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FINAL REMEDIAL INVESTIGATION SAMPLING AND ANALYSIS PLAN ADDENDUM SITE 43 TOLUENE DISPOSAL AREA NSWC INDIAN HEAD MD

5/1/2013

TETRA TECH

REMEDIAL INVESTIGATION
FINAL SAP ADDENDUM

SITE 43-TOLUENE DISPOSAL AREA

NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

Naval Facilities Engineering Command
Washington

Contract Number N62472-03-D-0057
Contract Task Order 114

May 2013

TETRA TECH

SAP Worksheet (WS) #1 -- Title and Approval Page

REMEDIAL INVESTIGATION
FINAL SAMPLING AND ANALYSIS PLAN ADDENDUM

SITE 43 – TOLUENE DISPOSAL

NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

MAY 2013

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Naval Facilities Engineering Command Washington
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INDIAN HEAD, MARYLAND

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Appendix A Groundwater - Background and Statistical Evaluation
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ACRONYMS

1,1-DCA	1,1-dichloroethane
1,1-DCE	1,1-dichloroethene
1,2-DCA	1,2-dichloroethane
bgs	below ground surface
cis-1,2-DCE	cis-1,2-dichloroethene
CLEAN	Comprehensive Long-Term Environmental Action Navy
COPC	chemical of potential concern
CSM	conceptual site model
CTO	Contract Task Order
DNAPL	dense non-aqueous phase liquid
DO	dissolved oxygen
DPT	direct push technology
DQO	data quality objective
DSR	data summary report
E/A&H	ENSAFE/Allen & Hoshall
EPA	Environmental Protection Agency
EPC	exposure point concentration
EU	exposure unit
FOL	field operations leader
FS	feasibility study
FTMR	Field Task Modification Request
HHRA	human health risk assessment
HI	hazard index
HQ	hazard quotient
HSA	hollow stem auger
IDW	investigation-derived waste
IH	Indian Head
ILCR	incremental life-time cancer risk
LOD	limit of detection
MCL	Maximum Contaminant Level
MDE	Maryland Department of the Environment
MDL	method detection limit
MIP	membrane interface probe
mg/kg	milligrams per kilogram
NAD	North American Datum

ACRONYMS (CONTINUED)

NAVFAC	Naval Facilities Engineering Command
NSF	Naval Support Facility
ORP	oxygen reduction potential
PA	preliminary assessment
PAL	project action level
PID	photoionization detector
PSL	Project Screening Level
QL	quantitation limit
RCRA	Resource Conservation and Recovery Act
RI	Remedial Investigation
RME	reasonable maximum exposure
RSL	Regional Screening Level
SAP	Sampling and Analysis Plan
SI	Site Investigation
SOP	standard operating procedure
SSP	site screening process
TCE	trichloroethene
UCL	upper confidence limit
UFP	Uniform Federal Policy
USGS	United States Geologic Survey
VC	vinyl chloride
VOC	volatile organic compound
µg/L	microgram per liter
$\mu g/m^3$	micrograms per cubic meter

1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) Addendum was prepared by Tetra Tech Inc. for the Washington Division of the Naval Facilities Engineering Command (NAVFAC) under Contract Task Order (CTO) 114 under the Comprehensive Long-Term Environmental Action Navy (CLEAN) contract number N62472-03-D-0057. This document is an addendum to the Final SAP (Tetra Tech, 2011) for Site 43 – Toluene Disposal Area (the "Site") at Naval Support Facility (NSF) Indian Head (IH) in Indian Head, Maryland (**Figures 1-1 and 1-2**). An Interim Data Summary Report (DSR) (Tetra Tech, 2012) for Site 43 was issued in April 2012 and presented the investigative results from 2011 and also included a SAP Addendum outlining additional Remedial Investigation (RI) fieldwork needed to address the original study questions presented the Final SAP.

1.1 SCOPE AND OBJECTIVES

This SAP Addendum summarizes the RI results and presents the revised CSM for Site 43. The primary objective of this SAP Addendum is to present the revised CSM, new study goals and additional sampling necessary to complete the RI at Site 43. A crosswalk is presented in **Table 1-1** which provides a guide to the revised Uniform Federal Policy (UFP) SAP worksheets, where they can be found in this SAP Addendum, and reference to the unchanged portions of the final UFP SAP. The format of this SAP Addendum mirrors the previous SAP Addendum for Site 43 for consistency and to facilitate team review.

1.2 SAP ORGANIZATION

This SAP Addendum consists of three sections. **Section 1.0** provides an introduction. **Section 2.0** provides the revised CSM, including facility description, environmental investigative history, geology and hydrogeology, nature and extent of contamination, and project quality objectives. **Section 3.0** summarizes the project tasks for additional monitoring well installation, soil gas sampling, groundwater sampling, and surface water and sediment sampling proposed to complete the RI and support the feasibility Study for Site 43.

2.0 CONCEPTUAL SITE MODEL

2.1 FACILITY DESCRIPTION

NSF-IH is located in Charles County, Maryland, 30 miles south of Washington, D.C. The facility is positioned along the Potomac River at the confluence of Mattawoman Creek, as shown on **Figures 1-1 and 1-2**, and comprises about 3,500 acres. The Main Area, on the Cornwallis Neck Peninsula, is approximately 2,500 acres. The Stump Neck Annex is approximately 1,000 acres and is separated from the Main Area by Mattawoman Creek. NSF-IH has been active since 1890 and assumed its current name in 2005.

NSF-IH is generally surrounded by commercial, residential, and State Park land to the east and south of the Main Area and Stump Neck Annex. The town of Indian Head is located just east of the facility where mostly residential neighborhoods are located. The Indian Head Highway (Route 210) extends eastward from NSF-IH main gate, attracting businesses and providing access to residential areas off the main highway. The Potomac River borders the main installation to the north and west, and Stump Neck area to the west. Mason Neck National Wildlife Refuge is located across the Potomac River, north of the Main Area. The Mattawoman Natural Environment Area is state owned property located along the southern edge of Mattawoman Creek east of the Main Area.

2.2 SITE BACKGROUND

Site 43 - Toluene Disposal includes two separate areas in the southwestern portion of the Main Area (**Figure 1-3**). The first is near a utility pole approximately 30 feet northwest of and across the road from Building 1041, and the second is the northern corner of Building 1040 (**Figure 1-3**). The Site is an industrial area where the handling of explosives and rocket production still occurs in Building 720.

A relatively steep earthen, grass-covered bunker is located immediately northwest of the utility pole near Building 1041. The area northeast of this vicinity is relatively flat and covered with mowed grasses and a few trees. The area near Building 1040 also is relatively flat. The area surrounding the building southwest of Schuyler Road is mostly covered with mowed grasses. The area northeast of Schuyler Road is mostly covered with unmowed grasses and trees. Refer to the Tetra Tech (2009a) Site Screening Progress (SSP) Report for additional information and Site photographs.

Facilities within Site 43 were reportedly active between the late 1950s and 1989 when approximately 23,000 to 30,000 gallons of spent solvents, were used in the Cast Plant (Buildings 1040 and 1041) to remove propellants and oily residues from various metal parts, and were disposed of directly on the

ground outside Building 1040 (where acetone contamination has been detected) at the drainage ditch and at the utility pole across from Building 1041 (where acetone and toluene contamination has been detected) (NEESA, 1992). This dumping took place two or three times per week for a period of more than 2 years in Building 1041. No signs of stressed vegetation or solvent dumping stains have been observed at Site 43; however, the historical dumping activities have impacted soil and surficial aquifer groundwater (Tetra Tech, 2011).

Operational history and/or functions of nearby buildings were reviewed to evaluate other potential sources of groundwater contamination (Tetra Tech, 2009a). According to the ENSAFE/Allen & Hoshall (E/A&H) (1994) Site Inspection (SI) Report, acetone was known to have been used in Building 1040 (Parts Cleaning House). Based on information provided by NSF-IH, trichloroethene (TCE) was reportedly used for production processes in Building 720 (Grain Manufacture and Motor Load Building), which is adjacent to Building 1040 (**Figure 1-3**). The duration of use and quantities of TCE are not documented. Other buildings near Building 1040 include Building 1360 (Scrap Powder Shed; approximately 80 feet south), Building 1257 (Transformer Station; approximately 110 feet north-northwest), and Building 821 (Toilet Building; approximately 150 feet west-southwest). It is not likely that these other buildings are the source of TCE detected in shallow groundwater.

A review of sewer lines was conducted to determine whether a leaking sewer could be a source of TCE detected in shallow groundwater (Tetra Tech, 2007). There are no sanitary sewers in the immediate vicinity of Building 1040 (**Figure 1-3**). The nearest sanitary sewer begins at Building 821 (Toilet Building), located across Gallery Road from Buildings 720 and 1040, and runs approximately 300 feet northwest and north to the former sewage treatment plant. Another sanitary sewer is located approximately 200 feet northwest of and parallel to Gallery Road. There are also sanitary sewers associated with Building 713 (offices – not shown on figures), Building 714 (change house, laundry, and offices), and Buildings 812 and 813 (lunch houses), all located north to north-northwest of Building 1040 near the former sewage treatment plant. The distance between the former sewage treatment plant and Building 1040 is approximately 400 feet. These observations indicate that the sewers are not a likely source of the localized shallow TCE contamination near Building 1040.

A short length of storm sewer exists emanating from Building 720 that empties into a drainage swale /ditch adjacent to Building 1040 and Schuyler Road (**Figure 1-3**). Based on topography and visual observations in the field, water in the ditch flows to the southeast.

The building descriptions above are from the report entitled *Water Pollution Study at the Naval Ordnance Station* (Dodohara, 1976). Although the building uses may have changed since 1976, this older report provides a historical perspective of nearby activities and sewer lines that are a potential source of

groundwater contamination. The uses of the buildings served by nearby sanitary sewers do not appear to be sources of the contaminants detected in shallow groundwater.

2.3 SITE ENVIRONMENTAL INVESTIGATIVE HISTORY

A list of important Site 43 historical events and relevant dates in the site chronology through 2012 is shown below. The identified events are illustrative, not comprehensive, and a written summary is provided below.

DATE	EVENT	REPORT
1991	Preliminary Assessment Basewide	Preliminary Assessment (NEESA, 1992)
1993	Site Inspection Site 43	Site Inspection (E/A&H, 1994)
2005	Site 43 SSP	Site Screening Process Report (Tetra Tech, 2009a)
2005-2007	Site 43 Supplemental Phase 1 SSP	Site Screening Process Report (Tetra Tech, 2009a)
2009	Site 43 Supplemental Phase 1A SSP	Site Screening Process Report (Tetra Tech, 2009a)
2011-2012	Site 43 Phase 1 and 2 RI	Phase 1 and 2 RI (Tetra Tech, 2012)

A multi-site Preliminary Assessment (PA) was performed at NSF-IH in 1991 (NEESA, 1992). The PA recommended a SI for the area near the utility pole across the road from Building 1041. Sampling was not recommended for Building 1040 because acetone would readily volatize under ambient conditions rather than migrate through soil to groundwater, so the SI was performed in the Building 1041 area in 1993 (E/A&H, 1994). Toluene and chlorinated solvents were detected at soil-gas sampling locations near the utility pole. The SI recommended additional sampling to determine whether volatile organic compounds (VOCs) were present in subsurface soil over a larger area.

A multi-phase SSP Investigation began in 2005 and continued through 2008 to determine whether residual contamination in soil or groundwater at the Site posed potential risks to human health or the environment (Tetra Tech, 2009a). TCE (54,000 micrograms per liter [µg/L]) and cis-1,2-dichloroethene (cis-1,2-DCE) (530 µg/L) were detected in shallow groundwater in monitoring well S43MW01 (near Building 1040), posing potential risks to human health under a residential use scenario; however, a contaminant source area was not found. The presence of cis-1,2-DCE is an indicator of biotic TCE degradation which, if complete, will ultimately generate vinyl chloride that degrades further to produce innocuous carbon dioxide, chloride, and water.

Supplemental SSP efforts continued (termed Supplemental Phase 1 SSP) and membrane interface probe (MIP) technology was used to investigate and delineate VOCs in the subsurface. Several VOCs were

detected near Building 1040; however, TCE was the only constituent detected at a significant concentration (>36,000 µg/L). TCE was detected again at Building 1041 in well S43MW02 at 3.2 µg/L.

The next phase of the supplemental SSP effort (termed Supplemental Phase 1A SSP) was additional delineation of VOCs in shallow groundwater at the Site, utilizing MIP technology. The results of this delineation indicated the potential for unacceptable human health risk from exposure to VOCs in groundwater. Therefore, a recommendation was made for a RI and Feasibility Study (FS).

The Phase 1 RI began in 2011 with the intention of characterizing the nature and extent of VOCs, explosives, and metals contamination in soil and groundwater near Building 1040 (Tetra Tech, 2012). During the Phase 1 RI, 13 soil borings were advanced at selected locations and soil samples were collected from each of the soil boring locations above the water table. The samples were analyzed for VOCs, metals, and explosives. TCE was detected at one location with a concentration of 3,940 milligram per kilogram (mg/kg). Soil gas samples were collected around Buildings 1040 and 720 at approximately 5 feet below ground surface (bgs) to evaluate whether vapor intrusion posed a potential concern for these buildings. Three VOCs were detected at elevated levels including acetone, cis-1,2-DCE, and vinyl chloride (VC). Groundwater grab samples were also collected from six temporary well locations and were analyzed for VOCs, total and dissolved metals, and explosives. In addition, field test kits were used to evaluate ferrous iron and presence of dense non-aqueous phase liquid (DNAPL) at each of the temporary well locations. TCE was detected at five of the temporary well locations. In addition, four permanent monitoring wells were installed during the investigation and all six monitoring wells were analyzed for VOCs, total and dissolved metals, and explosives. TCE was detected in groundwater at all of the monitoring well locations with a maximum concentration of 10,900 µg/L at S43MW01.

Based on the results of the Phase 1 RI, an Interim DSR and SAP Addendum was submitted presenting the results and additional sampling needed to address the existing study goals and to further define nature and extent of contamination (Tetra Tech, 2012). The sampling results identified a potential soil contaminant source area along a ditch (south side of Schuyler Road) east of the suspected disposal area (Building 1040). The extent of groundwater contamination (VOCs, explosives, and metals) near Building 1040 had not been delineated and local groundwater flow could not be determined. In addition, the VOC detections in soil gas (acetone, cis-1,2-DCE, and VC) indicated potential concern for vapor intrusion into Building 720 and the elevated cobalt detection in groundwater at S43MW002, near Building 1041 was confirmed.

The Phase 2 RI began in 2012 to further define the potential soil source area along the ditch, delineate groundwater contamination, determine local groundwater flow direction in both portions of Site 43

(Building 1040 and 1041), further evaluate potential vapor intrusion into Building 720, and further investigate cobalt in groundwater near Building 1041.

As a part of the Phase 2 RI, 12 soil samples were collected from six locations along the ditch north and east of building 1040, two indoor air samples were collected inside Building 720 to evaluate vapor intrusion into Building 720, and 81 groundwater grab samples were collected from 48 temporary well locations at various depths and analyzed for VOCs only. VOC results were quickly evaluated to determine where additional temporary well sampling locations were needed to define the groundwater plume. Once the plume was adequately defined, 23 new monitoring wells were installed. A total of 29 monitoring wells were sampled for VOCs, total and dissolved metals, and explosives. TCE was detected at 19 of the 29 monitoring well locations with a maximum concentration of 17,500 µg/L observed at S43MW01. In addition, two seep grab samples were collected from two drainage channels west of Strauss Avenue and analyzed for VOCs. TCE was detected at a maximum concentration of 354 µg/L at S43SP01-01.

Further detail regarding the sampling results and contamination at Site 43 is provided in **Section 2.6**, Nature and Extent of Contamination.

2.4 GEOLOGY

NSF-IH is underlain by unconsolidated sand, gravel, and clay from the Pleistocene and Cretaceous Periods. The geology varies both laterally and vertically, due to the fluvial depositional environment, and many units swell/pinch depending on the direction. The soil in this area consists of silty and sandy loams, with minor amounts of gravel. The soils tend to have low permeability and low shrink-swell potential.

The geology at Site 43, down to 50 feet bgs, can be characterized by clay and sand layers, with very little dip, which overlie an organic rich basal clay or hard expanding silty clay. **Figure 2-1** illustrates on plan view transects A-A', B-B', C-C', D-D', and E-E'. **Figures 2-2** (A-A' and B-B'), **2-3** (C-C' and D-D'), and **2-4** (E-E') show each cross-section with the associated geology, monitoring wells, and sampled intervals.

Across Site 43, the lithology of the first 20 feet is fairly consistent except in areas where the subsurface has been reworked. In undisturbed areas the first 7 feet is predominately a silty clay with varying amounts of sand and in areas that have been disturbed (roads, buildings, regarding, etc.) is a sandy gravel. From 7 to 20 feet bgs, the lithology is predominately sand with a clay layer that can pinch out or swell depending on the direction. The clay layer ranges in thickness from less than a foot to in excess of 10 feet thick (southeast corner) and shows varying amounts of silt with a smaller percentage of sand. The clay is gray in color and has a high plasticity. The sand is poorly sorted fine to medium grained with varying amounts of silt and very little gravel and is tan, brown, or grayish-brown in color. A few deviations

from this generalization do exist. In the southeastern corner of the Site around S43TW10, S43TW26/S43MW10, and S43TW39/S43MW09S the sand is found in thin layers (less than a foot thick) or consists of a higher percentage of silt/clay than seen elsewhere (**Figure 2-2**). In the southwestern corner of the Site around S34TW46/S43MW24, S43TW47/S43MW25, S43TW48, and S43TW52 the area appears to have been reworked with 5 to 16 feet of fill consisting of large gravel (>2 inches in diameter), sand, and silt (**Figures 2-3 and 2-4**).

Below 20 feet, the lithology is drastically different across Site 43. In the northern and central portions of the Site the lithology is predominately sand with a clay layer that pinches out or swells depending on the direction. This clay layer exhibits the same properties as the clay seen from 7 to 20 feet bgs. The sand deeper than 20 feet is generally coarser, better sorted, and contains less silt. Gravel, anywhere from half an inch to in excess of two inches, was found within the sand but at varying percentages and of limited extents. Underlying this sand is a dark gray to black organic rich clay (basal clay) which is found at depths ranging from 28 (north) to 37 (central) feet bgs across the study area. The top of this basal clay dips slightly to the south and was found to be in excess of 15 feet thick. Plant debris, including wood and decaying leaves, was seen in almost all cases to varying degrees. In the southern portion of the Site a dense gray, sometimes greenish-gray or reddish-gray, hard expanding silty clay was encountered anywhere from 15 to 20 feet bgs. The transition from the lithology observed in the northern and central portion to the southern portion seems to take place very abruptly along a WNW to ESE trending line from just north of S43TW52 (**Figure 2-4**) to just south of S43TW45 (**Figure 2-3**). The silty clay had much lower moisture content than the shallower silty clay layers or basal clay seen elsewhere. Drilling into this silty clay proved very difficult. The drill rig either hit refusal and/or the silty clay expanded in the tooling which made collecting lithology information and deeper groundwater grab samples unfeasible. Although no water bearing zones were seen in macrocore samples below this silty clay, multiple attempts were made to collect deeper groundwater grab samples. Screen-point samplers were advanced into the subsurface until refusal at these locations, and most attempts were not successful in obtaining groundwater for sampling. Only one groundwater grab sample was collected at S43TW48 from 40 to 44 feet bgs (**Figure 2-3**).

2.5 HYDROGEOLOGY

The shallow groundwater beneath Site 43 is unconfined. The water table at Site 43 was consistently encountered between 8 and 26 feet bgs and is recharged by precipitation that infiltrates the ground surface (**Figure 2-5**). Drainage ditches parallel almost all the roads at Site 43. These ditches are mostly dry, with most of the precipitation infiltrating into the soil except during heavy or prolonged rain events. During dry periods, standing water does occur in the ditches just north of Leary Road, west of Gallery Road, and south of Schuyler Road due to leaking water utilities. A groundwater divide is assumed to

exist somewhere near Gallery Road and has been observed in other areas of the Cornwallis Neck Peninsula. The surficial ditches east of the divide flow to the east, surficial flow ultimately continues into a small stream and eventual downgradient discharge to Mattawoman Creek. The stream starts approximately 100 feet north of the Schuyler and Gallery intersection and flows southeast just north of the S43MW09 well cluster. Brown and Root Environmental (1997) reported that combined runoff at the Site flows toward and eventually discharges to Mattawoman Creek through a series of drainage ditches and storm water pipes. The discharge point is more than 2,000 feet south of the Site. On the west side of the groundwater divide, the surficial ditches are all dry except during significant rain events. These ditches drain through culverts into drainage channels west of Strauss Avenue, and ultimately into the Potomac River. These drainage channels are fed not only by storm water runoff but also by shallow groundwater daylighting in seeps. Evidence of heavy rain events is seen on the sandy beach between the cliff and the Potomac River where several rills scour the surface before entering the river.

Shallow groundwater flow is in two directions from the groundwater divide that is located approximately along Gallery Rd. Groundwater flow is predominantly west towards the Potomac River and to the southeast toward Mattawoman Creek. A potable water line leaking near the Site may also be affecting the local water table and actual groundwater flow. **Figure 2-5** presents the potentiometric surface map for July 2012.

2.6 NATURE AND EXTENT OF CONTAMINATION

The nature and extent of contamination at Site 43 is described in the following sections. Investigative results of laboratory analyses of soil and groundwater have been compiled to detail the known extent of contamination observed at Site 43. A sample location map of soil and groundwater sampling locations is presented as **Figure 2-6**.

2.6.1 SOIL

Between the late 1950s and 1989, approximately 23,000 to 30,000 gallons of spent solvents, used in the Cast Plant (Buildings 1040 and 1041) to remove propellants and oily residues from various metal parts, were disposed of directly on the ground outside Building 1040, at the drainage ditch and at the utility pole across from Building 1041. Soil sampling along the drainage ditch located next to Building 1040 has indicated a soil source area of VOC-contamination. VOC analytical results have confirmed the presence of surface and subsurface soil contamination at depths of up to 10 feet bgs (**Figure 2-7**).

Between 2005 and 2012, 12 surface soil and 25 subsurface soil borings were collected at Site 43 and analyzed for explosives, metals and VOCs. There were no Project Screening Level (PSL) exceedances of

explosives; however, aluminum, arsenic, chromium, cobalt, iron, manganese, and vanadium exceeded the PSLs for soil (Environmental Protection Agency [EPA] Regional Screening Levels [RSLs] or Maryland Department of the Environment [MDE] Cleanup Standards for residential soil). Aluminum, chromium, and vanadium PSL exceedances were consistent with background values for the Site. The soil analytical results are provided in **Table 2-1**.

One surface soil sample exceeded the TCE PSL of 440 mg/kg at a concentration of 2,720 mg/kg at S43SB027-0001, also the location of the highest subsurface TCE PSL exceedance of 3,940 mg/kg, confirming the presence of TCE at ground surface to 8 feet bgs (**Figure 2-7**). Subsurface soil samples S43SB11, S43SB17, S43SB18, S43SB24, S43SB25, S43SB26, S43SB27 and S43SB28 (located along the source area drainage ditch) exceeded the TCE PSL of 440 mg/kg at concentrations ranging from 470 to 3,940 mg/kg, confirming the presence of TCE at depths from 5 to 10 feet bgs (to the water table) and the source of VOC-contamination.

Based on the sampling results, the soil source area has been adequately defined and risk will be evaluated as a part of the RI.

2.6.2 GROUNDWATER

Due to the presence of VOC-contaminated soil from improperly disposed spent solvents, groundwater investigation activities were performed from 2005 to 2012 to determine the extent of groundwater contamination at Site 43. Eight VOCs were identified as chemicals of potential concern (COPCs) because the maximum concentrations exceeded the PSLs. The analytical results for groundwater collected from monitoring wells and groundwater grab samples are provided in **Tables 2-2 and 2-3**, respectively. EPA RSLs for tap water (2012) and MDE Cleanup Standards for groundwater are included for comparison and the most conservative values for each medium are used as the PSL for each constituent.

In 2005, in order to determine if solvents had leached into the groundwater near the areas of suspected contamination, monitoring wells S43MW01 and S43MW02 were installed near Buildings 1040 and 1041, respectively, and analyzed for explosives, total and dissolved metals, and VOCs. Groundwater in S43MW01 exceeded the PSL of 2.8 µg/L for cis-1,2-DCE at 580 µg/L and the PSL of 0.44 µg/L for TCE at 53,000 µg/L, initiating further groundwater investigation along the drainage ditch located next to Building 1040 and throughout Site 43 (**Table 2-2** and **Figure 2-8**).

From 2005 through 2012, as part of the continuing groundwater investigation, a total of 94 groundwater grab samples were collected from 59 temporary well locations throughout Site 43 at depths ranging from

6 to 37 feet bgs. The samples were analyzed for VOCs in order to define the extent of groundwater contamination and determine additional placement of permanent monitoring wells (**Table 2-3** and **Figure 2-9**).

Temporary wells were initially installed in the area surrounding S43MW01 (S43MW03 to S43MW09). As a result of the concentrations of TCE in the groundwater grab samples from S43TW06 to S43TW09; additional temporary wells were installed throughout the site to the southeast and west of these locations (**Table 2-3** and **Figure 2-9**). TCE exceedances in groundwater grab samples collected from S43TW20 to S43TW23 (in the approximate area of the groundwater divide) defined an area of groundwater contamination flowing west southwest. Therefore, additional groundwater grab samples were collected from temporary well locations extending to the west near Buildings 715 and 716, to the southwest near Buildings 1134 and 717 extending to the Potomac River, and farther along the source area drainage ditch to the southeast (S43TW39).

Based on the analytical results from the groundwater grab samples, an additional 27 monitoring wells (S43MW03 to S43MW26) were installed throughout Site 43 in 2012 (**Figure 2-6**). All monitoring wells were sampled and analyzed for explosives, total and dissolved metals and VOCs, with the exception of the monitoring wells located near Building 1041 that were sampled for select VOCs and metals based on the historical sampling at S43MW02. Groundwater analytical results for explosives exhibited low level PSL exceedances of 0.15 µg/L for 3-Nitrotoluene in 13 of the 29 monitoring wells at concentrations ranging from 0.222 µg/L to 0.871 µg/L (**Table 2-4** and **Figure 2-10**).

Groundwater analytical results for total and dissolved metals exhibited PSL exceedances of arsenic, barium, beryllium, cadmium, chromium, cobalt, iron, manganese, nickel, and vanadium (**Table 2-5** and **Figure 2-11**). Arsenic, barium, beryllium, cadmium, chromium, manganese and vanadium concentrations were consistent with background values for the Site (**Appendix A**). However, cobalt and iron exceeded the background values of 15.6 µg/L and 19,900 µg/L, respectively, in several monitoring wells. In addition, all monitoring wells excluding S43MW09D, S43MW10, and S43MW19D exhibited concentrations of cobalt in total and dissolved groundwater samples exceeding the PSL of 0.47 µg/L, ranging from 1.95 µg/L to 130 µg/L. **Figure 2-12** represents the distribution of cobalt throughout the study area. The distribution of iron (**Figure 2-11**) shows the highest concentrations within the footprint of the VOC plume and is an indicator of increase biological activity and that biodegradation is likely occurring.

Groundwater analytical results for monitoring wells exhibited PSL exceedances of the following VOCs 1,1-dichloroethane (1,1-DCA), 1,1-dichloroethene (1-1-DCE), cis-1,2-DCE, TCE and VC (**Table 2-2** and **Figure 2-8**). TCE concentrations in groundwater exceeded the PSL of 0.44 µg/L in 19 of the 29 monitoring wells ranging from 0.54 µg/L to 17,500 µg/L in S43MW01. Cis-1,2-DCE concentrations in

groundwater exceeded the PSL of 2.4 µg/L in 8 of the 29 monitoring wells, ranging from 26.4 µg/L to 400 µg/L in S43MW01.

Figure 2-13 shows the estimated horizontal extent of TCE-contaminated groundwater at Site 43. The TCE isoconcentration lines depict the overall footprint of the groundwater plume and also encompasses the extent of other observed VOC exceedances. Two distinct areas of TCE greater than 1,000 µg/L are located east and west of the groundwater divide. On the east side, S43MW01 and S43MW06 are located along the source area drainage ditch and flow southeast towards S43MW09S and S43MW09D. On the west side S43MW17S, S43MW17D, S43MW19S, S43MW19D, and S43MW20 encompass a larger area and flow west towards the Potomac River. The plume area measures approximately 11 acres. The width, thickness, and depth of the TCE-contaminated groundwater varies based on location. The width ranges from approximately 550 feet to the west of the Site along the Potomac River where the plume is undefined, to 100 feet near Building 1040 (the source), then widens to the east of Building 1040 to 300 feet, finally tapering off to the southeast to a width of 50 feet in an area that requires additional delineation. The depth ranges from approximately 8 to 38 feet bgs, where the Basal Clay unit is encountered.

Figure 2-14 shows the estimated vertical extent of the VOC-contaminated groundwater on cross-section E-E'. VOC-contaminated groundwater is encountered entirely through the cross-section view beginning at approximately 8 feet bgs and extending to approximately 38 feet bgs. The area surrounding Building 1040 (the source area) includes the highest concentration of VOCs and the plume follows the sand (varying silt/clay) layers underneath the silt/clay to the east and west, including the zone above the Basal Clay unit as it flows towards the Potomac River.

2.6.3 SOIL GAS/INDOOR AIR

Soil gas samples were collected in the vadose zone around Buildings 1040 and 720 in 2011. Samples were collected at approximately 5 feet bgs and analyzed for VOCs via TO-15. Acetone with a concentration of 690,000 micrograms per cubic meter (µg/m^3), cis-1,2-DCE with a concentration of 29,000 µg/m^3, and vinyl chloride with a concentration of 30,000 µg/m^3 exceeded the PSLs of 3,200 µg/m^3, 6.3 µg/m^3 and 0.16 µg/m^3, respectively. These soil gas results indicated a potential for vapor intrusion to occur into these buildings (**Figure 2-15** and **Table 2-6**).

Therefore, indoor air samples were collected inside Building 720 to determine whether vapor intrusion may be occurring and whether the exposure pathway was complete. Building 1040 is currently used for chemical storage with no full-time occupants and therefore indoor air was not sampled. 1,2-dichloroethane (1,2-DCA) with a concentration 0.098 µg/m^3 and benzene with a concentration of

0.045 µg/m^3 exceeded the PSLs of 0.094 µg/m^3 and 0.31 µg/m^3, respectively (**Figure 2-15** and **Table 2-6**). However, both of these compounds were detected in the ambient (outside) air sample collected outside of Building 720. These detections in ambient air likely affected the measured concentrations in the building due to infiltration.

2.7 LIMITATIONS OF PREVIOUS DATA

Data collected during historical investigations and the ongoing RI have adequately defined the soil source area near Building 1040, delineated the vertical extent of contaminated groundwater, and determined approximately 90 percent of the horizontal extent of groundwater contamination at Site 43. The following data gaps or limitations have been identified and require further investigation to adequately define the nature and extent of contamination, evaluate potential risks, and support the feasibility study at Site 43.

1) The extent of shallow groundwater contamination east of Building 1040 has not been delineated.

2) Potential impacts of contaminated groundwater on downgradient surface water/sediment have not been evaluated.

3) Potential for vapor intrusion into Building 716 (building above downgradient portion of groundwater plume) has not been assessed.

2.8 CONCEPTUAL SITE MODEL SUMMARY

The basic components of the revised CSM representing a current understanding of contamination at Site 43 are presented below. An interpretive CSM is presented as **Figure 2-16**. This CSM will be updated as necessary in the RI Report with new data and information collected during this additional RI field effort presented in this SAP Addendum.

2.8.1 CONTAMINANT SOURCES

Based on the analytical data collected at Site 43, VOCs represent the primary COPCs. A revised COPC list containing the updated PSLs and reference limits can be found in **Table 2-7** and provides the revised Worksheet #15 from the UFP-SAP. VOC-contaminated groundwater has resulted from historical dumping activities at Site 43 which consisted of the dumping of spent solvents directly onto the ground and in the drainage ditch adjacent to Building 1040 (source area). Elevated levels of cobalt have been observed in groundwater near where dumping was reported at the utility pole across from Building 1041 and in other portions of the study area at Site 43. Ancillary metals contamination could have resulted from the cleaning of metal parts with the solvents or may be naturally occurring (background) in groundwater.

Based on data collected to date, the localized area of soil contamination near Building 1040 has been adequately defined and may continue to leach into groundwater.

2.8.2 CONTAMINANT MIGRATION PATHWAYS

Contaminant migration pathways potentially affecting current conditions at Site 43 consist of the following:

- Migration of VOCs and/or inorganics via leaching of soils to groundwater and subsequent groundwater impacts on downgradient surface water/sediment.
- Migration of VOCs and/or inorganics from surface water to Potomac River
- Migration of dissolved VOCs and/or inorganics in groundwater being influenced by groundwater flow and, if present, by preferential flow pathways.
- Migration of VOCs via vaporization/volatilization. Volatile VOCs (e.g., TCE) in the groundwater may volatilize and migrate through building foundations as vapor into indoor air.

Following the initial infiltration of the VOCs (dumping and subsequent flushing from precipitation), the VOC transport migration pathway follows two groundwater migration pathways (**Figure 2-16**) where an apparent groundwater divide exists near Building 1040. To the east of Building 1040, shallow groundwater flows southeast following the easterly flow of the surficial drainage channels and eventually discharging into Mattawoman Creek. To the west of Building 1040, groundwater flows west towards the Potomac River.

The rates of horizontal advective flow in the groundwater system will be evaluated for the RI (hydraulic conductivity, calculated groundwater seepage velocity, etc.) and used to support remedy evaluations in the FS. The rate of migration for TCE is affected very little by retardation in the aquifer (retardation coefficients are just over 1), but is significantly affected by natural attenuation mechanisms (degradation, dispersion, dilution, etc.); based on the presence of cis-1,2-DCE and VC in groundwater, biodegradation of TCE is occurring.

2.8.3 EXPOSURE PATHWAYS

There is potential for human exposure to VOCs under current site conditions, considering the potential vapor intrusion into Building 716 and potential direct exposure to groundwater discharging to surface water and sediment. The media at the Site through which future potential residential receptors could be exposed to VOCs are soil, groundwater (which would also be the source of any soil gas/indoor air vapor),

surface water, and sediment. See **Figure 2-16** for a graphical representation of the current CSM. Potential exposure routes under a hypothetical future exposure scenario include dermal contact, ingestion, and inhalation.

2.8.4 POTENTIAL RECEPTORS

Receptors that may be exposed to contaminated media at Site 43 include site workers and hypothetical future residents via ingestion, inhalation, or dermal contact with contaminated soils, surface water, or groundwater. Future site use is uncertain, but is anticipated to be industrial considering the base mission. **Table 2-8** presents the receptors and exposure routes to be evaluated in the risk assessment.

In addition, groundwater seeps have been identified in drainage ditches located in the downgradient area of the plume and lead directly to the Potomac River where ecological receptors may be affected. An ecological risk screening will be conducted for surface water, sediment, and porewater as part of the RI to determine if ecological risk is a concern at Site 43 and if further evaluation or consideration is necessary during the FS.

2.8.5 COPC REFINEMENT

Based on the analytical data obtained from investigative activities at Site 43, refinement of the COPCs is possible at this time and the analytical data were evaluated to determine which COPCs to retain for future sampling. The analytical data were compared to the current PSLs and site background data, and a risk ratio evaluation was conducted for groundwater. This risk evaluation compared individual chemical concentration for a particular receptor and exposure route to the corresponding PSL and summed the computed ratios to determine whether risks were likely to be unacceptable. Occurrence and distribution of the COPCs was also used to determine which COPCs to carry forward into further investigation at Site 43.

Tables 2-9 and **2-10** present the occurrence and distribution of site contaminants and results of the risk ratio evaluation for groundwater, respectively. Groundwater is the primary medium still being investigated in the RI and the potential impact of groundwater on surface water and sediments will be evaluated. As presented in **Table 2-9**, a total of 13 explosives, 20 metals, and 14 VOCs were detected in groundwater at Site 43. Of these constituents, six explosives, eight metals, and eight VOCs were observed above the PSLs and selected for the risk ratio evaluation.

For the risk ratio evaluation, the selected VOCs, metals, and explosives were evaluated for carcinogenic and non-carcinogenic risks using exposure point concentrations (EPC) calculated by ProUCL version

4.1.01 (95 percent upper confidence limit [UCL]) or maximum detected concentration if there were less than 3 detections in the data set. Based on the EPC for each constituent, the estimated incremental lifetime carcinogenic risk (ILCR) and non- carcinogenic hazard quotient (HQ) was determined.

VOCs in groundwater are the primary COPCs and all eight compounds evaluated in the risk ratio will be retained due to estimated unacceptable level of risk or association with degradation of chlorinated solvents. A total of 10 VOCs will be included in future analysis with the addition of, 1,2-DCA, and trans-1,2-dichloroethene to the eight VOC compounds listed on **Table 2-10**.

Of the eight metals evaluated in the risk ratio, four of them indicated potential unacceptable risk and will be included in future sampling. The four metals retained will be arsenic, cobalt, iron, and manganese.

Six explosives were evaluated in the risk ratio. Of those six, only nitrobenzene and nitroglycerin indicated adverse risk associated with the EPC calculated for these compounds. However, nitroglycerin was only detected in 6 of the 35 samples and the two highest concentrations driving the EPC, detected in MW01 and MW06 in 2011, were not reproduced and nitroglycerin was not detected in either of these wells during the 2012 sampling event. Nitrobenzene showed a very limited occurrence and was only detected in 3 of 35 samples. Two of these detections were above the PSL in MW24 (0.865 B µg/L) and MW10 (0.234 B µg/L) and both of these monitoring wells are not located in the source area and nitrobenzene was also detected at 0.45 µg/L in the associated field blank for these samples. Based on the limited occurrence, distribution, and non-reproducibility of the suspect detections of explosives, explosives will not be retained for future sampling.

2.8.6 SUMMARY

A continuing source of VOCs from site soils has been identified along the drainage ditch near Building 1040. Groundwater contamination has been detected both east and west of the source area and the plume footprint is larger than originally anticipated after the SSP investigations and Phase 1 RI. There is a groundwater divide located in the vicinity of Building 1040, where groundwater to the east of the divide flows southeast to Mattawoman creek and west of the divide groundwater flows west to the Potomac River. In addition, a leaking potable water line and other utilities may be affecting groundwater flow and contaminant migration. The horizontal extent of VOCs in groundwater has not been fully delineated with the existing monitoring well network. Shallow groundwater contamination has not been defined in the eastern portion of the plume. Site lithological data suggests a basal clay unit at approximately 25 to 35 feet bgs likely prevents any downward migration of VOCs into lower aquifers. There are no current potential receptors or exposure routes, except for the potential vapor intrusion pathway into Building 716 and potential exposure to surface water or sediment.

2.9 PROJECT QUALITY OBJECTIVES

2.9.1 PROBLEM DEFINITION

Historical dumping activities at Site 43 have impacted soil and the surficial aquifer, and could impact surface water and sediment. Preliminary risk screening conducted at the Site has shown some contaminants result in an unacceptable level of human health risk. Based on the revised CSM and consensus of the project team, further characterization of the nature and extent of select VOCs and inorganics contamination in groundwater, surface water, and sediment is needed to complete the RI and evaluate remedial alternatives at Site 43. This characterization includes a determination of:

1. Determine extent of VOC-contaminated groundwater to the east of the source area (Building 1040 drainage ditch) and near the Potomac River.

2. Determine the potential for a complete VOC vapor intrusion pathway into Building 716 and if further assessment is needed for vapor intrusion.

3. Evaluate potential impacts of groundwater discharge to downgradient surface water and sediments

4. Evaluate groundwater/surface water interface near the Potomac River to determine impact from groundwater plume.

5. A baseline human health risk assessment (HHRA) is needed to evaluate human health risks on soil, surface water, sediment, and groundwater.

6. An ecological screening is needed to evaluate potential ecological risks on surface water, sediment, and porewater.

Once the remaining RI fieldwork presented in this SAP Addendum is complete, new data will be evaluated as a part of the RI and the team will proceed with the FS for Site 43.

2.9.2 INFORMATION INPUTS

Additional groundwater, surface water, and sediment data are needed for VOCs and inorganics. The data to be collected will be used to conduct a HHRA and ecological risk screening for the RI.

The following inputs are needed to attain project objectives:

1. Field Screening Data:

a. Visual observations and photoionization detector (PID) measurements to help identify contamination. Visual and PID data inputs are considered qualitative because they are not necessarily an accurate measure of VOC concentrations. These qualitative data are of sufficient quality to facilitate consideration of actual sample locations selected for laboratory analysis as necessary. The tendency will be to collect samples for VOC analysis at locations identified by the field operation leader (FOL) as the location of greatest contaminant levels based on the highest PID reading and visible staining. No soil sampling is anticipated during this phase of the RI, but soil borings will be visually logged and screened with a PID.

b. Field water quality data to estimate geochemical conditions in the surficial aquifer (pH, dissolved oxygen [DO], turbidity, iron II, and oxidation-reduction potential [ORP]).

2. Chemical Data: Groundwater, surface water, sediment, and soil gas concentration data for select VOCs and select metals. These analytical fractions are included on the basis of the revised CSM to cover the known potential contaminants that may have impacted groundwater, surface water, and sediment (and potentially soil gas) based on risk ratio screening described in **Section 2.8**. See **Table 2-6** for the retained list of analytes and revised PSLs.

Resource Conservation and Recovery Act (RCRA) characteristic analyses must be conducted on soil and aqueous investigation-derived waste (IDW) in accordance with 40 Code of Federal Regulations (CFR) 261. The data will be used to determine proper disposal procedures for IDW.

3. Physical Data: The physical data to help characterize groundwater at the Site.

a. Groundwater stabilization parameters (mentioned above in Field Screening Data, and collected in accordance with Standard Operating Procedure (SOP) SA-1.1) to include pH, specific conductance, turbidity, temperature, DO, and ORP, and will be measured before groundwater sampling to ensure representative samples are collected.

b. Groundwater levels measured at Site monitoring wells prior to the collection of samples to determine groundwater depths and flow direction.

4. Project Screening Limits: PSLs were used to ensure that analytical methods were selected with enough sensitivity to support the risk assessment governed by this SAP Addendum. Surface water, sediment, groundwater, and soil gas data must be compared to the PSLs presented in **Table 2-7**. Primary PSLs/criteria are the “Selected PSLs,” which are lowest, most conservative values for each medium. These primary PSLs will be used to screen concentrations and determine which compounds must be included in the HHRA. Other PSL candidates will serve as secondary PSLs/criteria in many cases and will include background criteria for inorganics in soil (Tetra Tech, 2002) as presented in **Table 2-7**. In this case, background criteria will be used to screen out concentrations of inorganics that are naturally

occurring after the initial risk assessment is conducted. Statistics used to represent exposure point concentrations are identified in Section 11.4 of the UFP SAP (Tetra Tech, 2011). Project action levels (PALs) will be used to determine whether additional investigation or corrective action is needed to mitigate risks. For non-carcinogens the PAL is associated with hazard index (HI) of 1 (same target organ effect); for carcinogens it is associated with an exceedance of 1×10-5 ILCR.

Table 2-7 lists the PSLs as well as the laboratory quantitation limits (QLs) and statistically-derived method detection limits (MDLs). The laboratories are able to achieve QLs that are below the PSLs for most analytes. For purposes of selecting COPCs in the HHRA, one-half the limit of detection (LOD) will be used when the LOD is above the PSL.

2.9.3 STUDY BOUNDARIES

The primary populations of interest for further evaluating the extent of contamination are groundwater, surface water, sediment, and soil gas. The horizontal boundary for this investigation has expanded and now includes the westernmost area along the Potomac River (near Pump House Lane and north approximately 1000 feet) and eastern area including the area north of Johnson Road, and approximately 600 feet east of Glennon Road. An isolated area in the southern portion of the area is limited to the vicinity of Building 1041 (an approximately 200-foot radius from Building 1041). The vertical boundaries for soil and groundwater are the ground surface to the basal clay aquitard. For a typical risk assessment the soil population of interest is soil within the defined source area to a depth of 10 feet bgs. This constitutes the soil exposure unit (EU). The groundwater population of interest is groundwater within the contaminant plume which can be essentially defined by the occurrence of the primary COPC of TCE in groundwater.

For evaluating the potential for a complete vapor migration pathway from soil or groundwater to indoor air (specifically Building 716), the population of interest is soil gas and groundwater within and near the footprint of Building 716 that may be acting as a source of indoor air contamination.

Seep water is surface water at the moment of discharge from groundwater and prior to dilution in the channel. It thus represents the greatest risk to potential receptors exposed to surface water. For this reason, seep water samples will be used with other surface water samples to estimate risks from exposure to surface water.

To establish/confirm the vertical boundaries, lithological data will be collected as necessary where historical data does not exist. Groundwater upper vertical boundaries are established by the water table (ranging from 5 to 15 feet bgs) and the Basal Clay aquitard. Soil gas vertical boundaries are established from the ground surface to the water table. Subsurface soil data represents the subsurface soil population

contained within the study area boundaries described above. The vertical boundary for surface water is limited to the existing depth and flow (to be measured) in the drainage channels and culvert discharges and the sediment boundary will be defined by the ground surface to 6 inches bgs.

2.9.4 ANALYTIC APPROACH

To resolve the problem statement of **Section 2.9.1**, two decision rules were developed and are presented below.

For the HHRA, if any Site-related contaminant in any sediment, groundwater, surface water or soil gas sample exceeds the COPC screening value (selected PSL), the target chemical will be selected as a COPC for inclusion in the risk assessment. Chemical-specific UCLs will be calculated using EPA's latest ProUCL software. If the UCL is greater than the maximum detected concentration for that medium, the maximum detected concentration will represent the EPC. EPCs established in this manner represent a reasonable maximum exposure (RME). The most recent groundwater data collected in 2012 and the proposed additional round of sampling in this SAP Addendum will be used to evaluate risk in the HHRA. An ecological risk screening will be conducted with newly obtained data from surface water, sediment, and porewater (collected from the piezometers along the Potomac River).

To determine whether the vapor intrusion pathway is potentially complete, VOC concentrations in soil gas will be used to calculate expected indoor air concentrations. The Johnson and Ettinger model (Johnson, et al.,1991) will be used to calculate representative indoor air concentrations from soil gas results.

The following decision rules are based on evaluation of each population which represents the decision unit for that media type. For example, groundwater data and soil gas data represent the groundwater and soil gas population contained within the study area boundaries; and the groundwater and soil gas populations within the study area represent their respective decision units.

