be preserved to have a pH of <2.0. If the pH of these samples is >2.0 upon receipt, the client must be notified immediately. Upon client approval, the sample should then be adjusted to <2.0. Samples for sulfide analysis must have a pH >9.
 - 3.3.4.4 If a sample is not properly preserved, log-in personnel must either do the following:
 - 3.3.4.4.1 To meet project specific requirements, the client must be notified before preserving or adding additional preservative to the sample unless otherwise instructed. If the client instructs us to add chemical preservatives to a sample, complete traceability of the preservatives used is required.
 - 3.3.4.4.2 For other projects it may be acceptable to preserve the sample accordingly before the sample is placed in storage. Complete traceability of the preservatives used is required.
 - 3.3.4.4.3 All metals samples preserved upon receipt must be held 24 hours before proceeding with analysis. The client must be notified to see if the lab is to proceed with analysis. Notes to sample analytical comments indicating date/time/initials preserved.
 - 3.3.4.5 In some instances it may not be possible to adjust the sample to the proper pH due to matrix problems which cause excessive foaming or require an unusually large amount of acid. Do not continue to add acid if a few mL's of acid does not lower the pH. Notify the Project Manager, Metals Manager and/or analyst. They will make the decision if the sample will be diluted, not analyzed, etc. Make sure you note on the cooler receipt form and in the LIMS notes that the sample is not at the proper pH as well as any useful information (i.e., foaming, strong odor, etc.).

- 3.3.4.6 Samples may be generated in the Aquatic Toxicology Laboratories and brought directly to Sample Receiving after they are collected but before they are preserved. Log-in personnel must preserve the samples accordingly before they are placed in storage. Complete traceability of the preservatives used is required.
- 3.3.5 Check to make sure samples are in proper containers and that there is adequate volume for all the parameters requested and no leakage.
- 3.3.6 If VOA vials are present, each vial must be inverted and checked for head space. "Pea-sized" bubbles (i.e. bubbles not exceeding 1/4 inch or 6 mm in diameter) are acceptable and should be noted. Large bubbles or head space is not acceptable and this information must be documented on the cooler receipt form. If this occurs, the client must be contacted. The samples can be analyzed with their approval, however the report will be qualified and the data may be questionable. All VOA vials will be preserved with Na₂S₂O₃ (0.2g) when chlorine is known to be, or suspected to be present.
- 3.3.7 All chlorinated effluent samples received for Cyanide must be checked for residual chlorine. The one liter sample container should initially contain 1 to 2g/L of Ascorbic Acid. Potassium Iodide starch indicator paper will be used for detecting the presence of residual chlorine. DO NOT PUT THE TEST PAPER DIRECTLY INTO THE SAMPLE CONTAINER. Pour up a small aliquot, neutralize, test and dispose of this volume after the sample is checked. If the test paper turns blue, the sample must be treated for residual chlorine. Add ascorbic acid approximately 0.6g at a time and recheck the sample until there is no residual chlorine present. If the sample required this treatment this information must be included on the cooler receipt form. This must be done by log-in personnel before leaving the receiving area. It may be necessary to notify the Inorganic Manager and/or analyst.
- 3.3.8 Be aware of holding time requirements.
- 3.4 Notify the proper analyst if samples have been logged in for analyses which have a 24-48 hour holding time or if a 1-2 day turnaround has been requested.

4.0 Sample Storage

- 4.1 After samples have been correctly logged in they are then transferred to one of the following cold storage areas and arranged in numerical order by the assigned log in/LIMS sample number. ***Note that aqueous VOC samples must be segregated from all other samples.***
- 4.1.1 The refrigerator in the MS Lab: All aqueous VOC's must be stored in this refrigerator. Storage blanks consisting of organic free water from the laboratory may be required for specific projects. These will be analyzed for VOCs only. ***Storage blanks are required for all DOD projects.***
- 4.1.2 Walk in Refrigerator: All aqueous samples for all analyses must be stored in this refrigerator.
- 4.1.3 Soil Walk-In Refrigerator: All quarantined and non-quarantined soil samples for all analyses must be stored in this refrigerator.
- 4.1.4 VOC Soil Freezer: All soil samples requiring VOC analysis with short hold prep times (Encores, Organic Free Water Terracores, etc.) must be stored in this freezer.

- 4.1.5 VOC Dry Storage Rack: All water VOCs that have exceeded double holding time can be stored on this rack. These samples are stored here segregated alone to ensure no cross contamination occurs between VOC samples and other non-VOC aqueous samples.
- 4.2 Quarantined soils are those quarantined by the US Department of Agriculture. A separate disposal log must be maintained for these soils including the location, date and quantity of the soil received and processed. Soil residues from quarantined samples must be treated according to regulations after testing. Quarantined soils are defined as:
- 4.2.1 Soil taken from much of the southeastern US and parts of New York and Maryland at a depth of three feet or less. *Soils from three feet or more are not regulated provided they are stored separately.* A map of the regulated areas in the United States entitled *Soil Movement Regulations [Attachment VIII]* is posted in the log-in room.
- 4.2.2 All soils taken from foreign sources, US Territories and Hawaii.
NOTE: All soils are treated as quarantined soils and are disposed of in accordance with USDA regulations. Above for information purposes only.
- 4.3 All samples must be stored in one of the four refrigerators detailed above with the following exceptions:
- 4.3.1 Matrices that may be adversely affected by the cold temperature. (E.g. surfactant samples, multi-phase samples).
- 4.3.2 Highly contaminated waste or product type samples that could jeopardize the integrity of other samples in the walk in cooler. Often these can be stored at room temperature. If these require refrigeration see the project manager for other options.
- 4.4 The temperature of each sample refrigerator must be monitored and recorded each day by Wet Chem personnel by the following method. A Mercury thermometer or digital min/max thermometer with 1° increments must be used. Each thermometer must be calibrated against a NIST certified thermometer once a year (**digital thermometers quarterly**) and this information recorded in a bound notebook. The thermometers must be tagged with a unique identification, the date calibrated and the correction factor.
- The tolerance range for all refrigerators is 1 to 6°C. If the temperature exceeds this range, corrective action measures must be put in place immediately. The Wet Chemistry Manager, Organic Manager, and Laboratory Director will be notified in order to assess the situation. It may be necessary to put a service call in to the refrigeration repair service.
- 4.5 All personnel removing samples from any refrigerator must sign them in and out. This is done by completing the *Sample Custody Form [Attachment IX]* which is attached to the door of each refrigerator. These completed forms are kept on file. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the samples are returned and signed back into the appropriate storage area. It must be noted if the entire sample volume was used and that the container was discarded.
- 4.6 The water walk in refrigerator in the sample room is the largest refrigerator and stores a large majority of the samples. A back up compressor is hooked into the system and scheduled to automatically come on if the main compressor fails. There is a digital min/max thermometer, which monitors the temperature 7 days a week.