Decision Rule 1: If the measured concentration of any Site-related contaminant in the sample collected from any new plume periphery well, surface water, or peripheral sediment sample location exceeds the EPA Maximum Contaminant Level (MCL) (or PSL in cases where no MCL exists) and background concentrations (metals only), then re-evaluate the CSM and data quality objectives (DQOs) to determine whether additional sampling is necessary to complete the delineation of contamination, otherwise stop delineation. Factors to be considered by the project team when re-evaluating the CSM, if necessary, include the extent of delineation achieved from the sampling versus the cost of additional sampling as well as the consequences of incomplete delineation. If all Site-related contaminant concentrations on the perimeter are less than criteria, then it will be concluded that the Site-related contamination is delineated and an FS will be conducted.

Note: Background evaluation for metals will include comparisons to the non-turbid unfiltered data set for groundwater and surface soil data set will be used to evaluate sediment.
Note: The project team may decide that perfect bounding of contamination in all directions is not necessary but this decision must be deferred until data are collected and spatial patterns of contamination are evident.

Decision Rule 2: If the modeled concentration of any VOC from soil gas exceeds the applicable PSL for indoor air, then recommend additional evaluation to determine if vapor intrusion is occuring, otherwise recommend no further action. DQOs for additional sampling and/or investigation, if necessary, would be developed and documented in a Field Task Modification Request (FTMR).

The new data collected as a part of this additional investigative work will be evaluated in the RI and used in the HHRA and ecological risk screening. Site 43 will then proceed to the FS and remedial alternatives will be evaluated to address contamination at the Site.

2.9.5 PERFORMANCE CRITERIA

Sample locations were selected to bound the shallow groundwater plume to the east and evaluate the potential impact of contaminants in groundwater discharging to surface water and sediment near the Potomac River. Biased sampling is appropriate to further establish the extent of contamination. The number of biased samples required to achieve full delineation was estimated by the project team based on visual patterns of known contamination and known behavior of released contaminants in the environment. After new data have been collected, the project team will use concentration patterns in the new data combined with previously collected data to determine whether the contamination is satisfactorily delineated. The sampling design **Table 2-11** presents the sampling required to further delineate groundwater and determine the potential impacts to surface water, sediment, and soil gas. Delineation with the planned number of samples will be considered satisfactory unless perimeter samples exceed criteria and the exceedances are sufficiently large in magnitude and number to render additional delineation cost-effective, or if data quality deficiencies are identified that prevent risk assessment or completion of delineation.

2.9.6 PLANS FOR OBTAINING DATA

See **Table 2-11** for a detailed sampling summary and **Section 3.0** for project tasks.

3.0 SUMMARY OF PROJECT TASKS

3.1 FIELD INVESTIGATION TASK PLAN

The field tasks are summarized below. A short description of these tasks is also provided. **Table 3-1** includes the sample IDs and sample nomenclature for each location. **Table 3-2** contains the sample matrix and number of quality control samples to be sent to the laboratory. Refer to the 2011 UFP SAP for SOP references (Tetra Tech, 2011).

- Mobilization/demobilization
- Utility clearance
- Field monitoring equipment calibration
- Surface water sampling
- Sediment sampling
- Seep sampling
- Soil Gas Sampling
- Monitoring well installation
- Groundwater sampling
- Indoor air contaminant concentration modeling
- Water level measurements
- Investigation-derived waste management
- Surveying
- Field equipment decontamination procedures
- Field documentation procedures
- Sample custody and shipment tasks

Additional project-related tasks include:

- Analytical tasks
- Data management
- Assessment and oversight
- Data review
- Project reports

3.2 FIELD TASKS

Also see the following appendices in the NSF-IH Master SAP (Tetra Tech, 2009b):

• Appendix B – Field and General Sampling Operations

• Appendix C – Example Forms

• Appendix D – Facility SOPs

3.2.1 MOBILIZATION/DEMOBILIZATION

Mobilization shall consist of the delivery of all equipment, materials, and supplies to the Site, the complete assembly in satisfactory working order of all such equipment at the Site, and the satisfactory storage at the Site of all such materials and supplies. Tetra Tech will coordinate with the facility to identify locations

for the storage of equipment and supplies. Site-specific health and safety training will be provided to all Tetra Tech subcontractors as part of the site mobilization.

Demobilization shall consist of the prompt and timely removal of all equipment, materials, and supplies from the Site following completion of the work. Demobilization includes the cleanup and removal of IDW generated during the investigation.

3.2.2 UTILITY CLEARANCE

Prior to the commencement of any intrusive activities, the Tetra Tech FOL will coordinate with the utility subcontractor to identify and mark-out utilities that may be present within the proposed drilling areas. Subsurface utilities will also be cleared by the drilling subcontractor by notifying the utility clearing service. See Facility SOP HS-1.0 for conducting subsurface soil investigations for further information.

3.2.3 FIELD/MONITORING EQUIPMENT CALIBRATION

These procedures are described in WS #22 of the UFP SAP (Tetra Tech, 2011).

3.2.4 SURFACE WATER SAMPLING

Four surface water and two culvert samples will be obtained from the two drainage channel locations. The outfall sample will be collected at the outfall location along the Potomac River. The proposed surface water, culvert, and outfall sample locations are presented on **Figure 3-1** (for a more detailed map see **Figure 3-2**). In the event the drainage channels do not contain enough water to obtain a surface water or culvert sample, appropriate actions will documented in the field and sampling may be postponed, locations changed, or sample eliminated. Surface water, culvert, and outfall samples will be collected by direct filling methods and field parameters will be collected prior to sampling.

Surface water sampling and sample handling, are discussed in Facility SOPs SA-1.2.

3.2.5 SEDIMENT SAMPLING

Four sediment samples will be obtained from the two drainage channels. Site specific background sediment samples will be collected in similar drainage channels to the north. The proposed sediment sample locations are presented on **Figure 3-1** (for a more detailed map see **Figure 3-2**). Sediment samples will be collected using a disposable scoop or trowel. Debris, rocks, twigs, and vegetation will be removed before collection.

Sediment will be screened with a PID for evidence of potential contamination. Any visual signs of potential contamination (such as staining) will be noted and samples will be collected for select VOCs and inorganics.

Soil sampling, soil logging, sample handling, are discussed in Facility SOPs SA-1.2. The use of the PID is described in the manufacturer's instructions.

3.2.6 SEEP SAMPLING

Two seep samples will be obtained from two drainage channels in the locations previously sampled. The proposed seep samples locations are presented on **Figure 3-1** (for a more detailed map see **Figure 3-2**). Seep samples will be collected by inserting a drive-point horizontally into the seep location (if possible), or by creating a sampling collection point by digging a hole at the seep location to facilitate sample collection.

3.2.7 SOIL GAS SAMPLING

Four soil gas samples will be collected around Building 716. The proposed soil gas sample locations are presented on **Figure 3-1** (for a more detailed map see **Figure 3-2**). Soil gas samples will be collected from a temporary sampling probe installed via DPT methods. The samples will be collected using 6-L SUMMA™ canisters equipped with flow controllers set to collect samples over a 30-minute time period.

Soil gas sampling, collection, and handling, are discussed in Facility SOP SA-2.4.

3.2.8 MONITORING WELL/PIEZOMETER INSTALLATION

Three monitoring wells and 4 piezometers will be installed to further delineate the extent of groundwater contamination and evaluate groundwater conditions east of the source area and near the Potomac River. Proposed locations are shown on **Figure 3-1**.

Soil borings will be drilled at the proposed monitoring well locations to confirm the subsurface lithology and depths to ensure proper depths for well screens. The soil cores will be screened visually and along their entire length with a PID in accordance with the PID manufacturer's instructions. The boring information will be recorded in accordance with Facility SOP SA-6.3. The monitoring wells will be installed using hollow-stem auger (HSA) drilling methods. The piezometers will be installed using DPT and pre-pack well screens.

Monitoring well and piezometer installation procedures are discussed in Facility SOP GH-2.8 and SA-2.5 and each of the new monitoring wells will be developed in accordance with SOP GH-2.8.

3.2.9 GROUNDWATER SAMPLING

A complete round of groundwater sampling will be conducted including each new monitoring well and piezometer location using low-flow sampling procedures, per Facility SOP SA-1.1. A peristaltic pump will be used for groundwater sample purging and collection activities, in combination with a continuous flow-through cell suitable for taking water quality measurements. Turbidity measurements will be made using a separate field turbidity meter that is specifically designed to measure turbidity only.

3.2.10 INDOOR AIR CONTAMINANT CONCENTRATION MODELING

The Johnson and Ettinger model (Johnson, et al., 1991) will be used to calculate representative indoor air concentrations from groundwater and soil gas results. Sample information such as groundwater concentrations, soil gas concentrations, sample depths, chemical of concern, soil type, and average soil and groundwater temperature will be entered into the model. The model will calculate an attenuation factor and estimate an expected indoor air concentration for the specific compound evaluated in the model. The estimated indoor air concentrations will be compared to PSLs to determine if there are potential risks to exposure of indoor air in Building 716.

3.2.11 WATER LEVEL MEASUREMENTS

Prior to the start of sampling, a synoptic round of groundwater level measurements will be made. Depth to groundwater will be measured at each monitoring well, per Facility SOP GH-1.2. Along with the subsequent survey effort, this will provide for new groundwater elevation contour maps and provide additional information on groundwater flow patterns and gradients. Water-level measurements will be completed within the shortest time possible on the same day, and no sooner than 24 hours after a significant precipitation event to minimize the precipitation effects on the data. Water level measurements will be recorded to the nearest 0.01 foot and referenced to a top of casing notch or north side of the well casing. The measurement instrument will be decontaminated prior to conducting the measurement and between each monitoring well.

3.2.12 IDW MANAGEMENT

Waste soils will be generated during the installation of the monitoring wells. The soil IDW consists of the excess soil cuttings from the soil borings and the soil cuttings produced during the drilling of the boreholes for monitoring well installation. The waste soil will be collected and placed in 55-gallon drums for waste characterization sampling and analysis. Drums will be moved inside the diked area next to Building 289 that has secondary containment. Based upon the results of the waste characterization, and upon Navy approval, the waste soil will be transported and appropriately disposed offsite at a Navy-approved disposal facility by the IDW subcontractor.

Waste water will be generated during well development, sampling of the monitoring wells, and during decontamination procedures. All aqueous IDW will be containerized pending waste characterization analyses. Drums will be stored on pallets inside the diked area next to Building 289. Based on the results of the waste characterization, and upon Navy approval, the water will be transported and appropriately disposed offsite at a Navy-approved disposal facility by the IDW subcontractor.

3.2.13 SURVEYING

A surveyor licensed in the state of Maryland will be subcontracted by Tetra Tech to survey the horizontal location and vertical elevation of each of the monitoring wells (to-be-installed). The horizontal measurements shall be accurate to 0.1 feet. The vertical elevation measurements shall be accurate to 0.01 ft at the top-of-riser at each monitoring well. Each of the locations will be surveyed in the North American Datum (NAD) of 1983, State Plane Coordinate System of Virginia (feet) relative to the coordinates of established Site benchmarks or the nearest United States Geological Survey (USGS) benchmark.

3.2.14 FIELD EQUIPMENT DECONTAMINATION

Decontamination of equipment will be conducted in accordance with Facility SOP SA-7.1. Decontamination fluids will be containerized and characterized for appropriate disposal with other IDW.

3.2.15 FIELD DOCUMENTATION

Field documentation will be performed in accordance with Facility SOP SA-6.3. A summary of all field activities will be properly recorded in a bound logbook with consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use. At a minimum, the following information will be recorded in the Site logbook:

- Name of the person to whom the logbook is assigned.
- Project name.
- Project start date.
- Names and responsibilities of onsite project personnel including subcontractor personnel.
- Arrival/departure of site visitors.
- Arrival/departure of equipment.
- Sampling activities and sample log sheet references.
- Description of subcontractor activities.
- Sample pick-up information, including chain-of-custody numbers, air bill numbers, carrier, time, and date.
- Description of borehole or monitoring well installation activities and operations.
- Health and safety issues.

All entries will be written in ink and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; the person making the correction will initial and date the change. Boring logs, sampling forms, and other field forms will be used to document field activities.

3.2.16 SAMPLE CUSTODY AND SHIPMENT

Data management and sample tracking tasks are described in WS #27 of the UFP-SAP (Tetra Tech 2011) and in Facility SOP CT-05 (Tetra Tech, 2011a and Tetra Tech, 2009).

3.3 PROJECT REPORT

A draft RI Report will be prepared and submitted to the Navy and regulators (i.e., the Partnering Team) for review. The report will include a summary of the work performed in the approved UFP-SAP and subsequent SAP Addendums, field modifications as documented by the Tetra Tech FOL, summary and analysis of the analytical results, updated CSM, baseline HHRA, ecological risk screening, and conclusions and/or recommendations for Site 43.

Tetra Tech will respond to comments received on the draft report and the final version of the report will be submitted in hardcopy and electronic format to the project team.

REFERENCES

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TABLES

TABLE 1-1
CROSSWALK TABLE
PHASE 3 REMEDIAL INVESTIGATION
SITE 43 - TOLUENE DISPOSAL AREA
NSF INDIAN HEAD, MARYLAND

UFP SAP Worksheet #	Required Information	Crosswalk to Related Information
A. Project Management and Objectives		
Documentation		
1	Title and Approval Page	See **Signature Page**
2	SAP Identifying Information	See WS # 2 of final UFP SAP, and this **Table 1-1** Crosswalk
3	Distribution List	See revised **WS # 3** in Appendix B
4	Project Personnel Sign-Off Sheet	See revised **WS # 4** in Appendix B
Project Organization		
5	Project Organizational Chart	See WS # 5 of final UFP SAP
6	Communication Pathways	See revised **WS # 6** in Appendix B
7	Personnel Responsibilities Table	See revised **WS # 7** in Appendix B
8	Special Personnel Training Requirements Table	See WS # 8 of final UFP SAP
Project Planning/Problem Definition		
9	Project Scoping Session Participants Sheet	See WS # 9 of final UFP SAP
10	Conceptual Site Model	See **Section 2.8** and **Figure 2-16**
11	Project Quality Objectives/Systematic Planning Process Statements	See **Section 2.9**
12	Field Quality Control Samples	See WS # 12 of final UFP SAP
13	Secondary Data Criteria and Limitations Table	See **Section 2.3** through **2.7** and associated Tables and Figures
14	Summary of Project Tasks	See **Section 3.0** and associated Tables and Figures
15	Reference Limits and Evaluation Tables	See **Table 2-7**
16	Project Schedule/Timeline Table	TBD
B. Measurement/Data Acquisition		
Sampling Tasks		
17	Sampling Design and Rationale	See **Table 2-10**
18	Location-Specific Sampling Methods/ SOP Requirements Table	See **Table 3-1**
19	Field Sampling Requirements Table	See WS # 19 of final UFP SAP
20	Field QC Sample Summary Table	See **Table 3-2**
21	Project Sampling SOP References Table	See WS # 21 of final UFP SAP
22	Field Equipment Calibration, Maintenance, Testing, and Inspection Table	See WS # 22 of final UFP SAP
Analytical Tasks		
23	Analytical SOP References Table	See revised **WS # 23** in Appendix B
24	Analytical Instrument Calibration Table	See revised **WS # 24** in Appendix B
25	Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table	See WS # 25 of final UFP SAP
Sample Collection		
26	Sample Handling System	See WS # 26 of final UFP SAP
27	Sample Custody Requirements	See WS # 27 of final UFP SAP

TABLE 1-1
CROSSWALK TABLE
PHASE 3 REMEDIAL INVESTIGATION
SITE 43 - TOLUENE DISPOSAL AREA
NSF INDIAN HEAD, MARYLAND

UFP SAP Worksheet #	Required Information	Crosswalk to Related Information
Quality Control Samples		
28	Laboratory QC Samples Table	See revised **WS # 28.1**, **28.6**, & **28.12** in Appendix B
Data Management Tasks		
29	Project Documents and Records Table	See WS # 29 of final UFP SAP
30	Analytical Services Table	See revised **WS # 30** in Appendix B
C. Assessment Oversight		
31	Planned Project Assessments Table	See revised **WS # 31** in Appendix B
32	Assessment Findings and Corrective Action Responses Table	See WS # 32 of final UFP SAP
33	QA Management Reports Table	See WS # 33 of final UFP SAP
D. Data Review		
34-36	Data Verification and Validation (Steps I and IIa/IIb) Process Table	See WS # 34-36 of final UFP SAP
37	Usability Assessment	See WS # 37 of final UFP SAP

Notes:
WS : Worksheet
COC : Chain of custody
QA : Quality Assurance
TBD : To be determined
UFP SAP : Uniform Federal Policy Sampling and Analysis Plan (Tetra Tech, 2011)

TABLE 2-1
SOIL ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL Reference	NSF-IH Background Subsurface Soil	43SA01	43SA01	43SA02	43SA03	43SA04	S43SB001		S43SB002		S43SB003				S43SB004	
SAMPLE ID				43SA01	43SA01-D	43SA02	43SA03	43SA04	S43SB0010101	S43SS0010001	S43SB0020101	S43SS0020001	S43SB0030101	S43SB0030101-DUP	S43SS0030001	S43SS0030001-DUP	S43SB0040101	S43SS0040001
SAMPLE DATE				8/1/1992	8/1/1992	8/1/1992	8/1/1992	8/1/1992	7/14/2005	7/14/2005	7/14/2005	7/14/2005	7/14/2005	7/14/2005	7/14/2005	7/14/2005	7/14/2005	7/14/2005
EXPLOSIVES (mg/kg)																		
1,3,5-TRINITROBENZENE	220	EPA RSL	--	--	--	--	--	--	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U
1,3-DINITROBENZENE	0.61	EPA RSL	--	--	--	--	--	--	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U
3-NITROTOLUENE	0.61	EPA RSL	--	--	--	--	--	--	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U
4-NITROTOLUENE	30	EPA RSL	--	--	--	--	--	--	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U
NITROCELLULOSE	18,000,000	EPA RSL	--	--	--	--	--	--	1.5 B	8 J	1.4 B	9 J	1.1 B	1.2 B	1.2 B	1.3 B	1.5 B	1.4 B
NITROGUANIDINE	610	EPA RSL	--	--	--	--	--	--	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U
METALS (mg/kg)																		
ALUMINUM	7,700	EPA RSL	**21,400**	--	--	--	--	--	3,490 J	4,410 J	3,380 J	7,230 J	**16,200 J**	**16,800 J**	**17,000 J**	**18,300 J**	**16,200 J**	**18,400 J**
ARSENIC	0.39	EPA RSL	**28.7**	--	--	--	--	--	**3.6**	**4.3**	**2.7**	**5.3 K**	**11.3 K**	**13.5 K**	**5.7 K**	**3.9**	**8.5**	**3.8**
BARIUM	1,500	EPA RSL	66.5	--	--	--	--	--	20.4	25.9 J	29.5	64.0 J	93.4	92.4	165 J	457 J	106	112 J
BERYLLIUM	16	EPA RSL	1.5	--	--	--	--	--	0.26	0.27	1.6	0.40	1.2	1.2	1.2	0.95	1.1	0.63
CADMIUM	3.9	MDE	--	--	--	--	--	--	0.16	0.95	0.40	0.82 K	0.41 K	0.40 K	0.35 K	0.13	0.16	0.22
CALCIUM	--	--	--	--	--	--	--	--	176	1610	1280	1170	143	194	279	256	116	350
CHROMIUM	0.29	EPA RSL	**59.1**	--	--	--	--	--	**18.2 J**	**19.9 J**	**17.3 J**	**37.5 J**	**18.5 J**	**22.1 J**	**20.2 J**	**17.1 J**	**16.7 J**	**20.2 J**
COBALT	2.3	EPA RSL	**14.7**	--	--	--	--	--	**3.5**	**7.0**	**4.6**	**19.3**	**3.0**	**3.0**	1.7 L	**2.4**	**4.9**	**3.2**
COPPER	310	EPA RSL	47.6	--	--	--	--	--	9.7	22.8 J	21.9	25.7 J	16.8	17.7	20.2 J	14.0 J	14.8	13.9 J
IRON	5,500	EPA RSL	**35,200**	--	--	--	--	--	**14,100**	**13,500**	**18,700**	**23,400**	**41,200**	**41,500**	**34,600**	**16,800**	**19,200**	**16,500**
LEAD	400	EPA RSL	38.6	--	--	--	--	--	6.1	91.0	7.7	42.3	15.6	16.8	22.9	19.7	17.0	15.8
MAGNESIUM	--	--	--	--	--	--	--	--	196	3860	777	765	586	637	469	535	607	675
MANGANESE	160	MDE	155	--	--	--	--	--	110 J	**228 J**	**227 J**	**813 J**	10.9 J	12.3 J	9.9 J	9.9 J	10.2 J	15.9 J
MERCURY	0.78	MDE	0.14	--	--	--	--	--	0.045 U	0.05 U	0.048 U	0.05 U	0.056 U	0.06 U	0.062 K	0.063 U	0.065 U	0.34
NICKEL	150	EPA RSL	15.9	--	--	--	--	--	7.0	38.8	5.5	12.6	7.5 L	7.9 L	5.1 L	7.4	9.7	7.1
POTASSIUM	--	--	--	--	--	--	--	--	311	299	380	441	420 L	504 L	366 L	466	408	530
SELENIUM	39	EPA RSL	3.8	--	--	--	--	--	0.49 UR	0.56 UR	0.58 UR	0.54 UR	0.60 UR	0.67 UR	0.62 UR	0.72 UR	0.71 UR	0.56 UR
SILVER	39	EPA RSL	--	--	--	--	--	--	0.31 B	0.46 B	0.14 U	0.55 B	1.5 B	1.6 B	1.7 B	2.0 B	1.8 B	2.0 B
SODIUM	--	--	--	--	--	--	--	--	288	281	265	465	128 K	129 K	99.9 K	111	263	102
THALLIUM	0.078	EPA RSL	--	--	--	--	--	--	0.33 U	0.38 U	0.39 U	0.36 U	0.40 U	0.45 U	0.41 U	**0.88 B**	0.48 U	0.38 U
VANADIUM	7.8	MDE	**102**	--	--	--	--	--	**16.2**	**39.4**	**25.3**	**36.0**	**49.2**	**55.8**	**26.3**	**24.2**	**38.7**	**31.0**
ZINC	2,300	EPA RSL	49.7	--	--	--	--	--	23.1 K	70.8 K	13.1 K	58.4 K	16.7 K	18.7 K	14.5 K	16.2 K	22.6 K	19.4 K
MISCELLANEOUS PARAMETERS																		
CYANIDE (mg/kg)	2.2	EPA RSL	--	--	--	--	--	--	1.4	0.095 U	0.097 U	0.089 U	0.13 U	0.12 U	0.12 U	0.12 U	0.12 U	0.12 U
TOTAL ORGANIC CARBON (mg/kg)	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
PERCHLORATE (µg/kg)	5,500	EPA RSL	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
VOLATILES (µg/kg)																		
2-BUTANONE	2,800,000	EPA RSL	--	12 U	13 U	12 U	11 U	2 U	10 U	15 U	12 U	20 U	9 J	6 J	8 J	13 U	7 J	14 U
2-HEXANONE	21,000	EPA RSL	--	12 U	13 U	12 U	11 U	11 U	10 U	15 U	12 U	20 U	14 U	13 U	13 U	13 U	14 U	14 U
4-METHYL-2-PENTANONE	530,000	EPA RSL	--	12 U	13 U	12 U	11 U	11 U	10 U	15 U	12 U	20 U	14 U	13 U	13 U	13 U	14 U	14 U
ACETONE	6,100,000	EPA RSL	--	29 U	340	10 U	5 U	10 U	10 U	15 U	12 U	20 U	12 J	13 J	31 J	48 J	10 J	12 J
CARBON DISULFIDE	82,000	EPA RSL	--	12 U	13 U	12 U	11 U	11 U	10 U	15 U	12 U	20 U	14 U	13 U	13 U	13 U	14 U	14 U
CIS-1,2-DICHLOROETHENE	16,000	EPA RSL	--	--	--	--	--	--	10 U	15 U	12 U	20 U	14 U	13 U	13 U	13 U	14 U	14 U
METHYLENE CHLORIDE	56,000	EPA RSL	--	2 U	13 U	12 U	3 U	3 U	8 B	8 B	5 B	210 J	150 J	130 J	130 J	6 B	140 J	130 J
TETRACHLOROETHENE	1,200	MDE	--	12 U	13 U	12 U	11 U	11 U	10 U	15 U	12 U	20 U	14 U	13 U	13 U	13 U	14 U	14 U
TOLUENE	500,000	EPA RSL	--	12 U	13 U	12 U	11 U	11 U	10 U	15 U	12 U	20 U	14 U	13 U	13 U	13 U	14 U	14 U
TRICHLOROETHENE	910	EPA RSL	--	12 U	13 U	12 U	11 U	11 U	8 J	15 U	12 U	20 U	14 U	13 U	13 U	13 U	14 U	14 U
VINYL CHLORIDE	60	EPA RSL	--	12 U	13 U	12 U	11 U	11 U	10 U	15 U	12 U	20 U	14 U	13 U	13 U	13 U	14 U	14 U

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/kg : micrograms per kilogram
mg/kg : milligram per kilogram

J : Estimated value
B : Compound detected in blank sample
K : Biased high
L : Biased Low
R : The chemical was rejected.
U : The chemical was not detected.

-- : The chemical was not analyzed or no value was available.

Bold and shaded values indicate an exceedance of PSLs

EPA RSL : U.S. Environmental Protection Agency May 2012 Regional Screening Level for soil under residential scenario. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for chromium VI used as surrogate for [total] chromium. RSL for methylmercury used as surrogate for mercury.

MDE : Maryland Department of Environment Cleanup Standards for residential soil. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

TABLE 2-1
SOIL ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND
PAGE 2 OF 4

LOCATION	PSL [1]	PSL Reference	NSF-IH Background Subsurface Soil	S43SB005		S43SB006		S43SB008		S43SB009			S43SB010	S43SB011		
SAMPLE ID				S43SB0050101	S43SS0050001	S43SB0060101	S43SS0060001	S43SB008-0608	S43SB008-0607	S43SB009-0608	S43SB009-0708	S43SB009-0708-DUP	S43SB010-0709	S43SB011-0709	S43SB011-0709-DUP	S43SB011-0507
SAMPLE DATE				7/14/2005	7/14/2005	7/14/2005	7/14/2005	6/4/2011	6/10/2011	6/4/2011	6/10/2011	6/10/2011	6/3/2011	6/4/2011	6/4/2011	6/10/2011
EXPLOSIVES (mg/kg)																
1,3,5-TRINITROBENZENE	220	EPA RSL	--	0.25 U	0.25 U	0.25 U	0.25 U	0.0392 U	--	0.0385 U	--	--	0.0396 U	0.0396 U	0.0367 U	--
1,3-DINITROBENZENE	0.61	EPA RSL	--	0.25 U	0.25 U	0.25 U	0.25 U	0.0392 U	--	0.0375 J	--	--	0.0396 U	0.0396 U	0.0367 U	--
3-NITROTOLUENE	0.61	EPA RSL	--	0.25 U	0.043 J	0.25 U	0.25 U	0.0392 U	--	0.0385 U	--	--	0.0396 U	0.0396 U	0.0367 U	--
4-NITROTOLUENE	30	EPA RSL	--	0.25 U	0.25 U	0.25 U	0.25 U	0.0392 U	--	0.0385 U	--	--	0.0396 U	0.064 J	0.0367 U	--
NITROCELLULOSE	18,000,000	EPA RSL	--	1.5 B	1.4 B	1.5 B	1 B	--	--	--	--	--	--	--	--	--
NITROGUANIDINE	610	EPA RSL	--	0.25 U	0.25 U	0.25 U	0.25 U	0.37 UJ	--	0.455 UJ	--	--	0.345 U	0.455 UJ	0.37 UJ	--
METALS (mg/kg)																
ALUMINUM	7,700	EPA RSL	**21,400**	**18,300 J**	7190 J	**18,500 J**	3,370 J	5,890	--	**8,690**	--	--	5,970	**8,040**	7,100	--
ARSENIC	0.39	EPA RSL	**28.7**	**2.6**	**5.2 K**	**3.0**	**4.6**	**1.53 J**	--	**3.01 J**	--	--	**0.925 J**	**2.25 J**	**5.24 J**	--
BARIUM	1,500	EPA RSL	66.5	133	37.8 J	134	15.1 J	28.2 J	--	51.1 J	--	--	30.8	39.6 J	16.7 J	--
BERYLLIUM	16	EPA RSL	1.5	0.88	0.37	0.91	0.19	0.361	--	0.777	--	--	0.455 J	0.557	0.613	--
CADMIUM	3.9	MDE	--	0.058	0.20 K	0.10	0.16 K	0.117 U	--	0.118 U	--	--	0.239 U	0.119 U	0.115 U	--
CALCIUM	--	--	--	296	1,140	131	20,000	--	--	--	--	--	--	--	--	--
CHROMIUM	0.29	EPA RSL	**59.1**	**17.1 J**	**27.5 J**	**17.0 J**	**23.5 J**	**8.03 K**	--	**15.8 K**	--	--	**10**	**13.3 K**	**14.5 K**	--
COBALT	2.3	EPA RSL	**14.7**	**3.8**	**5.7**	**2.8**	**4.1**	**3.36**	--	**5.28**	--	--	**2.54**	**6.12**	**5.13**	--
COPPER	310	EPA RSL	47.6	12.0	13.0 J	11.6	17.1 J	4.8	--	14	--	--	10	8.13	11.6	--
IRON	5,500	EPA RSL	**35,200**	**8,550**	**19,700**	**13,400**	**12,100**	**9,430**	--	**14,500**	--	--	**8,670**	**9,380**	**15,500**	--
LEAD	400	EPA RSL	38.6	18.5	9.5	17.2	5.5	5.34	--	11.6	--	--	5.99	9.23	9.19	--
MAGNESIUM	--	--	--	629	369	579	11500	--	--	--	--	--	--	--	--	--
MANGANESE	160	MDE	155	29.9 J	156 J	43.4 J	102 J	23.3 K	--	16.9 K	--	--	11.5	28.6 K	30 K	--
MERCURY	0.78	MDE	0.14	0.056 U	0.051 U	0.049 U	0.043 U	0.0128 J	--	0.0347 U	--	--	0.0418 U	0.0292 U	0.0383 U	--
NICKEL	150	EPA RSL	15.9	8.2	6.6	7.4	7.7	6.02	--	10.3	--	--	5.92	10.4	9.5	--
POTASSIUM	--	--	--	357	351	412	366	--	--	--	--	--	--	--	--	--
SELENIUM	39	EPA RSL	3.8	0.65 UR	0.52 UR	0.64 UR	0.54 UR	0.293 UL	--	0.343 J	--	--	0.597 U	0.223 J	0.258 J	--
SILVER	39	EPA RSL	--	2.0 B	0.60 B	2.0 B	0.13 U	0.117 U	--	0.118 U	--	--	0.239 U	0.119 U	0.115 U	--
SODIUM	--	--	--	344	173	260	133	--	--	--	--	--	--	--	--	--
THALLIUM	0.078	EPA RSL	--	0.43 U	0.34 U	**0.61 B**	0.36 U	0.235 U	--	0.472 U	--	--	0.478 U	0.474 U	0.461 U	--
VANADIUM	7.8	MDE	**102**	**18.3**	**37.3**	**22.2**	**42.0**	**11.3 J**	--	**27.1 J**	--	--	**12.7**	**16.4 J**	**29.9 J**	--
ZINC	2,300	EPA RSL	49.7	20.5 K	13.2 K	15.2 K	9.7 K	18.8 K	--	35.7 K	--	--	16.9	27.8 K	31.4 K	--
MISCELLANEOUS PARAMETERS																
CYANIDE (mg/kg)	2.2	EPA RSL	--	0.11 U	0.10 U	0.21	0.096 U	0.292 U	--	0.266 U	--	--	0.266 U	0.285 U	0.291 U	--
TOTAL ORGANIC CARBON (mg/kg)	--	--	--	--	--	--	--	1,180 J	--	1,660	--	--	702 J	1,210 J	900 J	--
PERCHLORATE (µg/kg)	5,500	EPA RSL	--	--	--	--	--	3.86 J	--	2.38 UJ	--	--	2.45 U	2.42 UJ	4.84 J	--
VOLATILES (µg/kg)																
2-BUTANONE	2,800,000	EPA RSL	--	8 J	10 U	10 U	7 U	--	5 U	--	4.91 U	5.07 U	5.62 U	--	--	208 U
2-HEXANONE	21,000	EPA RSL	--	12 U	10 U	10 U	7 U	--	2.5 U	--	2.45 U	2.53 U	2.81 U	--	--	104 U
4-METHYL-2-PENTANONE	530,000	EPA RSL	--	12 U	10 U	10 U	7 U	--	2.5 U	--	2.45 U	2.53 U	2.81 U	--	--	104 U
ACETONE	6,100,000	EPA RSL	--	17 J	25 J	19 J	16 J	--	20 J	--	14.5 J	13.3 J	11.2 UR	--	--	416 U
CARBON DISULFIDE	82,000	EPA RSL	--	12 U	10 U	10 U	0.8 J	--	2.5 U	--	2.45 U	2.53 U	2.81 U	--	--	104 U
CIS-1,2-DICHLOROETHENE	16,000	EPA RSL	--	12 U	10 U	10 U	7 U	--	2.5 U	--	2.45 U	2.53 U	2.81 U	--	--	104 U
METHYLENE CHLORIDE	56,000	EPA RSL	--	120 J	6 B	8 B	9 B	--	5 U	--	4.91 U	5.07 U	9.47 B	--	--	208 U
TETRACHLOROETHENE	1,200	MDE	--	12 U	10 U	10 U	7 U	--	2.5 U	--	2.45 U	2.53 U	1.65 J	--	--	104 U
TOLUENE	500,000	EPA RSL	--	12 U	10 U	10 U	0.8 J	--	2.5 U	--	2.45 U	2.53 U	2.81 U	--	--	104 U
TRICHLOROETHENE	910	EPA RSL	--	12 U	10 U	10 U	7 U	--	2.5 U	--	2.45 U	2.53 U	2.81 U	--	--	470
VINYL CHLORIDE	60	EPA RSL	--	12 U	10 U	10 U	7 U	--	2.5 U	--	2.45 U	2.53 U	2.81 U	--	--	104 U

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/kg : micrograms per kilogram
mg/kg : milligram per kilogram

J : Estimated value
B : Compound detected in blank sample
K : Biased high
L : Biased Low
R : The chemical was rejected.
U : The chemical was not detected.

-- : The chemical was not analyzed or no value was available.

Bold and shaded values indicate an exceedance of PSLs

EPA RSL : U.S. Environmental Protection Agency May 2012 Regional Screening Level for soil under residential scenario. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for chromium VI used as surrogate for [total] chromium. RSL for methylmercury used as surrogate for mercury.

MDE : Maryland Department of Environment Cleanup Standards for residential soil. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

TABLE 2-1
SOIL ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND
PAGE 3 OF 4

LOCATION	PSL [1]	PSL Reference	NSF-IH Background Subsurface Soil	S43SB012		S43SB013	S43SB014	S43SB015	S43SB016		S43SB017	S43SB018	S43SB019	S43SB022	S43SB023	
SAMPLE ID				S43SB012-0507	S43SB012-0507-DUP	S43SB013-0608	S43SB014-0709	S43SB015-0810	S43SB016-0608	S43SB016-0607	S43SB017-0608	S43SB018-0507	S43SB019-0607	S43SB022-0708	S43SB023-0001	S43SB023-0001-DUP
SAMPLE DATE				6/11/2011	6/11/2011	6/3/2011	6/4/2011	6/3/2011	6/5/2011	6/12/2011	6/10/2011	6/10/2011	6/11/2011	6/11/2011	5/13/2012	5/13/2012
EXPLOSIVES (mg/kg)																
1,3,5-TRINITROBENZENE	220	EPA RSL	--	0.0367 U	0.0396 U	0.0367 U	0.0377 U	0.0381 U	0.0392 U	--	0.0377 U	0.0381 U	--	--	0.0392 U	0.0385 U
1,3-DINITROBENZENE	0.61	EPA RSL	--	0.0367 U	0.0396 U	0.0367 U	0.0377 U	0.0381 U	0.032 J	--	0.0377 U	0.0381 U	--	--	0.0392 U	0.0385 U
3-NITROTOLUENE	0.61	EPA RSL	--	0.0367 U	0.0396 U	0.0367 U	0.0377 U	0.0381 U	0.0392 U	--	0.0377 U	0.0381 U	--	--	0.0392 U	0.0385 U
4-NITROTOLUENE	30	EPA RSL	--	0.0367 U	0.0396 U	0.0367 U	0.0234 J	0.0381 U	0.0239 J	--	0.0377 U	0.0381 U	--	--	0.0392 U	0.0385 U
NITROCELLULOSE	18,000,000	EPA RSL	--	--	--	--	--	--	--	--	--	--	--	--	--	--
NITROGUANIDINE	610	EPA RSL	--	0.476 UJ	0.455 UJ	0.435 U	0.385 UJ	0.417 U	0.37 UJ	--	0.476 UJ	0.455 UJ	--	--	0.182 U	0.2 U
METALS (mg/kg)																
ALUMINUM	7,700	EPA RSL	**21,400**	**10,500**	**8,440**	2,180	**12,300**	**10,600**	**9,680**	--	7,220	7,260	--	--	**20,200**	**20,000**
ARSENIC	0.39	EPA RSL	**28.7**	**2.91**	**2.92**	**0.834 J**	**2.48 J**	**2.14 L**	**1.19 J**	--	**1.28**	**0.895**	--	--	**2.69 L**	**4.28 L**
BARIUM	1,500	EPA RSL	66.5	221	165	19	115 J	148	55.4 J	--	33.2	25.6	--	--	76 L	80.2 L
BERYLLIUM	16	EPA RSL	1.5	0.713	0.666	0.295 J	0.855	0.844	0.675	--	0.545	0.605	--	--	0.815	0.859
CADMIUM	3.9	MDE	--	0.284 U	0.292 U	0.232 U	0.112 U	0.233 U	0.113 U	--	0.298 U	0.288 U	--	--	0.459 J	2.13 J
CALCIUM	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
CHROMIUM	0.29	EPA RSL	**59.1**	**16.2**	**13.4**	**18.5**	**15.7 K**	**13.1**	**14.2 K**	--	**12.7**	**11.9**	--	--	**21.3 K**	**24.4 K**
COBALT	2.3	EPA RSL	**14.7**	**6.16**	**5.32**	**1.47**	**5.45**	**4.17**	**5.29**	--	**5.04**	**4.71**	--	--	**3.64**	**4.38**
COPPER	310	EPA RSL	47.6	12.1	10.2	7.38	15.3	12.9	10.4	--	6.77	9.81	--	--	21.1	32.7
IRON	5,500	EPA RSL	**35,200**	**9,620**	**9,590**	4,010	**10,600**	**8,630**	**8,290**	--	**9,110**	**10,500**	--	--	**25,900 J**	**32,100 J**
LEAD	400	EPA RSL	38.6	10.7	9.36	5.02	13.3	11.4	9.87	--	8.21	8.1	--	--	14.8 J	28.2 J
MAGNESIUM	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
MANGANESE	160	MDE	155	30.9 J	18.1 J	23	17 K	10.3	9.75 K	--	16.5 J	15.4 J	--	--	26.8 K	36.6 K
MERCURY	0.78	MDE	0.14	0.0376 U	0.036 U	0.0368 U	0.0133 J	0.0364 U	0.0388 U	--	0.0351 U	0.0365 U	--	--	0.0259 J	0.0207 J
NICKEL	150	EPA RSL	15.9	11.2	9.88	5.26	11.8	10.1	9.09	--	8.38	8.97	--	--	9.2	9.72
POTASSIUM	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
SELENIUM	39	EPA RSL	3.8	0.569 UL	0.585 UL	0.58 U	0.28 UL	0.583 U	0.284 UL	--	0.596 UL	0.575 UL	--	--	0.54 J	0.565 J
SILVER	39	EPA RSL	--	0.569 UL	0.585 UL	0.232 U	0.112 U	0.233 U	0.113 U	--	0.596 UL	0.575 UL	--	--	0.133 U	0.086 J
SODIUM	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
THALLIUM	0.078	EPA RSL	--	0.455 U	0.468 U	0.464 U	0.392 U	0.466 U	0.454 U	--	0.477 U	0.46 U	--	--	0.266 UL	0.257 UL
VANADIUM	7.8	MDE	**102**	**18.3**	**18**	7.53	**20.3 J**	**20.1**	**12.6 J**	--	**17.8**	**16.7**	--	--	**32**	**33.8**
ZINC	2,300	EPA RSL	49.7	35.2	33.8	9.98	46.6 K	26.3	36.2 K	--	23.1	22.7	--	--	47.1 J	110 J
MISCELLANEOUS PARAMETERS																
CYANIDE (mg/kg)	2.2	EPA RSL	--	0.883 U	0.873 U	0.268 U	0.294 U	0.272 U	0.265 U	--	0.877 U	0.884 U	--	--	--	--
TOTAL ORGANIC CARBON (mg/kg)	--	--	--	1,260 J	1,690	1,100 J	1,030 J	828 J	1,020 J	--	588 J	1,130 J	--	--	--	--
PERCHLORATE (µg/kg)	5,500	EPA RSL	--	2.36 UJ	2.33 U	2.38 U	1.08 J	2.35 U	2.35 UJ	--	2.34 UJ	2.36 UJ	--	--	--	--
VOLATILES (µg/kg)																
2-BUTANONE	2,800,000	EPA RSL	--	5.04 U	5.04 U	4.6 U	--	564 J	--	4.73 U	239 U	236 U	4.52 U	837	5.28 U	4.93 U
2-HEXANONE	21,000	EPA RSL	--	2.52 U	2.52 U	2.3 U	--	16	--	2.37 U	120 U	118 U	2.26 U	132 U	2.64 U	2.47 U
4-METHYL-2-PENTANONE	530,000	EPA RSL	--	2.52 U	2.52 U	2.3 U	--	5190 J	--	2.37 U	120 U	118 U	2.26 U	19,100 J	2.64 U	2.47 U
ACETONE	6,100,000	EPA RSL	--	12.1 B	12 J	9.2 UR	--	157000 J	--	18.3 J	478 U	472 U	23.6 J	4,660,000	26.1 B	19.1 B
CARBON DISULFIDE	82,000	EPA RSL	--	2.52 U	2.52 U	2.3 U	--	2.44 U	--	2.37 U	120 U	118 U	2.26 U	132 U	2.64 U	2.47 U
CIS-1,2-DICHLOROETHENE	16,000	EPA RSL	--	2.52 U	2.52 U	2.3 U	--	2.44 U	--	2.37 U	397	118 U	2.26 U	585	2.64 U	2.47 U
METHYLENE CHLORIDE	56,000	EPA RSL	--	5.04 U	5.04 U	6.81 J	--	8.6 J	--	4.73 U	239 U	236 U	4.52 U	265 U	5.28 U	4.93 U
TETRACHLOROETHENE	1,200	MDE	--	2.52 U	2.52 U	2.3 U	--	2.44 U	--	2.37 U	120 U	118 U	2.26 U	132 U	2.64 U	2.47 U
TOLUENE	500,000	EPA RSL	--	2.52 U	2.52 U	1.29 J	--	2.44 U	--	2.37 U	120 U	118 U	2.26 U	132 U	2.64 U	2.47 U
TRICHLOROETHENE	910	EPA RSL	--	2.52 U	2.52 U	2.3 U	--	2.44 U	--	2.37 U	**3,940**	776	2.26 U	132 U	2.64 U	2.47 U
VINYL CHLORIDE	60	EPA RSL	--	2.52 U	2.52 U	2.3 U	--	2.44 U	--	2.37 U	120 U	118 U	14.3	132 U	2.64 U	2.47 U

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/kg : micrograms per kilogram
mg/kg : milligram per kilogram

J : Estimated value
B : Compound detected in blank sample
K : Biased high
L : Biased Low
R : The chemical was rejected.
U : The chemical was not detected.

-- : The chemical was not analyzed or no value was available.

Bold and shaded values indicate an exceedance of PSLs

EPA RSL : U.S. Environmental Protection Agency May 2012 Regional Screening Level for soil under residential scenario. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for chromium VI used as surrogate for [total] chromium. RSL for methylmercury used as surrogate for mercury.

MDE : Maryland Department of Environment Cleanup Standards for residential soil. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

TABLE 2-1
SOIL ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL Reference	NSF-IH Background Subsurface Soil	S43SB023	S43SB024			S43SB025		S43SB026		S43SB027		S43SB028	
SAMPLE ID				S43SB023-0607	S43SB024-0001	S43SB024-0607	S43SB024-0607-DUP	S43SB025-0001	S43SB025-0607	S43SB026-0001	S43SB026-0506	S43SB027-0001	S43SB027-0708	S43SB028-0001	S43SB028-0405
SAMPLE DATE				5/13/2012	5/13/2012	5/13/2012	5/13/2012	5/13/2012	5/13/2012	5/13/2012	5/13/2012	5/13/2012	5/13/2012	5/13/2012	5/13/2012
EXPLOSIVES (mg/kg)															
1,3,5-TRINITROBENZENE	220	EPA RSL	--	0.0385 U	0.0385 U	0.0385 U	0.04 U	0.0396 U	0.0392 U	0.04 U	0.0396 U	0.0385 U	0.0388 U	0.032 J	0.0388 U
1,3-DINITROBENZENE	0.61	EPA RSL	--	0.0385 U	0.0385 U	0.0385 U	0.04 U	0.0396 U	0.0392 U	0.04 U	0.0396 U	0.0385 U	0.0277 J	0.0388 U	0.0388 U
3-NITROTOLUENE	0.61	EPA RSL	--	0.0385 U	0.0385 U	0.0385 U	0.04 U	0.0396 U	0.0392 U	0.04 U	0.0396 U	0.0385 U	0.0388 U	0.0388 U	0.0388 U
4-NITROTOLUENE	30	EPA RSL	--	0.0385 U	0.0385 U	0.0385 U	0.04 U	0.0396 U	0.0392 U	0.04 U	0.0396 U	0.0385 U	0.0388 U	0.0388 U	0.0388 U
NITROCELLULOSE	18,000,000	EPA RSL	--	--	--	--	--	--	--	--	--	--	--	--	--
NITROGUANIDINE	610	EPA RSL	--	0.19 U	0.211 U	0.19 U	0.182 U	0.182 U	0.19 U	0.19 U	0.211 U	0.2 U	0.19 U	0.429 J	0.174 U
METALS (mg/kg)															
ALUMINUM	7,700	EPA RSL	**21,400**	**11,200**	**17,400**	**8,860**	**8,640**	**15,700**	**8,390**	**16,000**	**9,620**	**13,100**	7,170	**17,600**	**10,400**
ARSENIC	0.39	EPA RSL	**28.7**	**1.88 L**	**1.95 L**	**1.41 L**	**2.08 L**	**2.27 L**	**1.45 L**	**1.6 L**	**2.34 L**	**2.68 L**	**1.15 L**	**12.3**	**2.53 L**
BARIUM	1,500	EPA RSL	66.5	408 K	60.9 L	33.5 L	31.6 L	56.1 L	53.7 L	70.3 L	177 L	233 L	14.9 L	132 L	43.5 L
BERYLLIUM	16	EPA RSL	1.5	0.704	0.691	0.557	0.552	0.572	0.507	0.524	0.462	0.534	0.435	0.883	0.742
CADMIUM	3.9	MDE	--	0.118 U	0.471	0.122 U	0.121 U	0.072 J	0.119 U	0.0996 J	0.124 U	0.0774 J	0.121 U	12.2	0.0771 J
CALCIUM	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
CHROMIUM	0.29	EPA RSL	**59.1**	**16.4 K**	**19.1 K**	**14.3 K**	**13.4 K**	**18.3 K**	**13.3 K**	**19.5 K**	**14.3 K**	**17.1 K**	**10.7 K**	**30.3 K**	**18.5 K**
COBALT	2.3	EPA RSL	**14.7**	**6.28**	**3.34**	**4.58**	**3.81**	**2.94**	**3.69**	**3.76**	**4.38**	**4.74**	**3.73**	**11.7**	**4.38**
COPPER	310	EPA RSL	47.6	15.1	16.6	10.8	10.2	14.9	11.3	12.3	11.9	10.6	10.4	57.4	14.4
IRON	5,500	EPA RSL	**35,200**	**12,300 J**	**18,500 J**	**13,700 J**	**12,200 J**	**15,400 J**	**10,400 J**	**12,700 J**	**14,400 J**	**12,500 J**	**14,600 J**	**23,900 J**	**17,300 J**
LEAD	400	EPA RSL	38.6	9.2	12.3	7.8	7.85	11	7.79	10.4	8.96	9.69	6.63	74	10.9
MAGNESIUM	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
MANGANESE	160	MDE	155	17.8 K	14.7 K	17.3 K	19.6 K	17.2 K	20.4 K	28.2 K	18.3 K	22.2 K	11.5 K	391 K	106 K
MERCURY	0.78	MDE	0.14	0.03 U	0.034 U	0.029 U	0.037 U	0.035 U	0.032 U	0.033 U	0.035 U	0.033 U	0.030 U	0.12	0.0221 J
NICKEL	150	EPA RSL	15.9	14.5	7.66	8.23	7.63	7.1	8.06	8.25	8.22	9.14	6.12	24.3	7.26
POTASSIUM	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
SELENIUM	39	EPA RSL	3.8	0.295 UL	0.331 J	0.253 J	0.353 J	0.396 J	0.227 J	0.31 J	0.358 J	0.256 J	0.279 J	0.801 J	0.327 J
SILVER	39	EPA RSL	--	0.118 U	0.0673 J	0.122 U	0.121 U	0.0848 J	0.119 U	0.121 U	0.124 U	0.129 U	0.121 U	0.262 J	0.133 U
SODIUM	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
THALLIUM	0.078	EPA RSL	--	0.295 UL	0.369 UL	0.304 UL	0.303 UL	0.31 UL	0.298 UL	0.242 UL	0.247 UL	0.258 UL	0.303 UL	**0.664 J**	0.266 UL
VANADIUM	7.8	MDE	**102**	**25.4**	**29.5**	**17**	**19.8**	**32.7**	**22.4**	**32.4**	**20.2**	**34.6**	**13.3**	**44.6**	**34**
ZINC	2,300	EPA RSL	49.7	23.5 J	33.5 J	24.4 J	21.4 J	19.4 J	20.8 J	19.7 J	21.2 J	23.8 J	16.5 J	402 J	31 J
MISCELLANEOUS PARAMETERS															
CYANIDE (mg/kg)	2.2	EPA RSL	--	--	--	--	--	--	--	--	--	--	--	--	--
TOTAL ORGANIC CARBON (mg/kg)	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--
PERCHLORATE (µg/kg)	5,500	EPA RSL	--	--	--	--	--	--	--	--	--	--	--	--	--
VOLATILES (µg/kg)															
2-BUTANONE	2,800,000	EPA RSL	--	5020 U	4.59 U	256 U	257 U	4.34 U	250 U	6.03 U	233 U	229 U	278 U	486 U	5.12 U
2-HEXANONE	21,000	EPA RSL	--	2,510 U	2.3 U	128 U	128 U	2.17 U	125 U	3.02 U	117 U	114 U	139 U	243 U	2.56 U
4-METHYL-2-PENTANONE	530,000	EPA RSL	--	2,510 U	2.3 U	128 U	128 U	2.17 U	125 U	3.02 U	117 U	114 U	139 U	243 U	2.56 U
ACETONE	6,100,000	EPA RSL	--	195,000 J	20.4 B	512 U	513 U	8.95 B	500 U	14.8 B	467 U	457 U	556 U	972 U	10.2 U
CARBON DISULFIDE	82,000	EPA RSL	--	2,510 U	2.3 U	128 U	128 U	2.17 U	125 U	3.02 U	117 U	114 U	139 U	243 U	2.56 U
CIS-1,2-DICHLOROETHENE	16,000	EPA RSL	--	2,510 U	14.4	442	397	2.17 U	100 J	23.7	117 U	175 J	447	243 U	2.56 U
METHYLENE CHLORIDE	56,000	EPA RSL	--	3,590 J	4.59 U	256 U	257 U	4.34 U	250 U	6.03 U	233 U	229 U	278 U	486 U	5.12 U
TETRACHLOROETHENE	1,200	MDE	--	2,510 U	2.3 U	128 U	128 U	2.17 U	125 U	3.02 U	117 U	114 U	139 U	243 U	2.56 U
TOLUENE	500,000	EPA RSL	--	2,510 U	2.3 U	128 U	128 U	2.17 U	125 U	3.02 U	117 U	114 U	139 U	243 U	2.56 UJ
TRICHLOROETHENE	910	EPA RSL	--	2,510 U	2.3 U	**2,080**	**1,700**	1.91 J	**2,380**	69.4	**1,150**	**2,720**	**4,790**	243 U	2.56 U
VINYL CHLORIDE	60	EPA RSL	--	2,510 U	2.3 U	128 U	128 U	2.17 U	125 U	2.18 J	117 U	114 U	139 U	243 U	2.56 U

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/kg : micrograms per kilogram
mg/kg : milligram per kilogram

J : Estimated value
B : Compound detected in blank sample
K : Biased high
L : Biased Low
R : The chemical was rejected.
U : The chemical was not detected.

-- : The chemical was not analyzed or no value was available.

Bold and shaded values indicate an exceedance of PSLs

EPA RSL : U.S. Environmental Protection Agency May 2012 Regional Screening Level for soil under residential scenario. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for chromium VI used as surrogate for [total] chromium. RSL for methylmercury used as surrogate for mercury.

MDE : Maryland Department of Environment Cleanup Standards for residential soil. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

TABLE 2-2
MONITORING WELL
VOLATILE ORGANIC COMPONDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	S43MW001							S43MW002					S43MW003	
SAMPLE ID			S43MW0010101	S43MW0010101-DUP	S43MW0010102	S43MW0010102-DUP	S43MW00103	S43MW0104	S43MW0104-DUP	S43MW0020101	S43MW0020102	S43MW00203	S43MW0204	S43MW0204-DUP	S43MW00301	S43MW0302
SAMPLE DATE			20050725	20050725	20071111	20071111	20110617	20120722	20120722	20050727	20071111	20110617	20120722	20120722	20110617	20120722
VOLATILES (µg/L)																
1,1,1-TRICHLOROETHANE	200	MDE	500 U	500 U	800 U	800 U		50 U	50 U	0.51	1.3				0.5 U	0.5 U
1,1-DICHLOROETHANE	2.4	EPA RSL	500 U	500 U	800 U	800 U	25 U	50 U	50 U	**2.7**	**15**				0.5 U	0.5 U
1,1-DICHLOROETHENE	7	MDE	500 U	500 U	800 U	800 U	25 U	50 U	50 U	1.1	3.9				0.5 U	0.5 U
1,2-DICHLOROETHANE	0.15	EPA RSL	500 U	500 U	800 U	800 U	25 U	50 U	50 U	0.5 U	0.5 U				0.5 U	0.5 U
2-BUTANONE	490	EPA RSL	5000 U	5000 U	8000 U	8000 U	250 U	500 U	500 U	5 U	5 U				5 U	5 U
ACETONE	550	MDE	5000 U	5000 U	8000 U	8000 U	250 U	500 U	500 U	5 U	5 U				5 U	5 U
BROMOFORM	7.9	EPA RSL	500 U	500 U	800 U	**960**	25 U	50 U	50 U	0.5 U	0.5 U				0.5 U	0.5 U
CARBON DISULFIDE	72	EPA RSL	500 U	500 U	800 U	800 U	25 U	50 U	50 U	0.5 U	0.5 U				0.5 U	0.5 U
CHLOROFORM	0.19	EPA RSL	500 U	500 U	800 U	800 U	**8.32 J**	50 U	50 U	0.5 U	0.5 U				0.5 U	0.5 U
CHLOROMETHANE	19	EPA RSL	500 U	500 U	800 U	800 U	25 U	50 U	50 U	0.5 U	0.5 U				6.35	0.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	**580**	**480 J**	800 U	800 U	**284**	**400**	**355**	0.5 U	0.5 U				0.896 J	0.77 J
METHYLENE CHLORIDE	5	MDE	500 U	**730 B**	800 U	800 U	50 U	100 U	100 U	0.5 U	0.5 U				1 U	1 U
TETRACHLOROETHENE	5	MDE	500 U	500 U	800 U	800 U	25 U	50 U	50 U	0.5 U	0.5 U				0.5 U	0.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	500 U	500 U	800 U	800 U	25 U	50 U	50 U	0.5 U	0.5 U				0.5 U	0.5 U
TRICHLOROETHENE	0.44	EPA RSL	**53000 J**	**55000 J**	**36000**	**36000**	**10900**	**17500**	**16200**	**1.3 J**	**3.2**	0.398 J	0.5 U	0.5 U	**1.74**	**0.66 J**
VINYL CHLORIDE	0.015	EPA RSL	500 U	500 U	800 U	800 U	25 U	50 U	50 U	0.5 U	0.5 U				**1.16**	**1.23**

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
U : The chemical was not detected.
Blank cells or -- : The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs
[1] Selected PSL is the lowest (most conservative) of the applicable screening criter

EPA RSL: U.S. Environmental Protection Agency (EPA) (November 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-2
MONITORING WELL
VOLATILE ORGANIC COMPONDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	S43MW004			S43MW005		S43MW006		S43MW007	S43MW008	S43MW009D	S43MW009S	S43MW010	S43MW011	S43MW012	S43MW013
SAMPLE ID			S43MW00401	S43MW00401-DUP	S43MW0402	S43MW00501	S43MW0502	S43MW00601	S43MW0602	S43MW0701	S43MW0801	S43MW09D01	S43MW09S01	S43MW1001	S43MW1101	S43MW1201	S43MW1301
SAMPLE DATE			20110617	20110617	20120722	20110617	20120722	20110617	20120721	20120722	20120721	20120722	20120722	20120722	20120721	20120722	20120722
VOLATILES (µg/L)																	
1,1,1-TRICHLOROETHANE	200	MDE	2.5 U	2.5 U	2.5 U	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	1 U	0.5 U	0.5 U	0.5 U	
1,1-DICHLOROETHANE	2.4	EPA RSL	2.5 U	2.5 U	2.5 U	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	1 U	0.5 U	0.5 U	0.5 U	
1,1-DICHLOROETHENE	7	MDE	2.56 J	2.43 J	2.95 J	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	4.36	0.5 U	0.5 U	0.5 U	
1,2-DICHLOROETHANE	0.15	EPA RSL	2.5 U	2.5 U	2.5 U	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	1 U	0.5 U	0.5 U	0.5 U	
2-BUTANONE	490	EPA RSL	25 U	25 U	25 U	5 U	5 U	50 U	125 U	5 U	5 U	5 U	10 U	5 U	5 U	5 U	
ACETONE	550	MDE	25 U	25 U	25 U	5 U	5 U	50 U	125 U	5 U	5 U	5 U	10 U	5 U	5 U	5 U	
BROMOFORM	7.9	EPA RSL	2.5 U	2.5 U	2.5 U	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	1 U	0.5 U	0.5 U	0.5 U	
CARBON DISULFIDE	72	EPA RSL	2.5 U	2.5 U	2.25 J	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	2.36	2.65	0.5 U	0.55 J	
CHLOROFORM	0.19	EPA RSL	2.5 U	2.5 U	2.5 U	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	1 U	0.5 U	0.5 U	0.5 U	
CHLOROMETHANE	19	EPA RSL	5.05	2.5 U	2.5 U	3.41	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	1 U	0.5 U	0.5 U	0.5 U	
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	**152**	**156**	**203**	0.5 U	0.5 U	**41.6**	**153 J**	0.5 U	0.5 U	0.5 U	**26.4**	0.5 U	0.5 U	0.5 U	
METHYLENE CHLORIDE	5	MDE	5 U	5 U	5 U	1 U	0.53 J	10 U	25 U	1 U	1 U	0.51 J	2 U	1 U	1 U	1 U	
TETRACHLOROETHENE	5	MDE	2.5 U	2.5 U	2.5 U	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	1 U	0.5 U	0.5 U	0.5 U	
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	2.5 U	2.5 U	1.4 J	0.5 U	0.5 U	5 U	12.5 U	0.5 U	0.5 U	0.5 U	0.58 J	0.5 U	0.5 U	0.5 U	
TRICHLOROETHENE	0.44	EPA RSL	**699**	**682**	**644 J**	0.5 U	0.5 U	**1130**	**4420**	0.31 J	0.5 U	0.5 U	**100**	0.5 U	0.5 U	**1.77**	**1.18**
VINYL CHLORIDE	0.015	EPA RSL	**15.7**	**14.1**	**37.9 J**	0.5 U	0.5 U	5 U	**7.5 J**	0.5 U	0.5 U	0.5 U	**2.7 J**	0.5 U	0.5 U	0.5 U	

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
U : The chemical was not detected.
Blank cells or -- : The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs
[1] Selected PSL is the lowest (most conservative) of the applicable screening criter

EPA RSL: U.S. Environmental Protection Agency (EPA) (November 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-2
MONITORING WELL
VOLATILE ORGANIC COMPONDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	S43MW014	S43MW015	S43MW016	S43MW017D	S43MW017S	S43MW018	S43MW019D	S43MW019S		S43MW020	S43MW021	S43MW022	S43MW023	S43MW024	S43MW025	S43MW026
SAMPLE ID			S43MW1401	S43MW1501	S43MW1601	S43MW17D01	S43MW17S01	S43MW1801	S43MW19D01	S43MW19S01	S43MW19S01-DUP	S43MW2001	S43MW2101	S43MW2201	S43MW2301	S43MW2401	S43MW2501	S43MW2601
SAMPLE DATE			20120722	20120722	20120722	20120721	20120721	20120721	20120721	20120721	20120721	20120719	20120721	20120719	20120720	20120720	20120720	20120718
VOLATILES (µg/L)																		
1,1,1-TRICHLOROETHANE	200	MDE				10 U	10 U	0.5 U	1 U	12.5 U	12.5 U	11.1	0.5 U	0.37 J	0.5 U	3.67	0.5 U	0.5 U
1,1-DICHLOROETHANE	2.4	EPA RSL				10 U	10 U	0.5 U	1 U	**9.75 J**	**10.2 J**	5 U	0.5 U	**3.49**	0.4 J	**39.6**	0.76 J	0.5 U
1,1-DICHLOROETHENE	7	MDE				6.2 J	6.6 J	0.5 U	1 U	**57**	**70**	5 U	0.5 U	2.04	0.5 U	**8.6**	0.8 J	0.5 U
1,2-DICHLOROETHANE	0.15	EPA RSL				10 U	10 U	0.5 U	1 U	12.5 U	12.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
2-BUTANONE	490	EPA RSL				100 U	100 U	5 U	10 U	125 U	125 U	50 U	5 U	5 U	5 U	4.04 J	5 U	5 U
ACETONE	550	MDE				100 U	100 U	5 U	10 U	125 U	125 U	50 U	5 U	5 U	5 U	7.56 J	5 U	5 U
BROMOFORM	7.9	EPA RSL				10 U	10 U	0.5 U	1 U	12.5 U	12.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CARBON DISULFIDE	72	EPA RSL				10 U	10 U	0.5 U	1 U	12.5 U	12.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CHLOROFORM	0.19	EPA RSL				10 U	10 U	0.5 U	1 U	12.5 U	12.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CHLOROMETHANE	19	EPA RSL				10 U	10 U	0.5 U	1 U	12.5 U	12.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL				**255**	**212**	1.59	**30.5**	**158**	**166**	2.5 J	0.5 U	0.5 U	0.5 U	0.5 U	1	0.5 U
METHYLENE CHLORIDE	5	MDE				20 U	20 U	1 U	2 U	25 U	25 U	10 U	1 U	1 U	1 U	1 U	1 U	1 U
TETRACHLOROETHENE	5	MDE				10 U	10 U	0.5 U	1 U	12.5 U	12.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL				10 U	10 U	0.5 U	1 U	12.5 U	12.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
TRICHLOROETHENE	0.44	EPA RSL	0.5 U	0.5 U	0.5 U	**2660**	**2740**	**2.78**	**72.8**	**3620**	**4090**	**1390**	**0.83 J**	**0.54 J**	**1.59**	0.5 U	**9.22**	**0.51 J**
VINYL CHLORIDE	0.015	EPA RSL				**65.6**	**45.2**	0.5 U	**9.12**	12.5 U	12.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
U : The chemical was not detected.
Blank cells or -- : The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criter

EPA RSL: U.S. Environmental Protection Agency (EPA) (November 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-3
GROUNDWATER GRAB
VOLATILE ORGANIC COMPUNDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL	S43NW1	S43W2	S43N4	S43N3	S43W4	S43TW006	S43TW007	S43TW007	S43TW008	S43TW009	S43TW011	S43TW012	S43TW013	S43TW014
SAMPLE ID		Reference	S43TW0010102	S43TW0020102	S43TW0030102	S43TW0040102	S43TW0050102	S43TW006-2832	S43TW007-2327	S43TW007-2327-DUP	S43TW008-2832	S43TW009-2125	S43TW011-3034	S43TW012-2630	S43TW13-2933	S43TW14-1317
VOLATILES (µg/L)																
1,1,1-TRICHLOROETHANE	200	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
1,1-DICHLOROETHANE	2.4	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
1,1-DICHLOROETHENE	7	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	4.5 J	4.75 J	0.364 J	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
1,2-DICHLOROETHANE	0.15	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
2-BUTANONE	490	EPA RSL	5 U	5 U	4.2 J	5 U	5 U	5 U	50 U	50 U	5 U	25 U	5 U	5 U	5 U	5 U
ACETONE	550	MDE	5 U	5 U	17.0	5 U	5 U	5 U	50 U	50 U	10.6 J	55.6 J	5 U	5 U	4.26 B	5 U
BROMOFORM	7.9	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
CARBON DISULFIDE	72	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
CHLOROFORM	0.19	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
CHLOROMETHANE	19	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	0.36 J	0.5 U	0.5 U	0.5 U	0.5 U	**4.49**	**56.5**	**61.3**	**21.8**	2.61 J	0.50 U	0.50 U	1.71	0.5 U
METHYLENE CHLORIDE	5	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1 U	**7.7 J**	**9.74 J**	0.748 J	4.84 J	1 U	1 U	1 U	1 U
TETRACHLOROETHENE	5	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	5 U	5 U	0.50 U	2.5 U	0.50 U	0.50 U	0.61 J	0.5 U
TRICHLOROETHENE	0.44	EPA RSL	**3.2**	0.5 U	0.5 U	0.5 U	0.5 U	**53.9**	**923**	**1010**	**41.9**	**563**	**1.68**	0.50 U	**20.3**	0.5 U
VINYL CHLORIDE	0.015	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	**0.724 J**	**26.2**	**25.8**	**16.2**	2.5 U	0.50 U	0.50 U	0.5 U	0.5 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
D : Diluted value
U : The chemical was not detected.
-- : The chemical was not analyzed or no value was available.

Bold and shaded values indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-3
GROUNDWATER GRAB
VOLATILE ORGANIC COMPUNDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL	S43TW014	S43TW015	S43TW015	S43TW016	S43TW016	S43TW016	S43TW017	S43TW017	S43TW018	S43TW018	S43TW019	S43TW019	S43TW020	S43TW020
SAMPLE ID		Reference	S43TW14-2933	S43TW15-1721	S43TW15-2933	S43TW16-1721	S43TW16-1721-DUP	S43TW16-2933	S43TW17-1923	S43TW17-2933	S43TW18-1923	S43TW18-3034	S43TW19-2024	S43TW19-2832	S43TW20-1923	S43TW20-2933
VOLATILES (µg/L)																
1,1,1-TRICHLOROETHANE	200	MDE	0.5 U	1 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1 U	2.5 U
1,1-DICHLOROETHANE	2.4	EPA RSL	0.5 U	1 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1 U	2.5 U
1,1-DICHLOROETHENE	7	MDE	0.5 U	0.56 J	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1.34 J	2.5 U
1,2-DICHLOROETHANE	0.15	EPA RSL	0.5 U	1 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1 U	2.5 U
2-BUTANONE	490	EPA RSL	5 U	10 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	10 U	25 U
ACETONE	550	MDE	5 U	10 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5.74 B	25 U
BROMOFORM	7.9	EPA RSL	0.5 U	1 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1 U	2.5 U
CARBON DISULFIDE	72	EPA RSL	0.4 J	1 U	0.63 J	0.5 U	0.5 U	0.65 J	0.5 U	0.5 U	0.5 U	0.5 U	0.29 J	0.5 U	1 U	2.5 U
CHLOROFORM	0.19	EPA RSL	0.5 U	1 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1 U	2.5 U
CHLOROMETHANE	19	EPA RSL	0.5 U	1 U	0.5 U	0.37 J	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.43 J	0.5 U	0.5 U	1 U	2.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	**3.35**	**25.7**	1.17	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	**81.8**	**50.2**
METHYLENE CHLORIDE	5	MDE	1 U	2 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1.28 J	5 U
TETRACHLOROETHENE	5	MDE	0.5 U	1 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1 U	2.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	0.5 U	1 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.56 J	2.5 U
TRICHLOROETHENE	0.44	EPA RSL	**79.9**	**158**	**0.64 J**	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	**298**	**298**
VINYL CHLORIDE	0.015	EPA RSL	**0.74 J**	**1.4 J**	**0.58 J**	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	**13.1**	**12.3**

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
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L : Biased Low
D : Diluted value
U : The chemical was not detected.
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Bold and shaded values indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-3
GROUNDWATER GRAB
VOLATILE ORGANIC COMPUNDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL	S43TW020	S43TW021	S43TW021	S43TW22	S43TW22	S43TW22	S43TW23	S43TW23	S43TW23	S43TW024	S43TW024	S43TW025	S43TW025	S43TW026
SAMPLE ID		Reference	S43TW20-2933-DUP	S43TW21-1923	S43TW21-2630	S43TW22-1014	S43TW22-2226	S43TW22-3337	S43TW23-1014	S43TW23-2226	S43TW23-3236	S43TW24-2327	S43TW24-3236	S43TW25-1115	S43TW25-2933	S43TW26-1014
VOLATILES (µg/L)																
1,1,1-TRICHLOROETHANE	200	MDE	2.5 U	0.5 U	0.5 U	0.5 U	25 U	1 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
1,1-DICHLOROETHANE	2.4	EPA RSL	2.5 U	0.5 U	0.5 U	0.5 U	25 U	1 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
1,1-DICHLOROETHENE	7	MDE	2.5 U	0.5 U	0.5 U	0.5 U	25 U	1.7 J	0.5 U	5.5 J	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
1,2-DICHLOROETHANE	0.15	EPA RSL	2.5 U	0.5 U	0.5 U	0.5 U	25 U	1 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
2-BUTANONE	490	EPA RSL	25 U	5 U	5 U	5 U	250 U	10 U	5 U	50 U	5 U	5 U	5 U	5 U	5 U	5 U
ACETONE	550	MDE	25 U	5 U	5 U	5 U	250 U	10 U	4.94 J	50 U	3.3 J	3.97 B	12.5 B	2.5 B	11.6 B	3.31 B
BROMOFORM	7.9	EPA RSL	2.5 U	0.5 U	0.5 U	0.5 U	25 U	1 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CARBON DISULFIDE	72	EPA RSL	2.5 U	0.5 U	0.5 U	0.5 U	25 U	0.88 J	0.59 J	5 U	0.44 J	0.31 J	0.39 J	0.5 U	0.77 J	0.25 J
CHLOROFORM	0.19	EPA RSL	2.5 U	0.5 U	0.5 U	**0.37 J**	25 U	1 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CHLOROMETHANE	19	EPA RSL	2.5 U	0.5 U	0.5 U	0.5 U	25 U	1 U	0.5 U	5 U	0.5 U	0.5 U	0.5 J	0.5 U	0.65 J	0.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	**47**	0.69 J	0.5 U	0.64 J	**288**	**170**	0.37 J	**197**	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
METHYLENE CHLORIDE	5	MDE	5 U	1 U	1 U	1 U	50 U	2 U	1 U	10 U	0.57 J	1 U	0.55 B	1 U	1 U	1 U
TETRACHLOROETHENE	5	MDE	2.5 U	0.5 U	0.5 U	0.5 U	25 U	1 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	2.5 U	0.5 U	0.5 U	0.5 U	25 U	0.88 J	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
TRICHLOROETHENE	0.44	EPA RSL	**287**	**1.04**	0.35 J	**1.64**	**4440**	**384**	**1.21**	**833**	**1.61**	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
VINYL CHLORIDE	0.015	EPA RSL	**11.5**	0.5 U	0.5 U	0.5 U	**54**	**47.2**	0.5 U	**29.7**	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
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L : Biased Low
D : Diluted value
U : The chemical was not detected.
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Bold and shaded values indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

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TABLE 2-3
GROUNDWATER GRAB
VOLATILE ORGANIC COMPUNDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL	S43TW026	S43TW027	S43TW028	S43TW028	S43TW028	S43TW029	S43TW029	S43TW030	S43TW030	S43TW030	S43TW030	S43TW031	S43TW031	S43TW031
SAMPLE ID		Reference	S43TW26-3236	S43TW27-2933	S43TW28-1721	S43TW28-2933	S43TW28-2933-DUP	S43TW29-2226	S43TW29-3236	S43TW30-1317	S43TW30-1317-DUP	S43TW30-2226	S43TW30-3236	S43TW31-1014	S43TW31-2226	S43TW31-3236
VOLATILES (µg/L)																
1,1,1-TRICHLOROETHANE	200	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
1,1-DICHLOROETHANE	2.4	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
1,1-DICHLOROETHENE	7	MDE	0.5 U	0.5 U	0.43 J	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.29 J	0.52 J	0.5 U	0.5 U	0.5 U
1,2-DICHLOROETHANE	0.15	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
2-BUTANONE	490	EPA RSL	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U
ACETONE	550	MDE	19.8 B	2.93 B	5 U	5 U	5 U	11.4 B	7.81 J	5 U	5 U	4.09 B	5 U	5 U	5 U	12.5 B
BROMOFORM	7.9	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CARBON DISULFIDE	72	EPA RSL	0.5 U	0.5 U	0.26 J	0.38 J	0.5 U	0.5 U	0.72 J	0.5 U	0.26 J	0.5 U	0.32 J	0.5 U	0.5 U	0.47 J
CHLOROFORM	0.19	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CHLOROMETHANE	19	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	1.24 J	0.5 U	0.36 J	0.5 U	0.5 U	0.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	0.5 U	0.5 U	**120**	0.5 U	0.5 U	0.32 J	**10.5**	0.5 U	0.5 U	**25.9**	**66.8**	0.5 U	0.5 U	0.5 U
METHYLENE CHLORIDE	5	MDE	0.54 B	0.59 B	1 U	1 U	1 U	0.55 B	1 U	1 U	1 U	1 U	1 U	1 U	1 U	0.55 B
TETRACHLOROETHENE	5	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	0.5 U	0.5 U	0.82 J	0.5 U	0.5 U	0.5 U	0.67 J	0.5 U	0.5 U	0.5 U	0.57 J	0.5 U	0.5 U	0.5 U
TRICHLOROETHENE	0.44	EPA RSL	0.5 U	0.5 U	**2.52**	0.5 U	0.5 U	**1.36**	**23.7**	0.5 U	0.5 U	**68.5**	**93.7**	0.5 U	**0.63 J**	0.5 U
VINYL CHLORIDE	0.015	EPA RSL	0.5 U	0.5 U	**3.46**	0.5 U	0.5 U	0.5 U	**0.49 J**	0.5 U	0.5 U	**2.25**	**54.5**	0.5 U	0.5 U	0.5 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
D : Diluted value
U : The chemical was not detected.
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TABLE 2-3
GROUNDWATER GRAB
VOLATILE ORGANIC COMPUNDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL	S43TW032	S43TW032	S43TW032	S43TW033	S43TW033	S43TW034	S43TW034	S43TW034	S43TW035	S43TW035	S43TW036	S43TW036	S43TW037	S43TW037
SAMPLE ID		Reference	S43TW32-2226	S43TW32-2226-DUP	S43TW32-3236	S43TW33-2226	S43TW33-3236	S43TW34-2226	S43TW34-3236	S43TW34-3236-DUP	S43TW35-2226	S43TW35-3236	S43TW36-2024	S43TW36-3034	S43TW37-2024	S43TW37-2024-DUP
VOLATILES (µg/L)																
1,1,1-TRICHLOROETHANE	200	MDE	0.5 U	0.5 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
1,1-DICHLOROETHANE	2.4	EPA RSL	0.5 U	0.5 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
1,1-DICHLOROETHENE	7	MDE	0.5 U	0.5 U	0.5 U	5.8 J	0.5 U	0.5 U	0.25 J	0.28 J	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
1,2-DICHLOROETHANE	0.15	EPA RSL	0.5 U	0.5 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
2-BUTANONE	490	EPA RSL	5 U	5 U	5 U	50 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U
ACETONE	550	MDE	5 U	5 U	5 U	50 U	5 U	5 U	5 U	5 U	5 U	2.95 J	5 U	5 U	5 U	5 U
BROMOFORM	7.9	EPA RSL	0.5 U	0.5 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CARBON DISULFIDE	72	EPA RSL	0.5 U	0.5 U	0.5 U	5 U	0.75 J	0.5 U	0.5 U	0.5 U	0.5 U	0.42 J	0.5 U	0.83 J	0.28 J	0.5 U
CHLOROFORM	0.19	EPA RSL	0.5 U	0.5 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CHLOROMETHANE	19	EPA RSL	0.75 J	0.86 J	1.04 J	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	0.5 U	0.5 U	0.5 U	**164**	**5.8**	1.8	**23.9**	**25**	0.3 J	**3.35**	0.5 U	**4.38**	**4.18**	**4.2**
METHYLENE CHLORIDE	5	MDE	1 U	1 U	1 U	10 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
TETRACHLOROETHENE	5	MDE	0.5 U	0.5 U	0.5 U	5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	0.5 U	0.5 U	0.5 U	5 U	0.29 J	0.5 U	0.5 U	0.29 J	0.5 U	0.26 J	0.5 U	0.51 J	0.5 U	0.5 U
TRICHLOROETHENE	0.44	EPA RSL	0.5 U	0.5 U	0.5 U	**1270**	**5.81**	**8.98**	**80.6**	**78.7**	**1.48**	**0.52 J**	**0.51 J**	0.31 J	**24.6**	**24.3**
VINYL CHLORIDE	0.015	EPA RSL	0.5 U	0.5 U	0.5 U	**39.1**	**4.02**	0.5 U	**8.01 J**	**11 J**	0.5 U	**0.39 J**	0.5 U	0.5 U	**0.5 J**	**0.47 J**

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
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TABLE 2-3
GROUNDWATER GRAB
VOLATILE ORGANIC COMPUNDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL	S43TW037	S43TW038	S43TW038	S43TW039	S43TW040	S43TW040	S43TW041	S43TW041	S43TW041	S43TW042	S43TW042	S43TW043	S43TW044	S43TW045
SAMPLE ID		Reference	S43TW37-3135	S43TW38-1822	S43TW38-2832	S43TW39-0812	S43TW40-3236	S43TW40-2226	S43TW41-1822	S43TW41-1822-DUP	S43TW41-3337	S43TW42-1822	S43TW42-3337	S43TW43-1519	S43TW44-1317	S43TW45-2024
VOLATILES (µg/L)																
1,1,1-TRICHLOROETHANE	200	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	0.5 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.96 J	2.54	1.95 J
1,1-DICHLOROETHANE	2.4	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	0.5 U	0.74 J	0.72 J	0.50 U	0.50 U	0.50 U	**8.89**	**3.37**	1.55 J
1,1-DICHLOROETHENE	7	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	0.5 U	0.25 J	0.28 J	0.50 U	0.76 J	0.50 U	3.27	4.97	2.5 U
1,2-DICHLOROETHANE	0.15	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	0.5 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.5 U	0.5 U	2.5 U
2-BUTANONE	490	EPA RSL	5 U	5 U	5 U	3.38 B	5 U	5 U	5 U	5 U	3.49 J	5 U	5 U	5 U	5 U	25 U
ACETONE	550	MDE	5 U	5 U	5 U	26.4 B	8.63 J	5 U	5 U	5 U	23.8	14.3	19.8	5 U	5 U	25 U
BROMOFORM	7.9	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	0.5 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.5 U	0.5 U	2.5 U
CARBON DISULFIDE	72	EPA RSL	0.95 J	0.5 U	0.5 U	0.88 J	0.50 U	0.5 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.5 U	0.5 U	3 J
CHLOROFORM	0.19	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	0.5 U	0.50 U	0.50 U	0.50 U	0.13 J	0.50 U	0.15 J	**0.25 J**	2.5 U
CHLOROMETHANE	19	EPA RSL	0.5 U	0.5 U	0.46 J	0.92 J	0.50 U	0.5 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.5 U	0.5 U	2.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	**23.3**	**3.06**	2.45	**8.64**	0.50 U	1.57	**13.4**	**13.6**	0.50 U	**30.7**	**10.7**	0.5 U	0.87 J	2.5 U
METHYLENE CHLORIDE	5	MDE	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1.55 J	1.33 J	1.4 J	1 U	1 U	5 U
TETRACHLOROETHENE	5	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.50 U	0.5 U	0.50 U	0.50 U	0.50 U	0.50 U	0.50 U	0.5 U	0.5 U	2.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	0.8 J	0.5 U	0.46 J	0.5 U	0.50 U	0.5 U	0.50 U	0.50 U	0.50 U	0.64 J	0.42 J	0.5 U	0.5 U	2.5 U
TRICHLOROETHENE	0.44	EPA RSL	**29**	**0.94 J**	**4.63**	**17.9**	0.50 U	**102**	**22.2**	**22.5**	0.50 U	**137**	**8.75**	**1.11**	**8.14**	**731 J**
VINYL CHLORIDE	0.015	EPA RSL	**20.6 J**	**0.73 J**	0.5 U	**0.36 J**	0.50 U	0.5 U	**2.88**	**2.81**	0.50 U	**2.37**	**6.25**	0.5 U	0.5 U	2.5 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
D : Diluted value
U : The chemical was not detected.
-- : The chemical was not analyzed or no value was available.

Bold and shaded values indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-3
GROUNDWATER GRAB
VOLATILE ORGANIC COMPUNDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL	S43TW047	S43TW048	S43TW049	S43TW049	S43TW050	S43TW051	S43TW052	S43TW053	S43TW053	S43TW053	S43TW054	S43TW055	S43TW056	S43TW057
SAMPLE ID		Reference	S43TW47-1014	S43TW48-4044	S43TW49-1721	S43TW49-1721-DUP	S43TW50-1822	S43TW51-1721	S43TW52-1014	S43TW53-1317	S43TW53-2428	S43TW53-2428-DUP	S43TW54-1721	S43TW55-1822	S43TW56-1620	S43TW57-1923
VOLATILES (µg/L)																
1,1,1-TRICHLOROETHANE	200	MDE	0.88 J	0.5 U	2.01	1.93	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
1,1-DICHLOROETHANE	2.4	EPA RSL	**5.84**	0.5 U	**23.3**	**23**	0.5 U	0.5 U	0.5 U	0.55 J	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
1,1-DICHLOROETHENE	7	MDE	3.47	0.5 U	5.74	5.66	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
1,2-DICHLOROETHANE	0.15	EPA RSL	0.5 U	0.5 U	**0.62 J**	**0.62 J**	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
2-BUTANONE	490	EPA RSL	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	5 U	50 U
ACETONE	550	MDE	31.4 B	4.45 J	5 U	5 U	5 U	5 U	19.7 B	12.2 B	5 U	5 U	5 U	5 U	5 U	50 U
BROMOFORM	7.9	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
CARBON DISULFIDE	72	EPA RSL	0.69 J	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
CHLOROFORM	0.19	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
CHLOROMETHANE	19	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	1.32	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	**12.4**	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	**87.7**
METHYLENE CHLORIDE	5	MDE	0.63 J	1 U	1 U	1 U	1 U	1 U	0.53 J	1 U	1 U	1 U	1 U	1 U	1 U	5.1 B
TETRACHLOROETHENE	5	MDE	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U
TRICHLOROETHENE	0.44	EPA RSL	**1.58**	0.5 U	**1.53**	**1.43**	0.5 U	0.5 U	0.5 U	**1**	0.5 U	0.5 U	**1.79**	0.5 U	0.5 U	**766**
VINYL CHLORIDE	0.015	EPA RSL	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	5 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
D : Diluted value
U : The chemical was not detected.
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Bold and shaded values indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

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TABLE 2-3
GROUNDWATER GRAB
VOLATILE ORGANIC COMPUNDS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL	S43TW057	S43TWSA01	S43TWSA02	S43TWSA02	S43SP01	S43SP02
SAMPLE ID		Reference	S43TW57-2933	S43TWSA01-3034	S43TWSA02-1418	S43TWSA02-3034	S43SP01-01	S43SP02-01
VOLATILES (µg/L)								
1,1,1-TRICHLOROETHANE	200	MDE	2.5 U	0.5 U	0.5 U	5 U	2.36	0.5 U
1,1-DICHLOROETHANE	2.4	EPA RSL	2.5 U	0.5 U	0.5 U	5 U	1 U	0.49 J
1,1-DICHLOROETHENE	7	MDE	2.5 U	0.5 U	0.5 U	5 U	1 U	4.43
1,2-DICHLOROETHANE	0.15	EPA RSL	2.5 U	0.5 U	0.5 U	5 U	1 U	0.5 U
2-BUTANONE	490	EPA RSL	25 U	5 U	5 U	50 U	10 U	5 U
ACETONE	550	MDE	25 U	5 U	5 U	50 U	10 U	25.8 B
BROMOFORM	7.9	EPA RSL	2.5 U	0.5 U	0.5 U	5 U	1 U	0.5 U
CARBON DISULFIDE	72	EPA RSL	2.5 U	0.5 U	0.5 U	5 U	1 U	0.5 U
CHLOROFORM	0.19	EPA RSL	2.5 U	0.5 U	0.5 U	5 U	1 U	0.5 U
CHLOROMETHANE	19	EPA RSL	2.5 U	0.5 U	0.5 U	5 U	1 U	0.5 U
CIS-1,2-DICHLOROETHENE	2.8	EPA RSL	**205**	0.46 J	**4.8**	**62.6**	**3.62**	**195**
METHYLENE CHLORIDE	5	MDE	5 U	1 U	1 U	10 U	2 U	0.65 J
TETRACHLOROETHENE	5	MDE	2.5 U	0.5 U	0.5 U	5 U	0.24 J	0.5 U
TRANS-1,2-DICHLOROETHENE	8.6	EPA RSL	3.75 J	0.5 U	0.5 U	5 U	1 U	1.09
TRICHLOROETHENE	0.44	EPA RSL	**259**	**15.4**	**135**	**1730**	**354**	**28.7**
VINYL CHLORIDE	0.015	EPA RSL	**47.9**	0.5 U	0.5 U	**4.1 J**	1 U	**1.16**

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
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[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

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TABLE 2-4
MONITORING WELL
EXPLOSIVES ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION					S43MW001							S43MW002			
SAMPLE ID	PSL [1]	PSL REFERENCE	Unfiltered Background Values	Total Background Values	S43MW0010101	S43MW0010101-D	S43MW0010102	S43MW0010102-D	S43MW00103	S43MW0104	S43MW0104-DUP	S43MW0020101	S43MW0020102	S43MW00203	S43MW0204
SAMPLE DATE					20050725	20050725	20071111	20071111	20110617	20120722	20120722	20050727	20071111	20110617	20120722
EXPLOSIVES (µg/L)															
1,3,5-TRINITROBENZENE	46	EPA RSL	--	--	0.1 U	0.1 U			0.15 U	0.152 U	0.168 U	0.1 U			
1,3-DINITROBENZENE	0.15	EPA RSL	--	--	0.1 U	0.1 U			0.15 J	0.152 U	0.168 U	0.1 U			
2,4,6-TRINITROTOLUENE	2.2	EPA RSL	--	--	0.1 U	0.1 U			0.15 U	0.152 U	0.168 U	0.1 U			
2,4-DINITROTOLUENE	0.2	EPA RSL	--	--	0.1 U	0.1 U			0.15 U	0.152 U	0.168 U	0.1 U			
2,6-DINITROTOLUENE	1.5	EPA RSL	--	--	0.1 U	0.1 U			0.15 U	0.185 J	0.168 U	0.1 U			
2-AMINO-4,6-DINITROTOLUENE	3	EPA RSL	--	--	0.1 U	0.1 U			0.274 J	0.152 U	0.168 U	0.1 U			
2-NITROTOLUENE	0.27	EPA RSL	--	--	0.5 U	0.5 U			0.236 J	0.152 U	0.168 U	0.5 U			
3-NITROTOLUENE	0.13	EPA RSL	--	--	0.5 U	0.5 U			0.723 J	0.511 J	0.609 J	0.5 U			
4-AMINO-2,6-DINITROTOLUENE	3	EPA RSL	--	--	0.1 U	0.1 U			0.15 U	0.152 U	0.168 U	0.1 U			
4-NITROTOLUENE	3.7	EPA RSL	--	--	0.5 U	0.5 U			0.15 U	0.394 J	0.173 J	0.5 U			
HMX	78	EPA RSL	--	--	0.1 U	0.1 U			0.15 U	0.152 U	0.168 U	3.4			
NITROBENZENE	0.12	EPA RSL	--	--	0.1 U	0.1 U			0.15 U	0.152 U	0.168 U	0.1 U			
NITROCELLULOSE	4700000	EPA RSL	--	--	500 U	120 B						500 U			
NITROGLYCERIN	0.15	EPA RSL	--	--	0.65 U	0.65 U			25.2 J	0.381 U	0.421 U	0.65 U			
NITROGUANIDINE	160	EPA RSL	--	--	20 U	20 U			72 UJ	10 U	10 U	20 U			
RDX	0.61	EPA RSL	--	--	0.17	0.22			0.15 U	0.215 B	0.225 B	0.11			
TETRYL	6.1	EPA RSL	--	--	0.1 U	0.1 U			0.15 U	0.152 U	0.168 U	0.1 U			

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
U : The chemical was not detected.
Blank cells or -- : The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs

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TABLE 2-4
MONITORING WELL
EXPLOSIVES ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE		S43MW003		S43MW004			S43MW005		S43MW006		S43MW007	S43MW008	S43MW009D	S43MW009S	S43MW010
SAMPLE ID			S43MW0204-DUP	S43MW00301	S43MW0302	S43MW00401	S43MW00401-DUP	S43MW0402	S43MW00501	S43MW0502	S43MW00601	S43MW0602	S43MW0701	S43MW0801	S43MW09D01	S43MW09S01	S43MW1001
SAMPLE DATE			20120722	20110617	20120722	20110617	20110617	20120722	20110617	20120722	20110617	20120721	20120722	20120721	20120722	20120722	20120722
EXPLOSIVES (µg/L)																	
1,3,5-TRINITROBENZENE	46	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.157 U	0.168 U	0.168 U	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U
1,3-DINITROBENZENE	0.15	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.131 J	0.168 U	0.244 J	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U
2,4,6-TRINITROTOLUENE	2.2	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.157 U	0.168 U	0.168 U	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U
2,4-DINITROTOLUENE	0.2	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.157 U	0.168 U	0.168 U	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U
2,6-DINITROTOLUENE	1.5	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.157 U	0.168 U	0.168 U	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.504 J
2-AMINO-4,6-DINITROTOLUENE	3	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.157 U	0.168 U	0.168 U	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U
2-NITROTOLUENE	0.27	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.157 U	0.168 U	0.168 U	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U
3-NITROTOLUENE	0.13	EPA RSL		0.15 U	0.168 U	2.91	3.57 J	0.28 J	0.157 U	0.439 J	0.168 U	0.871	0.227 J	0.107 J	0.168 U	0.365 J	0.168 U
4-AMINO-2,6-DINITROTOLUENE	3	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.0929 J	0.168 U	0.128 J	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U
4-NITROTOLUENE	3.7	EPA RSL		0.15 U	0.291 J	0.15 U	0.16 U	0.148 J	0.157 U	0.168 U	0.195 J	0.142 J	0.0861 J	0.155 U	0.168 U	0.381	0.101 J
HMX	78	EPA RSL		0.15 U	0.168 U	0.125 J	0.125 J	0.155 U	0.157 U	0.168 U	0.904	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U
NITROBENZENE	0.12	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.157 U	0.168 U	0.168 U	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.234 B
NITROCELLULOSE	4700000	EPA RSL															
NITROGLYCERIN	0.15	EPA RSL		0.262 J	0.421 U	0.374 U	0.4 U	0.388 U	0.392 U	0.421 U	32.3 J	0.396 U	0.388 U	0.388 U	0.421 U	0.385 U	0.421 U
NITROGUANIDINE	160	EPA RSL		5 U	10 U	5 U	5 U	10 U	5 U	10 U	5 U	10 U	10 U	10 U	10 U	10 U	10 U
RDX	0.61	EPA RSL		0.15 U	0.168 U	0.212 J	0.415 J	0.546 B	0.0886 J	0.305 B	1.7 J	0.398 B	0.217 B	0.229 B	0.168 U	0.583 B	0.791 B
TETRYL	6.1	EPA RSL		0.15 U	0.168 U	0.15 U	0.16 U	0.155 U	0.157 U	0.168 U	0.168 U	0.158 U	0.155 U	0.155 U	0.168 U	0.154 U	0.168 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
U : The chemical was not detected.
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TABLE 2-4
MONITORING WELL
EXPLOSIVES ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	S43MW011	S43MW012	S43MW013	S43MW014	S43MW015	S43MW016	S43MW017D	S43MW017S	S43MW018	S43MW019D	S43MW019S		S43MW020	S43MW021	S43MW022
SAMPLE ID			S43MW1101	S43MW1201	S43MW1301	S43MW1401	S43MW1501	S43MW1601	S43MW17D01	S43MW17S01	S43MW1801	S43MW19D01	S43MW19S01	S43MW19S01-DUP	S43MW2001	S43MW2101	S43MW2201
SAMPLE DATE			20120721	20120722	20120722	20120722	20120722	20120722	20120721	20120721	20120721	20120721	20120721	20120721	20120719	20120721	20120719
EXPLOSIVES (µg/L)																	
1,3,5-TRINITROBENZENE	46	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.168 U	0.16 U
1,3-DINITROBENZENE	0.15	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.168 U	0.16 U
2,4,6-TRINITROTOLUENE	2.2	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.168 U	0.16 U
2,4-DINITROTOLUENE	0.2	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.168 U	0.16 U
2,6-DINITROTOLUENE	1.5	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.168 U	0.16 U
2-AMINO-4,6-DINITROTOLUENE	3	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.168 U	0.16 U
2-NITROTOLUENE	0.27	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.0967 B	0.157 U	0.0797 B	0.168 U	0.16 U
3-NITROTOLUENE	0.13	EPA RSL	0.418 J	0.507					0.168 U	0.419	0.168 U	0.366 J	0.222 J	0.157 U	0.151 U	0.168 U	0.16 U
4-AMINO-2,6-DINITROTOLUENE	3	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.168 U	0.16 U
4-NITROTOLUENE	3.7	EPA RSL	0.164 U	0.174 J					2.13 J	0.6 J	0.168 U	0.0904 J	0.303 J	0.0968 J	0.137 J	0.168 U	0.179 J
HMX	78	EPA RSL	0.164 U	0.168 U					0.135 J	0.236 J	0.123 J	0.16 U	0.13 J	0.125 J	0.151 U	0.168 U	0.965
NITROBENZENE	0.12	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.117 B	0.16 U
NITROCELLULOSE	4700000	EPA RSL															
NITROGLYCERIN	0.15	EPA RSL	0.318 B	0.421 U					0.421 U	0.377 U	0.421 U	0.4 U	0.392 U	0.392 U	0.377 U	0.421 U	0.4 U
NITROGUANIDINE	160	EPA RSL	10 U	10 U					10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U	10 U
RDX	0.61	EPA RSL	0.164 U	0.168 U					0.103 B	0.499 B	0.348 B	0.239 B	0.497 B	0.369 B	0.151 U	0.168 U	0.16 U
TETRYL	6.1	EPA RSL	0.164 U	0.168 U					0.168 U	0.151 U	0.168 U	0.16 U	0.157 U	0.157 U	0.151 U	0.168 U	0.16 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
U : The chemical was not detected.
Blank cells or -- : The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (November 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-4
MONITORING WELL
EXPLOSIVES ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	S43MW023	S43MW024	S43MW025	S43MW026
SAMPLE ID			S43MW2301	S43MW2401	S43MW2501	S43MW2601
SAMPLE DATE			20120720	20120720	20120720	20120718
EXPLOSIVES (µg/L)						
1,3,5-TRINITROBENZENE	46	EPA RSL	0.16 U	0.16 U	0.178 U	0.157 U
1,3-DINITROBENZENE	0.15	EPA RSL	0.16 U	0.212 J	0.186 J	0.157 U
2,4,6-TRINITROTOLUENE	2.2	EPA RSL	0.16 U	0.16 U	0.178 U	0.157 U
2,4-DINITROTOLUENE	0.2	EPA RSL	0.16 U	0.16 U	0.178 U	0.157 U
2,6-DINITROTOLUENE	1.5	EPA RSL	0.16 U	0.16 U	0.178 U	0.157 U
2-AMINO-4,6-DINITROTOLUENE	3	EPA RSL	0.16 U	0.118 B	0.178 U	0.157 U
2-NITROTOLUENE	0.27	EPA RSL	0.16 U	0.371 B	0.353 B	0.157 U
3-NITROTOLUENE	0.13	EPA RSL	0.16 U	0.642	0.301 J	0.157 U
4-AMINO-2,6-DINITROTOLUENE	3	EPA RSL	0.16 U	0.473	0.178 U	0.157 U
4-NITROTOLUENE	3.7	EPA RSL	0.16 U	0.115 J	0.114 J	0.157 U
HMX	78	EPA RSL	0.16 U	0.16 U	0.178 U	0.157 U
NITROBENZENE	0.12	EPA RSL	0.16 U	0.865 B	0.178 U	0.157 U
NITROCELLULOSE	4700000	EPA RSL				
NITROGLYCERIN	0.15	EPA RSL	0.4 U	0.307 B	0.295 B	0.392 U
NITROGUANIDINE	160	EPA RSL	10 U	10 U	10 U	10 U
RDX	0.61	EPA RSL	0.223 B	0.16 U	0.178 U	0.157 U
TETRYL	6.1	EPA RSL	0.16 U	0.198 J	0.178 U	0.157 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
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Blank cells or -- : The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (November 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-5
MONITORING WELL
METALS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	Unfiltered Background Values	Total Background Values	S43MW001							S43MW002			
SAMPLE ID					S43MW0010101	S43MW0010101-DUP	S43MW0010102	S43MW0010102-DUP	S43MW00103	S43MW0104	S43MW0104-D	S43MW0020101	S43MW0020102	S43MW00203	S43MW0204
SAMPLE DATE					20050725	20050725	20071111	20071111	20110617	20120722	20120722	20050727	20071111	20110617	20120722
TOTAL METALS (µg/L)															
ALUMINUM	1600	EPA RSL	9620	73400	134 B	153			1550	156	143	215			
ANTIMONY	0.6	EPA RSL	--	0	**2 L**	2 UL			2 U	2 U	2 U	**2.1 L**			
ARSENIC	0.045	EPA RSL	--	19.1	2 UL	2 UL			**2.65**	**1.18 J**	**1.15 J**	2 UL			
BARIUM	290	EPA RSL	139	688	203	197			30.8	27.1	27	**354**			
BERYLLIUM	1.6	EPA RSL	--	11	0.69	0.66			0.385 J	0.321 J	0.306 J	**4.6**			
CADMIUM	0.69	EPA RSL	2.8	9.8	0.3	0.26			0.558 J	0.5 U	0.383 J	0.61			
CALCIUM	NC	NC	--	--	3890	3760						3450			
CHROMIUM	0.031	EPA RSL	16.4	191	**0.71**	**0.59**			**11.6**	**0.927 J**	**0.841 J**	0.5 U			
COBALT	0.47	EPA RSL	15.6	641	**35.2**	**34.2**			**9.03**	**13.3**	**13.2**	**96.4**		**70.4**	**33.6**
COPPER	62	EPA RSL	22.4	166	9.3 B	5.9 B			14.3	1.2 J	1.33 J	8.2 B			
IRON	1100	EPA RSL	19900	252000	**4230**	**4030**			**2420 K**	**5360 K**	**5150 K**	37.3 B			
LEAD	15	MDE	--	51	2.8 K	2.2 K			4.57	0.785	1.08	0.9 U			
MAGNESIUM	NC	NC	--	--	2340	2270						4640			
MANGANESE	32	EPA RSL	824	2290	**306**	**296**			**101**	**106**	**106**	**270**			
MERCURY	0.063	EPA RSL	--	--	0.13 U	0.13 U				0.16 U	0.16 U	0.13 U			
NICKEL	30	EPA RSL	16.6	166	**40.6 J**	**39.5 J**			11.7	13.4	13.5	**78.3 J**			
POTASSIUM	NC	NC	--	--	1660	1680						1690			
SELENIUM	7.8	EPA RSL	--	--	3 U	3 U			0.786 J	1.25 U	1.25 U	3 U			
SODIUM	NC	NC	--	--	40100	39100						60500			
THALLIUM	0.016	EPA RSL	--	--	**3.7 B**	**2.7 B**			1 UL	1 U	1 U	2 U			
VANADIUM	3.7	MDE	20.9	281	0.49	0.72			**14.2**	2.29 J	2.23 J	0.4 U			
ZINC	470	EPA RSL	45.2	483	51.9 J	49.9 J			102	38.6	38.9	134 J			
FILTERED METALS (µg/L)															
ALUMINUM	1600	EPA RSL	9620	73400					195	51.6	49.4 J				
ANTIMONY	0.6	EPA RSL	--	0					2 U	2 U	2 U				
ARSENIC	0.045	EPA RSL	--	19.1					**2.13 J**	**1.23 J**	**0.807 J**				
BARIUM	290	EPA RSL	139	688					25.3	27.8	26.3				
BERYLLIUM	1.6	EPA RSL	--	11					0.5 U	0.305 J	0.276 J				
CADMIUM	0.69	EPA RSL	2.8	9.8					0.427 J	0.5 U	0.5 U				
CHROMIUM	0.031	EPA RSL	16.4	191					**6.58**	**0.581 J**	1 U				
COBALT	0.47	EPA RSL	15.6	641					**8.28**	**13.7**	**13.1**			**73.5**	**33.8**
COPPER	62	EPA RSL	22.4	166					8.94	2 U	1.04 J				
IRON	1100	EPA RSL	19900	252000					768 K	**5370 K**	**4820 K**				
LEAD	15	MDE	--	51					0.444 J	0.75 U	0.75 U				
MANGANESE	32	EPA RSL	824	2290					**90**	**109 L**	**103 L**				
MERCURY	0.063	EPA RSL	--	--						0.16 U	0.16 U				
NICKEL	30	EPA RSL	16.6	166					10.2	13.8	13.2				
SELENIUM	7.8	EPA RSL	--	--					0.916 J	1.25 U	1.25 U				
THALLIUM	NC	NC	--	--					1 UL	1 U	1 U				
VANADIUM	3.7	MDE	20.9	281					**9.48**	1.6 J	1.55 J				
ZINC	470	EPA RSL	45.2	483					60.6	38.8	38.2				

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
U :The chemical was not detected.
Blank cells or -- = The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for Chromium VI used for chromium. RSL for methylmercury used for mercury.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-5
MONITORING WELL
METALS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	Unfiltered Background Values	Total Background Values		S43MW0003		S43MW0004			S43MW0005		S43MW0006		S43MW007	S43MW008	S43MW009D
SAMPLE ID					S43MW0204-DUP	S43MW00301	S43MW0302	S43MW00401	S43MW00401-DUP	S43MW0402	S43MW00501	S43MW0502	S43MW00601	S43MW0602	S43MW0701	S43MW0801	S43MW09D01
SAMPLE DATE					20120722	20110617	20120722	20110617	20110617	20120722	20110617	20120722	20110617	20120721	20120722	20120721	20120722
TOTAL METALS (µg/L)																	
ALUMINUM	1600	EPA RSL	9620	73400		22.5 J	14.8 J	18.2 J	14.1 J	16.5 J	1090	129	**4050**	1190	16.2 J	19.3 J	290
ANTIMONY	0.6	EPA RSL	--	0		2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U
ARSENIC	0.045	EPA RSL	--	19.1		**16.9**	**12.2**	**1.72 J**	**1.7 J**	**1.31 J**	**1.58 J**	**2.11 J**	**2.76**	**6.47**	1.5 U	1.5 U	**5.71**
BARIUM	290	EPA RSL	139	688		132	91.8	96.6	90.9	69.5	106	88.4	138	167	109	56.3	167
BERYLLIUM	1.6	EPA RSL	--	11		0.5 U	0.5 U	0.5 U	0.5 U	0.355 J	0.386 J	0.911 J	0.536 J	0.311 J	1.11 J	0.323 J	0.5 U
CADMIUM	0.69	EPA RSL	2.8	9.8		0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
CALCIUM	NC	NC	--	--													
CHROMIUM	0.031	EPA RSL	16.4	191		1 U	1 U	1 U	1 U	1 U	**2.76**	1 U	**9.79**	**3.13**	1 U	**1.43 J**	**0.906 J**
COBALT	0.47	EPA RSL	15.6	641	**34.3**	**4.25**	**6.96**	**24.7**	**23.4**	**24.3**	**31.1**	**50.3**	**40.7**	**26.4**	**60**	**14.2**	2.5 U
COPPER	62	EPA RSL	22.4	166		2 U	2 U	2 U	2 U	2 U	2.92	1.09 J	9.93	2.87	1.02 J	1.04 J	1.01 J
IRON	1100	EPA RSL	19900	252000		**42600 K**	**42200 K**	**16600 K**	**16200 K**	**12400 K**	**10400**	**8510 K**	**35800 K**	**54900 K**	**11600**	**5000 K**	**29800 K**
LEAD	15	MDE	--	51		0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	1.41 L	0.75 U	3.73	0.69 J	0.75 U	0.75 U	0.75 U
MAGNESIUM	NC	NC	--	--													
MANGANESE	32	EPA RSL	824	2290		**602**	**763**	**409**	**383**	**352**	**345**	**298**	**611**	**713**	**423**	**206**	**185**
MERCURY	0.063	EPA RSL	--	--			0.16 U			0.16 U		0.16 U		0.16 U	0.16 U	0.16 U	0.16 U
NICKEL	30	EPA RSL	16.6	166		0.875 J	2.05 J	14.3	14	14.3	25.5	**45.5**	28.8	15.1	**46.5**	8.2	0.761 J
POTASSIUM	NC	NC	--	--													
SELENIUM	7.8	EPA RSL	--	--		1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U
SODIUM	NC	NC	--	--													
THALLIUM	0.016	EPA RSL	--	--		1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
VANADIUM	3.7	MDE	20.9	281		2.5 U	2.19 J	2.5 U	2.5 U	1.33 J	**4.61**	1.69 J	**15.7**	**6.68**	1.59 J	2.5 U	1.64 J
ZINC	470	EPA RSL	45.2	483		11.6	4.1 J	14.8	13.2	15.8	38.3	57.9	33.2	16.4	53.2	12	2.06 J
FILTERED METALS (µg/L)																	
ALUMINUM	1600	EPA RSL	9620	73400		16.2 J	25 U	25 U	25 U	25 U	26.5 J	15.6 J	17 J	21 J	18.5 J	25 U	46.5 J
ANTIMONY	0.6	EPA RSL	--	0		2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U
ARSENIC	0.045	EPA RSL	--	19.1		**15.2**	**12.3**	**1.67 J**	**1.53 J**	**1.65 J**	**1.22 J**	**1.65 J**	**2.09 J**	**3.61**	1.5 U	1.5 U	**5.76**
BARIUM	290	EPA RSL	139	688		129	94.8	91.9	94.1	68.5	110	86.6	126	163	112	52.2	163
BERYLLIUM	1.6	EPA RSL	--	11		0.5 U	0.5 U	0.5 U	0.5 U	0.344 J	0.5 U	0.838 J	0.5 U	0.5 U	1.15 J	0.297 J	0.5 U
CADMIUM	0.69	EPA RSL	2.8	9.8		0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.262 J	0.5 U	0.5 U
CHROMIUM	0.031	EPA RSL	16.4	191		1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	**1.25 J**	1 U
COBALT	0.47	EPA RSL	15.6	641	**33.7**	**4.21**	**7.1**	**23.9**	**24.5**	**23.5**	**33.5**	**48.9**	**35.4**	**25.9**	**61.2**	**12.8**	2.5 U
COPPER	62	EPA RSL	22.4	166		2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U	2 U
IRON	1100	EPA RSL	19900	252000		**38600**	**43800 K**	**15900 K**	**16400 K**	**12500**	**10900**	**8400 K**	**34800 K**	**44500 K**	**11800**	**5100 K**	**29000 K**
LEAD	15	MDE	--	51		0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U	1.5 U	1.5 U	1.5 U	0.75 U	0.75 U	1.5 U
MANGANESE	32	EPA RSL	824	2290		**572**	**795 L**	**394**	**396**	**335 L**	**383**	**294**	**616**	**753**	**430 L**	**192**	**180**
MERCURY	0.063	EPA RSL	--	--			0.16 U			0.16 U		0.16 U		0.16 U	0.16 U	0.16 U	0.16 U
NICKEL	30	EPA RSL	16.6	166		1.5 U	2.13 J	14.4	14.5	14.4	27.7	**44.3**	24.9	14.4	**47**	7.43	1.5 U
SELENIUM	7.8	EPA RSL	--	--		1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U
THALLIUM	NC	NC	--	--		1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U	1 U
VANADIUM	3.7	MDE	20.9	281		2.5 U	2.23 J	2.5 U	2.5 U	2.5 U	2.5 U	2.5 U	2.5 U	2.24 J	1.67 J	2.5 U	2.5 U
ZINC	470	EPA RSL	45.2	483		11.5	5.06	14.1	13.9	15.3	36	56.3	13.9	10.7	56.3	11.6	2.14 J

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
U :The chemical was not detected.
Blank cells or -- = The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for Chromium VI used for chromium. RSL for methylmercury used for mercury.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-5
MONITORING WELL
METALS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	Unfiltered Background Values	Total Background Values	S43MW009S	S43MW010	S43MW011	S43MW012	S43MW013	S43MW014	S43MW015	S43MW016	S43MW017D	S43MW017S	S43MW018	S43MW019D	S43MW019S	
SAMPLE ID					S43MW09S01	S43MW1001	S43MW1101	S43MW1201	S43MW1301	S43MW1401	S43MW1501	S43MW1601	S43MW17D01	S43MW17S01	S43MW1801	S43MW19D01	S43MW19S01	S43MW19S01-DUP
SAMPLE DATE					20120722	20120722	20120721	20120722	20120722	20120722	20120722	20120722	20120721	20120721	20120721	20120721	20120721	20120721
TOTAL METALS (µg/L)																		
ALUMINUM	1600	EPA RSL	9620	73400	358	121	25 U	321					25 B	30.5 J	92.7	28 B	124 K	122 K
ANTIMONY	0.6	EPA RSL	--	0	2 U	2 U	2 U	2 U					2 U	2 U	2 U	2 U	2 U	2 U
ARSENIC	0.045	EPA RSL	--	19.1	1.5 U	**5.86**	**1.56 J**	**5.11**					**2.21 J**	**2.01 J**	1.5 U	**1.2 J**	1.5 U	1.5 U
BARIUM	290	EPA RSL	139	688	**352**	116	51.9	102					109	80.9	143	123	248	246
BERYLLIUM	1.6	EPA RSL	--	11	**1.91**	0.5 U	0.5 U	0.5 U					0.5 U	0.5 U	0.701 J	0.5 U	**1.95**	**1.94**
CADMIUM	0.69	EPA RSL	2.8	9.8	0.5 U	0.5 U	0.5 U	0.5 U					0.5 U	0.5 U	**0.909 J**	0.5 U	0.652 J	0.614 J
CALCIUM	NC	NC	--	--														
CHROMIUM	0.031	EPA RSL	16.4	191	1 U	1 U	1 U	**0.842 J**					1 U	1 U	1 U	1 U	1 U	1 U
COBALT	0.47	EPA RSL	15.6	641	**54.8**	2.5 U	**2.22 J**	**8.32**	**130**	**36**	**5.1**	**108**	**6.2**	**15.4**	**43.1**	2.5 U	**124**	**124**
COPPER	62	EPA RSL	22.4	166	1.38 J	2 U	2 U	2 U					2 U	2 U	1.1 J	2 U	1.4 J	1.29 J
IRON	1100	EPA RSL	19900	252000	**7640 K**	**33600 K**	**20900**	**33000 K**					**32900**	**34900 K**	192 K	**27000**	**6210**	**5970**
LEAD	15	MDE	--	51	0.75 U	0.75 U	0.75 U	0.75 U					0.75 U	0.75 UL	0.75 UL	0.75 U	0.75 U	0.75 U
MAGNESIUM	NC	NC	--	--														
MANGANESE	32	EPA RSL	824	2290	**405**	**196**	**497**	**456**					**498**	**561**	**326**	**522**	**1140**	**1110**
MERCURY	0.063	EPA RSL	--	--	0.16 U	0.16 U	0.16 UL	0.16 U					0.16 UL	0.16 UL	0.16 UL	0.16 UL	0.16 UL	0.16 UL
NICKEL	30	EPA RSL	16.6	166	**40.2**	1.5 U	1.5 U	4.65					1.35 J	4.47	**52.9**	1.5 U	**46.6**	**45.7**
POTASSIUM	NC	NC	--	--														
SELENIUM	7.8	EPA RSL	--	--	1.25 U	1.25 U	1.25 U	1.25 U					1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U
SODIUM	NC	NC	--	--														
THALLIUM	0.016	EPA RSL	--	--	1 U	1 U	1 U	1 U					1 U	1 U	1 U	1 U	**0.799 J**	**0.803 J**
VANADIUM	3.7	MDE	20.9	281	1.38 J	2.5 U	1.47 J	2.19 J					1.49 J	1.83 J	1.27 J	1.72 J	3.12	3.03 J
ZINC	470	EPA RSL	45.2	483	86.9	1.65 J	1.34 J	6.34					4.06 J	13.4	71.7	1.32 J	92.4	91.1
FILTERED METALS (µg/L)																		
ALUMINUM	1600	EPA RSL	9620	73400	357	31.4 J	25 U	63.7					25 B	25 U	79.6	25 B	101 B	108 B
ANTIMONY	0.6	EPA RSL	--	0	2 U	2 U	2 U	2 U					2 U	2 U	2 U	2 U	2 U	2 U
ARSENIC	0.045	EPA RSL	--	19.1	1.5 U	**4.91 K**	**1.49 J**	**5.58**					**2.3 J**	**2.11 J**	1.5 U	1.5 U	1.5 U	1.5 U
BARIUM	290	EPA RSL	139	688	**347**	104	54.5	100					111	82.9	144	117	242	232
BERYLLIUM	1.6	EPA RSL	--	11	**1.9**	0.5 U	0.5 U	0.5 U					0.5 U	0.5 U	0.734 J	0.5 U	**1.86**	**1.85**
CADMIUM	0.69	EPA RSL	2.8	9.8	0.5 U	0.5 U	0.5 U	0.5 U					0.5 U	0.5 U	**0.942 J**	0.5 U	0.596 J	**0.765 J**
CHROMIUM	0.031	EPA RSL	16.4	191	1 U	1 U	1 U	1 U					1 U	1 U	1 U	1 U	1 U	1 U
COBALT	0.47	EPA RSL	15.6	641	**54.2**	2.5 U	**2.39 J**	**8.11**	**127**	**34.8**	**5.1**	**108**	**6.24**	**15.7**	**44.2**	2.5 U	**121**	**117**
COPPER	62	EPA RSL	22.4	166	2.17 J	2 U	2 U	2 U					2 U	1.2 J	1.52 J	2 U	1.28 J	1.13 J
IRON	1100	EPA RSL	19900	252000	**7470 K**	**30600 K**	**21400**	**33200 K**					**32100**	**36100 K**	192 K	**25700**	**5930**	**5800**
LEAD	15	MDE	--	51	0.75 U	0.75 U	0.75 U	0.75 U					0.75 U	0.75 UL	0.75 UL	0.75 U	0.75 U	0.75 U
MANGANESE	32	EPA RSL	824	2290	**406**	**187 L**	**503**	**445 L**					**500**	**572**	**326**	**502**	**1090**	**1060**
MERCURY	0.063	EPA RSL	--	--	0.16 U	0.16 U	0.16 UL	0.16 U					0.16 UL	0.16 UL	0.16 UL	0.16 UL	0.16 UL	0.16 UL
NICKEL	30	EPA RSL	16.6	166	**39.7**	1.5 U	1.5 U	4.41					1.55 J	4.63	**54.4**	1.5 U	**45.1**	**43.3**
SELENIUM	7.8	EPA RSL	--	--	1.25 U	1.25 U	1.25 U	1.25 U					1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U
THALLIUM	NC	NC	--	--	1 U	1 U	1 U	1 U					1 U	1 U	1 U	1 U	1 U	0.751 J
VANADIUM	3.7	MDE	20.9	281	1.34 J	2.5 U	1.47 J	1.41 J					1.46 J	1.73 J	2.5 U	1.54 J	2.89 J	2.82 J
ZINC	470	EPA RSL	45.2	483	88.2	2.08 J	2.5 U	6.61					5.51	16.2	74.4	1.41 J	89.1	86.2

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
U :The chemical was not detected.
Blank cells or -- = The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs
[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for Chromium VI used for chromium. RSL for methylmercury used for mercury.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-5
MONITORING WELL
METALS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	Unfiltered Background Values	Total Background Values	S43MW020	S43MW021	S43MW022	S43MW023	S43MW024	S43MW025	S43MW026
SAMPLE ID					S43MW2001	S43MW2101	S43MW2201	S43MW2301	S43MW2401	S43MW2501	S43MW2601
SAMPLE DATE					20120719	20120721	20120719	20120720	20120720	20120720	20120718
TOTAL METALS (µg/L)											
ALUMINUM	1600	EPA RSL	9620	73400	122	70.5	57.9	47.7 J	127	39.3 J	49.1 J
ANTIMONY	0.6	EPA RSL	--	0	2 U	2 U	2 U	2 U	2 U	2 U	2 U
ARSENIC	0.045	EPA RSL	--	19.1	1.5 U	1.5 U	1.5 U	1.5 U	1.5 U	1.5 U	1.5 U
BARIUM	290	EPA RSL	139	688	84.5	86.8	170	47.9	48.9	92.9	45.1
BERYLLIUM	1.6	EPA RSL	--	11	0.709 J	0.422 J	**1.61**	0.479 J	0.5 U	0.5 U	0.42 J
CADMIUM	0.69	EPA RSL	2.8	9.8	0.368 J	0.5 U	0.29 J	0.5 U	0.5 U	0.5 U	0.5 U
CALCIUM	NC	NC	--	--							
CHROMIUM	0.031	EPA RSL	16.4	191	1 U	1 U	1 U	1 U	**4.45**	1 U	**1.53 J**
COBALT	0.47	EPA RSL	15.6	641	**19.9**	**33**	**73.2**	**4.31**	**1.95 J**	**2.35 J**	**4.85**
COPPER	62	EPA RSL	22.4	166	2 U	2 U	1.35 J	2 U	8.18	1.37 J	2 U
IRON	1100	EPA RSL	19900	252000	**1180**	**5990 K**	827	**1660**	115	**1510**	209
LEAD	15	MDE	--	51	0.75 U	0.75 UL	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U
MAGNESIUM	NC	NC	--	--							
MANGANESE	32	EPA RSL	824	2290	**234**	**888**	**511**	**160**	**79.5**	**184**	**32.7**
MERCURY	0.063	EPA RSL	--	--	0.16 U	0.16 UL	0.16 U	0.16 U	0.16 U	0.16 U	0.16 U
NICKEL	30	EPA RSL	16.6	166	22.5	15.3	**42.5**	9.21	1.6 J	3.09	7.88
POTASSIUM	NC	NC	--	--							
SELENIUM	7.8	EPA RSL	--	--	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U	1.25 U
SODIUM	NC	NC	--	--							
THALLIUM	0.016	EPA RSL	--	--	1 U	1 U	1 U	1 U	1 U	1 U	1 U
VANADIUM	3.7	MDE	20.9	281	2.5 U	2.95 J	1.56 J	2.5 U	2.5 U	2.5 U	2.5 U
ZINC	470	EPA RSL	45.2	483	35.6	22.2	57	12.1	7.21	4.86 J	13.2
FILTERED METALS (µg/L)											
ALUMINUM	1600	EPA RSL	9620	73400	49.9 B	17.3 J	73.2 B	30.8 B	67.7 B	41.7 B	54.3 B
ANTIMONY	0.6	EPA RSL	--	0	2 U	2 U	2 U	2 U	2 U	2 U	2 U
ARSENIC	0.045	EPA RSL	--	19.1	1.5 U	1.5 U	1.5 U	1.5 U	**1.14 J**	1.5 U	1.5 U
BARIUM	290	EPA RSL	139	688	78.7	88.4	160	42.8	48.7	85.7	44.5
BERYLLIUM	1.6	EPA RSL	--	11	0.629 J	0.414 J	1.51	0.426 J	0.5 U	0.5 U	0.409 J
CADMIUM	0.69	EPA RSL	2.8	9.8	0.348 J	0.5 U	0.257 J	0.5 U	0.5 U	0.5 U	0.5 U
CHROMIUM	0.031	EPA RSL	16.4	191	1 U	1 U	1 U	1 U	**4.5 K**	1 U	**1.4 J**
COBALT	0.47	EPA RSL	15.6	641	**19.7**	**33.6**	**71.8**	**3.85**	**2 J**	**2.31 J**	**5.08**
COPPER	62	EPA RSL	22.4	166	2 U	2 U	1.49 J	2 U	8.61	1.42 J	1.17 J
IRON	1100	EPA RSL	19900	252000	1020	**6050 K**	821	**1410**	66	**1580**	150
LEAD	15	MDE	--	51	0.75 U	0.75 UL	0.75 U	0.75 U	0.75 U	0.75 U	0.75 U
MANGANESE	32	EPA RSL	824	2290	**223**	**907**	**499**	**142**	**83.3**	**179**	**33.4**
MERCURY	0.063	EPA RSL	--	--	0.16 U	0.16 U	0.16 U	0.16 U	0.16 U	0.16 U	0.16 U
NICKEL	30	EPA RSL	16.6	166	20.6	15.6	**39.5**	8.32	2.03 J	2.82	7.76
SELENIUM	7.8	EPA RSL	--	--	1.25 U	1.25 U	1.25 U	1.25 U	0.855 J	1.25 U	1.25 U
THALLIUM	NC	NC	--	--	1 U	1 U	1 U	1 U	1 U	1 U	1 U
VANADIUM	3.7	MDE	20.9	281	2.5 U	2.52 J	1.35 J	2.5 U	2.5 U	2.5 U	2.5 U
ZINC	470	EPA RSL	45.2	483	34.8	22.7	59.8	12.5	11.4	5.91	15.7

Notes:

PSL : Project Screening Level
DUP : Duplicate sample
µg/L : micrograms per liter
J : Estimated value
B : Compound detected in blank sample
K : Biased High
L : Biased Low
U :The chemical was not detected.
Blank cells or -- = The chemical was not analyzed or no value was available.
Bold and Shaded cells indicate an exceedance of PSLs

[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for Chromium VI used for chromium. RSL for methylmercury used for mercury.

MDE: Maryland Department of Environment (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-6
INDOOR AIR AND SOIL GAS ANALYTICAL DATA
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

LOCATION	PSL [1]	PSL REFERENCE	S43IND01		S43IND02	S43ODA01	S43ODA02	S43SG01	S43SG03	S43SG04
SAMPLE ID			S43IND01	S43IND01-DUP	S43IND02	S43ODA01	S43ODA02	S43SG0106	S43SG0305	S43SG0406
SAMPLE DATE			20120420	20120420	20120420	20110611	20120420	20110611	20110611	20110611
Volatle Organic Compounds (µg/m3)										
1,1,1-TRICHLOROETHANE	520	EPA RSL	0.072 J	0.035 J	0.04 J	0.071 J	0.036 J	45 U	0.24 J	1,000 U
1,1-DICHLOROETHANE	1.5	EPA RSL	0.014 U	0.014 U	0.015 U	0.029 U	0.015 U	**140**	0.069 J	760 U
1,1-DICHLOROETHENE	21	EPA RSL	0.014 U	0.014 U	0.015 U	0.029 U	0.014 U	33 U	0.19	740 U
1,2-DICHLOROETHANE	0.094	EPA RSL	**0.098 B**	0.08 B	0.088 B	0.046 J	0.074 B	33 U	0.07 J	760 U
1,4-DIOXANE	0.32	EPA RSL	0.52 U	0.52 U	0.55 U	0.27 U	0.52 U	67 U	**0.68 J**	1,500 U
2-BUTANONE	520	EPA RSL	1 J	0.91 J	1 J	6.1	1.2 J	160 J	100	**1,600 J**
2-HEXANONE	3.1	EPA RSL	0.59 U	0.59 U	0.62 U	0.86 J	0.59 U	76 U	**12**	1,700 U
4-METHYL-2-PENTANONE	310	EPA RSL	0.59 U	0.59 U	0.62 U	0.3 U	0.59 U	76 U	0.6 U	**1,600 J**
ACETONE	3,200	EPA RSL	6.5 J	5 J	5.7 J	17	12 J	3,100	190	**690,000**
BENZENE	0.31	EPA RSL	**0.45**	0.28 J	0.27 J	**0.31**	0.3	26 U	**1.1**	600 U
CARBON DISULFIDE	73	EPA RSL	0.44 U	0.44 U	0.48 U	0.55 J	0.45 U	26 U	2.8 J	580 U
CARBON TETRACHLORIDE	0.41	EPA RSL	0.9 U	0.9 U	0.96 U	**0.66 J**	0.91 U	52 U	0.92 U	1,200 U
CHLOROETHANE	1,000	EPA RSL	0.38 U	0.38 U	0.4 U	0.31 U	0.38 U	180 J	0.64 J	1,100 U
CHLOROMETHANE	9.4	EPA RSL	0.91	0.68 J	1.1 J	1.8	1.2	**100 J**	2.3	870 U
CIS-1,2-DICHLOROETHENE	6.3	EPA RSL	0.015 B	0.014 U	0.015 U	0.029 U	0.017 B	**140**	**59**	**29,000**
DICHLORODIFLUOROMETHANE	10	EPA RSL	2.8	2.7 J	2.4 J	2.6	2.5	41 U	2.7	920 U
ETHYLBENZENE	0.97	EPA RSL	0.13 J	0.11 J	0.12 J	0.068 J	0.064 J	36 U	0.53	810 U
M+P-XYLENES	10	EPA RSL				0.11 J		**100**	1.1	810 U
METHYL TERT-BUTYL ETHER	9.4	EPA RSL	0.1 U	0.1 U	0.11 U	0.026 J	0.1 U	30 U	0.083 J	670 U
METHYLENE CHLORIDE	97	EPA RSL	0.5 U	0.5 U	0.53 U	0.68 J	0.5 U	29 U	5	650 U
O-XYLENE	10	EPA RSL				0.055 J		36 U	0.68	810 U
TETRACHLOROETHENE	9.4	EPA RSL	0.17 J	0.074 J	0.076 J	0.1 J	0.075 J	56 U	0.19 J	1,300 U
TOLUENE	520	EPA RSL	1.3	0.63 J	0.66 J	0.52	0.61	**790**	2	700 U
TRANS-1,2-DICHLOROETHENE	6.3	EPA RSL	0.014 U	0.014 U	0.015 U	0.029 U	0.014 U	33 U	0.44 J	740 U
TRICHLOROETHENE	0.43	EPA RSL	0.16 J	0.019 U	0.023 B	0.029 U	0.03 B	44 U	0.33	1,000 U
TRICHLOROFLUOROMETHANE	73	EPA RSL	1.1	1.2 J	1.3 J	1.6	1.2	46 U	1.6 J	1,000 U
VINYL CHLORIDE	0.16	EPA RSL	0.0091 U	0.0091 U	0.011 J	0.015 U	0.0092 U	**30,000**	**100**	1,100 U

Notes:
PSL : Project Screening Level
DUP : Duplicate sample
µg/m^3 : micrograms per cubic meter
J : Estimated value
U : The chemical was not detected.
B : Compound detected in blank sample
Blank cells = The chemical was not analyzed.
Bold and shaded values indicate an exceedance of PSLs
[1] Selected PSL is the lowest (most conservative) of the applicable screening criteria

EPA RSL: U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water.
RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

TABLE 2-7
REFERENCE LIMITS AND EVALUATION
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

PALs for all matrices:

• Carcinogenic Risk PAL = Incremental Lifetime Cancer Risk > 1×10^{-5}

• Non-carcinogenic Risk (for chemicals affecting the same target organ) PAL = Hazard index > 1

Matrix: Soils

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)		Selected PSL [3, 7]	PSL Reference [4]	PQL Goal [5]	Laboratory-Specific QLs		
		EPA RSL Residential Soil [1]	MDE Standard Residential Soil [2]				LOQ [6, 7]	LOD [6, 7]	MDL [6]
VOCs (mg/kg)									
Bromoform	75-25-2	62	81	62	EPA RSL	20	0.005	0.0025	0.00125
Chloroform	667-66-3	0.29	78	0.29	EPA RSL	0.1	0.005	0.0025	0.00125
Dichloroethane (DCA), 1,1-	75-34-3	3.3	1,600	3.3	EPA RSL	1	0.005	0.0025	0.00125
Dichloroethane (DCA), 1,2-	107-06-2	0.43	7	0.43	EPA RSL	0.1	0.005	0.0025	0.00125
Dichloroethene (DCE), 1,1-	75-35-4	24	390	24	EPA RSL	8	0.005	0.0025	0.00125
Dichloroethene (DCE), cis-1,2-	156-59-2	16	78	16	EPA RSL	5	0.005	0.0025	0.00125
Dichloroethene (DCE), trans-1,2-	156-60-5	15	160	15	EPA RSL	5	0.005	0.0025	0.00125
Methylene Chloride	75-09-2	56	85	56	EPA RSL	19	0.01	0.005	0.0025
Trichloroethene (TCE)	79-01-6	0.91	1.6	0.91	EPA RSL	0.3	0.005	0.0025	0.00125
Vinyl Chloride (VC)	75-01-4	0.06	0.09	0.06	EPA RSL	0.02	0.005	0.0025	0.00125
Inorganics (mg/kg)									
Arsenic [8]	7440-38-2	0.39	0.43	0.39	EPA RSL	0.1	0.5	0.3	0.15
Cobalt	7440-48-4	2.3	*none*	2.3	EPA RSL	0.8	0.75	0.5	0.25
Iron	7439-89-6	5,500	5,500	5,500	EPA RSL	1,833	20	12	6
Manganese	7439-96-5	180	160	160	MDE	53	3	1.2	0.6

Notes:

This table is an updated/revised version of Worksheet #15 from the final UFP SAP (Tetra Tech, 2011)

mg/kg : milligrams per kilogram CAS : Chemical Abstracts Service [number] LOQ : Limit of Quantitation LOD: Limit of Detection MDL: Method Detection Limit

Note: In most cases where the LOQ exceeds the PSL, the DL is less than PSL. Therefore data usability is not expected to be affected. The target analytes will be detectable if not accurately quantifiable. By including the analytes on the target compound list and striving to quantify as low a concentration as possible with conventional analytical methods, the project team ensures that gross contamination will not be overlooked even though the PSL is less than the LOQ.

[1] U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for soil under residential scenario. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for chromium VI used as surrogate for [total] chromium. RSL for methylmercury used as surrogate for mercury.

[2] Maryland Department of Environment (MDE) (2008) Cleanup Standards for residential soil. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

[3] Selected PSL is the lowest (most conservative) of the evaluated PSLs

[4] PSL References consist of EPA RSLs or MDE Cleanup Standards (see Notes 1 and 2 above)

[5] Project Quantitation Limit (PQL) Goal is set at 1/3 the PSL.

[6] The Laboratory QLs and MDLs from the subcontracted laboratory(ies) are presented and are current as of June 2010. The QLs and MDLs are subject to change. The actual limits will be evaluated during the data usability assessment to ensure that the actual QLs and MDLs are satisfactory to support the data evaluations.

[7] All PSLs are above QLs, except as noted: Shading indicates the Laboratory-Specific LOQ(s) is(are) greater than the Selected PSL. For purposes of selecting COPCs in the baseline human health risk assessment, ½ the LOQ will be used.

[8] Laboratory will perfom a 4x concentration on these metals

TABLE 2-7
REFERENCE LIMITS AND EVALUATION
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

Matrix Soil: (Secondary/Candidate Criteria)
Analytical Group: Inorganics

Chemical / Analyte	CAS Number	Carcinogenic (c) or Non-Carcinogenic (n)	NSF Indian Head (NSF-IH)	
			NSF-IH Background Surface Soil (mg/kg)	NSF-IH Background Subsurface Soil (mg/kg)
Inorganics				
Arsenic	7440-38-2	c	14.9	28.7
Cobalt	7440-48-4	n	22.3	14.7
Iron	7439-89-6	n	38,500	35,200
Manganese	7439-96-5	n	1,390	155

TABLE 2-7
REFERENCE LIMITS AND EVALUATION
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

Matrix: Groundwater

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)				Selected PSL [5, 9]	PSL Reference [6]	Project Quantitation Limit Goal [7]	Laboratory-Specific QLs		
		EPA RSL	MDE	Federal	Residential RSL-Based				LOQ [8, 9]	LOD [8, 9]	MDL [8]
		Tapwater [1]	Standard Groundwater [2]	MCL [3]	V.I. Screening Value [4]						
VOCs (µg/L)											
Bromoform	75-25-2	7.9	80	none	100	7.9	EPA RSL	2.6	1	0.5	0.25
Chloroform	667-66-3	0.19	80	none	0.7	0.19	EPA RSL	0.06	1	0.5	0.13
Dichloroethane (DCA), 1,1-	75-34-3	2.4	90	*none*	6.5	2.4	EPA RSL	0.8	1	0.5	0.25
Dichloroethane (DCA), 1,2-	107-06-2	0.15	5	5	2	0.15	EPA RSL	0.05	1	0.5	0.13
Dichloroethene (DCE), 1,1-	75-35-4	26	7	7	191	7	MDE	2	1	0.5	0.25
Dichloroethene (DCE), cis-1,2-	156-59-2	2.8	70	70	371	2.8	EPA RSL	2	1	0.5	0.25
Dichloroethene (DCE), trans-1,2-	156-60-5	8.6	100	100	371	8.6	EPA RSL	4	1	0.5	0.25
Methylene Chloride	75-09-2	9.9	5	5	4	5	MDE	1.6	2	1	0.5
Trichloroethene (TCE)	79-01-6	0.44	5	5	3	0.44	EPA RSL	0.7	1	0.5	0.25
Vinyl Chloride (VC)	75-01-4	0.015	2	2	0.15	0.015	EPA RSL	0.005	1	0.5	0.25
Inorganics (µg/L)											
Arsenic[10]	7440-38-2	0.045	10	10	*none*	0.045	EPA RSL	0.02	2.5	1.5	0.75
Cobalt[10]	7440-48-4	0.47	*none*	*none*	*none*	0.47	EPA RSL	0.4	3.125	2.5	1.25
Iron	7439-89-6	1,100	2,600	*none*	*none*	1,100	EPA RSL	867	100	60	30
Manganese	7439-96-5	32	73	*none*	*none*	32	MDE	24	15	6	3

Notes:

This table is an updated/revised version of Worksheet #15 from the final UFP SAP (Tetra Tech, 2011)

µg/L : micrograms per liter CAS : Chemical Abstracts Service [number] LOQ : Limit of Quantitation LOD: Limit of Detection MDL : Method Detection Limit

Note: In most cases where the LOQ exceeds the PSL, the DL is less than PSL. Therefore data usability is not expected to be affected. The target analytes will be detectable if not accurately quantifiable. By including the analytes on the target compound list and striving to quantify as low a concentration as possible with conventional analytical methods, the project team ensures that gross contamination will not be overlooked even though the PSL is less than the LOQ.

For groundwater, inorganics will also be evaluated with base values to determine what concentrations are within background concentrations.

[1] U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL

[2] Maryland Department of Environment (MDE) (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.

[3] Federal Maximum Contaminant Level (MCL) per National Primary Drinking Water Regulations.

[4] Vapor Intrusion (V.I.) screening values for groundwater calculated using the EPA (December 2009) RSLs for indoor air and methodology detailed in Appendix D of the EPA (2002) Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils.

[5] Selected PSL is the lowest (most conservative) of the evaluated PSLs.

[6] PSL References indicate the source of the selected PSL from the evaluated PSL list (see Notes 1 through 4 above), or if the PSL is selected as a Performance Indicator Level (PIL) determined for chemicals and analytes in the "OTHER" category. The OTHER chemicals and analytes provide information about the aquifer conditions and have no inherent risk (except for Nitrate, which has an EPA RSL of 5,800 µg/L and MCL of 10,000 µg/L; however, the

[7] Project Quantitation Limit (PQL) Goal is set at 1/3 the PSL.

[8] The Laboratory LOQs and MDLs from the subcontracted laboratory(ies) are presented and are current as of June 2010. The QLs and MDLs are subject to change. The actual limits will be evaluated during the data usability assessment to ensure that the actual LOQs and MDLs are satisfactory to support the data evaluations.

[9] All PSLs are above QLs, except as noted: Shading indicates the Laboratory-Specific LOQ(s) is(are) greater than the Selected PSL. For purposes of selecting COPCs in the baseline human health risk assessment, ½ the LOQ will be used.

[10] Laboratory will perfom a 4x concentration on these metals.

TABLE 2-7
REFERENCE LIMITS AND EVALUATION
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

Matrix: Soil Gas

Chemical / Analyte	CAS Number	Selected PSL [1, 4]	PQL Goal [2]	Laboratory-Specific QLs		
		EPA RSL Residential Indoor Air [1]		LOQ [3, 4]	LOD [3, 4]	MDL [3]
VOCs (µg/m^3)						
Bromoform	75-25-2	2.2	0.7	1	0.165	0.165
Chloroform	667-66-3	0.11	0.04	0.49	0.068	0.068
Dichloroethane (DCA), 1,1-	75-34-3	1.5	0.5	0.081	0.004	0.004
Dichloroethane (DCA), 1,2-	107-06-2	0.094	0.03	0.081	0.024	0.024
Dichloroethene (DCE), 1,1-	75-35-4	21	7	0.04	0.008	0.008
Dichloroethene (DCE), cis-1,2-	156-59-2	6.3	2.1	0.079	0.008	0.008
Dichloroethene (DCE), trans-1,2-	156-60-5	6.3	2.1	0.4	0.004	0.004
Methylene Chloride	75-09-2	96	32	0.69	0.076	0.076
Trichloroethene (TCE)	79-01-6	0.43	0.4	0.11	0.005	0.005
Vinyl Chloride (VC)	75-01-4	0.16	0.05	0.026	0.005	0.005

Notes:

This table is an updated/revised version of Worksheet #15 from the final UFP SAP (Tetra Tech, 2011)

µg/m^3 : micrograms per cubic meter

PSL : Project Screening Limit LOQ : Limit of Quantitation LOD : Limit of Detection MDL : Method Detection Limit PQL : Project Quantitation Limit

EPA RSL : U.S. Environmental Protection Agency Regional Screening Level NC: No Criteria

Note: In most cases where the LOQ exceeds the PSL, the DL is less than PSL. Therefore data usability is not expected to be affected. The target analytes will be detectable if not accurately quantifiable. By including the analytes on the target compound list and striving to quantify as low a concentration as possible with conventional analytical methods, the project team ensures that gross contamination will not be overlooked even though the PSL is less than the LOQ.