- 4.7 As stated above the temperatures for all refrigerators that samples are stored are checked each day Monday-Friday and monitored seven days a week with min/max thermometers. Pay close attention to these readings and watch for signs of possible problems.
- 4.8 A temperature maintenance record book is kept for each refrigerator.
- 4.9 Samples must be held for a minimum of 30 days after the final report unless specified otherwise. For USACE projects, samples must be held for a minimum of 45 days after the final report unless otherwise specified. See SOP QS14 entitled Analytical Laboratory Waste Disposal SOP for guidance on disposal of samples.

5.0 Laboratory Information Management System (LIMS)

- 5.1 Log the sample information into the LIMS for each sample. Every attempt should be made to get every sample logged into the LIMS by the end of the day. All information entered should be clearly stated and recorded on the COC provided. After opening the main menu of the LIMS, select the 'Work Orders' tab from the 'Sample Control' drop down menu. Now click on the 'Add' button to create a new Work Order. You will see the following:
 - 5.1.1 Client: Select the client I.D. by clicking on the pull-down and choosing from the client list. This list is in alphabetical order. If the desired client is not on the list, a new client must be created by the project manager or I.T. director.
 - 5.1.2 Project: Click on 'Projects' and choose the project I.D. The projects will be client specific. After the project is chosen the "project information" areas should populate. The 'Project Name,' 'Project Number,' 'TAT,' 'Client Project Manager,' 'Lab Project Manager,' and 'Comments' information should also appear. If there are no applicable project choices, a project must be created by the project manager or I.T. director. There are two types of projects:
 - 5.1.2.1 Internal – Empirical Laboratories projects;
 - 5.1.2.2 External – direct laboratory clients.
 - 5.1.3 Comments: This area is to be used to note any information from the project manager for all work orders of this project. It can also be used to list any work order specific notes; this includes but is not limited to information concerning rush turnaround, deliverables or other QC requirements, analyte concentrations, safety issues, quarantined soils, preservation or matrix problems, etc.
 - 5.1.4 Received By: Enter the name of the person who received the samples.
 - 5.1.5 Logged In By: Enter the name of the person who logged in the samples.
 - 5.1.6 Received: Enter the date and time received separated by a space and using military time. Example: 08/02/2008 08:30.
 - 5.1.7 Project/Package Date Due: After the date and time received have been entered, the date due for both of these fields will be calculated. If this information is not correct or needs to be amended later, check with the project manager before doing so.
 - 5.1.8 Shipping Containers: Click on the 'Coolers' button and enter the temperature and condition upon receipt. If more than one cooler was received, each cooler must be assigned a different name. For example, if these came in by dedicated courier, enter the last four numbers of the Tracking Number as the name. After all of a cooler's information has been entered (received on ice, where custody seals present, preservation confirmed, COC/container labels agree, sample containers in-tact) click the 'Save' button. If more than one cooler was received, click the 'Add' button and repeat the process above, then click 'Done' after all the coolers' info has been saved.
 - 5.1.9 COC Number: If an identifiable COC number is listed, record that ID here.

- 5.1.10 Shipped By: Enter the courier used to deliver the samples. If the samples were picked up by a lab employee or dropped off by the client/representative, enter 'Hand-Delivered.'
- After these items have been completed, click 'Save,' then the 'Samples' button to continue. To begin entering information for a sample, click the 'Add' button on the bottom of the Samples screen.*
- 5.1.11 Sample Name:
- 5.1.11.1 Only abbreviate if description is too long for the spaces allotted in the LIMS. This information should come directly from the chain of custody. The sample ID entered into the LIMS will be the sample ID on the final report.
- 5.1.11.2 If no sample ID is provided, or is indistinguishable from other samples listed, contact the project manager to ascertain distinction in the samples. Include date as part of the description if this is the only way to differentiate the samples.
- 5.1.11.3 When logging in trip blanks that do not have an ID assigned by the client, list them as "Trip Blank # ____". This information should be on the containers. A log book must be kept in the sample kit room which lists all trip blanks and the date they were filled. This will ensure consistency with the descriptions for trip blanks.
- 5.1.12 Collection Date: Enter the date and time the sample was collected. You must use military time and separate by a space. Often the time collected is not given. Although this is a sampling requirement, this information may not be crucial unless a parameter with a short holding time or a data deliverables package is required. In the event that a sample collection time is not listed on the COC or the sample container, a default time of 00:01 can be used temporarily until client verification. Once verified, then the correct sample collection time must be input into LIMS. If the COC and sample containers do not list a collection date and time, this must be documented on the cooler receipt form and the project manager must be notified. All attempts should be made to get all our clients to supply this information.
- 5.1.13 Lab/Report Matrix: Click on pull down and select matrix. Many times it is difficult to discern the matrix if it is not specified on the COC, and log-in personnel must use their best judgment with regard to analytes/methods requested. Keep in mind that the detection limits and units on the LIMS reports are linked to the matrix. In some cases it may be necessary to ask the Section Managers about the matrix selection. Log-in may do a dilution test to distinguish water samples from oil samples if the COC does not clarify a sample matrix if need be.
- 5.1.14 Sample Type: This is used to differentiate between special types of samples (i.e. Field Duplicates, Equipment Blanks, Trip Blanks, etc.). If there is no definite way to determine that a sample should be classified as something else, then "SAMP-Client Sample" will be selected as the sample type. Do not list a sample as anything other than a Client Sample unless noted on the COC or are instructed by the client to do so.
- 5.1.15 Container: Click on the drop down list and select the appropriate bottle type. If multiple bottles are received for the same sample, move down to the next line and select all other containers as required. Repeat this process until all containers for the sample are listed. As each container is entered, an individual number is

- assigned to it by the LIMS system. This number is also listed on the container labels that are printed from the LIMS, and is placed on the corresponding bottle for container tracking purposes.
- 5.1.16 pH (Container Preservative): Use this to document the pH check information taken during sample unpacking. If no preservative was used, then nothing is required in this field.
 - 5.1.17 Comments: Enter any information that is applicable at the sample level.
 - 5.1.18 Field Analysis: Click on field analysis tab and enter field information when provided.
 - 5.1.19 Work Analyses: Select all parameters requested for the sample from this list.
 - 5.1.19.1 If the required test code is not listed, and the sample matrix is not a contributing factor, click the Work Analyses tab to open the All Analyses list. When selecting from this expanded list, be careful to select the proper method as all methods available for the current matrix will be selectable.
 - 5.1.19.2 If any analyses are selected from the All Analyses list, the project manager in charge should be notified so that the correctness of methods and pricing can be checked and updated as needed. Put selected analysis on hold and then let project manager know what was done so they can correct it and/or take it off hold.
 - 5.1.19.3 All preparation codes for analytes are entered and stored by the system independently of the test codes selected, except in the cases of Dry Weight analysis, and TCLP/SPLP preparation (tumbling). In the case of the TCLP/SPLP prep codes, these are entered alongside the other required analyses automatically by the LIMS when a TCLP/SPLP analyte is selected. As for Dry Weight, it is required for all solids testing except in the cases of TCLP/SPLP analysis, Explosives only analysis, and/or any pure product/non-soil based sample when specified by the client.
 - 5.1.20 Analyses Comments: These comments should be used for any notes that only apply to that particular test code.
 - 5.1.21 RTAT: If the Rush Turn-Around Time for this sample is known at the time of log-in, this information should be updated here.
 - 5.1.22 Save: Once all applicable information is entered for a sample, click the save button. At this time the LIMS applies the Laboratory Sample ID to the sample. This is a four part ID code composed of the following:
 - 5.1.22.1 A 2-digit numeral of the year. Example (0811248-06).
 - 5.1.22.2 A 2-digit numeral of the month. Example (0811248-06).
 - 5.1.22.3 A 3-digit numeral of the work order number. This number reset to 001 at the beginning of each month. Example (0811248-06).
 - 5.1.22.4 A 2-digit numeral of the sample number separated by a dash. Example (0811248-06). This number is different for each sample in a work order.
 - 5.1.23 Add/Edit/Copy: Use these selections to add more samples to the work order, or to change existing information prior to label printing.

Once all the tests have been selected and all samples have been added in the work order, a work order summary and all container labels are printed. Labels are checked for accuracy against the containers while being labeled. At this point log-in of this group of samples is complete.

- 5.2 After log-in of a work order is complete, the COC can then be scanned into the system and attached to the work order on the Work Order screen. The work order then must be updated to Available status so as to be seen by the analysts.

6.0 Daily Follow Up for Sample Receiving/Log In

- 6.1 Wipe out the inside of coolers and return all Empirical Laboratories coolers to the sample kit room.
- 6.2 At the end of the day organize all paperwork received and generated for the day. The following should be given to the Project Managers:
 - 6.2.1 The original chains of custody and yellow original or copy of each. The Cooler Receipt Forms will accompany the COC for the project.
 - 6.2.2 Any information (letters, regulatory limits, etc.) from a client which was received with any samples.
- 6.3 All the above information from the day will be reviewed as soon as possible.
 - 6.3.1 If any corrections or changes are required, all laboratory personnel will be notified by distributing a *Sample Log Change Form [Attachment XIII]* through email distribution. A *Sample Log Change Form* by the project manager will also be sent out if a client adds or deletes any parameters, changes sample IDs, etc.
- 6.4 Sample Receiving will distribute the following to the appropriate laboratory personnel:
 - 6.4.1 Copies of the LIMS receiving reports to necessary laboratory personnel.
 - 6.4.2 Original (white copy) chains of custody are given to the project manager.
 - 6.4.3 Copies of any project/sample specific information to the Section Manager and analysts.
- 6.5 Information will be filed as follows:
 - 6.5.1 Chains of custody:
 - 6.5.1.1 Original (white copy) is returned to the customer with the final report along with the CRF.
 - 6.5.1.2 Pink copies should be retained by the sampler.
 - 6.5.2 Sample Log Change Forms
 - 6.5.2.1 Sample Log Change Forms are distributed through email to all laboratory personnel.

7.0 Miscellaneous

- 7.1 All projects which require deliverables or other QC requirements should be listed in the notes section of the LIMS.
- 7.2 If samples are received from a new client or a new job number that is not in the LIMS, a new client code must be set up. This information should be on the chain of custody or it may be necessary to contact the customer if the information is incomplete.
- 7.3 Samples from the Aquatic Toxicity Laboratory (ATL) are logged into the LIMS for billing and long-term tracking purposes. The receiving information and proper assignment of tests are reviewed by the ATL manager. The samples are then logged in by ATL personnel.
- 7.4 All log books used in the Sample Receiving and Sample Storage Areas are numbered. The following log books are presently maintained. All log books must be "Z"ed out. The Sample Receiving Group Leader will review the log books each week to check for completeness.

Log Book ID	Log Book Description
LI014	Trip Blank Prep Log Book

LI009	Tracking of VOC Trip Blanks Shipped
LI011	Quarantined Soil Treatment Log Book
LI012	Acid Neutralization Log Book
LI015	Sample Receiving and Disposal Log Book
LI010	Kit Room Preservation Preparation Log Book

8.0 Sample Storage, Secure Areas and Sample Custody

- 8.1 Empirical Laboratories, LLC is located at 621 Mainstream Dr. suite 270 Nashville, TN 37228 on the first floor. This building is locked and monitored by an alarm system after normal business hours. No unauthorized personnel are permitted within the facility without a proper escort and a visitor's badge. During non business hours, all doors to the building are locked and secured by an alarm system. All front and back doors are locked and only Empirical Laboratories, LLC personnel have a key to access the building. Upon unlocking the door and entering into the laboratory, then the employee is to deactivate the alarm system using the assigned 4 digit alarm code assigned to them by Human Resources. Each employee is assigned their own designated alarm code, with no code being assigned twice. There is a buzzer at the door to Log-in to allow entry for sample and supply deliveries.
- 8.2 Log-in is also responsible for maintaining a Sample Receiving Custody and Disposal Form for samples received. This form is to be filled out electronically and kept on file before the actual disposing of any sample in house. The following information must be logged onto this form:
 - 8.2.1 Client and Log #s
 - 8.2.2 Date/Time Unpacked
 - 8.2.3 Logged In/Numbered By (Initials)
 - 8.2.4 2nd Checked By (Initials)
 - 8.2.5 Date/Time Placed in Cold Storage
 - 8.2.6 Storage Area (Walk In, Blue Air-VOCs, Quarantined Soils, Quarantined-VOC, Other)
 - 8.2.7 Disposed of By/Date
 - 8.2.8 Method of Disposal
- 8.3 Sample extracts and digestates are stored in the following areas:
 - 8.3.1 All metals digestates are stored in the metals instrument laboratory. The transfer from the digestion analysts to the ICAP analysts is documented in the metals digestion log book.
 - 8.3.2 Non - ZHE TCLP extracts are returned to the refrigerator in which the original samples are stored. For ZHE samples, the extract is returned to the refrigerator in which the original VOC sample containers are stored.
 - 8.3.3 Extracts from medium level VOC analyses are also stored in the Soil Walk – in or VOC sample freezer in the VOC Lab.
 - 8.3.4 All Organic extracts are stored in a Beverage Air side by side refrigerator in the organic extraction laboratory.
- 8.4 The generation of all sample extracts/digests and their movement through the laboratory will also be tracked on a laboratory custody sheet or in a log book. The individual performing the processing becomes responsible for the samples at this point. The samples are maintained in the secure possession of the individual processing the samples. When the processing is completed, the extracts are returned and signed back into the appropriate storage area. The metals digestates are not removed from the metals instrument laboratory.