[1] Selected PSL is the U.S. Environmental Protection Agency (EPA) (Novmber 2012) Regional Screening Level (RSL) for indoor air. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for trans-1,2-DCE used as surrogate for cis-1,2-DCE.

[2] Project Quantitation Limit (PQL) Goal is set at 1/3 the PSL.

[3] The Laboratory QLs and MDLs from the subcontracted laboratory(ies) are presented and are current as of June 2010. The QLs and MDLs are subject to change. The actual limits wi be evaluated during the data usability assessment to ensure that the actual QLs and MDLs are satisfactory to support the data evaluations.

[4] All PSLs are above QLs.

TABLE 2-7
REFERENCE LIMITS AND EVALUATION
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

Matrix: Surface Water

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)		Selected PSL [3, 4]	PSL Reference [5]	Project Quantitation Limit Goal [6]	Laboratory-Specific QLs		
		EPA BTAG Freshwater [1]	MDE Standard Groundwater[2]				LOQ [7, 4]	LOD [7, 4]	MDL [7]
VOCs (µg/L)									
Bromoform	75-25-2	320	*none*	320	EPA BTAG	105	1	0.5	0.25
Chloroform	667-66-3	1.8	*none*	1.8	EPA BTAG	0.6	1	0.5	0.13
Dichloroethane (DCA), 1,1-	75-34-3	47	*none*	47	EPA BTAG	16	1	0.5	0.25
Dichloroethane (DCA), 1,2-	107-06-2	100	*none*	100	EPA BTAG	33	1	0.5	0.13
Dichloroethene (DCE), 1,1-	75-35-4	25	*none*	25	EPA BTAG	8	1	0.5	0.25
Dichloroethene (DCE), cis-1,2-	156-59-2	970	*none*	970	EPA BTAG	320	1	0.5	0.25
Dichloroethene (DCE), trans-1,2-	156-60-5	970	*none*	970	EPA BTAG	320	1	0.5	0.25
Methylene Chloride	75-09-2	98.1	*none*	98.1	EPA BTAG	33	2	1	0.5
Trichloroethene (TCE)	79-01-6	21	*none*	21	EPA BTAG	7	1	0.5	0.25
Vinyl Chloride (VC)	75-01-4	930	*none*	930	EPA BTAG	310	1	0.5	0.25
Inorganics (µg/L)									
Arsenic [8]	7440-38-2	5	150	5	EPA BTAG	1.5	2.5	1.5	0.75
Cobalt [8]	7440-48-4	23	*none*	23	EPA BTAG	8	3.125	2.5	1.25
Iron	7439-89-6	300	*none*	300	EPA BTAG	100	100	60	30
Manganese	7439-96-5	120	*none*	120	EPA BTAG	40	15	6	3

Notes:

This table is an updated/revised version of Worksheet #15 from the final UFP SAP (Tetra Tech, 2011)

µg/L – micrograms per liter CAS – Chemical Abstracts Service [number] LOQ – Limit of Quantitation LOD – Limit of Detection MDL – Method Detection Limit

Note: In most cases where the LOQ exceeds the PSL, the DL is less than PSL. Therefore data usability is not expected to be affected. The target analytes will be detectable if not accurately quantifiable. By including the analytes on the target compound list and striving to quantify as low a concentration as possible with conventional analytical methods, the project team ensures that gross contamination will not be overlooked even though the PSL is less than the LOQ.

[1] U.S. Environmental Protection Agency (EPA) EPA Region 3 BTAG Freshwater Surface Water Screening Benchmarks (July 2006) http://www.epa.gov/reg3hwmd/risk/eco/btag/sbv/fw/screenbench.htm

[2] Maryland Department of Environment (MDE) (2008) Numerical Criteria for toxic substances in Surface Waters Table 1. http://www.dsd.state.md.us/comar/getfile.aspx?file=26.08.02.03-2.htm

[3] Selected PSL is the lowest (most conservative) of the evaluated PSLs.

[4] All PSLs are above QLs, except as noted: Shading indicates the Laboratory-Specific LOQ(s) is(are) greater than the Selected PSL. For purposes of selecting COPCs in the baseline human health risk assessment, ½ the LOQ

[5] PSL References indicate the source of the selected PSL from the evaluated PSL list (see Notes 1 through 4 above), or if the PSL is selected as a Performance Indicator Level (PIL) determined for chemicals and analytes in the "OTHER" category.

[6] Project Quantitation Limit (PQL) Goal is set at 1/3 the PSL.

[7] The Laboratory LOQs and MDLs from the subcontracted laboratory(ies) are presented and are current as of June 2010. The QLs and MDLs are subject to change. The actual limits will be evaluated during the data usability assessment to ensure that the actual LOQs and MDLs are satisfactory to support the data evaluations.

[8] Laboratory will perfom a 4x concentration on these metals.

TABLE 2-7
REFERENCE LIMITS AND EVALUATION
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

Matrix: Sediment

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)			Selected PSL [4, 5]	PSL Reference [6]	PQL Goal [7]	Laboratory-Specific QLs		
		EPA BTAG SEDIMENT [1]	EPA RSL Residential Soil [2]	MDE Standard Residential Soil [3]				LOQ	LOD	MDL
VOCs (mg/kg)										
Bromoform	75-25-2	0.654	62	81	0.654	EPA BTAG	0.22	0.005	0.0025	0.00125
Chloroform	667-66-3	*none*	0.29	78	0.29	EPA RSL	0.1	0.005	0.0025	0.00125
Dichloroethane (DCA), 1,1-	75-34-3	*none*	3.3	1,600	3.3	EPA RSL	1.1	0.005	0.0025	0.00125
Dichloroethane (DCA), 1,2-	107-06-2	*none*	0.43	7	0.43	EPA RSL	0.14	0.005	0.0025	0.00125
Dichloroethene (DCE), 1,1-	75-35-4	0.031	24	390	0.031	EPA BTAG	0.01	0.005	0.0025	0.00125
Dichloroethene (DCE), cis-1,2-	156-59-2	1.05	16	78	1.05	EPA BTAG	0.35	0.005	0.0025	0.00125
Dichloroethene (DCE), trans-1,2-	156-60-5	1.05	15	160	1.05	EPA BTAG	0.35	0.005	0.0025	0.00125
Methylene Chloride	75-09-2	*none*	56	85	56	EPA RSL	18	0.01	0.005	0.0025
Trichloroethene (TCE)	79-01-6	0.969	0.91	1.6	0.91	EPA RSL	0.3	0.005	0.0025	0.00125
Vinyl Chloride (VC)	75-01-4	*none*	0.06	0.09	0.06	EPA RSL	0.02	0.005	0.0025	0.00125
Inorganics (mg/kg)										
Arsenic [8]	7440-38-2	9.8	0.39	0.43	0.39	EPA RSL	0.13	0.5	0.3	0.15
Cobalt	7440-48-4	50	2.3	*none*	2.3	EPA RSL	0.07	0.75	0.5	0.25
Iron	7439-89-6	20,000	5,500	5,500	5,500	EPA RSL	1,800	20	12	6
Manganese	7439-96-5	460	180	160	160	MDE	50	3	1.2	0.6

Notes:

This table is an updated/revised version of Worksheet #15 from the final UFP SAP (Tetra Tech, 2011)

mg/kg– milligrams per kilogram CAS – Chemical Abstracts Service [number] LOQ – Limit of Quantitation LOD – Limit of Detection MDL – Method Detection Limit

Note: In most cases where the LOQ exceeds the PSL, the DL is less than PSL. Therefore data usability is not expected to be affected. The target analytes will be detectable if not accurately quantifiable. By including the analytes on the target compound list and striving to quantify as low a concentration as possible with conventional analytical methods, the project team ensures that gross contamination will not be overlooked even though the PSL is less than the LOQ.

[1] U.S. Environmental Protection Agency (EPA) EPA Region 3 BTAG Freshwater Sediment Screening Benchmarks (July 2006) http://www.epa.gov/reg3hwmd/risk/eco/btag/sbv/fwsed/screenbench.htm

[2] U.S. Environmental Protection Agency (EPA) (May 2012) Regional Screening Level (RSL) for soil under residential scenario. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for chromium VI used as surrogate for [total] chromium. RSL for methylmercury used as surrogate for mercury.

[3] Maryland Department of Environment (MDE) (2008) Numerical Criteria for toxic substances in Surface Waters Table 1. http://www.dsd.state.md.us/comar/getfile.aspx?file=26.08.02.03-2.htm

[4] Selected PSL is the lowest (most conservative) of the evaluated PSLs.

[5] The Laboratory LOQs and MDLs from the subcontracted laboratory(ies) are presented and are current as of June 2010. The QLs and MDLs are subject to change. The actual limits will be evaluated during the data usability assessment to ensure that the actual LOQs and MDLs are satisfactory to support the data evaluations.

[6] PSL References indicate the source of the selected PSL from the evaluated PSL list (see Notes 1 through 4 above), or if the PSL is selected as a Performance Indicator Level (PIL) determined for chemicals and analytes in the "OTHER" category. The OTHER chemicals and analytes provide information about the aquifer conditions and have no inherent risk (except for Nitrate, which has an EPA RSL of 5,800 µg/L and MCL of 10,000 µg/L; however, the preferred PSL/PIL for nitrate is 1,000 µg/L to evaluate natural biodegradation potential in the aquifer). (Aluminum, chloride, copper,iron, manganese, silver, sulfate, and zinc have Federal Secondary Drinking Water Standards, which are not evaluated herein).

[7] Project Quantitation Limit (PQL) Goal is set at 1/3 the PSL.

[8] Laboratory will perfom a 4x concentration on these metals.

TABLE 2-8
RECEPTORS AND EXPOSURE ROUTES FOR QUANTITATIVE EVALUATION
SITE 43 – TOLUENE DISPOSAL
NAVAL SUPPORT FACILITY, INDIAN HEAD
INDIAN HEAD MARYLAND

Receptors	Exposure Routes
Construction Worker (current/future land use)	• Soil dermal contact (surface/ subsurface) • Soil incidental ingestion (surface/ subsurface) • Inhalation of air/dust/emissions (surface/ subsurface) • Direct ingestion of groundwater • Groundwater dermal contact • Inhalation of vapors intruding into a building (soil gas) • Sediment dermal contact • Sediment incidental ingestion • Direct ingestion of surface water • Surface water dermal contact
Maintenance Worker (current/future land use)	• Soil dermal contact (surface/ subsurface) • Soil incidental ingestion (surface/ subsurface) • Inhalation of air/dust/emissions (surface/ subsurface) • Direct ingestion of groundwater • Groundwater dermal contact • Inhalation of vapors intruding into a building (soil gas) • Sediment dermal contact • Sediment incidental ingestion • Direct ingestion of surface water • Surface water dermal contact
Occupational Worker (current/future land use)	• Soil dermal contact (surface/subsurface) • Soil incidental ingestion (surface/subsurface) • Inhalation of air/dust/emissions (surface/subsurface) • Direct ingestion of groundwater • Groundwater dermal contact • Inhalation of vapors intruding into a building (soil gas)
On-Base Residents (Child/ Adult) (future land use)	• Soil dermal contact (surface/ subsurface) • Soil incidental ingestion (surface/ subsurface) • Inhalation of air/dust/emissions (surface/ subsurface) • Direct ingestion of groundwater • Groundwater dermal contact • Inhalation of vapors intruding into a building (soil gas) • Sediment dermal contact • Sediment incidental ingestion • Direct ingestion of surface water • Surface water dermal contact

TABLE 2-9
OCCURENCE AND DISTRIBUTION - GROUNDWATER
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

Chemical	Frequency of Detection	Range of Non-detected Concentrations		Minimum Detected Concentration	Maximum Detected Concentration	Sample of Maximum Detected Concentration	Concentration Used for Screening	USEPA Tapwater RSL [1]	Background Concentration	COPC	Rationale for COPC Selection
EXPLOSIVES (µg/L)											
1,3-DINITROBENZENE	5/35	0.1	0.168	0.131 J	0.244 J	S43MW00601	0.244	**0.15 N**	--	YES	ASL
2,6-DINITROTOLUENE	2/35	0.1	0.178	0.185 J	0.504 J	S43MW1001	0.504	1.5 N	--	NO	BSL
2-AMINO-4,6-DINITROTOLUENE	2/35	0.1	0.178	0.118 B	0.274 J	S43MW00103	0.274	3 N	--	NO	BSL
2-NITROTOLUENE	5/34	0.15	0.5	0.0797 B	0.371 B	S43MW2401	0.371	**0.27 C**	--	YES	ASL
3-NITROTOLUENE	18/35	0.15	0.5	0.107 J	3.57 J	S43MW00401-D	3.57	**0.13 N**	--	YES	ASL
4-AMINO-2,6-DINITROTOLUENE	3/35	0.1	0.178	0.0929 J	0.473	S43MW2401	0.473	3 N	--	NO	BSL
4-NITROTOLUENE	19/35	0.15	0.5	0.0861 J	2.13 J	S43MW17D01	2.13	3.7 C	--	NO	BSL
HMX	10/35	0.1	0.178	0.123 J	3.4	S43MW0020101	3.4	78 N	--	NO	BSL
NITROBENZENE	3/35	0.1	0.178	0.117 B	0.865 B	S43MW2401	0.865	**0.12 C**	--	YES	ASL
NITROCELLULOSE	1/3	500	500	120 B	120 B	S43MW0010101-D	120	4700000 N	--	NO	NSL
NITROGLYCERIN	6/35	0.374	0.65	0.262 J	32.3 J	S43MW00601	32.3	**0.15 N**	--	YES	ASL
RDX	23/35	0.15	0.178	0.0886 J	1.7 J	S43MW00601	1.7	**0.61 C**	--	YES	ASL
TETRYL	1/35	0.1	0.178	0.198 J	0.198 J	S43MW2401	0.198	6.1 N	--	NO	BSL
METALS (µg/L)											
ALUMINUM	34/35	25	25	14.1 J	4050	S43MW00601	4050	**1600 N**	9620	NO	BKG
ANTIMONY	2/35	2	2	2 L	2.1 L	S43MW0020101	2.1	**0.6 N**	--	YES	ASL
ARSENIC	19/35	1.5	2	1.15 J	16.9	S43MW00301	16.9	**0.045 C**	--	YES	ASL
BARIUM	35/35	-	-	27	354	S43MW0020101	354	**290 N**	**139**	YES	ASL
BERYLLIUM	22/35	0.5	0.5	0.306 J	4.6	S43MW0020101	4.6	**1.6 N**	--	YES	ASL
CADMIUM	10/35	0.5	0.5	0.26	0.909 J	S43MW1801	0.909	**0.69 N**	2.8	NO	BKG
CALCIUM	3/3	-	-	3450	3890	S43MW0010101	3890	NA	--	NO	NSL
CHROMIUM	13/35	0.5	1	0.59	11.6	S43MW00103	11.6	**0.031 C** [2]	16.4	NO	BKG
COBALT	39/42	2.5	2.5	1.95 J	130	S43MW1301	130	**0.47 N**	**15.6**	YES	ASL
COPPER	20/35	2	2	1.01 J	14.3	S43MW00103	14.3	62 N	22.4	NO	BSL
IRON	35/35	-	-	37.3 B	54900 K	S43MW0602	54900	**1100 N**	**19900**	YES	ASL
LEAD	8/35	0.75	0.9	0.69 J	4.57	S43MW00103	4.57	15	--	NO	BSL
MAGNESIUM	3/3	-	-	2270	4640	S43MW0020101	4640	NA	--	NO	NSL
MANGANESE	35/35	-	-	32.7	1140	S43MW19S01	1140	**32 N**	**824**	YES	ASL
NICKEL	32/35	1.5	1.5	0.761 J	78.3 J	S43MW0020101	78.3	**30 N**	**16.6**	YES	ASL
POTASSIUM	3/3	-	-	1660	1690	S43MW0020101	1690	NA	--	NO	NSL
SELENIUM	1/35	1.25	3	0.786 J	0.786 J	S43MW00103	0.786	7.8 N	--	NO	BSL
SODIUM	3/3	-	-	39100	60500	S43MW0020101	60500	NA	--	NO	NSL
VANADIUM	24/35	0.4	2.5	0.49	15.7	S43MW00601	15.7	**7.8 N**	20.9	NO	BKG
ZINC	35/35	-	-	1.32 J	134 J	S43MW0020101	134	470 N	**45.2**	NO	BSL

TABLE 2-9
OCCURENCE AND DISTRIBUTION - GROUNDWATER
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

Chemical	Frequency of Detection	Range of Non-detected Concentrations		Minimum Detected Concentration	Maximum Detected Concentration	Sample of Maximum Detected Concentration	Concentration Used for Screening	USEPA Tapwater RSL [1]	Background Concentration	COPC	Rationale for COPC Selection
VOLATILES (µg/L)											
1,1,1-TRICHLOROETHANE	5/40	0.5	800	0.37 J	11.1	S43MW2001	11.1	750 N	--	NO	BSL
1,1-DICHLOROETHANE	8/41	0.5	800	0.4 J	39.6	S43MW2401	39.6	**2.4 C**	--	YES	ASL
1,1-DICHLOROETHENE	14/41	0.5	800	0.42 J	70	S43MW19S01-D	70	**26 N**	--	YES	ASL
2-BUTANONE	1/41	5	8000	4.04 J	4.04 J	S43MW2401	4.04	490 N	--	NO	BSL
ACETONE	2/41	5	8000	5.61 B	7.56 J	S43MW2401	7.56	1200 N	--	NO	BSL
BROMOFORM	1/41	0.5	800	960	960	S43MW0010102-D	960	**7.9 C**	--	YES	ASL
CARBON DISULFIDE	5/41	0.5	800	0.53 J	2.65	S43MW1001	2.65	72 N	--	NO	BSL
CHLOROFORM	1/41	0.5	800	8.32 J	8.32 J	S43MW00103	8.32	**0.19 C**	--	YES	ASL
CHLOROMETHANE	4/41	0.5	800	0.48 J	6.35	S43MW00301	6.35	19 N	--	NO	BSL
CIS-1,2-DICHLOROETHENE	23/41	0.5	800	0.66 J	580	S43MW0010101	580	**2.8 N**	--	YES	ASL
METHYLENE CHLORIDE	3/41	0.5	800	0.51 J	730 B	S43MW0010101-D	730	**8.4 N** [3]	--	YES	ASL
TRANS-1,2-DICHLOROETHENE	3/41	0.5	800	0.58 J	1.4 J	S43MW0402	1.4	8.6 N	--	NO	BSL
TRICHLOROETHENE	36/48	0.5	0.5	0.31 J	55000 J	S43MW0010101-D	55000	**0.26 N** [3]	--	YES	ASL
VINYL CHLORIDE	10/41	0.5	800	1.16	65.6	S43MW17D01	65.6	**0.015 C**	--	YES	ASL

Notes:

µg/L : micrograms per liter
J : Estimated value
K : Biased High
L : Biased Low
ASL : Above Screening Level
BSL : Below Screening Level
BKG : Below Background Level
NSL : No Screening Level
B : Compound detected in blank sample (qualified data were evaluated as positive detections)
COPC : Compound of Potential Concern

Bold and shaded values indicate an exceeded of PSLs

[1] Based on USEPA Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites, November 2012

[2] Value is for Hexavalent Chromium

[3] The non-carcinogenic value divided by 10 is less than the carcinogenic value. Therefore the non-carcinogenic value is presente

TABLE 2-10
RISK RATION EVALUATION
SITE 43 - TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND
PAGE 1 OF 1

Chemical	Incremental Lifetime Carcinogenic Risk (ILCR)			Estimated Non-Carcinogenic Hazard Quotient (HQ)		
	EPC[1] (µg/L)	USEPA Residential Tapwater RSL[2] (µg/L)	Estimated ILCR	Primary Target Organs	USEPA Residential RSL[2] (µg/L)	Estimated HQ
EXPLOSIVES						
1,3-DINITROBENZENE	0.15	NA	NA	Spleen	1.5	0.10
2-NITROTOLUENE	0.13	0.27	5E-07	Bone	12	0.01
3-NITROTOLUENE	0.56	NA	NA	NA	1.3	0.43
NITROBENZENE	0.2	0.12	2E-06	Blood, Respiratory	11	0.02
NITROGLYCERIN	15.9	3.9	4E-06	Blood	1.5	10.60
RDX	0.38	0.61	6E-07	Reproductive	47	0.01
METALS						
ANTIMONY	2.1 [2]	NA	NA	Blood	6	0.35
ARSENIC	4	0.045	9E-05	Cardiovascular, Skin	4.7	0.85
BARIUM	146	NA	NA	Cardiovascular & Kidney	2900	0.05
BERYLLIUM	0.97	NA	NA	Gastrointestinal, Respiratory	16	0.06
COBALT	58.3	NA	NA	Thyroid and Respiratory	4.7	12.40
IRON	24400	NA	NA	Gastrointestinal	11000	2.22
MANGANESE	475	NA	NA	Neurological	320	1.48
NICKEL	35.1	NA	NA	Body Weight	300	0.12
VOLATILES						
1,1-DICHLOROETHANE	4.9	2.4	2E-06	Kidney	2900	0.00
1,1-DICHLOROETHENE	6.7	NA	NA	Liver	260	0.03
BROMOFORM	960 [2]	7.9	1E-04	Liver	290	3.31
CHLOROFORM	8.32 [2]	0.19	4E-05	Liver	84	0.10
CIS-1,2-DICHLOROETHENE	112	NA	NA	Blood	28	4.00
METHYLENE CHLORIDE	183	9.9	2E-05	Liver	84	2.18
TRICHLOROETHENE	18900	0.44	4E-02	Immune System, Developmental, Liver, Kidney	2.6	7269.23
VINYL CHLORIDE	10.9	0.015	7E-04	Liver	36	0.30
		Total ILCR	**4E-02**		Total HI	**7308**

Notes:

HI : Hazard Index
HQ : Hazard Quotient
ILCR : Incremental Lifetime Carcinogenic Risk
NA : Not Applicable
RSL : Regional Screening Level
UCL : Upper Confidence Limit
µg/L : micrograms per liter

[1] EPC is the 95% UCL calculated by ProUCL version 4.1.01 unless otherwise noted
[2] Based on USEPA Regional Screening Levels (RSLs) for Chemical Contaminants at Superfund Sites, November 2012
[3] Less than three detected concentration. UCL cannot be computed therefore the maximum detected concentration is used as the EPC

TABLE 2-11
SAMPLING DESIGN AND RATIONALE
PHASE 3 REMEDIAL INVESTIGATION
SITE 43 - TOLUENE DISPOSAL AREA
NSF INDIAN HEAD, MARYLAND

Location	Matrix	Depth of Samples	Analysis	Method	Number of Samples	Rationale	Sampling Strategy
Site-Wide	Monitoring well groundwater	Ranges from 9-34 ft bgs	VOCs, Metals[1]	See **WS #19, Final March 2011 UFP-SAP**	28	Samples will be collected from monitoring wells to evaluate the VOC plume. Data will be used in the baseline HHRA and to characterize the Site.	See **WSs #11 Final March 2011 UFP-SAP** and sections 2.9 and 3.1
Parallel to Glennon Rd at the end of Johnson Rd (Bldg 724)	Monitoring well groundwater	Approximately 20-35 ft bgs (Actual depths TBD)	VOCs, Metals[1]		3	Samples will be collected from monitoring wells to evaluate extent of the VOC plume east of the source area. Data will be used in the baseline HHRA and to characterize the Site.	
Along Potomac River/End of Pump House Ln/Parallel to Strauss Ave.	Outfall	Sample the water flowing into the Potomac River	VOCs, Metals[1]		1	A sample will be collected from the outfall discharging into the Potomac River to determine if the VOC plume has impacted the water in the outfall.	
	Surface Water	Sample the surface water in the drainage ditches leading to the Potomac River	VOCs, Metals[1]		4	Samples will be collected from the surface water drainage channels to determine if the VOC plume has impacted them. Data will be used in the baseline ERA and HHRA and to characterize the Site.	
	Sediment	Sample the sediment in the drainage ditches leading to the Potomac River. Obtain additional background sediment samples to the north	VOCs, Metals[1]		6	Samples will be collected from the sediment in the drainage area to determine if the VOC plume has impacted the sediment. Background samples will be collected to the north from similar darinage channels. Data will be used in the baseline ERA and HHRA and to characterize the Site.	
	Piezometers	Approximately 20-25 ft bgs (Actual depths TBD)	VOCs, Metals[1]		4	Samples will be collected from Piezometers to determine if the VOC plume has impacted the Potomac River. Data will be used in the baseline ERA and HHRA and to characterize the Site.	
	Culvert	Sample the water at the beginning drainage ditch leading to the Potomac River	VOCs, Metals[1]		2	Samples will be collected from the culverts to determine if the VOC plume has impacted water discharging into the Potomac River through the culverts. Data will be used in the baseline HHRA and to characterize the Site.	
Bldg 716	Soil Gas	Sample the soil gas outside and inside Building 716	VOCs		3	Samples will be collected around Building 716 to determibe if vapor intrusion maybe an issue.	

Note: The sampling design presented in the table above includes the collection of soil, groundwater, and indoor air samples needed to further characterize Site 43. The sampling will provide the data needed to evaluate risks to human health, vapor intrusion into Building 720, and support the FS. This table is an update to Worksheet #17 from the UFP-SAP (Tetra Tech, March 2011) and please refer to SAP for all references to Worksheet #'s.

[1] Aqueous "Metals" Analytical Group samples will only include a non-field-filtered (total metals) sample unless turbidity exceeds 10 Nephelometric Turbidity Units upon which a field-filtered (dissolved metals) sample will be collected.

TABLE 3-1
SAMPLE SUMMARY
PHASE 3 REMEDIAL INVESTIGATION
SITE 43 - TOLUENE DISPOSAL AREA
NSF INDIAN HEAD, MARYLAND

Sampling Location / ID Number [1]	Depth	Analytical Group	Number of Samples (identify field duplicates[2])	Sampling SOP Reference[3]
PIEZOMETER SAMPLES				
PZ01 / S43PZ001	20-25 ft bgs (TBD)	VOCs, Metals[4]	1 (plus 1 duplicate)	SA-1.1
PZ02 / S43PZ002	20-25 ft bgs (TBD)	VOCs, Metals[4]	2	SA-1.1
PZ03 / S43PZ003	20-25 ft bgs (TBD)	VOCs, Metals[4]	2	SA-1.1
PZ04 / S43PZ004	20-25 ft bgs (TBD)	VOCs, Metals[4]	2	SA-1.1
MONITORING WELL GROUNDWATER SAMPLES[5]				
MW01 / S43MW00105	9-19 ft bgs	VOCs, Metals[4]	1 (plus 1 duplicate)	SA-1.1
MW02 / S43MW00205	12-22 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW03 / S43MW00303	24-34 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW04 / S43MW00403	23-33 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW05 / S43MW00503	21-31 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW06 / S43MW00603	17-27 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW07 / S43MW00702	23-33 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW08 / S43MW00802	23-33 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW09S / S43MW009S02	6-16 ft bgs	VOCs, Metals[4]	1 (plus 1 duplicate)	SA-1.1
MW09D / S43MW009D02	28-33 ft bgs	VOCs, Metals[4]	1 (plus 1 duplicate)	SA-1.1
MW10 / S43MW01002	30-35 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW11 / S43MW01102	26-36 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW12 / S43MW01202	27.5-32.5 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW13 / S43MW01302	11-21 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW14 / S43MW01402	8-18 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW15 / S43MW01502	11-21 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW16 / S43MW01602	11-21 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW17S / S43MW017S02	18-28 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW17D / S43MW017D02	31-36 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW18 / S43MW01802	7-17 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW19S / S43MW019S02	14-24 ft bgs	VOCs, Metals[4]	1	SA-1.1

TABLE 3-1
SAMPLE SUMMARY
PHASE 3 REMEDIAL INVESTIGATION
SITE 43 - TOLUENE DISPOSAL AREA
NSF INDIAN HEAD, MARYLAND

Sampling Location / ID Number [1]	Depth	Analytical Group	Number of Samples (identify field duplicates[2])	Sampling SOP Reference[3]
MW19D / S43MW019D02	30-35 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW20 / S43MW02002	6-21 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW21 / S43MW02102	15-25 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW22 / S43MW02202	10-20 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW23 / S43MW02302	11-21 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW24 / S43MW02402	2-17 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW25 / S43MW02502	7-17 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW26 / S43MW02602	13-23 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW027 / S43MW02701	25-35 ft bgs	VOCs, Metals[4]	1 (plus 1 duplicate)	SA-1.1
MW028 / S43MW02801	25-35 ft bgs	VOCs, Metals[4]	1	SA-1.1
MW029 / S43MW02901	25-35 ft bgs	VOCs, Metals[4]	1	SA-1.1
SEEP SAMPLES [6]				
SP01 / S43SP0102	CULVERT OPENING	VOCs, Metals[4]	1	SA-1.2
SP02 / S43SP0202	CULVERT OPENING	VOCs, Metals[4]	1	SA-1.2
SURFACE WATER SAMPLES				
SW01 / S43SW01	SURFACE	VOCs, Metals[4]	1 (plus 1 duplicate)	SA-1.2
SW02 / S43SW02	SURFACE	VOCs, Metals[4]	1	SA-1.2
SW03 / S43SW03	SURFACE	VOCs, Metals[4]	1	SA-1.2
SW04 / S43SW04	SURFACE	VOCs, Metals[4]	1	SA-1.2
CULVERT SAMPLES				
CL01 / S43CL01	CULVERT OPENING	VOCs, Metals[4]	1	SA-1.2
CL02 / S43CL02	CULVERT OPENING	VOCs, Metals[4]	1	SA-1.2
OUTFALL SAMPLE				
OF01 / S43OF01	OUTFALL	Revised VOCs, Metals[4]	1	SA-1.2

TABLE 3-1
SAMPLE SUMMARY
PHASE 3 REMEDIAL INVESTIGATION
SITE 43 - TOLUENE DISPOSAL AREA
NSF INDIAN HEAD, MARYLAND

Sampling Location / ID Number [1]	Depth	Analytical Group	Number of Samples (identify field duplicates[2])	Sampling SOP Reference[3]
SEDIMENT SAMPLES				
SD01 / S43SD01	SURFACE	VOCs, Metals	1 (plus 1 duplicate)	SA-1.2
SD02 / S43SD02	SURFACE	VOCs, Metals	1	SA-1.2
SD03 / S43SD03	SURFACE	VOCs, Metals	1	SA-1.2
SD04 / S43SD04	SURFACE	VOCs, Metals	1	SA-1.2
BKGSD01 / S43BKGSD01	SURFACE	VOCs, Metals	1	SA-1.2
BKGSD02 / S43BKGSD02	SURFACE	VOCs, Metals	1	SA-1.2
SOIL GAS SAMPLES				
SG05 / S43SG005TBD	NEAR BUILDING 716. ~6 - 8 FT BGS	VOCs	1 (plus 1 duplicate)	SA-1.2
SD06 / S43SD06TBD	NEAR BUILDING 716. ~6 - 8 FT BGS	VOCs	1	SA-1.2
SD07 / S43SD07TBD	NEAR BUILDING 716. ~6 - 8 FT BGS	VOCs	1	SA-1.2
SD08 / S43SD08TBD	NEAR BUILDING 716. ~6 - 8 FT BGS	VOCs	1	SA-1.2

Note: This table is a supplement to Worksheet #18 from the Final Sampling and Analysis Plan (SAP) (Tetra Tech, March 2011) and please see final UFP SAP for all standard operating procedures (SOP) and analytical references.

[1] Refer to SOP CT-04 for sample numbering system.

[2] Field duplicate locations may change in the field based sampling order and samples per day. Duplicate samples will have "-D" added to the parent sample ID (this is a deviation from Facility SOP CT-04).

[3] Facility SOPs are provided in Appendix D of the NSF-IH Master SAP.

[4] Aqueous "Metals" Analytical Group samples will only include a non-field-filtered (total metals) sample unless turbidity exceeds 10 Nephelometric Turbidity Units upon which a fie filtered (dissolved metals) sample will be collected.

[5] Groundwater samples from monitoring wells MW01 and MW02 are being collected for the fifth time (Round 5), MW03 to MW06 will be sampled for the third time (Round 3), MW07 to MW26 will be sampled for the second time (Round 2), while the to-be-installed monitoring wells will be collected for the first time (Round 1). For sediment and soil gas sample IDs, the "TDBD" refers to the two-digit top depth and two-digit bottom depth of the screened interval from which the sample is collected.

[6] Seep samples from SP01 and sp02 are being collected for the second time (Round 2).

TBD : To be determined

VOC : volatile organic compounds

TCE : trichloroethane

TABLE 3-2
FIELD QUALITY CONTROL SAMPLE SUMMARY TABLE
PHASE 3 REMEDIAL INVESTIGATION
SITE 43 - TOLUENE DISPOSAL AREA
NSF INDIAN HEAD, MARYLAND

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates [1]	No. of MS/MSDs [2]	No. of Source Water Blanks [3]	No. of Equip. Blanks [4]	No. of VOA Trip Blanks [5]	Total No. of Samples to Lab
Groundwater	VOCs	37	4	2/2	2	4	4	55
	Metals[6]	37	4	2/2	2	4	0	51
Seep	VOCs	2	0	0/0	0	0	0	2
	Metals[6]	2	0	0/0	0	0	0	2
Surface Water	VOCs	4	1	1/1	0	0	1	8
	Metals[6]	4	1	1/1	0	0	0	7
Culvert	VOCs	2	0	0/0	0	0	0	2
	Metals[6]	2	0	0/0	0	0	0	2
Outfall	VOCs	1	0	0/0	0	0	0	1
	Metals[6]	1	0	0/0	0	0	0	1
Sediment	VOCs	6	1	1/1	0	0	1	10
	Metals	6	1	1/1	0	0	0	9
Soil Gas	VOCs	4	1	0	0	0	0	5

Note: This table is updated Worksheet #20 from the Final Sampling and Analysis Plan (SAP) (Tetra Tech, March 2011)

[1] One field duplicate for every 10 or fewer investigative samples.

[2] Although the matrix spike (MS)/matrix spike duplicate (MSD) is not typically considered a field QC sample, it is included here because location determination is often established in the field. One MS/MSD sample for every 20 or fewer investigative samples per sample matrix (e.g., groundwater, soil).

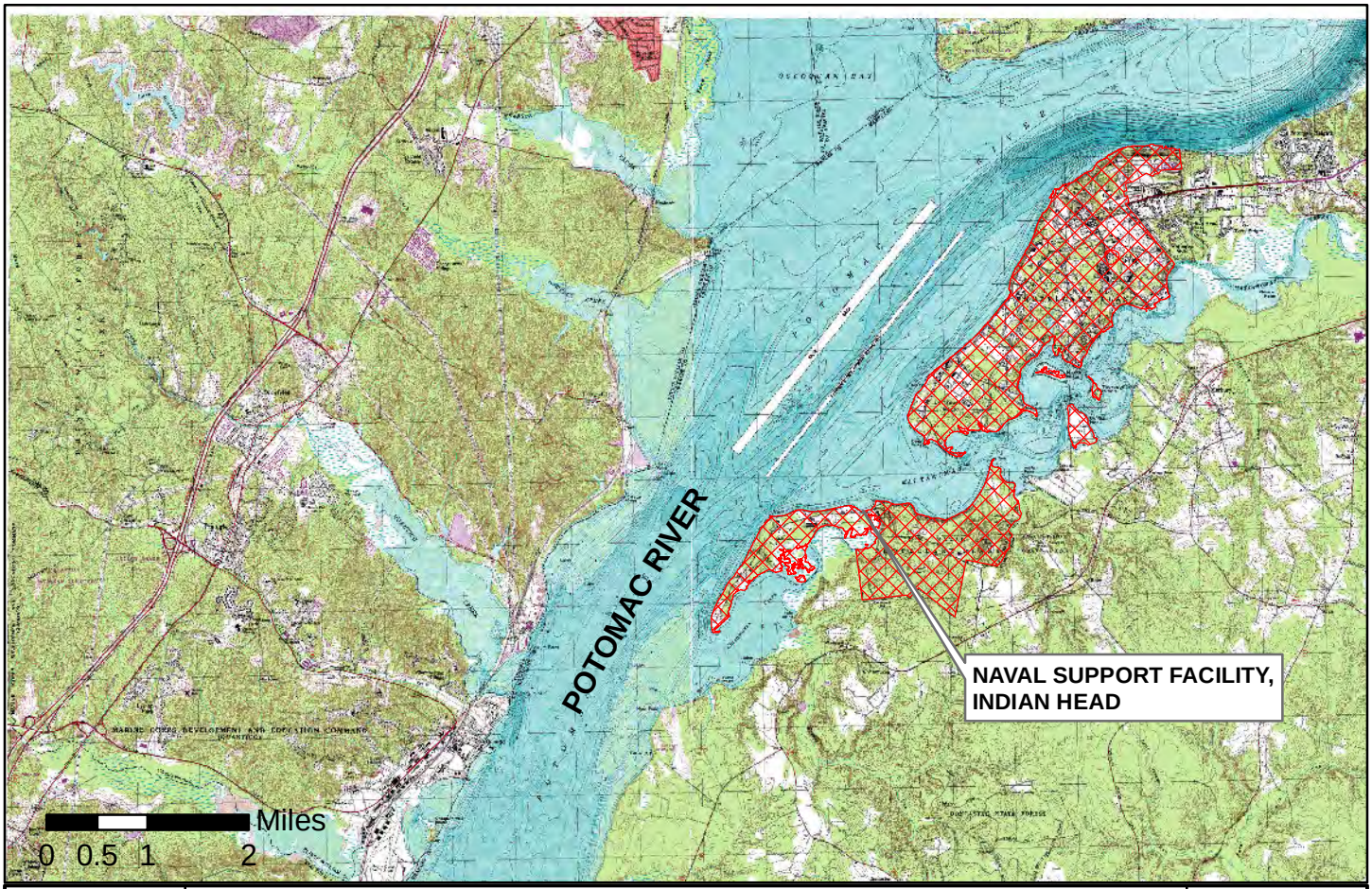
[3] One source water blank for VOC analysis per water source. It is not anticipated that mutipule sources of water will be utilized.

[4] For non-disposable equipment, one equipment blank per type of non-dedicated, decontaminated equipment, with a minimum of one per week per sampling device/instrument. For pre-cleaned, dedicated, and/or disposal sampling equipment, equipment blank samples will not be collected.

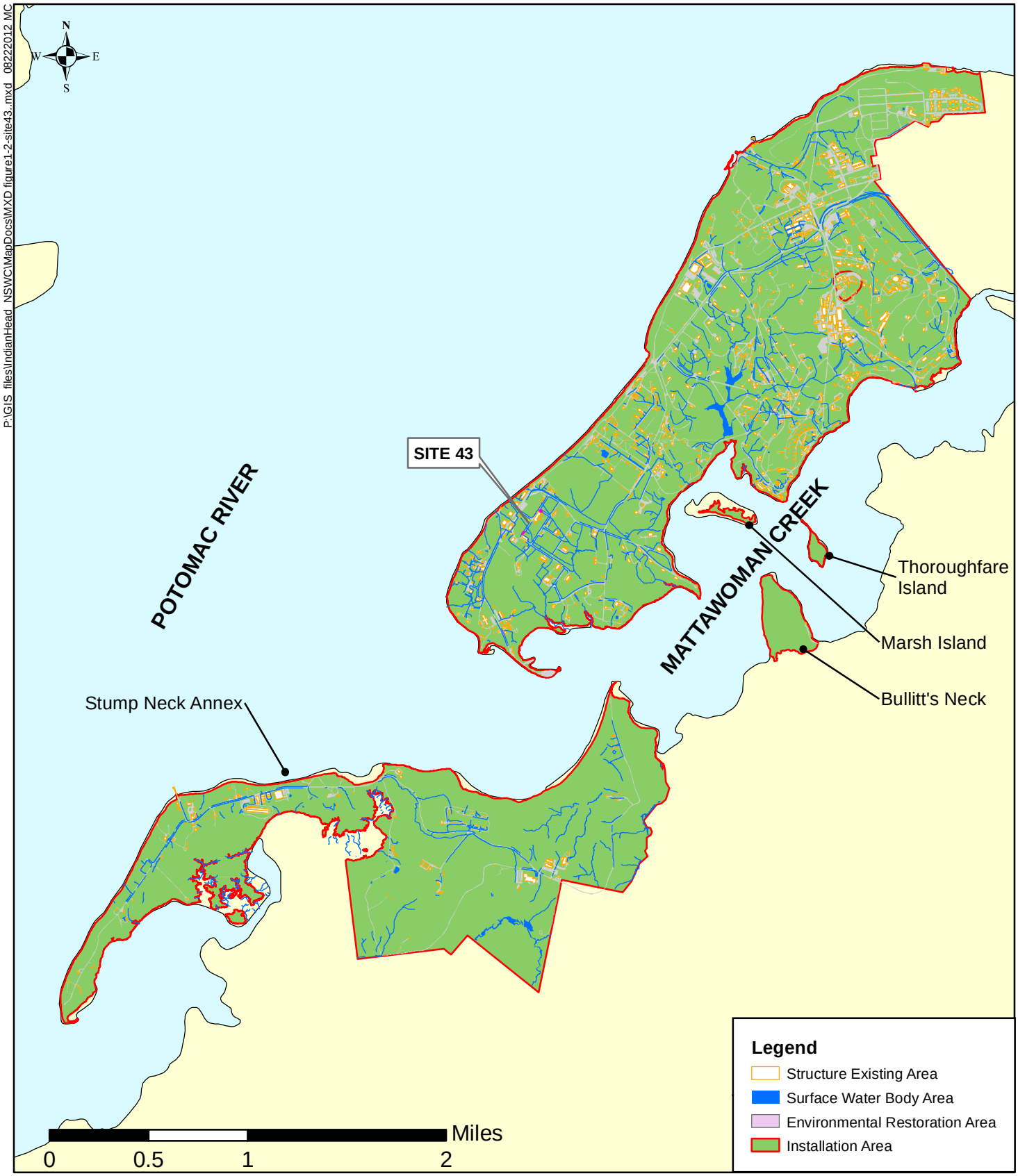
[5] One trip blank per cooler containing samples for VOCs analysis (not including methane, ethane, and ethene [dissolved gases]).

[6] Aqueous "Metals" Analytical Group samples will only include a non-field-filtered (total metals) sample unless turbidity exceeds 10 Nephelometric Turbidity Units upon which a field-filtered (dissolved metals) sample will be collected. Field-filtered (dissolved) metal samples not included in the total sample count.