After the analytical results have been reported, the original samples, sample extracts, and digestates will remain in secure storage until they are disposed of in accordance with the Waste Disposal Standard Operating Procedure (SOP QS14).

9.0 Sample Custodian's Duties and Responsibilities

- 9.1 The Sample Custodian is responsible for the receiving; log in, tracking and disposal of all samples. The duties of this position are performed by the persons in the sample receiving section of the laboratory. These individuals are the primary custodian, secondary custodian and section supervisor. Although other laboratory personnel may assist with the duties, this is done under supervision and direction of one of the three individuals listed above. The sample custodians are responsible for the following:
 - 9.1.1 Receive all samples for the analytical laboratory and maintain chain of custody. This includes documenting the validated time/date of receipt.
 - 9.1.2 Maintain the flow of samples through the log in process and make them available to the analysts on a timely manner. This includes prioritizing samples/projects based on turnaround requests and holding times.
 - 9.1.3 Assign the correct laboratory ID sample numbers and validate that this information is properly labeled on the containers and entered into the Laboratory Information Management System (LIMS).
 - 9.1.4 Validate that every sample proceed through all steps of the log in process. This includes checking the following to determine that the sample integrity has been upheld from the time the sample is collected until it is received in the laboratory: proper containers with ample sample volume, correct preservation, sample dates/times to ensure that holding times can be met, condition of the sample containers, headspace of vials for VOC analysis, sample ID discrepancies and completeness of the chain of custody.
 - 9.1.5 Communicate any information or specific requests by the client that are listed on the chain of custody, i.e., method information, detection limits, specific analytes, reporting information, turnaround information, potential hazards etc. They are also responsible for forwarding any additional information that may be received along with the samples, i.e. permit or regulatory information, letters, etc. to the laboratory managers.
 - 9.1.6 The sample custodian is personally responsible for continuing to uphold the sample integrity throughout the log in procedure and until the time when the samples are properly stored and disposed.
 - 9.1.7 Ensure that samples are transferred into the proper storage area and that these secure areas are locked after hours.
 - 9.1.8 Maintain all log books used in the section. These must be kept up to date, complete, neat and orderly.
 - 9.1.9 Maintain the sample receiving and sample disposal areas in a clean, orderly and safe manner.
 - 9.1.10 Follow good laboratory practices and safety procedures.
 - 9.1.11 Communicate all problems, discrepancies, etc. to the section supervisor and laboratory Director.
 - 9.1.12 In situations where the client cannot be contacted, the sample custodian along with the section supervisor must apply the best judgment on how to handle the samples or situation.

- 9.1.13 Complete all the necessary paperwork and section forms including Cooler Receipt Forms, LIMS daily print outs, Sample Receiving Custody and Disposal Form, etc. in a timely manner.
- 9.1.14 Dispose of all samples in a manner that is safe, cost efficient, timely, meets project requirements and is in accordance with hazardous waste regulations.
- 9.1.15 The sample custodian(s) are responsible for compliance of all procedures outlined in this SOP and the following SOPs. They must maintain personal copies of each SOP:
 - 9.1.15.1 SOP QS10 Laboratory Sample Receiving, Login and Storage
 - 9.1.15.2 SOP QS14 Analytical Laboratory Waste Disposal
 - 9.1.15.3 SOP QS11 Field Sampling & Bottle Kit Preparation

10.0 Procedure for Treatment of Soil Samples from Quarantined Areas

- 10.1 This summary is to explain the handling and treatment of soil samples that come from USDA quarantined areas of the United States, territories of the United States and foreign sources. This treatment is done to prevent the spread of pests to other areas.
 - 10.1.1 When soil samples are ready for disposal, separate out soils that are from quarantined areas that need to be treated. Quarantined areas are from the southern United States (see attached maps), from United States territories such as Puerto Rico, and from foreign countries.
 - 10.1.2 Only quarantined non-hazardous soil samples with containers that are less than three feet in depth will be treated by this procedure. Hazardous samples will have to be treated differently. A list of samples to be treated will be determined by the login supervisor.
 - 10.1.3 Log the samples to be treated in the Soil Treatment Logbook as to location, date, and quantity.
 - 10.1.4 Turn the oven on. Place soil samples in their containers uncovered in the oven. After oven reaches 180°C, heat samples for 2 hours. Treat container liners too. When time is up, remove soil samples with gloves or tongs and cool.
 - 10.1.5 After samples have cooled, put them in the non-hazardous soil barrel for disposal.

11.0 Subcontracting Laboratory Samples

- 11.1 Sample receiving is responsible for handling all aspects of shipment of subcontracted samples. Once samples have been confirmed as sub-outs, login then notifies the project manager that subout samples are in house. The project manager then generates a purchase order number for the specific subout samples. Once the purchase order is generated by the project manager, then login prints out a subcontracted chain of custody from LIMS that will accompany the subout samples during transit. Then login packs up the samples into a cooler, ices them down (if necessary) to keep the samples chilled during transit, and then the cooler is shipped to the subcontracted laboratory.
- 11.2 Chain of Custody/Shipping Requirements
 - 11.2.1 When the samples are sent out, a completed chain of custody must be sent with the samples. Make sure to include the following information:
 - 11.2.1.1 Be specific in your analyses request. List the method number if applicable and/or any specific analytes required. This should already have been discussed with the laboratory.
 - 11.2.1.2 List the name of sub contract laboratory and the date shipped or delivered.

- 11.2.1.3 List the Empirical Laboratories; LLC LIMS log # as the sample description on the chain of custody. Do not list the actual client name or actual project information.
- 11.2.1.4 Record the date and time that the samples were sampled on the chain of custody.
- 11.2.1.5 Results and invoice should be sent to the project manager.
- 11.2.2 Two copies of the sub contract chain of custody should be retained. One copy should be stapled to the original chain of custody received from the client and the other should be stapled to the copy in log in.
- 11.2.3 Make sure samples are packed well so they will not break or spill in shipment. Ice must be packed in the cooler to keep the samples cold if chilling is required.
- 11.2.4 A P.O. must be completed and approved by the project manager prior to sample shipment. Sample receiving should then keep a copy of this P.O. for their records.

Attachments to QS10

I	Chain of Custody Record
IV	Cooler Receipt Form
V	List of Short Holding Time Parameters
VII	Sample Receiving Custody and Disposal Form
VIII	Map of Quarantined Soil Areas in the U.S.
IX	Laboratory Sample Custody Form for Walk in Refrigerator
X	Container Codes for the LIMS
XIII	Sample Log Change Form (Green Sheet)

[Attachments II, III, VI, XI, XII, and XIV were removed during the editing process and not added to the QS.]

EMPIRICAL LABORATORIES
COOLER RECEIPT FORM

Cooler Received/Opened On: _____ @ _____
(date) (time)

1. Tracking # _____ (last 4 digits, FedEx)
Courier: ___FED-EX UPS ROUTE WALK-IN
2. Temperature of rep. sample or temp blank when opened: _____ *Degrees Celsius + correction factor 1.6* = _____
3. If Item #2 temperature is 0°C or less, was the representative sample or temp blank frozen? YES NO...NA
4. Were custody seals on outside of cooler? YES...NO...NA
If yes, how many and where: _____
5. Were the seals intact, signed, and dated correctly? YES...NO...NA
6. Were custody papers inside cooler? YES...NO...NA

I certify that I opened the cooler and answered questions 1-6 (initial) _____

7. Were custody seals on containers: YES NO and Intact YES...NO...NA
Were these signed and dated correctly? YES...NO...NA
8. Packing material used? Bubblewrap Plastic bag Peanuts Vermiculite Foam Insert Paper Other None
9. Cooling process: Ice Ice-pack Ice (direct contact) Dry ice Other None
10. Did all containers arrive in good condition (unbroken)? YES...NO...NA
11. Were all container labels complete (#, date, signed, pres., etc)? YES...NO...NA
12. Did all container labels and tags agree with custody papers? YES...NO...NA
13. a. Were VOA vials received? YES...NO...NA
b. Was there any observable headspace present in any VOA vial? YES...NO...NA
14. Was there a Trip Blank in this cooler? YES...NO...NA If multiple coolers, sequence # _____

I certify that I unloaded the cooler and answered questions 7-14 (initial) _____

15. a. On pres'd bottles, did pH test strips suggest preservation reached the correct pH level? YES...NO...NA
b. Did the bottle labels indicate that the correct preservatives were used YES...NO...NA
16. Was residual chlorine present? YES...NO...NA

I certify that I checked for chlorine and pH as per SOP and answered questions 15-16 (initial) _____

17. Were custody papers properly filled out (ink, signed, etc)? YES...NO...NA
18. Did you sign the custody papers in the appropriate place? YES...NO...NA
19. Were correct containers used for the analysis requested? YES...NO...NA
20. Was sufficient amount of sample sent in each container? YES...NO...NA

I certify that I entered this project into LIMS and answered questions 17-20 (initial) _____

I certify that I attached a label with the unique LIMS number to each container (initial) _____

21. Were there Non-Conformance issues at login? YES...NO Was a NCR generated? YES...NO...# _____

Additional Details:

Short Holding Time Parameters

(Immediate-72 hours)

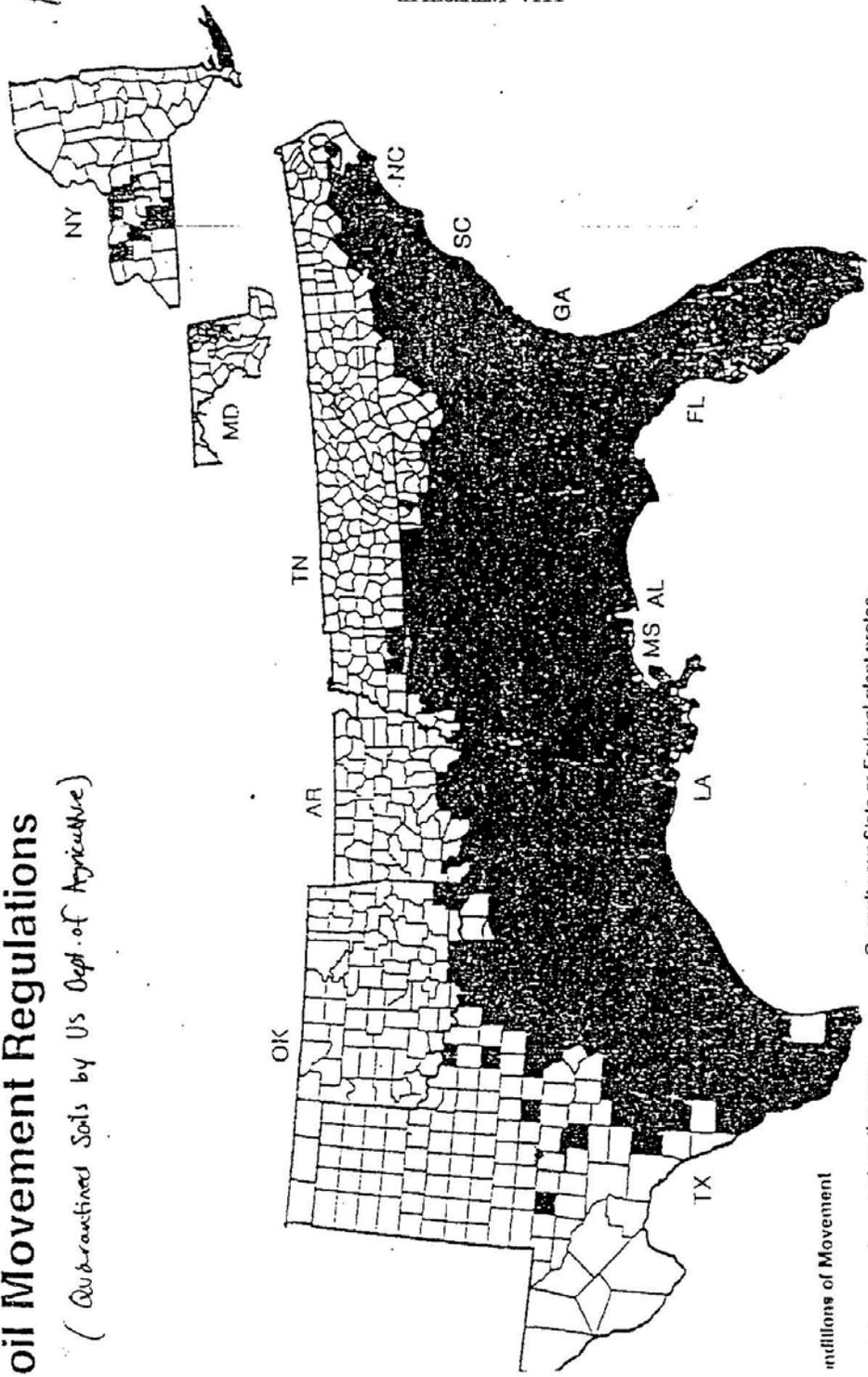
Parameter	Holding Time
pH	Immediate ^a
Sulfite	Immediate ^a
Temperature	Immediate ^a
Residual Chlorine	Immediate ^a
Coliform (Fecal and Total) RCRA/WW	6 hours
Hexavalent Chromium (Cr +6)	24 hours
Odor	24 hours
Coliform (Fecal and Total) <i>Drinking Water only</i>	30 hours
BOD	48 hours
Color	48 hours
Settleable Solids	48 hours
MBAS	48 hours
Orthophosphate	48 hours
Turbidity	48 hours
Nitrite	48 hours
Flashpoint	72 hours ^b