FIGURES



DRAWN BY MC	DATE 6/11/12	**FACILITY LOCATION MAP** **NAVAL SUPPORT FACILITY - INDIAN HEAD** **INDIAN HEAD, MARYLAND**	CONTRACT NUMBER 112G03107
CHECKED BY	DATE		APPROVED BY DATE
REVISED BY	DATE		APPROVED BY DATE
SCALE AS NOTED			FIGURE NO. **1-1** REV



DRAWN BY MC	DATE 8/22/12	**SITE LOCATION MAP** **SITE 43-TOLUENE DISPOSAL** **NAVAL SUPPORT FACILITY - INDIAN HEAD** **INDIAN HEAD, MARYLAND**	CONTRACT NUMBER 112G03107
CHECKED BY	DATE		APPROVED BY — DATE
REVISED BY	DATE		APPROVED BY — DATE
SCALE AS NOTED			FIGURE NO. 1-2 REV

P:\GIS_files\IndianHead_NSWC\MapDocs\MXD IH_xsectlocmap_100112.mxd 12132012 MC

Potomac River

812 LUNCH HOUSE
ADMIN/OFFICE BLDG
1856 OIL UNLOADING FACILITY
712 AUXILIARY STEAM PLANT
TRANSFORMER STATION
1646 AIR COMPRESSOR BLDG
1648 COOLING TOWER
1532
813 LUNCH HOUSE
714 CHANGE HOUSE, LAUNDRY, AND OFFICES
1180
1482
1531 TREATMENT PLANT - WELL WATER
N GREENSLADE RD
1612 SETTLING BASIN
C-135
C-136
1386
715 BEAKER WRAP MOTOR PREP BLDG
1257 TRANSFORMER STATION
1579 STRAINER BLDG
740 SUBSTATION
739 PUMP HOUSE - RIVER
Building 1040
1964 MECHANICAL / WATER TREATMENT B
1963
1040 PARTS CLEANING HOUSE
1962
C-197
3022
721 PRESS HOUSE - EXPLOSIVES
C-221
C-33
3007
821 TOILET BLDG
SCHUYLER RD
1360 SCRAP POWDER SHED
716 RETAIL OUTLET NO.8 / INSPECT.
STRAUSS AVE
LEARY RD
C-137
C-138
C-139
1916 STORAGE BLDG
720 GRAIN MANUFACTURE AND MOTOR LOAD BLDG
PUMP HOUSE LANE
1755 STORAGE SHED - TOMAHAWK
C-272
1921 STORAGE BLDG
1193 CONTROL BLDG
717 PROCESS BLDG - INERT
720A MOTOR TEST BAY
GALLERY RD
722 MOLD FILL BLDG NO.2
GLENNON RD
JOHNSON RD
1801
1853
1857
C-193
1134 MISSILE TRNG / REWORK / FABRICAT
C-194
C-204
VOEGELI RD
1629
Building 1041
1485 STORAGE BLDG
1041 PARTS CLEANING HOUSE
723 MOLD FILL BUILDING NO.3
1773

N

Legend

- Road
- Railroad
- Contour Line (feet msl)
- Surface Water Line
- Building
- Flora Special Species Area
- Wetland Area
- Forest Stand Area

0 62.5 125 250 Feet

TETRA TECH

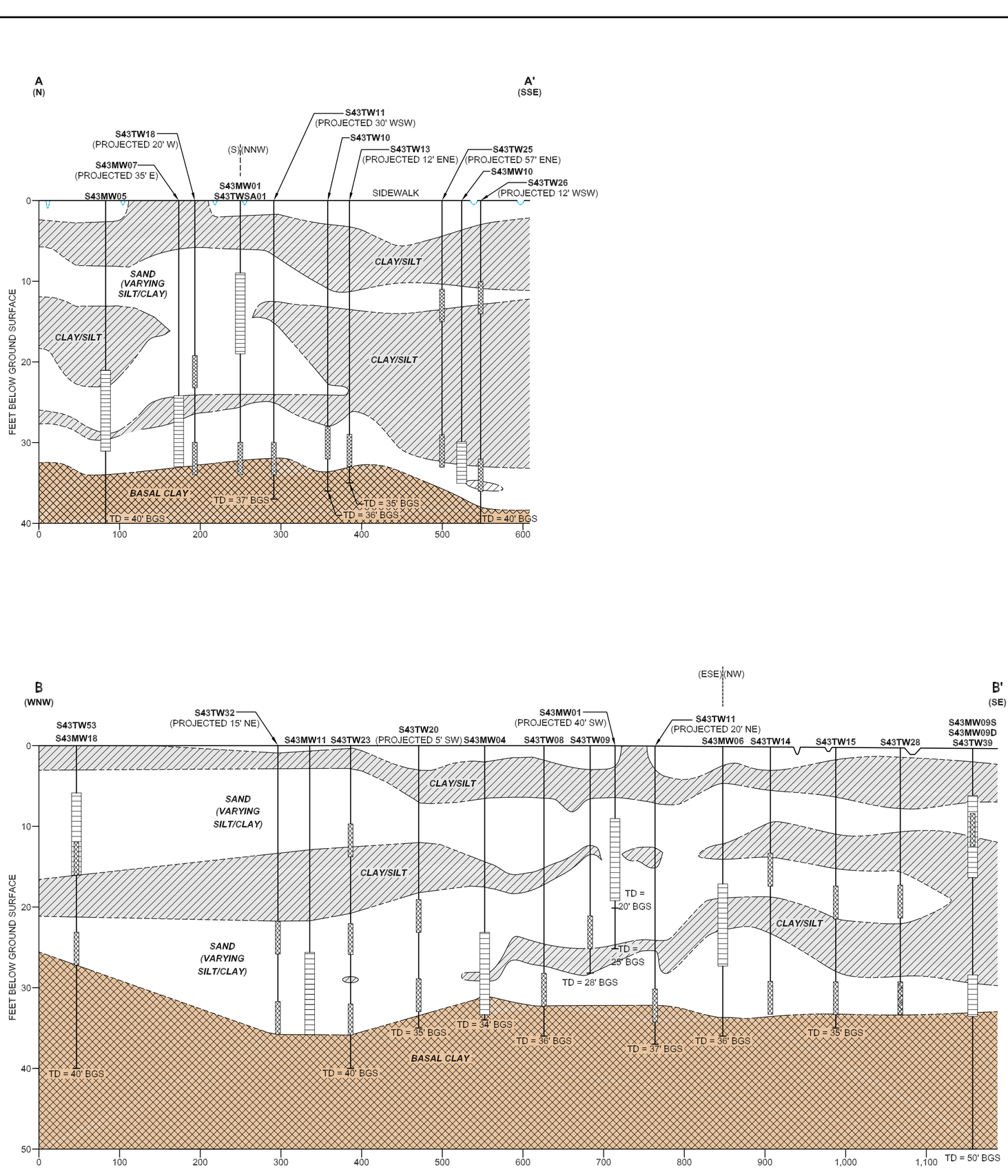
Site Layout
Site 43 Toluene Disposal Area
Naval Support Facility Indian Head
Indian Head, MD

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	**FIGURE 1-3**	REV	DATE 12/13/12

Cross Section Location Map
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

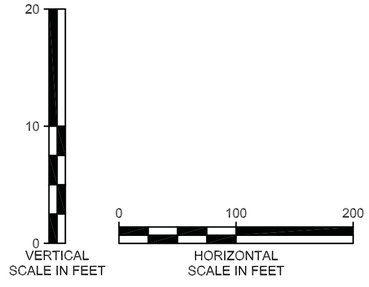
FILE	112G01358	SCALE	AS NOTED
FIGURE NO.	FIGURE 2-1	REV	DATE 1/17/13

112G01358/0620/112G01358GS02-1.DWG 09/24/12 MKB



LEGEND

S43MW04 S43TW08 (PROJECTED 5' S)	MONITORING WELL GROUNDWATER GRAB SAMPLE PROJECTED DISTANCE AND DIRECTION TO CROSS SECTION LINE
	GROUND SURFACE (APPROXIMATED TO BE FLAT) DITCH WITH STANDING WATER
	GEOLOGIC CONTACT (DASHED WHERE INFERRED)
	BOREHOLE
	GROUNDWATER GRAB SAMPLE
	MONITORING WELL SCREEN
TD = 40' BGS	TOTAL DEPTH
	SAND WITH VARYING AMOUNTS OF SILT, CLAY, AND GRAVEL
	CLAY OR SILT WITH VARYING AMOUNTS OF SAND
	BASAL CLAY

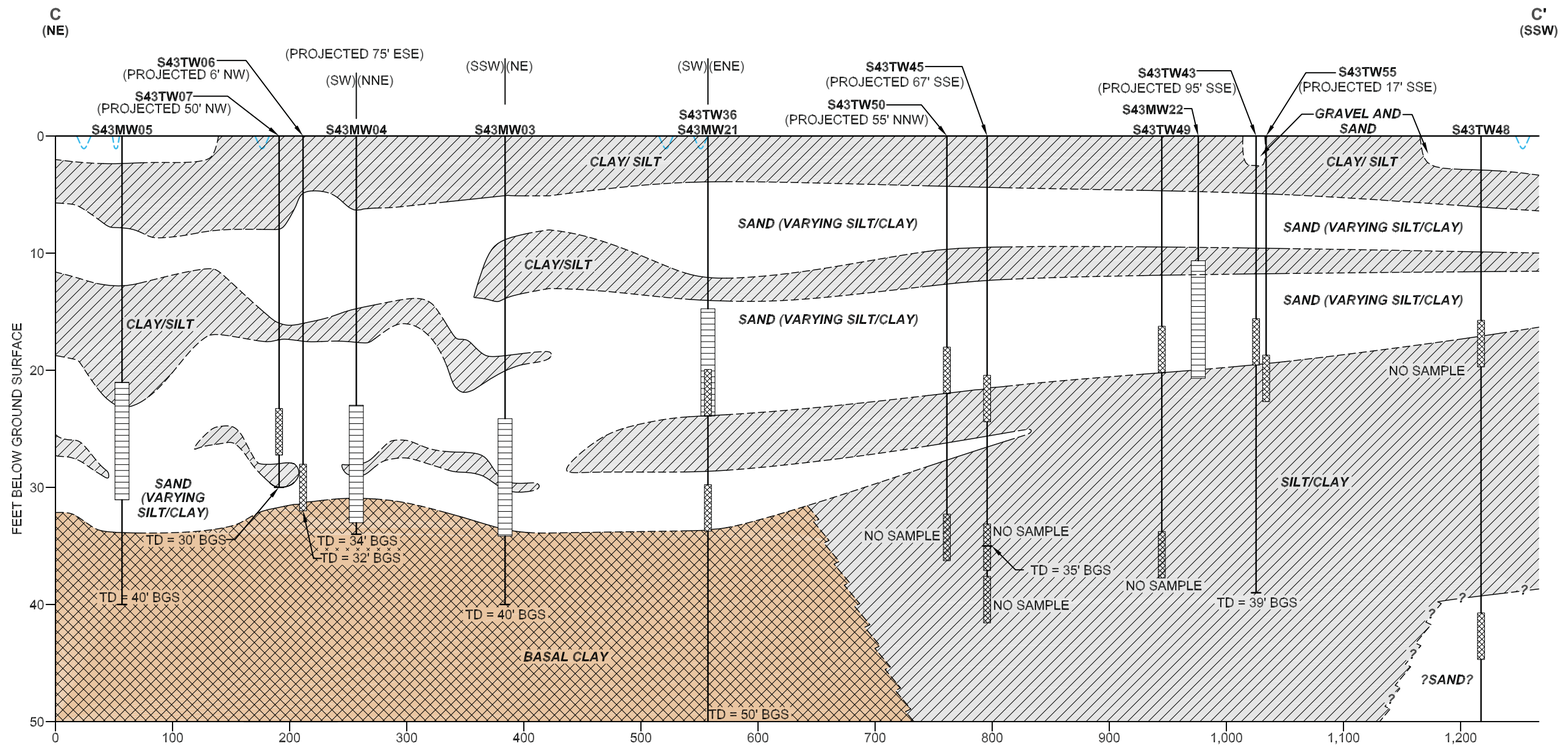


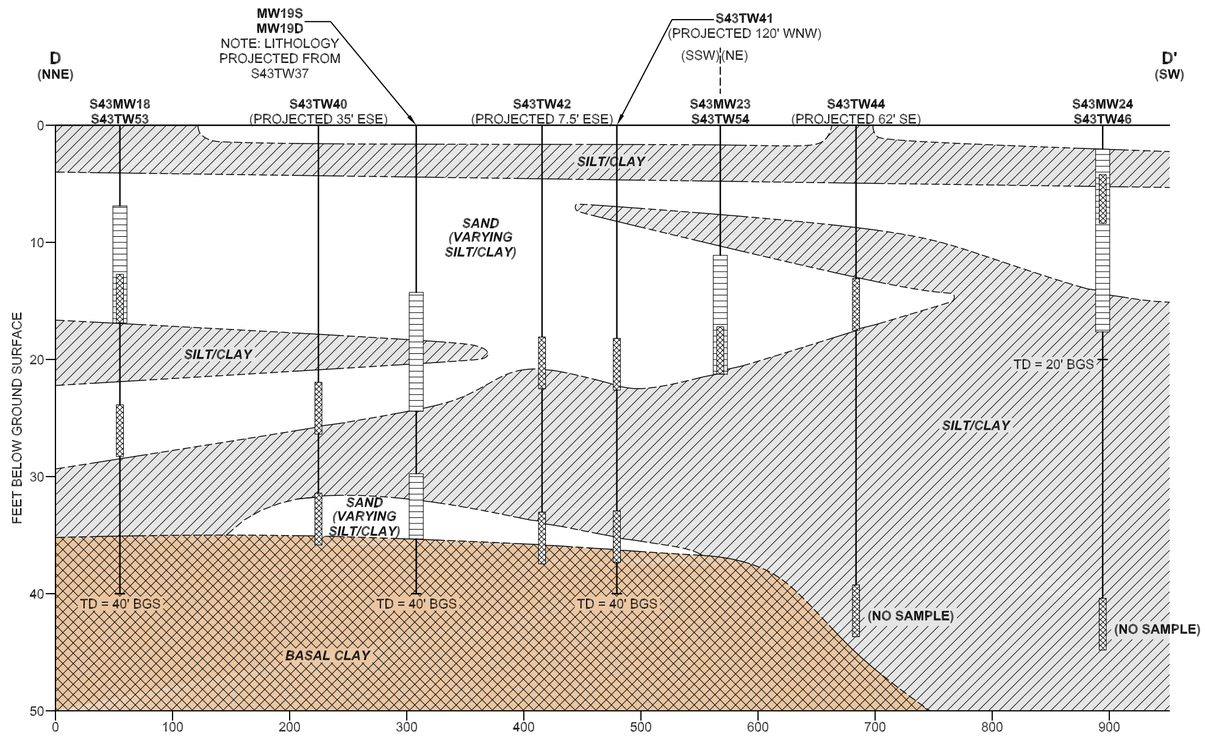
TETRA TECH

GEOLOGIC CROSS SECTIONS
A – A' AND B – B'
SITE 43 – TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

FILE 112G01358GS02-1	SCALE AS NOTED
FIGURE NUMBER 2-2	REV 0 DATE 09/24/12

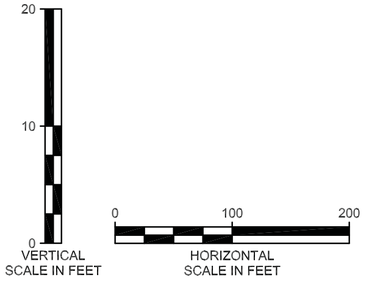
112G01358/0620/112G01358GS02-2.DWG 09/24/12 MKB





LEGEND

- **S43MW04 / S43TW08 (PROJECTED 5' S)** — MONITORING WELL; GROUNDWATER GRAB SAMPLE; PROJECTED DISTANCE AND DIRECTION TO CROSS SECTION LINE
- GROUND SURFACE (APPROXIMATED TO BE FLAT) DITCH WITH STANDING WATER
- GEOLOGIC CONTACT (DASHED WHERE INFERRED)
- BOREHOLE
- GROUNDWATER GRAB SAMPLE
- MONITORING WELL SCREEN
- TD = 40' BGS — TOTAL DEPTH
- SAND WITH VARYING AMOUNTS OF SILT, CLAY, AND GRAVEL
- CLAY OR SILT WITH VARYING AMOUNTS OF SAND
- BASAL CLAY

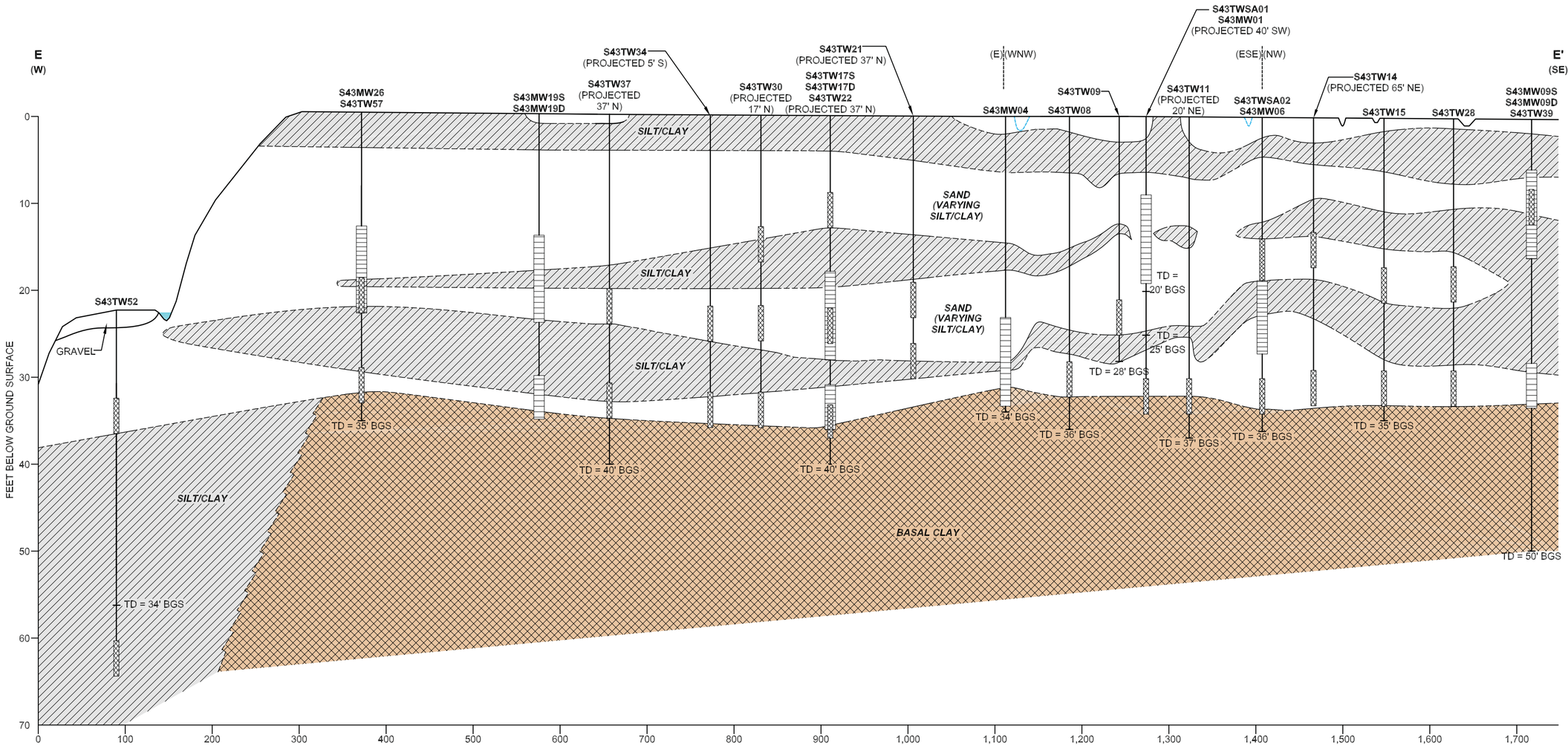


TETRA TECH

GEOLOGIC CROSS SECTIONS
C – C' AND D – D'
SITE 43 – TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

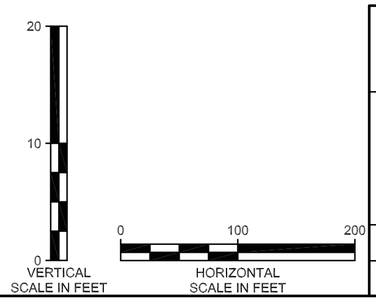
FILE	SCALE
112G01358GS02-2	AS NOTED
FIGURE 2-3	REV 0 / DATE 09/24/12

112G01358/0620/112G01358GS02_3.DWG 09/24/12 MKB



LEGEND

S43MW04	MONITORING WELL
S43TW08	GROUNDWATER GRAB SAMPLE
(PROJECTED 5' S)	PROJECTED DISTANCE AND DIRECTION TO CROSS SECTION LINE
	GROUND SURFACE (APPROXIMATED TO BE FLAT) DITCH WITH STANDING WATER
	GEOLOGIC CONTACT (DASHED WHERE INFERRED)
	BOREHOLE
	GROUNDWATER GRAB SAMPLE
	MONITORING WELL SCREEN
TD = 40' BGS	TOTAL DEPTH
	SAND WITH VARYING AMOUNTS OF SILT, CLAY, AND GRAVEL
	CLAY OR SILT WITH VARYING AMOUNTS OF SAND
	BASAL CLAY



TETRA TECH

GEOLOGIC CROSS SECTION E – E'
SITE 43 – TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

FILE 112G01358GS02-3	SCALE AS NOTED
FIGURE 2-4	REV 0 / DATE 09/24/12

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P:\GIS_files\IndianHead_NSWC\MapDocs\MXD IH_potmap_091912.mxd 0117213 MC

N

Surface Water Elevation
approximately -0.9 FT MSL

MW18 24.22
MW11 23.47
MW05 24.90
MW07 25.03
MW04 25.36
MW17S 22.06
MW17D 17.52
MW19S 21.50
MW19D 13.89
MW26 20.73
MW01 25.01
MW08 24.95
MW03 19.80
MW06 24.81
MW25 8.25
MW23 20.17
MW20 21.40
MW21 22.46
MW12 18.94
MW10 23.78
MW09S 24.55
MW09D 24.75
MW24 26.70
MW22 21.45
MW13 22.91
MW02 23.04
MW14 23.09
MW16 22.99
MW15 23.08

STRAUSS AVE
LEARY RD
PUMP HOUSE LANE
GALLERY RD
GLENNON RD
JOHNSON RD
VOEGELI RD

Legend

- Monitoring Well
- Groundwater Contours (Feet MSL)
- Groundwater Flow Direction
- 25.01 Groundwater Elevation (Feet MSL)
- Notes: Dashed lines are inferred

0 62.5 125 250 Feet

TETRA TECH

Potentiometric Surface Map
July 2012
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G01358	SCALE	AS NOTED
FIGURE NO.	**FIGURE 2-5**	REV	DATE 1/17/13

INH000027.cb

P:\GIS_files\IndianHead_NSWC\MapDocs\MXD IH_samplelocation_091712.mxd 01172013 MC

Bing Maps aerial:
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N

TW53 MW18 SB29 TW24 MW05 SB10 MW11 TW32 TW19 TW23 SB12 TW18 SP02 TW40 SB07 TW01 TW04 TW31 SB04 TW20 SB13 SB23 MW07 SB01 TWSA01 SB18 TW12 TW06 MW01 TW34 TW22 MW17S MW04 TW07 MW19S SB09 MW19D MW17D TW21 SB14 TW17 MW08 MW26 TW57 TW52 TW30 TW37 TW08 TW05 SB17 TW42 TW02 SB19 SB24 TW11 MW06 TWSA02 TW29 SB08 TW10A SB22 SB26 TW35 MW03 TW09 SB15 TW03 SB25 TW33 TW38 SB27 TW16 SB16 TW10 SB11 SP01 MW25 MW23 STRAUSS AVE LEARY RD TW13 SB28 TW41 TW54 TW14 TW36 TW47 MW12 TW15 MW20 MW21 TW44 TW25 TW27 TW28 TW56 TW45 MW10 TW26 MW09S TW39 TW43 MW09D TW46 TW49 TW50 GALLERY RD GLENNON RD TW55 MW22 MW24 TW51 JOHNSON RD MW13 TW48 MW14 SB06 MW16 MW02 MW15 VOEGELI RD POMPHOUSE LANE

715 714 716 717 718 720 720A 721 722 723 724 730 1134 1485 1604 1612 1531 1921 1963 1962 1755 1857 1853 1629 1789 1773 1787 1255 1055 1482 1386 1579 739 1916 1007 1193 1210

C-136 C-272 C-193 C-194b C-204 C-241 C-242 C-721 C-33 C-197 INH000027.cb

Legend

- Soil Boring
- Monitoring Well
- Temporary Well
- Seep Sample
- Surface Water Line
- Building
- Road Area

0 62.5 125 250 Feet

TETRA TECH

Sample Location Map
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	**FIGURE 2-6**	REV / DATE	1/17/13

P:\GIS_files\IndianHead_NSWC\MapDocs\MXD IH_soiltag_080211.mxd 01222013 MC

S43SB012-0507

	June 2011	June 2011 DUP
VOCs µg/kg		
Acetone	12.1 B	12 J

S43SB013-0608

	June 2011
VOCs µg/kg	
Methylene Chloride	6.81 J
Toluene	1.29 J

S43SB009-0708

	June 2011	June 2011 DUP
VOCs µg/kg		
Acetone	14.5 J	13.3 J

S43SB019-0607

	June 2011
VOCs µg/kg	
Acetone	23.6 J
Vinyl Chloride	14.3

S43SB008-0607

	June 2011
VOCs µg/kg	
Acetone	20 J

S43SB022-0708

	June 2011
VOCs µg/kg	
4-Methyl-2-Pentanone	19,100 J
2-Butanone	837
Acetone	4,660,000
Cis-1,2-Dichloroethene	585

S43SB015-0810

	June 2011
VOCs µg/kg	
4-Methyl-2-Pentanone	5,190
2-Butanone	564 J
2-Hexanone	16
Acetone	157,000 J
Methylene Chloride	8.6 J

S43SS004-0001

	July 2005
VOCs µg/kg	
Acetone	12 J
Methylene Chloride	130 J

S43SB004-0101

VOCs µg/kg	
2-Butanone	7 J
Acetone	10 J
Methylene Chloride	140 J

S43SB010-0709

	June 2011
VOCs µg/kg	
Methylene Chloride	9.47 B
Tetrachloroethene	1.65 J

S43SB023-0001

	May 2012	May 2012 (DUP)
VOCs µg/kg		
Acetone	26.1 B	19.1 B

S43SB023-0607

VOCs µg/kg		
Acetone	195,000 J	NA
Methylene Chloride	3,590 J	NA

S43SS001-0001

	July 2005
VOCs µg/kg	
Methylene Chloride	8 B

S43SB001-0101

VOCs µg/kg	
Methylene Chloride	8 B
Trichloroethene	8 J

S43SB024-0001

	May 2012	May 2012 (DUP)
VOCs µg/kg		
Acetone	20.4	NA
Cis 1,2-Dichloroethene	14.4	NA

S43SB024-0607

VOCs µg/kg		
Cis 1,2-Dichloroethene	442	397
Trichloroethene	**2,080**	**1,700**

S43SB018-0507

	June 2011
VOCs µg/kg	
Trichloroethene	776

S43SB017-0608

	June 2011
VOCs µg/kg	
Cis-1,2-Dichloroethene	397
Trichloroethene	**3,940**

S43SS002-0001

	July 2005
VOCs µg/kg	
Methylene Chloride	210 J

S43SB002-0101

VOCs µg/kg	
Methylene Chloride	5 B

S43SB027-0001

	May 2012
VOCs µg/kg	
Cis 1,2-Dichloroethene	175
Trichloroethene	**2,720**

S43SB027-0708

VOCs µg/kg	
Cis 1,2-Dichloroethene	447
Trichloroethene	**4,790**

S43SB016-0607

	June 2011
VOCs µg/kg	
Acetone	18.3 J

S43SB028-0001

	May 2012
VOCs µg/kg	ND

S43SB028-0405

VOCs µg/kg	ND

S43SB025-0001

	May 2012
VOCs µg/kg	
Acetone	8.95 B
Trichloroethene	1.91 J

S43SB025-0607

VOCs µg/kg	
Cis 1,2-Dichloroethene	100 J
Trichloroethene	**2,380**

S43SB011-0507

	June 2011
VOCs µg/kg	
Trichloroethene	470

S43SB026-0001

	May 2012
VOCs µg/kg	
Acetone	14.8 B
Cis 1,2-Dichloroethene	23.7
Trichloroethene	69.4
Vinyl Chloride	2.18 J

S43SB026-0506

VOCs µg/kg	
Trichloroethene	**1,150**

SB10, SB07, SB12, SB09, SB13, SB23, SB04, SB01, SB14, SB19, SB02, SB22, SB15, SB24, SB18, SB17, SB25, SB11, SB26, SB27, SB16, SB28, SB08

Gallery Rd

Glennon Rd

Schulyer Rd

Ditch

Ditch

1257 1964 1962 821 1040 1193 3022 C-197 C-205 C-221 C-33

N

Notes:
J=estimated value
B=Detected in Blank sample
ND=not detected
NA=not analyzed
VOCs=volatile organic compounds
µg/kg-microgram per kilogram
DUP=duplicate sample
Bold=Exceedances of Project Action Levels

* These soil boringshave the shallow and deeper samples offset by several feet due to drill rig access in the ditch. Shallow SB (0 to 1 ft bgs) samples were collected in the ditch while samples greater than 1 ft bgs were collected on the northside of the ditch

Legend

- Soil Boring
- Surface Water Line
- Building
- Road Area

0 25 50 100 Feet

Bing Maps aerial:
Aerial photograph from ESRI Bing Maps map service
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TETRA TECH

Soil Analytical Detections-VOCs
Site 43 Toluene Disposal Area
Naval Support Facility Indian Head
Indian Head, Maryland

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	**FIGURE 2-7**	REV	DATE 1/22/13

P:\GIS files\IndianHead_NSWC\MapDocs\MXD IH_mwtag_voc_091912.mxd MC 01222013

Notes:
* =Analyzed for Trichloroethene only
bgs=below ground surface
DUP=duplicate sample
J=estimated value
VOCs=volatile organic compounds
CIS-1,2-DCE= CIS-1,2-dichloroethene
µg/L-microgram per liter
ND=Not Detected
NX=No Exceedance
Bold=Exceedances of Project Action Levels

Bromoform, chloroform, and methylene chloride exceedances are not shown

N

N GREENSLADE RD

PUMP HOUSE LANE

STRAUSS AVE

GALLERY RD

SCHUYLER RD

JOHNSON RD

VOEGELI RD

GLENNON RD

S43MW18	July 2012
feet bgs	7 -17
VOCs µg/L	
Trichloroethene	2.78

S43MW19D	July 2012
feet bgs	30 - 35
VOCs µg/L	
CIS-1,2-DCE	30.5
Trichloroethene	72.8
Vinyl Chloride	9.12

S43MW19S	July 2012	July 2012 (DUP)
feet bgs	14 - 24	
VOCs µg/L		
1,1-Dichloroethane	9.75 J	10.2 J
1,1-Dichloroethene	57	70
CIS-1,2-DCE	158	166
Trichloroethene	3,620	4,090

S43MW26	July 2012
feet bgs	13 - 23 ft
VOCs µg/L	
Trichloroethene	0.51 J

S43MW25	July 2012
feet bgs	7 - 17
VOCs µg/L	
Trichloroethene	9.22

S43MW23	July 2012
feet bgs	11 - 21
VOCs µg/L	
Trichloroethene	1.59

S43MW17S	July 2012
feet bgs	18 - 28
VOCs µg/L	
CIS-1,2-DCE	212
Trichloroethene	2,740
Vinyl Chloride	45.2

S43MW24	July 2012
feet bgs	2 - 17
VOCs µg/L	
1,1-Dichloroethane	39.6
1,1-Dichloroethene	8.6

S43MW20	July 2012
feet bgs	6 - 21
VOCs µg/L	
Trichloroethene	1,390

*S43MW13	July 2012
feet bgs	11 - 21 ft
Trichloroethene µg/L	1.18

S43MW22	July 2012
feet bgs	10 - 20
VOCs µg/L	
1,1-Dichloroethane	3.49
Trichloroethene	0.54 J

*S43MW16	July 2012
feet bgs	11 - 21
Trichloroethene µg/L	ND

S43MW11	July 2012
feet bgs	26 - 36
VOCs µg/L	ND

S43MW04	June 2011	June 2011 (DUP)	July 2012
feet bgs	23 - 33		
VOCs µg/L			
CIS-1,2-DCE	152	156	203
Trichloroethene	699	682	644 J
Vinyl Chloride	15.7	14.1	37.9 J

S43MW17D	July 2012
feet bgs	31 - 36
VOCs µg/L	
CIS-1,2-DCE	255
Trichloroethene	2,660
Vinyl Chloride	65.6

S43MW03	June 2011	July 2012
feet bgs	24 - 34	
VOCs µg/L		
Trichloroethene	1.74	0.66 J
Vinyl Chloride	1.16	1.23

S43MW21	July 2012
feet bgs	15 - 25
VOCs µg/L	
Trichloroethene	0.83 J

S43MW12	July 2012
feet bgs	27.5 - 32.5
VOCs µg/L	
Trichloroethene	1.77

S43MW05	June 2011	July 2012
feet bgs	21 - 31	
VOCs µg/L	NX	NX

S43MW07	July 2012
feet bgs	23 - 33
VOCs µg/L	NX

S43MW08	July 2012
feet bgs	23 - 33
VOCs µg/L	ND

S43MW06	June 2011	July 2012
feet bgs	17 - 27	
VOCs µg/L		
CIS-1,2-DCE	41.6	153 J
Trichloroethene	1,130	4,420
Vinyl Chloride	ND	7.5 J

S43MW01	2005	2005 (DUP)	2007	2007 (DUP)	June 2011	July 2012	July 2012 (DUP)
feet bgs	9 - 19						
VOCs µg/L							
CIS-1,2-DCE	580	480 J	ND	ND	284	400	355
Trichloroethene	53,000 J	55,000 J	36,000	36,000	10,900	17,500	16,200

S43MW10	July 2012
feet bgs	30 - 35
VOCs µg/L	NX

S43MW09D	July 2012
feet bgs	28 - 33
VOCs µg/L	NX

S43MW09S	July 2012
feet bgs	6 - 16
VOCs µg/L	
CIS-1,2-DCE	26.4
Trichloroethene	100
Vinyl Chloride	2.7 J

*S43MW14	July 2012
feet bgs	8 - 18
Trichloroethene µg/L	ND

*S43MW02	2005	2007	June 2011	July 2012	July 2012 (DUP)
feet bgs	12 - 22				
Trichloroethene µg/L	1.3 J	3.2	NX J	ND	ND

*S43MW15	July 2012
feet bgs	11 - 21
Trichloroethene µg/L	ND

MW18 MW19D MW19S MW26 MW25 MW23 MW24 MW20 MW22 MW21 MW17D MW17S MW11 MW04 MW05 MW07 MW08 MW01 MW03 MW06 MW12 MW10 MW09D MW09S MW13 MW02 MW14 MW16 MW15

Legend
- Monitoring Well
- Surface Water Line
- Building
- Road Area

0 62.5 125 250 Feet

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TETRA TECH

Monitoring Well-VOCs Analytical Exceedances Site 43 Toluene Disposal Area Naval Support Facility Indian Head, MD

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	FIGURE 2-8	REV	DATE 1/22/13

P:\GIS_files\IndianHead_NSWC\MapDocs\MXD IH_twtag_12052012.mxd 01222013 MC

Notes:
All results are in µg/L
ND= Not Detected
NX=No exceedances
NS=Not Sampled
J=estimated value
VOCs=volatile organic compounds
µg/L-microgram per liter
DUP=duplicate sample
Bold=Exceedances of Project Action Levels
CIS-1,2-DCE= CIS-1,2-dichloroethene
1,1-DCA= 1,1-dichloroethane
1,2-DCA= 1,2-dichloroethane

Chloroform and methylene chloride exceedances are not shown

S43TW31	May 2012		
feet bgs	14 ft	26 ft	36 ft
Trichloroethene	ND	0.63 J	ND

S43TW20	April 2012		
feet bgs	23 ft	33 ft	33 ft (DUP)
CIS-1,2-DCE	81.8	50.2	47
Trichloroethene	298	298	287
Vinyl Chloride	13.1	12.3	11.5

S43TW01	Nov 2007	
feet bgs	32 ft	
Trichloroethene	3.2	

S43TW07	June 2011		
feet bgs	27 ft	27 ft (DUP)	
CIS-1,2-DCE	56.5	61.3	
Trichloroethene	923	1,010	
Vinyl Chloride	26.2	25.8	

S43TW53	June 2012		
feet bgs	17 ft	28 ft	28 ft (DUP)
CIS-1,2-DCE	12.4	ND	ND
Trichloroethene	1.00	ND	ND

S43TW23	April 2012		
feet bgs	14 ft	26 ft	36 ft
CIS-1,2-DCE	NX	197	ND
Trichloroethene	1.21	833	1.61
Vinyl Chloride	ND	29.7	ND

S43TW06	June 2011	
feet bgs	32 ft	
CIS-1,2-DCE	4.49	
Trichloroethene	53.9	
Vinyl Chloride	0.724 J	

S43TW34	May 2012		
feet bgs	26 ft	36 ft	36ft (DUP)
CIS-1,2-DCE	NX	23.9	25
Trichloroethene	8.98	80.6	78.7
Vinyl Chloride	ND	8.01 J	11 J

S43TW08	June 2011	
feet bgs	32 ft	
CIS-1,2-DCE	21.8	
Trichloroethene	41.9	
Vinyl Chloride	16.2	

S43TWSA01	May 2012	
feet bgs	34 ft	
Trichloroethene	15.4	

S43SP02-01	June 2012
CIS-1,2-DCE	195
Trichloroethene	28.7
Vinyl Chloride	1.16

S43TW40	June 2012	
feet bgs	26 ft	36 ft
Trichloroethene	102	ND

S43TW09	June 2011	
feet bgs	25 ft	
Trichloroethene	563	

S43TW37	June 2012		
feet bgs	24 ft	24 ft (DUP)	35 ft
CIS-1,2-DCE	4.18	4.20	23.3
Trichloroethene	24.6	24.3	29.0
Vinyl Chloride	0.5 J	0.47 J	20.6 J

S43TW57	July 2012	
feet bgs	23ft	33 ft
CIS-1,2-DCE	87.7	205
Trichloroethene	766	259
Vinyl Chloride	ND	47.9

S43TW42	June 2012	
feet bgs	22 ft	37 ft
CIS-1,2-DCE	30.7	10.7
Trichloroethene	137	8.75
Vinyl Chloride	2.37	6.25

S43SP01-01	June 2012
CIS-1,2-DCE	3.62
Trichloroethene	354

S43TW11	June 2011
feet bgs	34 ft
Trichloroethene	1.68

S43TW47	June 2012
feet bgs	14 ft
1,1-DCA	5.84 J
Trichloroethene	1.58

S43TWSA02	May 2012	
feet bgs	18 ft	34 ft
CIS-1,2-DCE	4.8	62.6
Trichloroethene	135	1,730
Vinyl Chloride	ND	4.1 J

S43TW44	June 2012
feet bgs	17 ft
1,1-DCA	3.37
Trichloroethene	8.14

S43TW28	May 2012		
feet bgs	21 ft	33 ft	33 ft (DUP)
CIS-1,2-DCE	120	ND	ND
Trichloroethene	2.52	ND	ND
Vinyl Chloride	3.46	ND	ND

S43TW54	June 2012
feet bgs	21 ft
Trichloroethene	1.79

S43TW39	June 2012
feet bgs	12 ft
CIS-1,2-DCE	8.64
Trichloroethene	17.9
Vinyl Chloride	0.360 J

S43TW41	June 2012		
feet bgs	22 ft	22 ft (DUP)	37 ft
CIS-1,2-DCE	13.4	13.6	ND
Trichloroethene	22.5	22.5	ND
Vinyl Chloride	2.88	2.81	ND

S43TW21	April 2012	
feet bgs	23 ft	30 ft
Trichloroethene	1.04	NX

S43TW38	June 2012	
feet bgs	22 ft	32 ft
CIS-1,2-DCE	3.06	NX
Trichloroethene	0.940 J	4.63
Vinyl Chloride	0.730 J	ND

S43TW14	April 2012	
feet bgs	17 ft	33 ft
CIS-1,2-DCE	ND	3.35
Trichloroethene	ND	79.9
Vinyl Chloride	ND	0.74 J

S43TW15	April 2012	
feet bgs	21 ft	33 ft
CIS-1,2-DCE	25.7	NX
Trichloroethene	158	0.64 J
Vinyl Chloride	1.4 J	0.58 J

S43TW43	June 2012
feet bgs	19 ft
1,1-DCA	8.89
Trichloroethene	1.11

S43TW29	May 2012	
feet bgs	26 ft	36 ft
CIS-1,2-DCE	NX	10.5
Trichloroethene	1.36	23.7
Vinyl Chloride	ND	0.490 J

S43TW49	June 2012		
feet bgs	21 ft	21 ft (DUP)	
1,1-DCA	23.3	23	
1,2-DCA	0.62 J	0.62 J	
Trichloroethene	1.53	1.43	

S43TW30	May 2012			
feet bgs	17 ft	17 ft (DUP)	26 ft	36 ft
CIS-1,2-DCE	ND	ND	25.9	66.8
Trichloroethene	ND	ND	68.5	93.7
Vinyl Chloride	ND	ND	2.25	54.5

S43TW36	June 2012	
feet bgs	24 ft	34 ft
CIS-1,2-DCE	ND	4.38
Trichloroethene	0.510 J	NX

S43TW45	June 2012
feet bgs	24 ft
Trichloroethene	731 J

S43TW13	April 2012
feet bgs	33 ft
Trichloroethene	20.3

S43TW33	May 2012		
feet bgs	26 ft	36 ft	
CIS-1,2-DCE	164	5.8	
Trichloroethene	1,270	5.81	
Vinyl Chloride	39.1	4.02	

S43TW35	May 2012	
feet bgs	26 ft	36 ft
CIS-1,2-DCE	NX	3.35
Trichloroethene	1.48	0.520 J
Vinyl Chloride	ND	0.390 J

S43TW22	April 2012		
feet bgs	14 ft	26 ft	37 ft
CIS-1,2-DCE	NX	288	170
Trichloroethene	1.64	4,440	384
Vinyl Chloride	ND	54	47.2

Legend
- Seep Sample
- Temporary Well
- Surface Water Line
- Building
- Road Area

0 50 100 200 Feet

TETRA TECH

Groundwater Grab
VOC Analytical Exceedances
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G01358	SCALE	AS NOTED
FIGURE NO.	FIGURE 2-9	REV	DATE 1/22/13

P:\GIS_files\IndianHead_NSWC\MapDocs\MXD IH_mwtag_expl_091912.mxd MC 01222013

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N

S43MW11 July 2012
feet bgs 26 - 36
Explosives µg/L
3-Nitrotoluene 0.418 J
Nitroglycerin 0.318 B

S43MW01 July 2005 June 2011 July 2012
feet bgs 9 - 19
Explosives µg/L
3-Nitrotoluene ND 0.723 J 0.511 J
Nitroglycerin ND 25.2 J ND

S43MW18 July 2012
feet bgs 7 -17
Explosives µg/L NX

S43MW05 July 2011 July 2012
feet bgs 21 - 31
Explosives µg/L
3-Nitrotoluene NX 0.439 J

S43MW07 July 2012
feet bgs 23 - 33
Explosives µg/L
3-Nitrotoluene 0.227 J

S43MW19S July 2012
feet bgs 14 - 24
Explosives µg/L
3-Nitrotoluene 0.222 J

S43MW17D July 2012
feet bgs 31 - 36
Explosives µg/L NX

S43MW04 June 2011 July 2012
feet bgs 23 - 33
Explosives µg/L
3-Nitrotoluene 2.91 0.28 J

S43MW08 July 2012
feet bgs 23 - 33
Explosives µg/L NX

S43MW17S July 2012
feet bgs 18 - 28
Explosives µg/L
3-Nitrotoluene 0.419

S43MW26 July 2012
feet bgs 13 - 23 ft
Explosives µg/L ND

S43MW19D July 2012
feet bgs 30 - 35
Explosives µg/L
3-Nitrotoluene 0.366 J

S43MW06 June 2011 July 2012
feet bgs 17 - 27
Explosives µg/L
1,3-Dinitrobenzene 0.244 J ND
3-Nitrotoluene ND 0.871
Nitroglycerin 32.3 J ND
RDX 1.7 J NX

S43MW25 July 2012
feet bgs 7 - 17
Explosives µg/L
1,3-Dinitrobenzene 0.186 J
2-Nitrotoluene 0.353 B
3-Nitrotoluene 0.301 J
Nitroglycerin 0.295 B

S43MW03 June 2011 July 2012
feet bgs 24 - 34
Explosives µg/L
Nitroglycerin 0.262 J ND

S43MW10 July 2012
feet bgs 30 - 35
Explosives µg/L
Nitrobenzene 0.234 B
RDX 0.791 B

S43MW23 July 2012
feet bgs 11 - 21
Explosives µg/L NX

S43MW20 July 2012
feet bgs 6 - 21
Explosives µg/L NX

S43MW21 July 2012
feet bgs 15 - 25
Explosives µg/L ND

S43MW12 July 2012
feet bgs 27.5 - 32.5
Explosives µg/L
3-Nitrotoluene 0.507

S43MW24 July 2012
feet bgs 2 - 17
Explosives µg/L
1,3-Dinitrobenzene 0.212 J
2-Nitrotoluene 0.371 B
3-Nitrotoluene 0.642
Nitrobenzene 0.865 B
Nitroglycerin 0.307 B

S43MW13 July 2012
feet bgs 11 - 21 ft
Explosives µg/L NA

S43MW09S July 2012
feet bgs 6 - 16
Explosives µg/L
3-Nitrotoluene 0.365 J

S43MW09D July 2012
feet bgs 28 - 33
Explosives µg/L ND

S43MW22 July 2012
feet bgs 10 - 20
Explosives µg/L NX

S43MW14 July 2012
feet bgs 8 - 18
Explosives µg/L NA

S43MW16 July 2012
feet bgs 11 - 21
Explosives µg/L NA

S43MW02 July 2005 June 2011 July 2012
feet bgs 12 - 22
Explosives µg/L NX NA NA

S43MW15 July 2012
feet bgs 11 - 21
Explosives µg/L NA

MW18 MW11 MW05 MW07 MW19S MW19D MW17S MW17D MW04 MW08 MW26 MW01 MW03 MW06 MW25 MW23 MW20 MW21 MW12 MW10 MW09S MW09D MW24 MW22 MW13 MW14 MW02 MW16 MW15

STRAUSS AVE
LEARY RD
SCHUYLER RD
PUMP HOUSE LANE
GALLERY RD
GLENNON RD

Notes:
J=estimated value
B=detected in blank sample
µg/L-microgram per liter
ND=Not Detected
NA=Not Analyzed
NX=No Exceedance
RDX=cyclotrimethylenetrinitramine
Bold=Exceedances of Project Action Levels

Legend
- Monitoring Well
- Surface Water Line
- Building
- Road Area

0 62.5 125 250 Feet

INH000027.cb

TETRA TECH

Monitoring Well-Explosives Analytical Exceedances
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	**FIGURE 2-10**	REV	DATE 1/22/13

P:\GIS files\IndianHead_NSWC\MapDocs\MXD IH_mwtag_metals_082312.mxd MC 01172013

S43MW19S July 2012
feet bgs 14 - 24
Metals µg/L
Beryllium 1.95
Cobalt 124
Iron 6,210
Manganese 1,140
Nickel 46.6
Thallium 0.799 J
Filtered Metals µg/L
Beryllium 1.86
Cadmium 0.765 J
Cobalt 121
Iron 5,930
Manganese 1,090
Nickel 45.1

S43MW18 July 2012
feet bgs 7 -17
Metals µg/L
Cadmium 0.909 J
Cobalt 43.1
Manganese 326
Nickel 52.9
Filtered Metals µg/L
Cadmium 0.942 J
Cobalt 44.2
Manganese 326
Nickel 54.4

S43MW11 July 2012
feet bgs 26 - 36
Metals µg/L
Arsenic 1.56 J
Cobalt 2.22 J
Iron 20,900
Manganese 497
Filtered Metals µg/L
Arsenic 1.49 J
Cobalt 2.39 J
Iron 21,400
Manganese 503

S43MW04 July 2012
feet bgs 23 - 33
Metals µg/L
Arsenic 1.31 J
Cobalt 24.3
Iron 12,400 K
Manganese 352
Filtered Metals µg/L
Arsenic 1.65 J
Cobalt 23.5
Iron 12,500
Manganese 335 L

S43MW05 July 2012
feet bgs 21 - 31
Metals µg/L
Arsenic 2.11 J
Cobalt 50.3
Iron 8,510 K
Manganese 298
Nickel 45.5
Filtered Metals µg/L
Arsenic 1.65 J
Cobalt 48.9
Iron 8,400 K
Manganese 294
Nickel 44.3

S43MW07 July 2012
feet bgs 23 - 33
Metals µg/L
Cobalt 60
Iron 11,600
Manganese 423
Nickel 46.5
Filtered Metals µg/L
Cobalt 61.2
Iron 11,800
Manganese 430 L
Nickel 47

S43MW01 July 2012
feet bgs 9 - 19
Metals µg/L
Arsenic 1.18 J
Chromium 0.927 J
Cobalt 13.3
Iron 5,360K
Manganese 106
Filtered Metals µg/L
Arsenic 1.23 J
Chromium 0.581 J
Cobalt 13.7
Iron 5,370 K
Manganese 109 L

S43MW19D July 2012
feet bgs 30 - 35
Metals µg/L
Arsenic 1.2 J
Iron 27,000
Manganese 522
Filtered Metals µg/L
Iron 25,700
Manganese 502

S43MW08 July 2012
feet bgs 23 - 33
Metals µg/L
Chromium 1.43 J
Cobalt 14.2
Iron 5,000 K
Manganese 206
Filtered Metals µg/L
Chromium 1.25 J
Cobalt 12.8
Iron 5,100 K
Manganese 192

S43MW26 July 2012
feet bgs 13 - 23 ft
Metals µg/L
Chromium 1.53 J
Cobalt 4.85
Manganese 32.7
Filtered Metals µg/L
Chromium 1.4 J
Cobalt 5.08
Manganese 33.4

S43MW23 July 2012
feet bgs 11 - 21
Metals µg/L
Cobalt 4.31
Iron 1,660
Manganese 160
Filtered Metals µg/L
Cobalt 3.85
Iron 1,410
Manganese 142

S43MW17D July 2012
feet bgs 31 - 36
Metals µg/L
Arsenic 2.21 J
Cobalt 6.2
Iron 32,900
Manganese 498
Filtered Metals µg/L
Arsenic 2.3 J
Cobalt 6.24
Iron 32,100
Manganese 500

S43MW09D July 2012
feet bgs 28 - 33
Metals µg/L
Arsenic 5.71
Chromium 0.906 J
Iron 29,800 K
Manganese 185
Filtered Metals µg/L
Arsenic 5.76
Iron 29,000 K
Manganese 180

S43MW06 July 2012
feet bgs 17 - 27
Metals µg/L
Arsenic 6.47
Chromium 3.13
Cobalt 26.4
Iron 54,900 K
Manganese 713
Vanadium 6.68
Filtered Metals µg/L
Arsenic 3.61
Cobalt 25.9
Iron 44,500 K
Manganese 753

S43MW25 July 2012
feet bgs 7 - 17
Metals µg/L
Cobalt 2.35
Iron 1,510
Manganese 184
Filtered Metals µg/L
Cobalt 2.31
Iron 1,580
Manganese 179

S43MW20 July 2012
feet bgs 6 - 21
Metals µg/L
Cobalt 19.9
Iron 1,180
Manganese 234
Filtered Metals µg/L
Cobalt 19.7
Manganese 223

S43MW17S July 2012
feet bgs 18 - 28
Metals µg/L
Arsenic 2.01 J
Cobalt 15.4
Iron 34,900 K
Manganese 561
Filtered Metals µg/L
Arsenic 2.11 J
Cobalt 15.7
Iron 36,100 K
Manganese 572

S43MW21 July 2012
feet bgs 15 - 25
Metals µg/L
Cobalt 33
Iron 5,990 K
Manganese 888
Filtered Metals µg/L
Cobalt 33.6
Iron 6,050 K
Manganese 907

S43MW09S July 2012
feet bgs 6 - 16
Metals µg/L
Cobalt 54.8
Iron 7,640 K
Manganese 405
Nickel 40.2
Filtered Metals µg/L
Barium 347
Beryllium 1.9
Cobalt 54.2
Iron 7,470 K
Manganese 406
Nickel 39.7

S43MW24 July 2012
feet bgs 2 - 17
Metals µg/L
Chromium 4.45
Cobalt 1.95 J
Manganese 79.5
Filtered Metals µg/L
Arsenic 1.14 J
Chromium 4.5 K
Cobalt 2.0 J
Manganese 83.3

S43MW22 July 2012
feet bgs 10 - 20
Metals µg/L
Beryllium 1.61
Cobalt 73.2
Manganese 511
Nickel 42.5
Filtered Metals µg/L
Cobalt 71.8
Manganese 499
Nickel 39.5

S43MW12 July 2012
feet bgs 27.5 - 32.5
Metals µg/L
Arsenic 5.11
Chromium 0.842 J
Cobalt 8.32
Iron 33,000 K
Manganese 456
Filtered Metals µg/L
Arsenic 5.58
Cobalt 8.11
Iron 33,200 K
Manganese 445 L

S43MW10 July 2012
feet bgs 30 - 35
Metals µg/L
Arsenic 5.86
Iron 33,600 K
Manganese 196
Filtered Metals µg/L
Arsenic 4.91 K
Iron 30,600 K
Manganese 187 L

***S43MW13 July 2012**
feet bgs 11 - 21 ft
Metals µg/L
Cobalt 130
Filtered Metals µg/L
Cobalt 127

***S43MW16 July 2012**
feet bgs 11 - 21
Metals µg/L
Cobalt 108
Filtered Metals µg/L
Cobalt 108

***S43MW14 July 2012**
feet bgs 8 - 18
Metals µg/L
Cobalt 36.0
Filtered Metals µg/L
Cobalt 34.8

***S43MW15 July 2012**
feet bgs 11 - 21
Metals µg/L
Cobalt 5.10
Filtered Metals µg/L
Cobalt 5.10

***S43MW02 July 2012**
feet bgs 12 - 22
Metals µg/L
Cobalt 33.6
Filtered Metals µg/L
Cobalt 33.8

S43MW03 July 2012
feet bgs 24 - 34
Metals µg/L
Arsenic 12.2
Cobalt 6.96
Iron 42,200 K
Manganese 763
Filtered Metals µg/L
Arsenic 12.3
Cobalt 7.1
Iron 43,800 K
Manganese 795 L

MW18 MW19S MW19D MW26 MW25 MW24 MW23 MW22 MW20 MW17S MW17D MW11 MW04 MW05 MW07 MW01 MW08 MW03 MW06 MW21 MW12 MW10 MW09S MW09D MW13 MW14 MW02 MW16 MW15

PUMP HOUSE LANE
LEARY RD
GALLERY RD
GLENNON RD

Notes:
J=estimated value
K=biased high
L=biased low
µg/L-microgram per liter
ND=Not Detected
Bold=Exceedances of Project Action Levels
*** Analyzed for Cobalt only**

Legend
Monitoring Well
Surface Water Line
Building
Road Area

0 62.5 125 250 Feet

Bing Maps aerial:
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TETRA TECH

Monitoring Well-Metals Analytical Exceedances
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE 112G00771	SCALE AS NOTED
FIGURE NO. **FIGURE 2-11**	REV DATE 1/17/13

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N

STRAUSS AVE

MW18
MW11
MW05
MW07
MW08
MW19S
MW19D
MW17D
MW17S
MW04
MW01
MW26
MW03
MW06
MW25
MW23
MW21
MW12
MW20
MW10
MW09S
MW09D
MW22
MW13
MW02
MW14
MW16
MW15
MW24

100
50
15
50
15
50
100
50

1040

Legend

- Cobalt contour µg/L
- Cobalt contour µg/L inferred
- Monitoring Well

0 75 150 300 Feet

TETRA TECH

Cobalt Iscoconcentration Map
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	FIGURE 2-12	REV / DATE	1/17/13

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Legend

- Temporary Well
- Existing Monitoring Well
- TCE contour µg/L
- TCE contour µg/L inferred

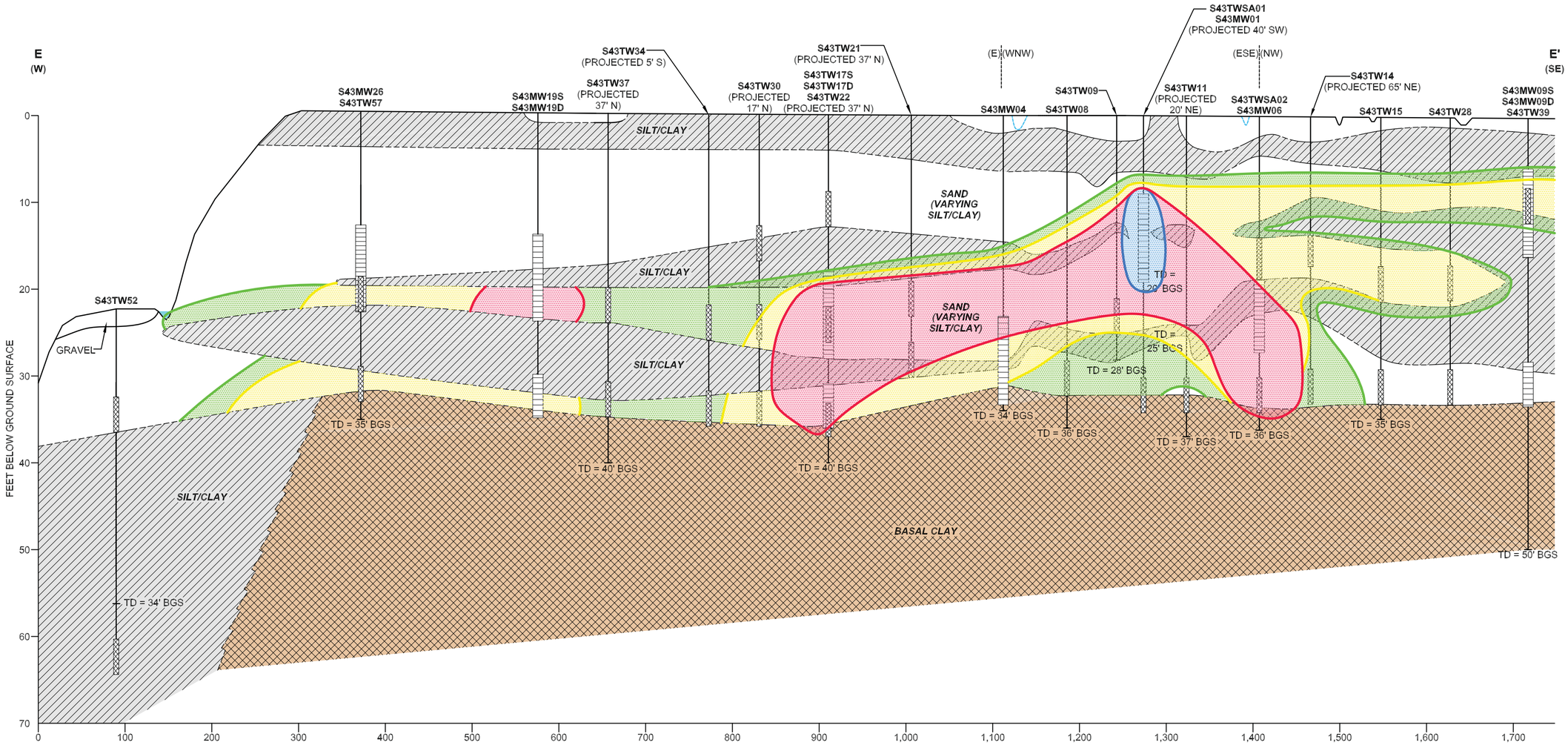
0 75 150 300 Feet

TETRA TECH

Trichloroethene Isoconcentration Map
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	FIGURE 2-13	REV / DATE	1/17/13

112G01358/0620/112G01358GS02_4.DWG 09/24/12 MKB



LEGEND

S43MW04	MONITORING WELL
S43TW08	GROUNDWATER GRAB SAMPLE
(PROJECTED 5' S)	PROJECTED DISTANCE AND DIRECTION TO CROSS SECTION LINE
	GROUND SURFACE (APPROXIMATED TO BE FLAT) DITCH WITH STANDING WATER
	GEOLOGIC CONTACT (DASHED WHERE INFERRED)
	BOREHOLE
	GROUNDWATER GRAB SAMPLE
	MONITORING WELL SCREEN
TD = 40' BGS	TOTAL DEPTH
	SAND WITH VARYING AMOUNTS OF SILT, CLAY, AND GRAVEL
	CLAY OR SILT WITH VARYING AMOUNTS OF SAND
	BASAL CLAY

	TOTAL CHLORINATED VOC ISOCONCENTRATION CONTOUR 5 µg/L
	TOTAL CHLORINATED VOC ISOCONCENTRATION CONTOUR 100 µg/L
	TOTAL CHLORINATED VOC ISOCONCENTRATION CONTOUR 1,000 µg/L
	TOTAL CHLORINATED VOC ISOCONCENTRATION CONTOUR 10,000 µg/L

NOTE:
1. µg/L = MICROGRAMS PER LITER

20
10
0
VERTICAL SCALE IN FEET

0 100 200
HORIZONTAL SCALE IN FEET

TETRA TECH

TOTAL CHLORINATED VOC ISOCONCENTRATION CONTOUR MAP
GEOLOGIC CROSS SECTION E – E'
SITE 43 – TOLUENE DISPOSAL AREA
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

FILE	112G01358GS02-4	SCALE	AS NOTED
FIGURE	2-14	REV	0
		DATE	09/24/12

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S43IND01

VOCs µg/m3	April 2012	DUP
1,1,1-Trichloroethane	0.72 J	0.35 J
1,2-Dichloroethane	**0.098 J**	0.08 B
2-Butanone	1 J	0.91 J
Acetone	6.5 J	5 J
Benzene	**0.45**	0.28 J
Chloromethane	0.91	0.68 J
CIS-1,2-Dichloroethene	0.15 B	ND
Dichlorodifluoromethane	2.8	2.7 J
Ethylbenzene	0.13 J	0.11 J
Tetrachloroethene	0.17 J	0.074 J
Toluene	1.3	0.63 J
Trichloroethene	0.16 J	ND
Trichlorofluoromethane	1.1	1.2 J

S43SG04-06

VOCs µg/m3	June 2011
2-Butanone	**1,600 J**
4-methyl-2-pentanone	**1,600 J**
Acetone	**690,000**
cis-1,2-Dichloroethene	**29,000**

S43ODA01

VOCs µg/m3	June 2011
Benzene	**0.31**
Carbon Tetrachloride	**0.66 J**

S43SG01-06

VOCs µg/m3	June 2011
1,1-Dichloroethane	**140**
Chloromethane	**100 J**
CIS-1,2-Dichloroethene	**140**
m,p-Xylene	**100**
Toluene	**790**
Vinyl Chloride	**30,000**

S43IND02

VOCs µg/m3	April 2012
1,1,1-Trichloroethane	0.04 J
1,2-Dichloroethane	0.088 B
2-Butanone	1 J
Acetone	5.7 J
Benzene	0.27 J
Chloromethane	1.1 J
Dichlorodifluoromethane	2.4 J
Ethylbenzene	0.12 J
Tetrachloroethene	0.076 J
Toluene	0.66 J
Trichloroethene	0.023 B
Trichlorofluoromethane	1.3 J
Vinyl Chloride	0.011 J

S43ODA02

VOCs µg/m3	April 2012
1,1,1-Trichloroethane	0.36 J
1,2-Dichloroethane	0.074 B
2-Butanone	1.2 J
Acetone	12 J
Benzene	0.3
Chloromethane	1.2
CIS-1,2-Dichloroethene	0.017 B
Dichlorodifluoromethane	2.5
Ethylbenzene	0.064
Tetrachloroethene	0.075 J
Toluene	0.61
Trichloroethene	0.03 B
Trichlorofluoromethane	1.2

S43SG03-05

VOCs µg/m3	June 2011
1,4-Dioxane	**0.68 J**
2-Hexanone	**12**
Benzene	**1.1**
cis-1,2-Dichloroethene	**59**
Vinyl Chloride	**100**

Notes:
J=estimated value
B=found in field blank
VOCs=volatile organic compounds
µg/m3-microgram per cubic meter of air
DUP= Duplicate Sample
Bold=Exceedances of Project Action Levels
ND=Not Detected
Soil gas results are exceedances of PALS
Indoor air results are detections
*** Canister could not pull soil gas sample due to a tight zone**

Legend

- Indoor Air
- Outdoor Air
- Soil Gas
- Surface Water Line
- Building
- Road Area

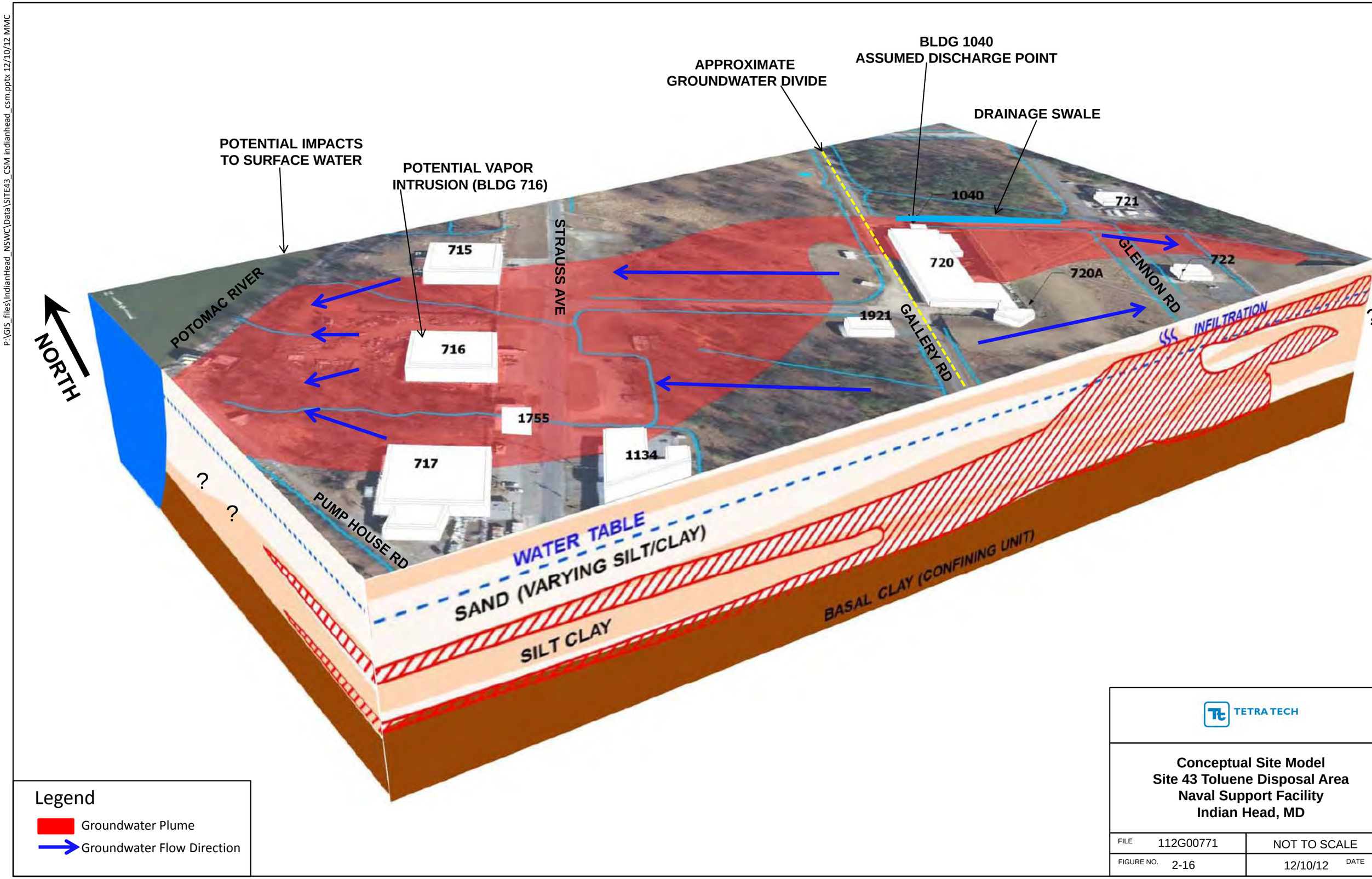
0 25 50 100 Feet

ODA01
SG01
SG04
SG02
SG03
*Not Sampled
IND01
IND02
ODA02
Ditch
Gallery Rd
Schulyer Rd
Glennon Rd
1257
1040
821
1916
720
1360
1193
1963
1962
3022
721
C-205
C-221
C-33

TETRA TECH

Indoor Air and Soil Gas Analytical Results
Site 43 Toluene Disposal Area
Naval Support Facility Indian Head
Indian Head, Maryland

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	FIGURE 2-15	REV	DATE 1/22/13



TETRA TECH

Conceptual Site Model
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G00771	NOT TO SCALE	
FIGURE NO.	2-16	12/10/12	DATE

P:\GIS_files\IndianHead_NSWC\Data\SITE43_CSM indianhead_csm.pptx 12/10/12 MMC

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PZ01 BKGSD01 PZ02 SD/SW04 MW18 PZ03 SP02 SD/SW03 CL02 OF01 MW19S MW19D MW26 SG01 SG04 SD/SW02 PZ04 SG03 SG02 SD/SW01 SP01 MW25 MW23 CL01 MW20 MW21 MW24 MW22 MW13 MW14 MW16 MW02 MW15 MW11 MW17S MW17D MW04 MW03 MW05 MW07 MW01 MW08 MW06 MW12 MW10 MW09S MW09D MW27 MW28 MW29

STRAUSS AVE
LEARY RD
PUMPHOUSE LANE
GALLERY RD
GLENNON RD
JOHNSON RD
VOEGELI RD

715 716 717 1134 1485 1604 730 720 720A 721 722 723 724

Legend

- Proposed Monitoring Well
- Proposed Piezometer
- Proposed Culvert Sample
- Proposed Sediment/Surface Water Sample
- Proposed Outfall Sample
- Proposed Soil Gas Sample
- Monitoring Well
- Seep Sample

0 62.5 125 250 Feet

TETRA TECH

Proposed Sample Location Map
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G01358	SCALE	AS NOTED
FIGURE NO.	FIGURE 3-1	REV	DATE 1/28/13

P:\GIS_files\IndianHead_NSWC\MapDocs\MXD IH_prosamplelocation_zoom.mxd 012813 MC

Bing Maps aerial:
Aerial photograph from ESRI Bing Maps map service
(© 2010 Microsoft Corporation and its data suppliers).

N

BKGSD02
PZ01
BKGSD01
PZ02
SD/SW04
MW18
PZ03
SP02
MW11
SD/SW03
OF01
CL02
MW19S
MW19D
MW17S
MW17D
MW04
SG01
MW26
SG04
SD/SW02
SG03
SG02
PZ04
MW03
SP01
MW25
SD/SW01
MW23
CL01
MW20
MW12
MW24
MW22

STRAUSS AVE
LEARY RD
PUMP HOUSE LANE

714
813
715
1386
740
1579
739
716
717
1755
1134
720
1916
821
1921
1257
1801
1853
1857

Legend

- Proposed Monitoring Well
- Proposed Piezometer
- Proposed Culvert Sample
- Proposed Surface Water/Sediment Sample
- Proposed Outfall Sample
- Proposed Soil Gas Sample
- Monitoring Well
- Seep Sample

0 62.5 125 250 Feet

TETRA TECH

Proposed Sample Location Map
Site 43 Toluene Disposal Area
Naval Support Facility
Indian Head, MD

FILE	112G00771	SCALE	AS NOTED
FIGURE NO.	FIGURE 3-2	REV / DATE	1/28/13

APPENDICES

APPENDIX A

Groundwater - Background and Statistical Evaluation

BACKGROUND COMPARISONS
NSWC INDIAN HEAD
INDIAN HEAD, MARYLAND

1. INTRODUCTION

The comparative statistical method was used to compare the 2012 Site 43 groundwater data to the non-turbid unfiltered groundwater data from the Indian Head background report. The comparative method utilizes several statistical techniques to compare site and background data.

The comparison of the site data with the background data involved a graphical evaluation and a hypothesis test comparing the central tendency (mean/median) concentrations and a hypothesis test comparing the right tails (largest values). The graphical evaluation consisted of visual inspection of boxplots, normal probability plots, and histograms. The statistical package R version 2.15.0 was used to conduct the statistical evaluations. A chemical was included in the background evaluation if it was detected in at least three samples in both the background and site dataset. Having less than three detections limits the ability to compute meaningful statistics therefore these chemicals were not included in the evaluation. The detection limit was used for non-detected concentrations for the graphical displays and the statistical hypothesis tests. The graphical displays and the hypothesis tests are described in Section 2 Statistical Methodology.

2. STATISTICAL METHODOLOGY

This section describes the graphical displays and hypothesis tests used to compare the site dataset to the background dataset.

2.1 Graphical Displays

Boxplots show the central tendency, degree of symmetry, range of variation, and potential outliers of a data set. The data set is shown as a rectangular box that represents the middle 50 percent of the data. The upper value of the box represents the 75th percentile and the lower value of the box represents the 25th percentile. The median is represented by the middle line in the box. Box plots for the same analyte were plotted on the same graph. The plots were visually inspected to see which data sets look similar and which ones differed. Particular attention was paid to see if the median from one data set fell within the 75th and 25th percentile range of the other data sets.

Probability plots are a useful first step for visually comparing two data sets in a single graph. If the site and background distributions were exactly identical, the plotted values would lie on a straight line through the origin. Deviations from this line show the differences between the two distributions. If the site and background distributions are similar the scattering of the two data sets will be mixed. If there is grouping of the two data sets then data sets are most likely different.

Histograms are a visual representation of the data collected into groups. The data range is divided into several bins or classes and the data are sorted into the bins. The x-axis displays the chemical concentration range for the bin and the y-axis shows the number of observations that fall within the bin. The histograms of the site and background datasets were plotted on top of each other to be able to compare the shapes of the two distributions, overall concentration ranges, and ranges of concentrations that have the most samples.

2.2 Hypothesis Tests

Two types of hypothesis tests were conducted one comparing the central tendency of the data sets and one comparing the right tails of the data sets. The Two sample T-test, Wilcoxon Rank Sum test, and the Gehan test used to compare the central tendency of the site and background data sets. The Slippage Test and Quantile test were used to compare the right tails of the site

and background datasets. All hypothesis tests were conducted using a five percent significance level meaning that if the p-value associated with the hypothesis test is less than 0.05 there is statistically significant evidence that the null hypothesis (H_o or assumed hypothesis) is false. The p-value of a test can be thought of as the credibility of the null hypothesis; p-values greater than 0.05 indicate that the null hypothesis is credible whereas p-values less than 0.05 indicate otherwise.

The Two Sample T-Test tests for a difference between two populations means when it can be assumed that the data are approximately normally distributed or sample sizes are large enough (m and n at least 30) and the data are all detected. If the variances of the two data sets are not equal than the Two sample T Test with unequal variance was computed. The null and alternative hypotheses were:

H_o: Site Average ≥ Background Average + Background Standard Deviation
H_A: Site Average < Background Average + Background Standard Deviation

The Wilcoxon Rank-Sum (WRS) nonparametric test is used to test for a difference between median concentrations between two independent populations. The WRS test was used when the data were not normally distributed and there were less than three reporting limits if non-detected concentrations were present. The null and alternative hypotheses were:

H_o: Site Median ≥ Background Media + Background Standard Deviation
H_A: Site Median < Background Median + Background Standard Deviation

The Gehan nonparametric test is used to test for a difference between median concentrations between two independent populations. The Gehan test can be used when the background or site datasets contain multiple nondetects with different reporting limits. The Gehan test was used when the nondetects had more than three reporting limits. The null and alternative hypotheses are the same as the null and alternative hypothesis for the WRS test.

The Slippage Test and Quantile tests are used to test for a shift to the right in the extreme right-tail of the site versus the background concentrations. This is equivalent to asking if a set of the largest values of the site distribution are larger than the maximum value of the background distribution. If the slippage test found the extreme right tail of the site data to be larger than the background data it was concluded that the site concentrations are greater than background. The null and alternative hypothesis were:

H_o: Right Tail of Site Dataset ≤ Right Tail of the Background Dataset
H_A: Right Tail of the Site Dataset > Right Tail of the Background Dataset

3. RESULTS OF BACKGROUND COMPARISONS

This section summarizes the results of the visual comparison of the site and background data sets based on the graphical displays described in Section 2.1 and the results of the hypothesis tests described in Section 2.2.

2012 site 43 metals concentrations were compared to the non-turbid unfiltered Indian Head background concentrations. There were less than three detected concentrations of antimony, arsenic, beryllium, cadmium, calcium, copper, lead, magnesium, mercury, selenium, silver, sodium, thallium, vanadium, and zinc in either the background or the site dataset therefore these chemicals were not evaluated in the background comparisons.

Figure 1 presents the graphical displays for aluminum. From the boxplots, it can be seen that the maximum site concentration falls between the background 75th percentile and the background median, indicating that site concentrations are not elevated above the background concentrations. The normal probability plots shows a mixing of the two data sets indicating that

the two data sets are from a similar population. The site histogram falls in the first bin of the background histogram indicating that site concentrations are not elevated above background concentrations. As presented in Table 1 the Gehan test was used to compare the site and background median concentrations. The p-value for the Test is less than 0.05 indicating that the site median is not greater than the background median by more than one background standard deviation. The Slippage and Quantile tests indicate that the right tail of the site data set is not elevated above the right tail of the background data set. Based on the graphical displays, and hypothesis tests the 2012 Site 43 aluminum groundwater concentrations are consistent with the non-turbid unfiltered Indian Head background concentrations.

Figure 2 presents the graphical displays for barium. From the boxplots, it can be seen that the background median is less than the site 25th percentile, indicating that site concentrations are slightly elevated above background concentrations. The normal probability plot shows a mixing of the two data sets; indicating that the combined data represent the same population. The overlapping histograms have similar shape however the background concentrations fall within the first three bins of the site histogram; indicating that the site data are elevated above the background data. The WRS test was used to compare site median and background median concentrations. The p-value for the WRS test is not less than 0.05 indicating that the site median concentration is greater than the background mean concentration by more than one background standard deviation. The Slippage and Quantile tests indicate that the right tail of the site data set is elevated above the right tail of the background data set. Based on the graphical displays, WRS test, Slippage and Quantile Test the 2012 Site 43 barium groundwater concentrations are greater than the non-turbid unfiltered Indian Head background concentrations.

Figure 3 presents the graphical displays for chromium. From the boxplots, it can be seen that the background 25th is greater than the site 75th percentile, indicating that site concentrations are not elevated above background concentrations. The normal probability plot shows that the background concentrations only fall in the upper tail of the combined datasets indicating that the background data are slightly elevated above the site data. This histograms show that the maximum concentrations of the background data set is elevated above the site data. The Gehan test was used to compare median concentrations. The p-value for the Gehan test is less than 0.05, therefore it can be concluded that site concentrations are not greater than background concentrations by more than one background standard deviation. The Slippage and Quantile test indicate that the right tails of the site data is not shifted above the right tail of the background data. Based on the graphical displays, Gehan test, Slippage and Quantile Test the 2012 Site 43 chromium concentrations are not greater than the non-turbid unfiltered Indian Head background chromium concentrations.

Figure 4 presents the graphical displays for cobalt. From the boxplots, it can be seen that the maximum background concentration is roughly equal to the site median concentration indicating that site concentrations are elevated above background concentrations. The normal probability plot shows that the background concentrations only fall in the lower half of the combined data indicating that the site data are elevated above the background data. The background histograms falls in the first bin of the site histogram indicating that the site concentrations are elevated above the background data. The WRS test was used to compare median concentrations. The p-value for the WRS test is greater than 0.05, therefore it can be concluded that site concentrations are greater than background concentrations by more than one background standard deviation. The Slippage and Quantile test indicate that the right tails of the site data is shifted above the right tail of the background data. Based on the graphical displays, WRS test, Slippage and Quantile test it can be concluded that the 2012 Site 43 groundwater cobalt concentrations are greater than the non-turbid background cobalt groundwater concentrations.

Figure 5 presents the graphical displays for iron. From the boxplots, it can be seen that the background and site 25th percentile and median are roughly equal however the site 75th percentile is greater than the maximum background concentrations, indicating that site concentrations are

greater than the background concentrations. The normal probability plot shows a mixing of the two data sets. The background histograms falls in the lower half of the site histogram indicating that the site concentrations are elevated above the background concentrations. The WRS test was used to compare site median and background median concentrations. The p-value for the WRS test is greater than 0.05 indicating that the site median is greater than the background median by more than one background standard deviation. The Slippage and Quantile tests indicate that the right tail of the site data set is elevated above the right tail of the background data set. Based on the graphical displays, WRS Test, Slippage and Quantile Test the 2012 Site 43 iron groundwater concentrations are greater than the non-turbid background iron concentrations.

Figure 6 presents the graphical displays for manganese. From the boxplots, it can be seen that the background median falls between the Site 25th percentile and median; indicating that site concentrations are similar to background concentrations. The normal probability plot shows a mixing of the two datasets indicating that the background and site concentrations are similar. The two histograms have similar spread indicating that the two datasets are similar. The T-Test was used to compare mean concentrations. The p-value for the T-Test test is less than 0.05, therefore it can be concluded that site concentrations are not greater than background concentrations by more than one background standard deviation. The Slippage and Quantile test indicate that the right tail of the site data is not shifted above the right tail of the background data. Therefore based on the graphical displays and all hypothesis tests 2012 Site 43 manganese concentrations are similar to the non-turbid unfiltered groundwater background concentrations.

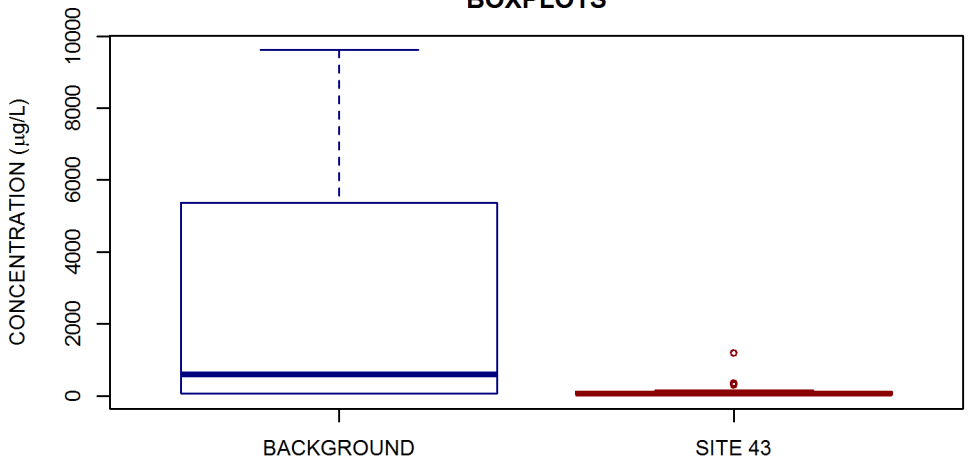
Figure 7 presents the graphical displays for nickel. From the boxplots, it can be seen that the background median falls between the site 25th percentile and median, indicating that site concentrations are slightly elevated above background concentrations. The normal probability plot shows a mixing of the two data sets with no inflection point; indicating that the combined data represent the same population. The background histogram falls in the first half of the site histogram indicating that the site concentrations are elevated above background. The WRS test was used to compare site median and background median concentrations. The p-value for the WRS test is not less than 0.05 indicating that the site median concentration is greater than the background median concentration by more than one background standard deviation. The Slippage and Quantile tests indicate that the right tail of the site data set is elevated above the right tail of the background data set. Based on the graphical displays, WRS test, Slippage and Quantile Test the 2012 Site 43 nickel groundwater concentrations are greater than the non-turbid unfiltered Indian Head background concentrations.

4. CONCLUSION

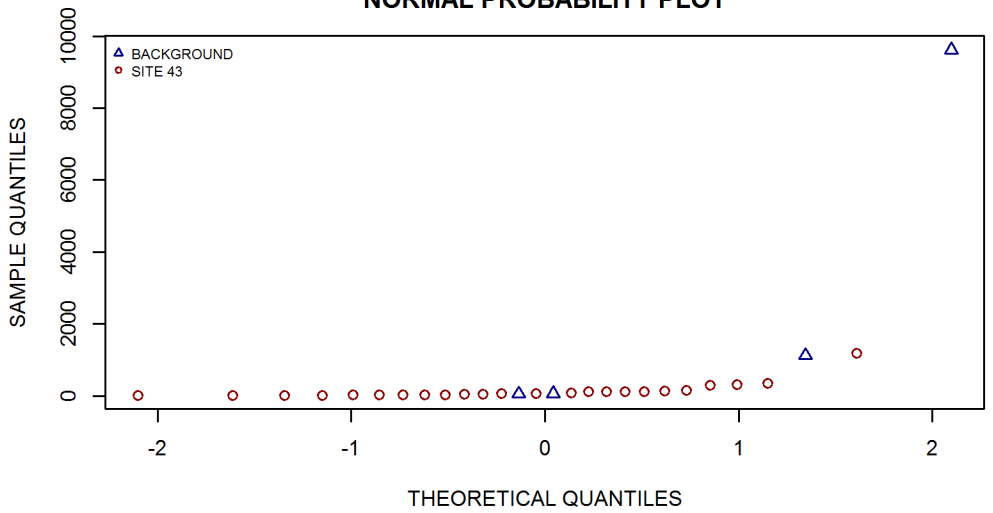
The comparative statistical method was used to 2012 Site 43 groundwater data to the non-turbid unfiltered groundwater data. The background comparison involved a graphical evaluation and a hypothesis test comparing the central tendency (mean/median) concentrations and a hypothesis test comparing the right tails (largest values). The graphical evaluation consisted of visual inspection of boxplots, normal probability plots, and histograms. Based on the evaluations 2012 Site 43 concentrations of aluminum, chromium, and manganese are similar to the non-turbid unfiltered Indian Head background concentrations while the 2012 Site 43 concentrations of barium, cobalt, iron, and nickel were greater than the non-turbid unfiltered Indian Head background concentrations.

ALUMINUM GRAPHICAL DISPLAYS
SITE 43
NSWC INDIAN HEAD, INDIAN HEAD, MARYLAND
FIGURE 1 OF 7

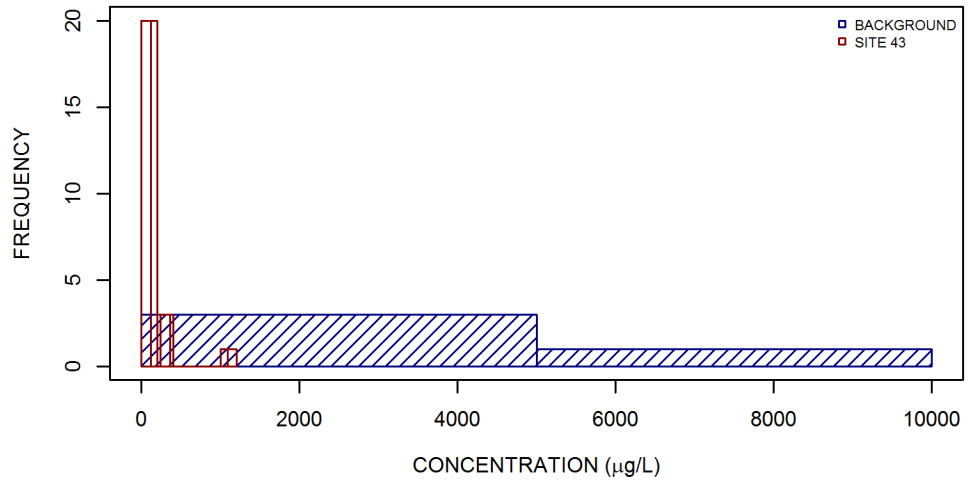
BOXPLOTS



NORMAL PROBABILITY PLOT



HISTOGRAMS



SUMMARY STATISTICS

	BACKGROUND	SITE 43
MINIMUM	63	15
25TH PERCENTILE	69	27
MEDIAN	600	64
MEAN	2700	140
75TH PERCENTILE	3300	130
MAXIMUM	9600	1200

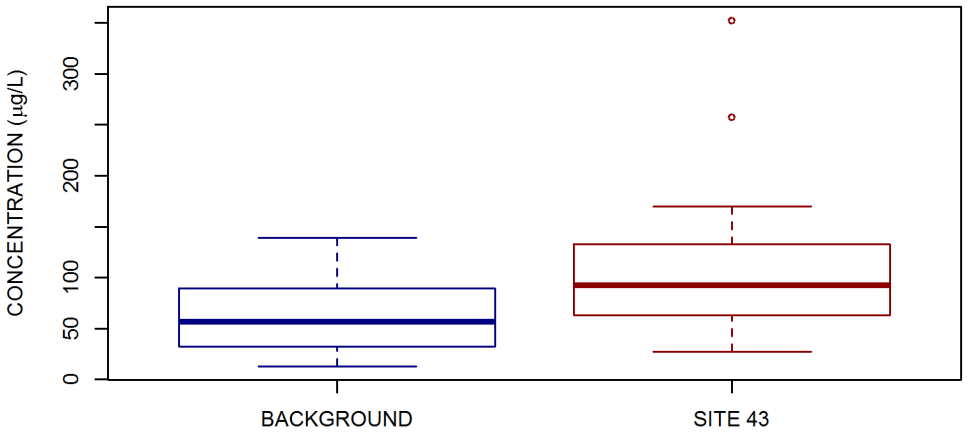
UNITS ARE µg/L

DETECTION LIMIT USED FOR NON-DETECTS

SUMMARY STATISTICS ROUNDED TO TWO SIGNIFICANT FIGURES

BARIUM GRAPHICAL DISPLAYS
SITE 43
NSWC INDIAN HEAD, INDIAN HEAD, MARYLAND
FIGURE 2 OF 7

BOXPLOTS



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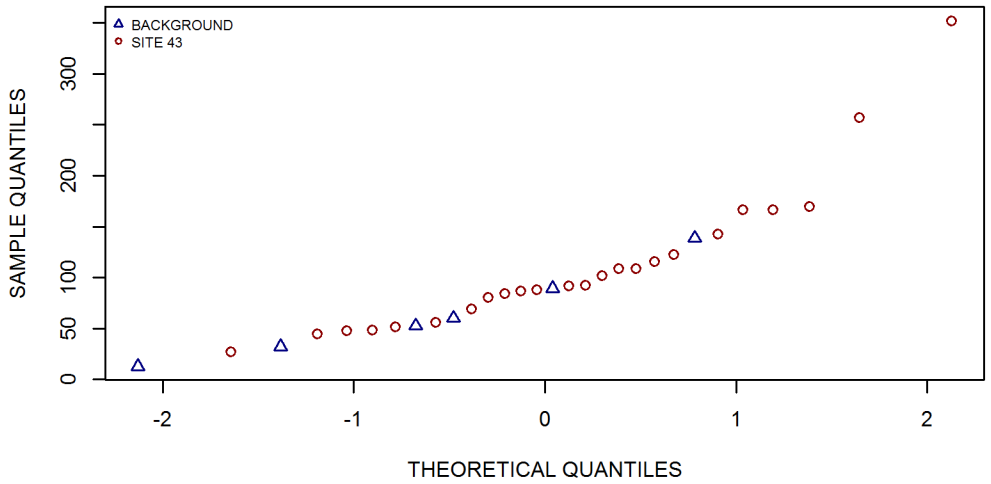

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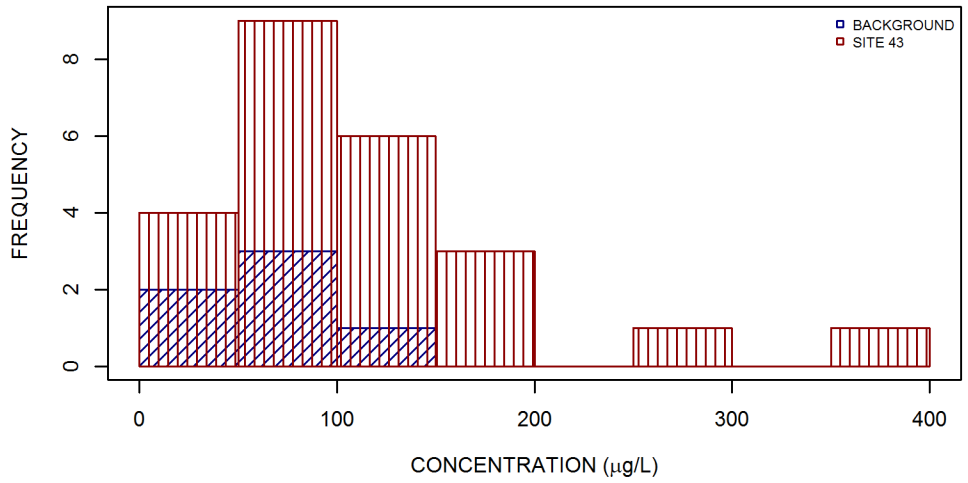


SUMMARY STATISTICS

	BACKGROUND	SITE 43
MINIMUM	13	27
25TH PERCENTILE	38	66
MEDIAN	57	92
MEAN	65	110
75TH PERCENTILE	82	130
MAXIMUM	140	350

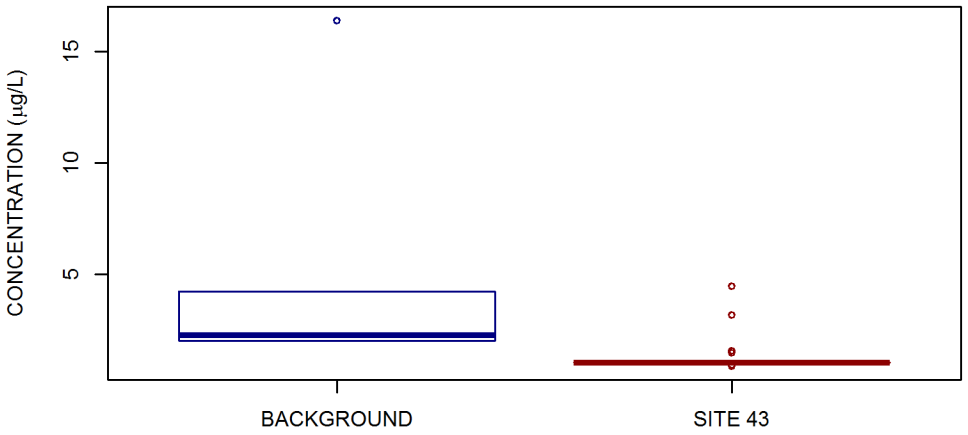
UNITS ARE µg/L

DETECTION LIMIT USED FOR NON-DETECTS

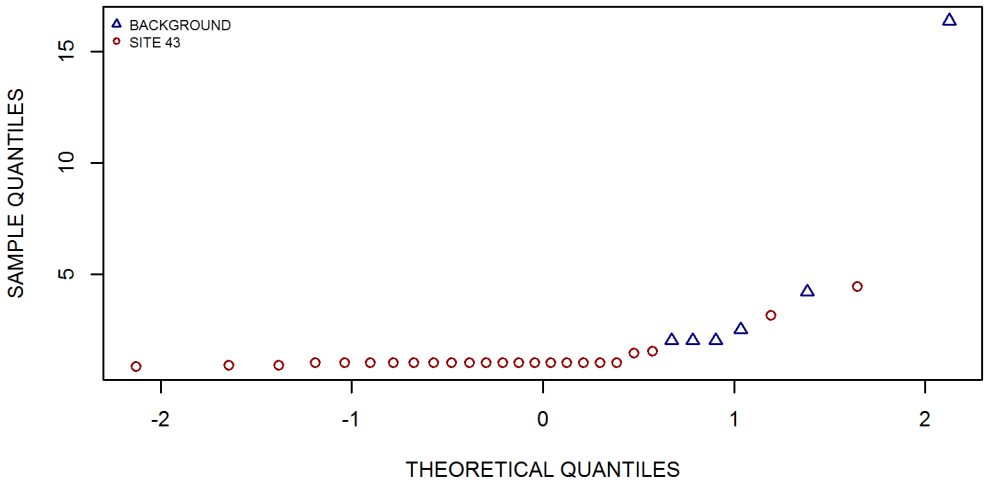
SUMMARY STATISTICS ROUNDED TO TWO SIGNIFICANT FIGURES

CHROMIUM GRAPHICAL DISPLAYS
SITE 43
NSWC INDIAN HEAD, INDIAN HEAD, MARYLAND
FIGURE 3 OF 7

BOXPLOTS



NORMAL PROBABILITY PLOT



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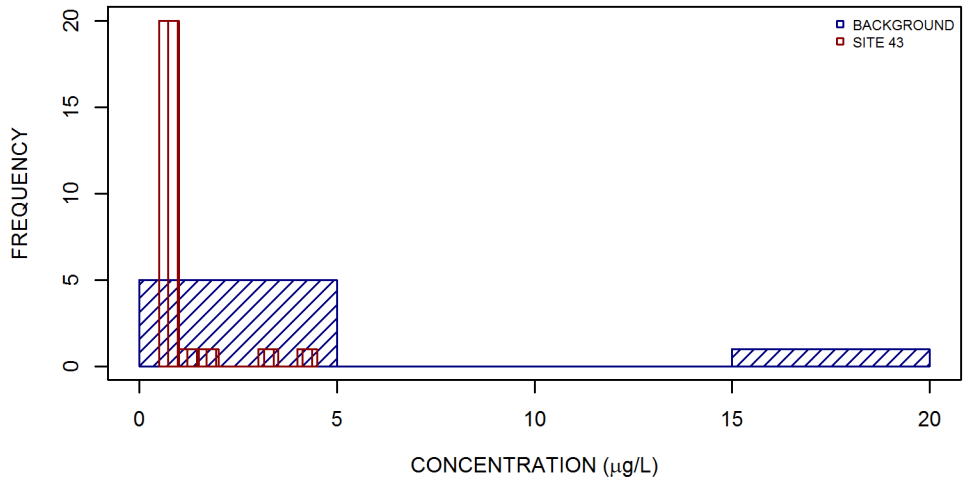