^a Immediate generally means within 15 minutes of sample collection.

^b This is an internal holding time. The method does not specify a holding time.

oil Movement Regulations

(Restrained Soils by U.S. Dept. of Agriculture)



millions of Movement

Restrictions are imposed on the movement of regulated articles from a regulated area as follows:

- 1. Movement into or through white areas.
- 2. Movement within red areas may be regulated.

Consult your State or Federal plant protection inspector or your county agent for assistance regarding exact areas under regulation and requirements for moving regulated articles.

 Regulated Area

Attachment X

Preservatives		Types of Container	
NI	<i>HNO3</i>	A	<i>1 LITER - PLASTIC</i>
NF	<i>HNO3 (Filtered)</i>	B	<i>500 mL - PLASTIC</i>
SU	<i>H2SO4</i>	C	<i>250 mL - PLASTIC</i>
SH	<i>NaOH</i>	D	<i>120 mL - PLASTIC</i>
ZN	<i>ZnAC / NaOH</i>	EN	<i>ENCORE PAK</i>
HY	<i>HCl</i>	F	<i>1 LITER - GLASS CLEAR WIDE MOUTH</i>
		G	<i>1 LITER - GLASS CLEAR BOSTON ROUND</i>
		H	<i>1 LITER - GLASS AMBER</i>
		I	<i>250 ml. - AMBER</i>
		J	<i>VOA VIALS - (40 ml.)</i>
		K	<i>500 ml. - (16 oz)</i>
		L	<i>250 ml. - (8 oz)</i>
		M	<i>125 ml. - (4 oz)</i>
		N	<i>60 ml. - (2 oz)</i>
		O	<i>OTHER</i>
		P	<i>PLASTIC BAG -1 Gallon</i>

Attachment XIII

SAMPLE LOG CHANGE FORM



DATE:

Workorder:

CLIENT:

PARAMETERS:



**EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE**

QUALITY SYSTEMS: QS14 REVISION #: 06 EFFECTIVE DATE: 20100831

ANALYTICAL LABORATORY WASTE DISPOSAL

APPROVALS:

Lab Director:  Date: 9/8/10

Data Quality Manager:  Date: 9/8/10

Section Supervisor:  Date: 9/8/10
mm
9/9/10

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Changes to this Revision – R06 08/31/2010

- Revision to SOP405 R05 dated 6/23/2009.
- Changed the document control and named this as QS14 R06.
- Minor cosmetic/grammatical changes made.

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Analytical Laboratory Waste Disposal Standard Operating Procedure

I. SCOPE AND APPLICATION:

Laboratory waste includes excess client sample waste and waste that is generated while performing an array of analytical services, some of which are hazardous. These wastes must be disposed of in a manner that is safe, cost efficient and in accordance with hazardous waste regulations.

A. Wastes can be broken down into the following categories:

1. Unused portions of actual samples received from outside clients
 - a. Unused aliquots of completed water samples
 - b. Unused aliquots of completed non-aqueous samples
2. Soils from quarantined areas
3. All other soils, sediments, building debris, wipes, oils, etc.
4. Hazardous waste generated within the laboratory as part of numerous analytical procedures.

II. SUMMARY OF PROCEDURES:

A. There are four options for disposing of unused sample portions:

1. Return completed samples and any generated waste from these samples to the client
2. Disposal of samples after confirming that they are non-hazardous.
3. Disposal through a waste vendor in either a sealed drum or lab pack.
4. Treat the sample to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

B. There are two options for disposing of laboratory generated waste:

1. Disposal through a waste vendor in either a sealed drum or lab pack. This is normally done twice a year. The waste must be stored properly until transported off site.
2. Treat the waste to make it non-hazardous and dispose of it as such. (Aqueous pH neutralization only.)

III. EQUIPMENT/APPARATUS:

- A. Proper safety equipment should be in good working condition. This includes gloves, lab coats, safety glasses/goggles, and face shields. Voluntary use of cartridge respirators is allowed (see area manager or QAO).**
- B. USDOT approved drums for storing and shipping hazardous waste.**
- C. Fume hoods.**

IV. PROCEDURE

Waste disposal is done under the management and coordination of the Sample Receiving Manager, Section Managers and the Safety Coordinator.

A. Disposal of completed aqueous samples:

Completed samples are kept in cold storage for a minimum of 45 days from receipt and sample extracts are held for 90 days minimum from receipt.

Engineering support projects involving CLP work, litigation cases, etc. may be saved for longer than three weeks at the request of the project manager.

No samples should be disposed of without approval from the responsible area manager or analyst. **At this point, the area manager and/or analyst will communicate information about samples deemed as hazardous.**

1. The majority of the water samples (ground, surface and drinking) is non-hazardous and is disposed of by pouring them down the sink.
 - a. This must be done under the hooded area located near the sink in sample receiving. Make sure that the sash is closed far enough to produce sufficient ventilation. The tap water should be turned on to supply copious wash for sample disposal.
 - b. Proper safety equipment **must** be used including safety glasses (face shield if necessary), lab coat, and gloves.
 - c. **Be alert to potential problems: for example, separate Cyanide waste from acid waste. Neutralize acid waste that will be poured down the drain and don't mix waste/samples thought to contain Cyanide with samples that are acidified. Also, look for things such as phase separation, odd color, odor, etc. Check with the area manager or Safety Coordinator before disposing of any questionable samples.**
 - d. Tap water must be running when samples are poured out for approximately 10 minutes in order for sufficient flushing and dilution to take place.
 - e. All containers must be rinsed out and thrown into the trash.
 - f. All samples disposed of in this manner must be documented in the bound sample disposal logbook.
2. If water samples are hazardous (known or suspected), one of the following steps must be taken:
 - a. Samples may be returned to the client. If you plan to ship the unused portion back to the client, check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vendor.**

- b. Treat the sample to make it non-hazardous. One example of this is if the sample is highly corrosive, the pH may be adjusted.
- c. Store the sample properly until either a sealed drum or lab pack is sent out.
- d. All samples disposed of in this manner must be documented in the bound sample disposal logbook.

B. Disposal of completed non-aqueous samples:

The majority of non-aqueous samples are soils and sediments. Although there may also be building debris, wipes, oils, and occasionally product type samples.

1. If samples are non-hazardous, they are placed in a sealed drum and destroyed. On specific projects we may also opt to return the unused portions to the client even if they are non-hazardous.
2. If non-aqueous samples are hazardous (known or suspected), one of the following steps must be taken.
 - a. Samples may be returned to the client. If you plan to ship the unused portion back to the client, check with shipping and receiving to make sure that the material can be shipped in accordance with USDOT regulations. **If the samples are not returned to the client they must be stored properly until picked up by a waste vender.**
 - b. Store the sample properly until a lab pack is sent out.
3. Soil samples taken at a depth of three feet or less from areas, which have been quarantined by the US Department of Agriculture (USDA), must first be treated at the laboratory to prevent the spread of any plant pests. The USDA has detailed proper treatment procedures of which we use the following:
 - a. The sample is heated to 180°C (356°F) in a vented oven for two hours.
 - b. After heating the samples they are removed from the oven to cool and then placed in a sealed drum and destroyed.
4. Once the samples have undergone treatment they can then be disposed of by one of the procedures for non-aqueous samples. **All samples disposed of in this manner must be documented in both the bound sample receiving disposal logbook, and soil quarantine logbook with the following information:**
 - a. Client
 - b. Work Order/Sample #s (from LIMS)
 - c. Date(s) treated
 - d. How much sample volume (in ounces) was treated

C. Disposal of laboratory generated waste:

Generated waste is stored outside the building until a waste pick up occurs. This area must be maintained properly.

1. Waste handling and disposal within each laboratory section:

NOTE: Each laboratory analyst and section manager is responsible in assuring that **handling** operations (within their area) are being followed according to the laboratory requirements.

a. General Chemistry/Inorganic

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety coordinator.

- Concentrated acid waste, (**>2% by volume**) and dilute mercury waste (mercury, chemical oxygen demand, total kjeldahl nitrogen and chloride analyses waste) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry.**
- Dilute acid waste (**<2% by volume or less**) are neutralized by using concentrated amounts of sodium hydroxide. Once the pH of the acid waste is neutralized, the acid waste is then poured down a sink drain within hooded ventilation with copious amounts of tap water. The amounts of acid waste treated, the amount of sodium hydroxide used to neutralize the acid waste, final pH of the acid waste, date performed, and date disposed of is then recorded into an acid waste neutralization log book that is kept in sample receiving.
- **All other non-hazardous sample waste, reagents, and standards are poured down the drain with copious amounts of tap water.**

b. Metals

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the inorganic manager or the safety coordinator.

- Concentrated acid waste, aqueous sample waste digestates, and old unused calibration standards (**>2% by volume**) are poured into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and**

amount of waste in the acid waste logbook, then initial and date the entry

- Non-aqueous sample digestate wastes are decanted off the soil/solid samples into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry**
- **Throw the soil/solids in the trash once the acid has been rinsed free.**
- **Cr6 digestates as with all concentrated metal/acid waste are poured into the Acid Satellite Waste drum. Document the type and amount of waste in the acid waste logbook, then initial and date the entry**

****Note: The laboratory 'Acid Waste Logbook' is located in Extractions.**

c. Organic Extraction Laboratory Area

Each analyst performing specific laboratory tests that generates waste is responsible to handle and dispose of the waste in a safe manner and under the guidelines listed below. If you have any questions left unanswered regarding waste disposal within your specific area contact the organic manager or the coordinator.

- Concentrated acid waste is discarded into the Acid Satellite Waste located outside the back of the building inside the caged fence.
- Non-chlorinated solvent waste (Acetone, Ether, Hexane, and Methanol ...etc...) is poured into the Non-Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.
- Chlorinated solvent waste (Methylene Chloride, Chloroform, chlorinated standard and spike waste) is poured into the Chlorinated Waste labeled bottle located in the hood in the Organic Extraction Laboratory.

****Note: Laboratory generated solvent waste is transferred to the appropriate Satellite Solvent Waste Drum (chlorinated or non-chlorinated) weekly or as deemed necessary. Disposal of solvent waste is done under the direction of the organic laboratory manager. These drums are**

located outside the back of the building inside the caged fence and only authorized laboratory staff are allowed to add waste solvent to these drums. The date of addition to the drum, type, and quantity of solvent is entered into the '*Organic Solvent Waste Logbook*' located in Extractions.

- Aqueous sample waste from extracted samples (once the extraction solvent has been removed) is poured down the drain and flush with copious amount of tap water.
- Sodium sulfate waste is dumped into a waste container under an extraction laboratory hood and left overnight or until evaporated. Then the waste is discarded into the trash.

d. **Gas Chromatography (GC)/High Performance Liquid Chromatography (HPLC) Laboratory:**

- Autosampler vials are discarded into the appropriate buckets located in the GC/HPLC Laboratory.
- Sample and spike extract vials are separated according to the contents in the vial. **Acid cleaned extracts** are combined into a separatory funnel and the acid layer separated from the solvent. The acid portion is discarded into the Acid Satellite Waste drum located outside the back of the building inside the caged fence. The solvent waste is discarded into the appropriate solvent waste bottle (chlorinated/non-chlorinated waste) located in the hood in the organic extraction laboratory and then transferred to the appropriate satellite drum when deemed necessary. **Document the type and amount of waste into the appropriate logbook, then initial and date the entry.**
- **Unused stock and working standards** are discarded into the chlorinated solvent waste bottle located in the organic extraction laboratory. The empty vials are rinsed several times with solvent, and the solvent rinsate poured into the solvent waste bottle. Then the vials are discarded into the glassware waste container.

e. **Gas Chromatography/Mass Spectrometry**

- **Volatile sample, standard, and reagent waste:**

Instrument Waste - Aqueous sample waste is collected in waste bottles via waste lines from the instrument. The bottles are emptied into buckets and poured down the drain (pH is < 2% by volume). A small amount of methanol used to clean glassware is also dumped into the bucket and poured down the drain. While disposing of sample waste always run the cold tap water 10-15 minutes. Non-aqueous waste from sample analyses is retained and disposed of in the same manner as the unused sample. Unused sample is held for sample disposal by the sample receiving area (see A and B listed above). Lachat instrument waste and COD waste is collected and poured into the acid satellite drum when deemed necessary. **Document the type and amount of waste in the acid waste logbook, then initial and date the entry.**

Standards - Unused stock and working standards are discarded into the chlorinated solvent waste bottle in the hood located in extractions. The empty vials are rinsed several times with solvent, and the solvent rinsate poured into the solvent waste bottle. Then the vials are discarded into the glassware waste container.

In conjunction with section managers, the sample receiving area disposes of solid sample waste, unused aqueous and unused solid samples (see procedures A and B listed above).

- **Semi-volatile sample and standard waste disposal:**

Methylene chloride waste solvent and standard waste in vials are poured into the chlorinated waste bottle in the hood in extractions. The empty vials are rinsed several times with solvent, and the solvent rinsate poured into the solvent waste bottle. Then the vials are discarded into the glassware waste container.

Auto sampler vials are collected in buckets, and then either consolidated in lab packs or the contents are transferred into the appropriate waste drums.

Lab packs for disposal are done by a licensed waste disposal company.

f. Bioassay Laboratory

- Aqueous sample waste and a small amount of methanol are poured down the drain with copious amounts of tap water. Larger amounts of methanol used for glassware cleaning are collected in beakers and evaporated at room temperature.
- Hazardous or product samples are returned to the client.

D. Consolidation of satellite waste for contractor disposal:

In conjunction with the Safety Coordinator, the sample receiving supervisor is responsible to coordinate waste disposal operations with outside waste disposal contractors.

1. Solvent waste from the areas discussed above is periodically consolidated into two drums located outside the back of the building inside the caged fence. A drum designated either chlorinated or non-chlorinated solvent waste is available to receive the appropriate solvent waste. When the drums become full (fluid surface six inches below the top of the drum), an authorized hazardous waste contractor will be scheduled to remove them to proper waste disposal.
2. The Acid Satellite Waste drum is also disposed through the authorized hazardous waste contractor once the drum is full to the level of six inches below the top of the drum.
3. Consolidated autosampler and standard vials are periodically Lab-Packed in drums and disposed through the authorized hazardous waste contractor.
4. The Safety Coordinator will administer the Waste Disposal Program and maintain current information to track quantities of waste generated and stored on-site.

It is the continuous objective of our laboratory to find ways to decrease the amount of waste generated.

**EMPIRICAL LABS, LLC.
TRAINING ATTENDANCE RECORD**

TRAINING TOPIC: QS14 Rev06 - Waste Disposal

Group: William Schwab

ATTENDEES:					
NAMES (print)	SIGNATURE	REMARK	DATE	TIME	INSTRUCTOR
1 Will Schwab	<i>Will Schwab</i>		9/21/10	15:19	W. Schwab
2 F. Rivera	<i>F. Rivera</i>		9/21/10	15:45	↓
3 Russell Townsend	<i>Russell Townsend</i>		9/21/10	16:00	
4 Veronica Mullen	<i>Veronica Mullen</i>		9-24-10	1300	
5 Penny Cormier	<i>Penny Cormier</i>		9/24/10	1444	
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September 21, 2011



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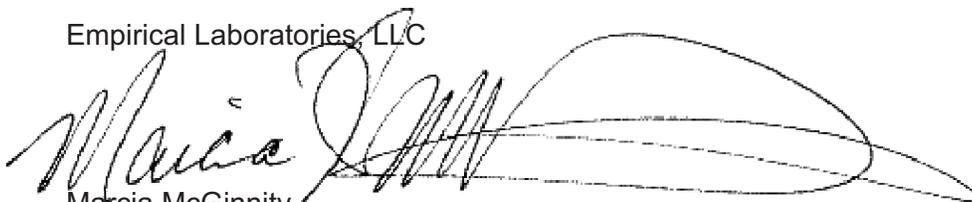
Dear Kelly:

Attached are the DL/LOD/LOQ for our Low-Level PAH analysis. Please let us know if can provide any additional information.

Matrix	Method	Analyte	LOQ	LOD	DL	Units
Solid	8270C/D low	1-Methylnaphthalene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	2-Methylnaphthalene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Acenaphthene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Acenaphthylene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Anthracene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Benzo(a)anthracene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Benzo(a)pyrene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Benzo(b)fluoranthene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Benzo(g,h,i)perylene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Benzo(k)fluoranthene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Chrysene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Dibenz (a,h) anthracene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Fluoranthene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Fluorene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Indeno (1,2,3-cd) pyrene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Naphthalene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Phenanthrene	6.67	3.33	1.67	ug/Kg
Solid	8270C/D low	Pyrene	6.67	3.33	1.67	ug/Kg
Water	8270C/D low	1-Methylnaphthalene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	2-Methylnaphthalene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Acenaphthene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Acenaphthylene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Anthracene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Benzo(a)anthracene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Benzo(a)pyrene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Benzo(b)fluoranthene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Benzo(g,h,i)perylene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Benzo(k)fluoranthene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Chrysene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Dibenz (a,h) anthracene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Fluoranthene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Fluorene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Indeno (1,2,3-cd) pyrene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Naphthalene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Phenanthrene	0.200	0.100	0.0500	ug/L
Water	8270C/D low	Pyrene	0.200	0.100	0.0500	ug/L

Sincerely,

Empirical Laboratories, LLC



Marcia McGinnity
Data Quality Manager