SUMMARY STATISTICS

	BACKGROUND	SITE 43
MINIMUM	2	0.84
25TH PERCENTILE	2	1
MEDIAN	2.2	1
MEAN	4.8	1.3
75TH PERCENTILE	3.8	1
MAXIMUM	16	4.4

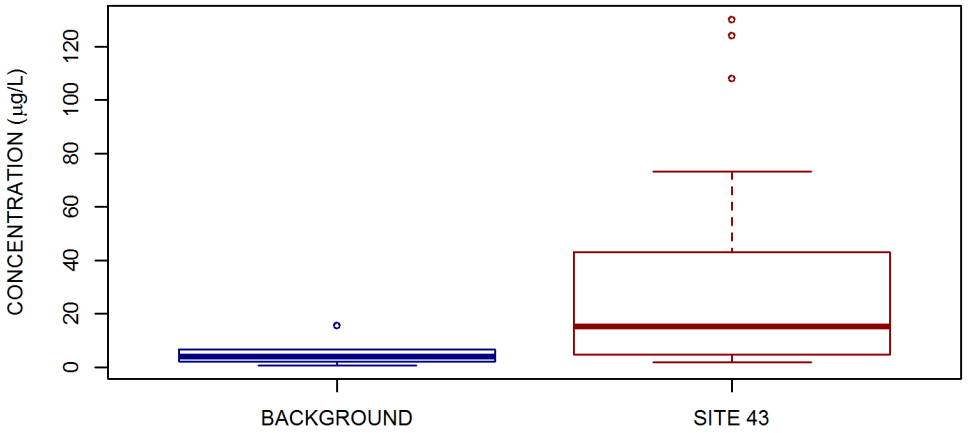
UNITS ARE µg/L

DETECTION LIMIT USED FOR NON-DETECTS

SUMMARY STATISTICS ROUNDED TO TWO SIGNIFICANT FIGURES

COBALT GRAPHICAL DISPLAYS
SITE 43
NSWC INDIAN HEAD, INDIAN HEAD, MARYLAND
FIGURE 4 OF 7

BOXPLOTS



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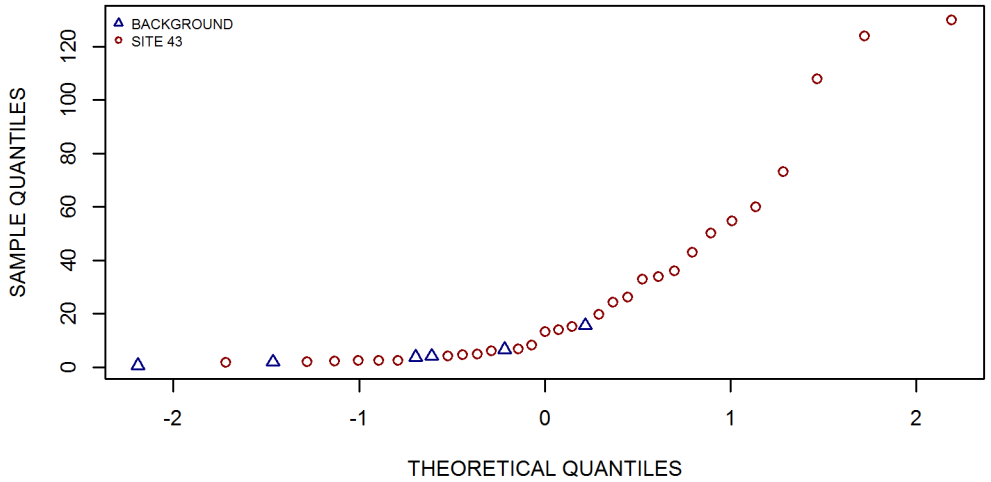

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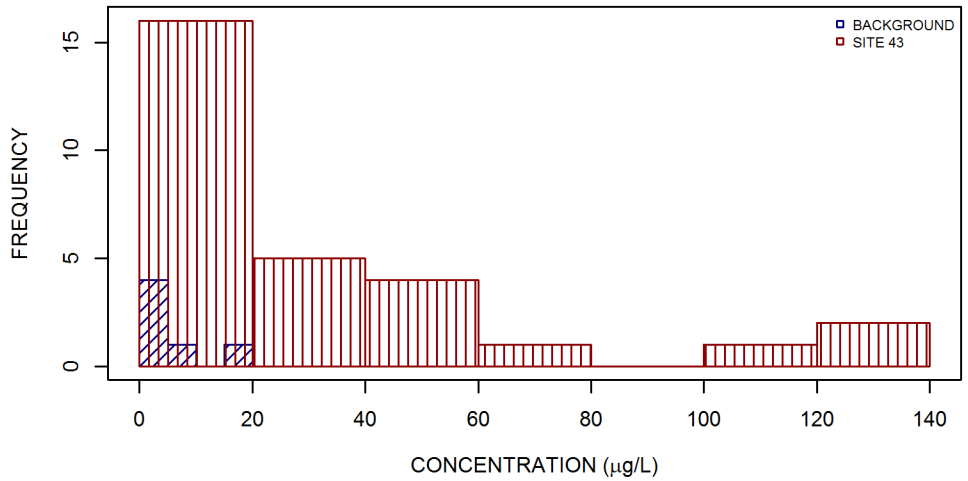


SUMMARY STATISTICS

	BACKGROUND	SITE 43
MINIMUM	0.7	2
25TH PERCENTILE	2.5	4.8
MEDIAN	4	15
MEAN	5.5	31
75TH PERCENTILE	6.1	43
MAXIMUM	16	130

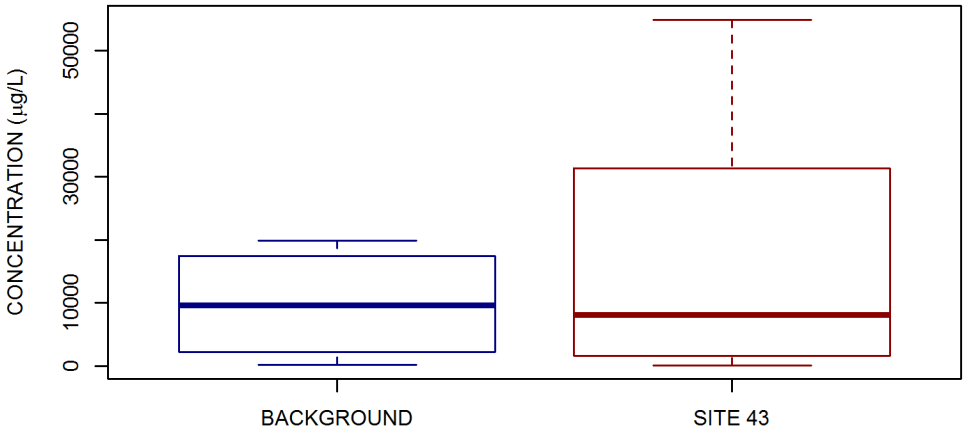
UNITS ARE µg/L

DETECTION LIMIT USED FOR NON-DETECTS

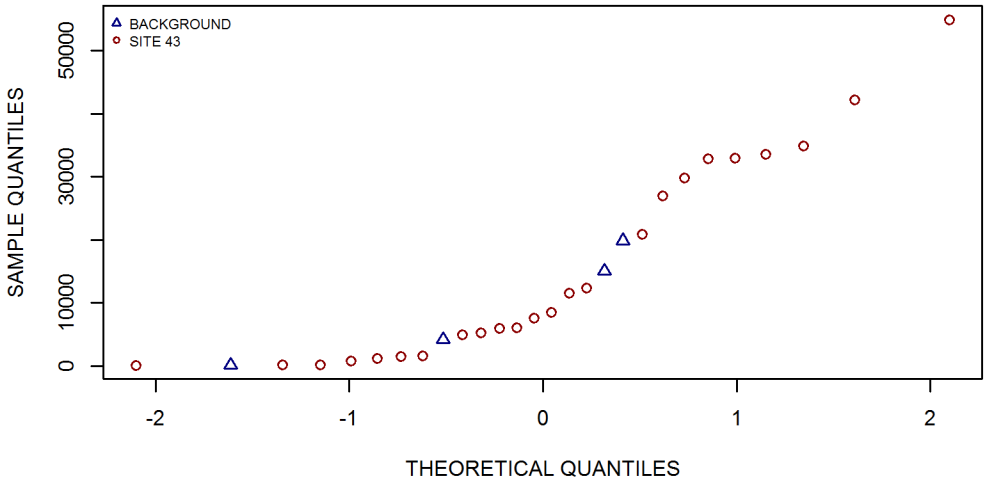
SUMMARY STATISTICS ROUNDED TO TWO SIGNIFICANT FIGURES

IRON GRAPHICAL DISPLAYS
SITE 43
NSWC INDIAN HEAD, INDIAN HEAD, MARYLAND
FIGURE 5 OF 7

BOXPLOTS



NORMAL PROBABILITY PLOT



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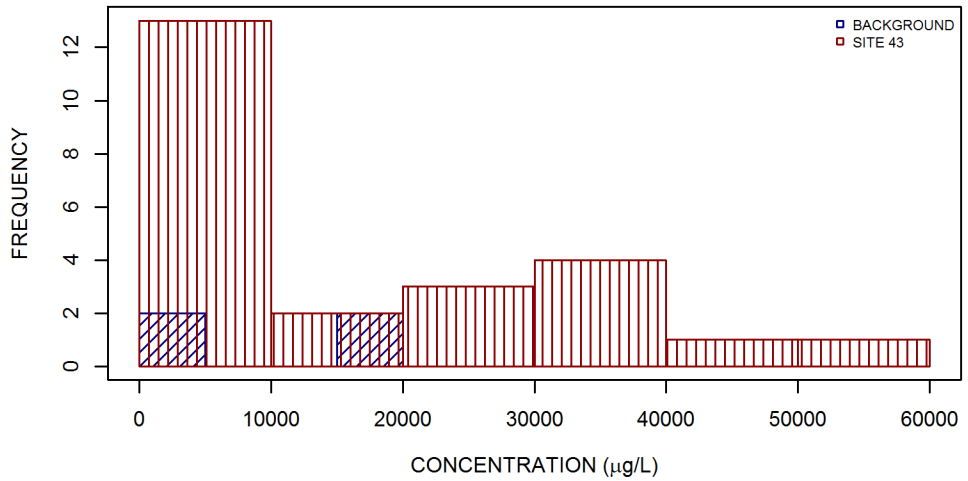


SUMMARY STATISTICS

	BACKGROUND	SITE 43
MINIMUM	160	120
25TH PERCENTILE	3200	1600
MEDIAN	9700	8100
MEAN	9800	16000
75TH PERCENTILE	16000	31000
MAXIMUM	20000	55000

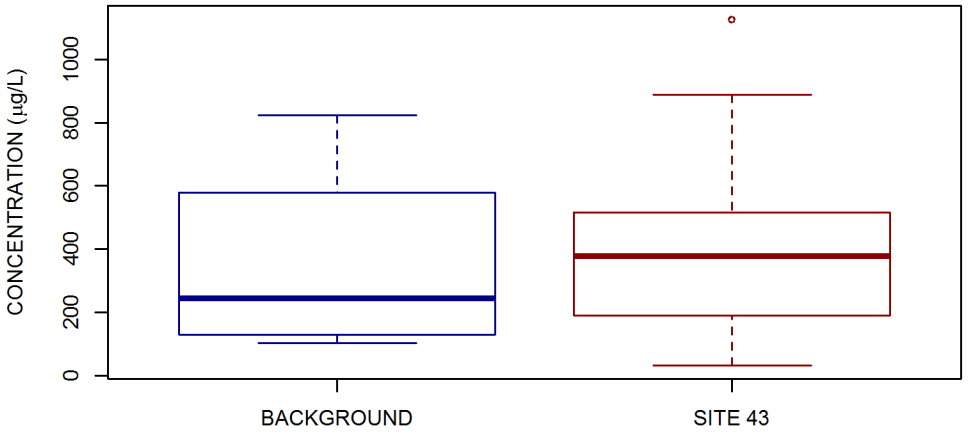
UNITS ARE µg/L

DETECTION LIMIT USED FOR NON-DETECTS

SUMMARY STATISTICS ROUNDED TO TWO SIGNIFICANT FIGURES

MANGANESE GRAPHICAL DISPLAYS
SITE 43
NSWC INDIAN HEAD, INDIAN HEAD, MARYLAND
FIGURE 6 OF 7

BOXPLOTS



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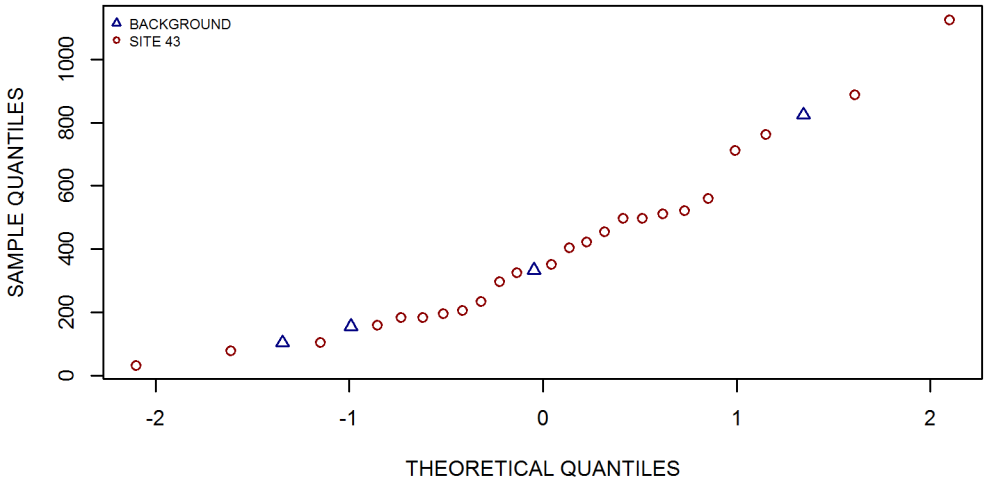

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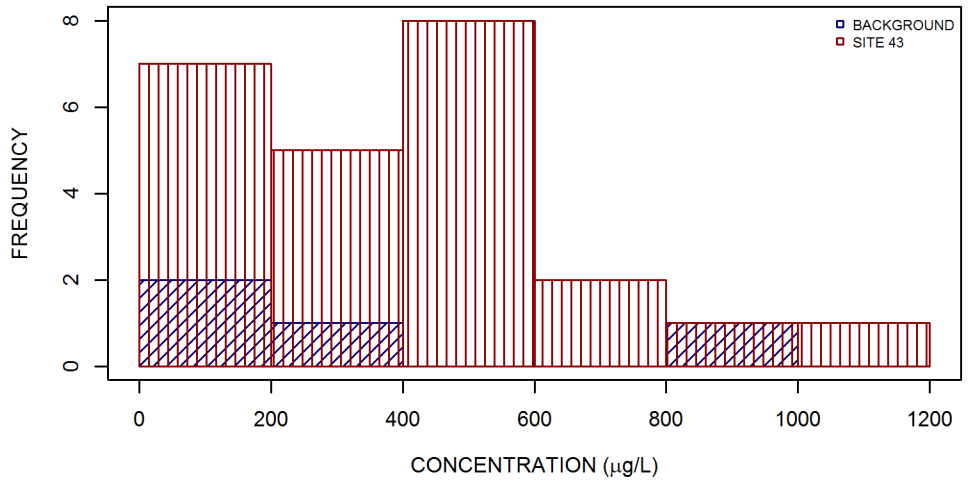


SUMMARY STATISTICS

	BACKGROUND	SITE 43
MINIMUM	100	33
25TH PERCENTILE	140	190
MEDIAN	240	380
MEAN	350	400
75TH PERCENTILE	460	510
MAXIMUM	820	1100

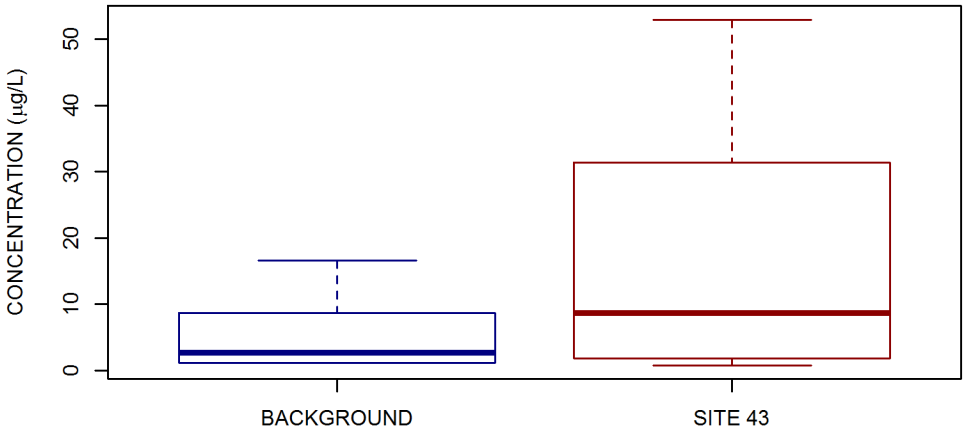
UNITS ARE µg/L

DETECTION LIMIT USED FOR NON-DETECTS

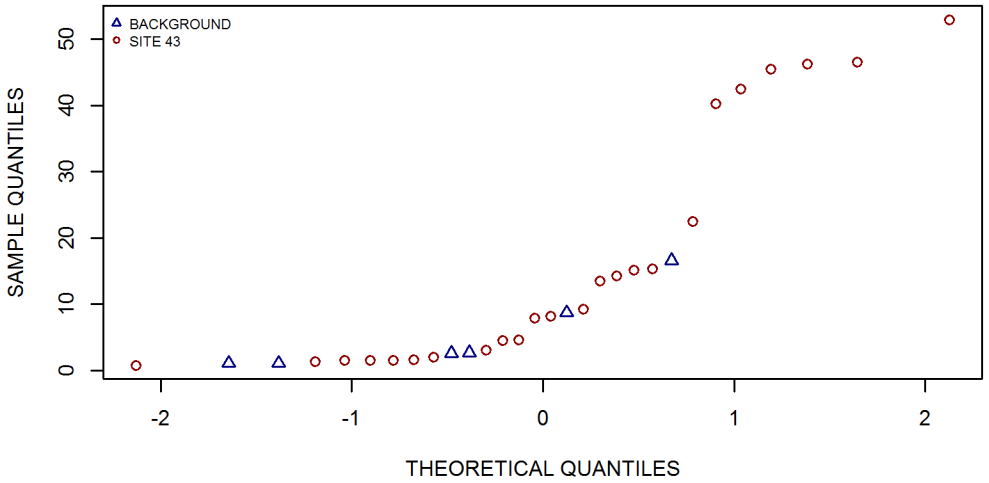
SUMMARY STATISTICS ROUNDED TO TWO SIGNIFICANT FIGURES

NICKEL GRAPHICAL DISPLAYS
SITE 43
NSWC INDIAN HEAD, INDIAN HEAD, MARYLAND
FIGURE 7 OF 7

BOXPLOTS



NORMAL PROBABILITY PLOT



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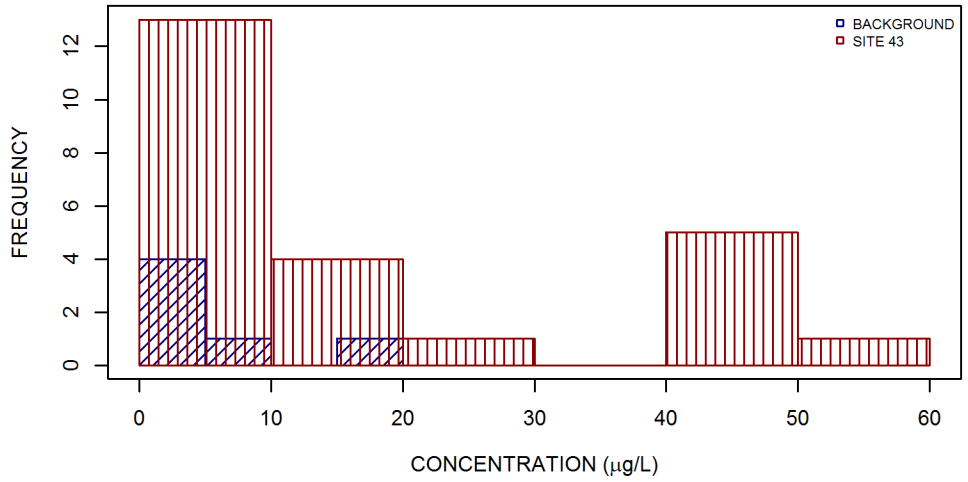

SUMMARY STATISTICS

	BACKGROUND	SITE 43
MINIMUM	1.1	0.76
25TH PERCENTILE	1.5	1.9
MEDIAN	2.6	8.7
MEAN	5.5	17
75TH PERCENTILE	7.2	27
MAXIMUM	17	53

UNITS ARE µg/L

DETECTION LIMIT USED FOR NON-DETECTS

SUMMARY STATISTICS ROUNDED TO TWO SIGNIFICANT FIGURES

TABLE 1
COMPARISON OF 2012 SITE 43 GROUNDWATER DATA TO NON-TURBID UNFILTERED BACKGROUND GROUNDWATER
NSWC INDIAN HEAD, INDIAN HEAD, MARYLAND

Parameter	Site FOD	Background FOD	Site Maximum Detected Concentration (µg/L)	Background Maximum Detected Concentration (µg/L)	Site Distribution	Background Distribution	Hypothesis Test	Hypothesis Test P-value	Hypothesis Test Conclusion	Slippage Test Conclusion	Quantile Test Conclusion	Site Greater than Background?
ALUMINUM	21 / 24	3 / 4	1190	9620	Nonparametric	Nonparametric	Gehan	0.0065	Site Consistent with Background	Site Consistent with Background	Site Consistent with Background	NO
ANTIMONY	0 / 24	0 / 6	--	--	Assumed Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
ARSENIC	12 / 24	0 / 6	12.2	--	Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
BARIUM	24 / 24	6 / 6	352	139	Nonparametric	Normal	WRS	0.3487	Site Greater than Background	Site Greater than Background	Site Greater than Background	YES
BERYLLIUM	14 / 24	0 / 6	1.945	--	Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
CADMIUM	5 / 24	1 / 6	0.909	2.8	Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	
CALCIUM	0 / 0	4 / 4	--	18100	Assumed Nonparametric	Normal	No Hypothesis Test	NA	NA	NA	NA	NA
CHROMIUM	7 / 24	3 / 6	4.45	16.4	Nonparametric	Nonparametric	Gehan	0.0038	Site Consistent with Background	Site Consistent with Background	Site Consistent with Background	NO
COBALT	26 / 29	5 / 6	130	15.6	Nonparametric	Normal	WRS	0.7513	Site Greater than Background	Site Greater than Background	Site Greater than Background	YES
COPPER	12 / 24	2 / 6	8.18	22.4	Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
IRON	24 / 24	4 / 4	54900	19900	Nonparametric	Normal	WRS	0.2134	Site Greater than Background	Site Greater than Background	Site Greater than Background	YES
LEAD	2 / 24	0 / 6	0.9325	--	Assumed Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
MAGNESIUM	0 / 0	4 / 4	--	3980	Assumed Nonparametric	Normal	No Hypothesis Test	NA	NA	NA	NA	NA
MANGANESE	24 / 24	4 / 4	1125	824	Normal	Normal	T-Test	0.03641	Site Consistent with Background	Site Consistent with Background	Site Consistent with Background	NO
MERCURY	0 / 24	1 / 6	--	0.13	Assumed Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
NICKEL	21 / 24	4 / 6	52.9	16.6	Nonparametric	Nonparametric	WRS	0.4077	Site Greater than Background	Site Greater than Background	Site Greater than Background	YES
SELENIUM	0 / 24	0 / 6	--	--	Assumed Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
SILVER	0 / 24	0 / 6	--	--	Assumed Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
SODIUM	0 / 0	4 / 4	--	33000	Assumed Nonparametric	Normal	No Hypothesis Test	NA	NA	NA	NA	NA
THALLIUM	1 / 24	0 / 6	0.801	--	Assumed Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
VANADIUM	17 / 24	1 / 6	6.68	20.9	Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA
ZINC	24 / 24	1 / 6	91.75	45.2	Nonparametric	Assumed Nonparametric	No Hypothesis Test	NA	NA	NA	NA	NA

Hypothesis tests were only computed if there were at least three detected concentrations in both the site and background datasets.

APPENDIX B

Revised UFP SAP Worksheets

WS #3 -- Distribution List

WS #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Each person in this table will be responsible for distributing copies of the SAP to appropriate personnel within their organization. For example, the Tetra Tech PM will be responsible for distributing copies of the SAP to all Tetra Tech personnel listed in **WS #4** (*Project Personnel Sign-Off Sheet*).

Name of SAP Recipients	Title / Role	Organization	Telephone Number (Optional)	Email Address or Mailing Address	Document Control Number (Optional)
Joe Rail	Navy Remedial Project Manager (RPM)	NAVFAC Washington	202-685-3105	joseph.rail@navy.mil	N/A
Nicholas Carros	NSF-IH ERP Lead / Facility Point of Contact (POC)	NAVFAC Washington	301-744-2263	nicholas.carros@navy.mil	N/A
Dennis Orenshaw	EPA Region 3 RPM	EPA Region 3	215-814-3361	orenshaw.dennis@epa.gov	N/A
Curtis DeTore	MDE RPM	MDE	410-537-3791	cdetore@mde.state.md.us	N/A
John Tucker (electronic only)	Navy Quality Assurance Officer (QAO)	NAVFAC Atlantic	757-322-8288	jonathan.tucker@navy.mil	N/A
Bonnie Capito (copy of final cover letter)	Librarian, Records Manager, Administrative Record Manager	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil	N/A
Scott Nesbit	Tetra Tech Activity Coordinator	Tetra Tech	412-921-8945	scott.nesbit@tetratech.com	N/A
Rob Sok	Tetra Tech Project Manager (PM)	Tetra Tech	757-466-4904	rob.sok@tetratech.com	N/A
tbd	Field Operations Leader (FOL) and Site Safety Officer (SSO)	Tetra Tech	tbd	tbd	N/A
Kelly Carper (electronic only)	Project Chemist	Tetra Tech	412-921-7273	kelly.carper@tetratech.com	N/A
Lee Leck (electronic only)	Data Manager and Database Manager	Tetra Tech	412-921-8856	lee.leck@tetratech.com	N/A
Joe Samchuck (electronic copy only)	Data Validation (DV) Manager (DVM)	Tetra Tech	412-921-8510	joseph.samchuck@tetratech.com	N/A
Glenn Wagner	NAVFAC Atlantic Administrative Record PM	Tetra Tech	412-921-8386	glenn.wagner@tetratech.com	N/A

Name of SAP Recipients	Title / Role	Organization	Telephone Number (Optional)	Email Address or Mailing Address	Document Control Number (Optional)
Tom Johnston (cover letter)	Navy CLEAN Quality Assurance Manager (QAM)	Tetra Tech	412-921-8615	tom.johnston@tetratech.com	N/A
John Trepanowski (cover letter)	Navy CLEAN Program Manager	Tetra Tech	610-491-9688	john.trepanowski@tetratech.com	N/A
Matt Soltis (electronic only)	Health and Safety (H&S) Manager (HSM)	Tetra Tech	412-921-8912	matt.soltis@tetratech.com	N/A
RAB Members (Final, electronic version only)	NSF-IH Restoration Advisory Board (RAB) Members	Indian Head local residential community	Various	Various	N/A
Ausha Scott (electronic only)	Laboratory Subcontractor PM	Eurofins - Air Toxics	916-985-1000 X 1044	AScott@airtoxics.com	N/A
Mark Dobday (electronic only)	Laboratory Subcontractor PM	Geotesting Express	978-635-0424	mdobday@geotesting.com	N/A
Brian Richard (electronic copy only)	Empirical Laboratories, LLC (Empirical) PM	Empirical	615- 345-1115	brichard@empirilabs.com	N/A
tbd (electronic only)	Utility Location Subcontractor	tbd	tbd	tbd	N/A
tbd (electronic only)	Drilling Subcontractor	tbd	tbd	tbd	N/A
tbd (electronic only)	Direct Push Technology (DPT) / Membrane Interface Probe (MIP) Subcontractor	tbd	tbd	tbd	N/A
tbd (electronic only)	Survey Subcontractor	tbd	tbd	tbd	N/A
tbd (electronic only)	Investigation-Derived Waste (IDW) Management Subcontractor	tbd	tbd	tbd	N/A

WS #4 -- Project Personnel Sign-Off Sheet

WS #4 -- Project Personnel Sign-Off Sheet

(UFP-QAPP Manual Section 2.3.2)

Certification that project personnel have read the text will be obtained by one of the following methods as applicable:

- In the case of regulatory agency personnel with oversight authority, approval letters or emails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters / emails will be retained in the project files as project records (see **WS #29**).
- Emails will be sent to the listed Navy, Tetra Tech, and subcontractor project personnel who will be requested to verify by email that they have read the applicable SAP sections and the date on which they were reviewed. Copies of the verification email will be included in the project files (see **WS #29**).
- A copy of this [signed] WS will be retained in the project files and identified as a project document in **WS #29**.

Key personnel will be instructed to read the SAP prior to attending an internal Site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and ensure that the completed sign-off sheet is included in the central project file.

Name [1]	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulatory Project Team					
Joe Rail	NAVFAC Washington / Navy RPM/Manages project	202-685-3105	*See **WS #1** for signature*	All	
Nicholas Carros	NAVFAC Washington / Facility POC/Manages project	301-744-2263		All	
Dennis Orenshaw	EPA Region 3 / Federal Regulatory RPM/ Manages project	215-814-3361		All	
Curtis DeTore	MDE / State Regulatory RPM/Manages project	410-537-3791		All	
Tetra Tech Project Team					
Scott Nesbit	Tetra Tech Activity Coordinator/Coordinates Tetra Tech activities at this facility	412-921-8945		All	
Rob Sok	Tetra Tech PM/ Manages project	757-466-4904	*See **WS #1** for signature*	All	
tbd	Tetra Tech / FOL and SSO/As FOL, manages field work; as SSO, oversees safety operations in the field	tbd		All	
Kelly Carper	Tetra Tech Project Chemist/oversees chemistry-related activities	412-921-7273		All	

Name [1]	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Joe Samchuck	Tetra Tech / DVM/manages data validation activities	412-921-8510		**WS #12, 14, 15, 19, 20, 23-28, 30, and 34-37**	
Lee Leck	Tetra Tech / Data Manager and Database Manager/ Manages database operations	412-921-8856		**WS #12, 14, 15, 19, 20, 23-28, 30, and 34-37**	
Matt Soltis	Tetra Tech / HSM/Manages Tetra Tech corporate Health and Safety Program	412-921-8912	*See H&S Plan (HASP) in* **Appendix A** *for signature* [2]	All	
Tom Johnston	Tetra Tech / Navy CLEAN QAM/Manages quality assurance activities for Tetra Tech CLEAN program	412-921-8615	*See* ***WS #1*** *for signature*	All	
Subcontractors					
Ausha Scott	Air Toxics Ltd. / PM/Manages daily project activities	916-605-3410		**WS #6, 12, 14, 15, 19, 20, 23-28, 30, 34-36**	
Mark Dobday	Geotesting Express / PM/ Manages daily project activities	978-635-0424			
Brian Richard	Laboratory PM/Manages project for Empirical	615-345-1115			
tbd	tbd / Utility Location Subcontractor/ Manages daily project activities	tbd		**WS #6, 14, 17,** and **Figures**	
tbd	tbd / Drilling Subcontractor/ Manages daily project activities	tbd		**WS #6, 14, 17**, and **Figures**	
tbd	tbd / DPT / MIP Subcontractor/ Manages daily project activities	tbd			
tbd	tbd / Survey Subcontractor/ Manages daily project activities	tbd		**WS #14** and **Figures**	

Name [1]	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
tbd	tbd / IDW Management Subcontractor/ Manages daily project activities	tbd		**WS #14**	

1. Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organization.
2. The HSM will be responsible for developing the Site-specific HASP based on field activities presented in this SAP.

WS #6 -- Communication Pathways

WS #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or email	Procedure
SAP amendments	Tetra Tech FOL	tbd	tbd	Tetra Tech FOL will verbally inform Tetra Tech PM within 24 hours of realizing a need for an amendment.
Changes in schedule	Tetra Tech PM	Rob Sok	757-466-4904	Tetra Tech PM will document the changes within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change.
	NAVFAC RPM	Joe Rail	202-685-3105	NAVFAC RPM will send scope change to Tetra Tech Program office before work is started.
	Tetra Tech FOL	tbd	tbd	Tetra Tech PM will verbally inform Navy RPM and Facility POC on the day that schedule change is known and document via schedule impact letter within 1 business-day of when impact is realized.
Issues in the field that result in changes in scope of field work	Tetra Tech PM	Rob Sok	757-466-4904	Tetra Tech FOL will verbally inform Tetra Tech PM within 1 business-day of when the issue is discovered.
	NAVFAC RPM	Joe Rail	202-685-3105	
	Facility POC	Nicholas Carros	301-744-2263	
	Tetra Tech FOL	tbd	tbd	
	Tetra Tech PM	Rob Sok	757-466-4904	Tetra Tech PM will inform Navy RPM (verbally or via email) by close of the next working day.
	NAVFAC RPM	Joe Rail	202-685-3105	NAVFAC RPM will issue scope change approval (verbally or via email at RPM discretion). If warranted (as determined by the NAVFAC RPM), scope change will be documented before work is executed. The Tetra Tech FOL will document the changes on a FTMR form within 2 days of identifying the need for change and obtain required approvals within five days of initiating the form.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or email	Procedure
Recommendations to stop work and initiate work upon corrective action (CA)	Tetra Tech FOL and SSO	tbd	tbd	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onSite personnel, subcontractor(s), the Navy RPM, the Facility POC, and the identified Project Team members within 1 hour (verbally or by email). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.
	Tetra Tech PM	Rob Sok	757-466-4904	
	Tetra Tech QAM	Tom Johnston	412-921-8615	
	Tetra Tech HSM	Matt Soltis	412-921-8912	
	NAVFAC RPM	Joe Rail	202-685-3105	
	Facility POC	Nicholas Carros	301-744-2263	
Corrective Action for field program	Tetra Tech QAM	Tom Johnston	412-921-8615	Tetra Tech QAM will notify Tetra Tech PM within one business day that the Corrective Action has been completed. Tetra Tech PM will then notify the Navy RPM within one business day
	Tetra Tech PM	Rob Sok	757-466-4904	
Field data quality issues	Tetra Tech FOL and SSO	tbd	tbd	Tetra Tech FOL will inform Tetra Tech PM verbally or by email (at FOL discretion) on the same day that a field data quality issue is discovered.
	Tetra Tech PM	Rob Sok	757-466-4904	
Analytical laboratory data quality issues	Laboratory PM	Ausha Scott Mark Dobday Brian Richard	916-605-3410 978-635-0424 615-345-1115	Laboratory PM will notify (verbally or via email as PM discretion) Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered.
	Project Chemist	Kelly Carper	412-921-7273	Tetra Tech Project Chemist will notify (verbally or via email at project chemist discretion) DV staff and Tetra Tech PM within one business day

WS #7 -- Personnel Responsibilities Table

WS #7 -- Personnel Responsibilities Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title / Role	Organizational Affiliation	Responsibilities
Joe Rail	NAVFAC Washington Remedial Project Manager/ Manages project f	Navy	Oversees project implementation, including scoping, data review, and evaluation.
Nicholas Carros	NAVFAC Washington / Serves as point of contact for fieldwork	Navy	Supports Navy RPM role and responsibilities; is Facility Point of Contact (POC) for scheduling and coordinating fieldwork.
Dennis Orenshaw	Remedial Project Manager / Manages project	EPA Region 3	Participates in scoping, data review, and evaluation.
Curtis DeTore	Remedial Project Manager / Manages project	MDE	Participates in scoping, data review, evaluation, and approves the SAP.
Scott Nesbit	Tetra Tech Activity Coordinator/Coordinates all facility work for Tetra Tech	Tetra Tech	Oversees all Tetra Tech project related work at the facility.
Tom Johnston	QAM / Oversees program and project quality assurance activities	Tetra Tech	Ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Kelly Carper	Project Chemist / Conducts data validation and reporting	Tetra Tech	Participates in project scoping, prepares laboratory scope of work (SOW), and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and quality assurance of data validation deliverables.
Rob Sok	PM / Manages project on a daily basis	Tetra Tech	Oversees project, financial, schedule, and technical day to day management of the project
tbd	FOL / Manages field operations	Tetra Tech	Supervises, coordinates, and performs field sampling activities.
	SSO / Oversees Site activities to ensure safety requirements are met		Responsible for on-Site project specific health and safety training and monitoring Site conditions. Details of these responsibilities are presented in the HASP.
Joseph Samchuck	DVM / Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including: • Ensure quality assurance of data validation deliverables. • Provide technical advice on data usability. • Coordinate and maintain data validation review schedule.

Name	Title / Role	Organizational Affiliation	Responsibilities
Lee Leck	Data Manager / Manages analytical and applicable field data	Tetra Tech	Manages Tetra Tech databases and ensures correct input of data.
Matt Soltis	HSM / Oversees H&S activities	Tetra Tech	Oversees Tetra Tech's Navy CLEAN Program H&S
Ausha Scott	Laboratory PM / Manages project	Air Toxics Ltd.	Coordinates analyses with laboratory chemists, lower tier subcontract laboratories, ensures laboratory SOW is followed, provides QA of data packages, and communicates with Tetra Tech project staff
Mark Dobday		Geotesting Express	
Brian Richard		Empirical	
tbd	Utility Subcontractor / Performs utility mark-out	tbd	Performs third-party utility clearance and mark-out (in addition to Miss Utility) at and near intrusive work according to SOW.
tbd	Drilling Subcontractor / Performs drilling activities	tbd	Performs drilling techniques to advance soil borings and installs monitoring wells according to SOW.
tbd	DPT / MIP Subcontractor / Performs DPT and MIP activities	tbd	Performs soil boring, macrocoring / soil sampling, air sampling preparation, and MIP data collection techniques according to SOW.
tbd	IDW Subcontractor / Handles IDW	tbd	Responsible for transport and disposal of IDW according to SOW.
tbd	Survey Subcontractor / Surveys locations	tbd	Determines location coordinates and elevation data for monitoring wells according to SOW.

In some cases, one person may be designated responsibilities for more than one position. For example, the FOL may also be responsible for SSO duties. This action will be performed only as credentials, experience, and availability permits.

WS #23 -- Analytical SOP References Table

WS #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Variance to QSM? (Y/N)	Modified for Project Work? (1)
Empirical SOP-100	Metals Digestion/ Preparation, Methods 3005A/ USEPA CLP ILMO 4.1 Aqueous, 3010A, 3030C, 3050B, USEPA CLP ILMO 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C (Revision 23, 03/07/12)	Definitive	Soil, groundwater, and aqueous QC samples/ Metals Digestion	None/Preparation	Empirical	N	N
Empirical SOP-103	Mercury Analysis in Water by Manual Cold Vapor Technique, Methods USEPA SW846 7470A and 245.1, CLP-M 4.1 (Revision 21, 08/13/12)	Definitive	Groundwater, and aqueous QC samples/ Mercury	Flow Injection Mercury Analyzer	Empirical	N	N
Empirical SOP-104	Mercury Analysis in Soil/Sediment by Manual Cold Vapor Technique, Methods SW846 7471A, 7471B, 245.5, and CLP-ILM 4.1 (Revision 21, 10/31/12)	Definitive	Soil / Mercury	Flow Injection Mercury Analyzer	Empirical	N	N
Empirical SOP-105	Metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) Technique, SW-846 Methods 6010B, 6010C, USEPA Method 200.7, Standard Methods 19th Edition 2340B, USEPA CLP ILMO 4.1 (Revision 19, 08/13/12)	Definitive	Soil, groundwater, and aqueous QC samples/ Metals	ICP-AES	Empirical	N	N
Empirical SOP-202	GC/MS Volatiles using USEPA Method 624 and SW846 Method 8260B, Including Appendix IX Compounds (Revision 26, 01/08/13)	Definitive	Soil, groundwater, and aqueous QC samples/ VOCs	Gas Chromatography/Mass Spectroscopy (GC/MS)	Empirical	N	N
Empirical SOP-225	GC/MS Volatile Non-Aqueous Matrix Extraction using SW-846 Method 5035 for 8260B Analysis (Revision 10, 03/06/12)	Definitive	Soil / VOCs Extraction	Purge and Trap instrumentation coupled with GC/MS	Empirical	N	N
SOP-83[1]	Analysis of VOCs in Summa Polished Canisters by GC/MS Low Level, Revision 10, January, 2013.	Definitive	Soil Gas, Indoor Air, and Outdoor Air/ VOCs	Agilent/HP GC/MS	Eurofins -Air Toxics	NA	No
SOP-38[1]	Analysis of VOCs in Summa Polished Canisters by GC/MS SIM, Revision 15, January, 2013	Definitive	Soil Gas, Indoor Air, and Outdoor Air/ VOCs	Agilent/HP GC/MS	Eurofins - Air Toxics	NA	No

SOP – standard operating procedure

1. Air Toxics Ltd. SOPs are proprietary and cannot be provided as part of this submittal but can be reviewed via the internet after the party wishing to review the SOP signs a nondisclosure agreement (NDA). The review process is as follows:
    - Air Toxics Ltd. sends the NDA in PDF to theparty wishing to view the SOP.
    - The potential viewer signs and returns the NDA to Air Toxics Ltd. via email.
    - Air Toxics Ltd. notifies the potential viewer that they will be allowed to view the SOP and provides a user name, password, web Site address and a notification that viewing will be supported for a very limited time (e.g., 0.5 hrs).
    - The viewer accesses the web Site and views the SOP. Downloading, copying, and printing of the SOP are prevented.

WS #24 -- Analytical Instrument Calibration Table

WS #24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference [1]
GC/MS VOCs	Bromofluoro-benzene (BFB) Tune	Prior to each Initial Calibration (ICAL) and at the beginning of each 12-hour period.	Must meet the ion abundance criteria required by the method (SW8260B; Section 7.3.1; Table 4).	Retune and/or clean or replace source. No samples may be accepted without a valid tune.	Analyst/ Supervisor	Empirical SOP-202
	ICAL – a minimum of a 5-point calibration is prepared for all target analytes (including surrogates)	Upon instrument receipt, for major instrument changes, or when continuing calibration verification (CCV) does not meet criteria.	1)The average response factor (RF) for System Performance Check Compound (SPCCs) must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.10 for chloromethane, bromoform, and 1,1-dichloroethane. 2) Each analyte must meet one of the three options below: Option 1: RSD for each analyte ≤ 15%; Option 2: linear least squares regression for each analyte: r2 ≥ 0.99; Option 3: non-linear least squares regression (quadratic) for each analyte: r2 ≥ 0.99 (minimum 6 points)	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	Retention Time (RT) Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst / Supervisor	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within ± 0.06 RRT units.	Correct problem, then rerun ICAL.	Analyst / Supervisor	
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL, prior to beginning a sample run.	The percent recovery (%R) for all target analytes must be within 80-120% of true values.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst/ Supervisor	
	CCV	Perform one per 12-hour analysis period after tune and before sample analysis.	The minimum RF for SPCCs must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent difference or percent drift (%D) for all target analytes and surrogates must be ≤ 20%.	If analyte exceeds with a positive bias and is non-detect, no corrective action will be performed. Detected analytes and analytes with negative bias will be requested for qualification/narration with client. If client approval is not received, correct problem, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference [1]
ICP-AES Metals	ICAL - a 1-point calibration per manufacturer's guidelines is prepared for all target analytes	At the beginning of each day, or if the QC is out of criteria, prior to sample analysis.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995 ($r2 \geq 0.99$) .	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	**Empirical SOP-100/105**
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R of all target analytes must be within 90-110% of true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, correct problem and repeat ICAL.	Analyst/ Supervisor	
	CCV	At the beginning and end of the sequence and after every 10 samples.	The %R of all target analytes must be within 90-110% of true value.	If analyte exceeds with a positive bias and is non-detect, results will be qualified. Detected analytes and analytes with negative bias will be requested for qualification/narration with client. If client approval is not received, correct problem, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze all affected samples.	Analyst/ Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst / Supervisor	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and all affected samples.	Analyst / Supervisor	
	Low-Level Check Standard (if using 1-point ICAL)	Daily after 1-point ICAL and before samples.	The %R of all target analytes must be within 80-120% of true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference [1]
	Interference Check Standards (ICS – ICS A and ICS B)	At the beginning of an analytical run.	The absolute value of ICS A recoveries for non-spiked analytes must be < LOD; and ICS B recoveries must be within 80-120 %R of true value.	Terminate analysis; locate and correct problem; reanalyze ICS.	Analyst / Supervisor	
	ICAL – A minimum of a 5-point calibration is prepared	Perform daily prior to sample analysis.	The %RSD for RFs must be ≤ 20%, or r must be ≥ 0.995 ($r^2 \geq 0.99$).	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	
Cold Vapor Mercury Analyzer	ICB	Before beginning a sample sequence.	No mercury detected > LOD.	Correct problem, then re-prepare and reanalyze.	Analyst / Supervisor	**Empirical SOP-103/104**
	ICV - Second Source	Once after each ICAL and prior to sample analysis	The %R must be within 90-110% of the true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst / Supervisor	
	CCB	After each CCV, after every 10 samples, and at the end of the sequence	No mercury detected > LOD.	Investigate source of contamination, rerun any samples not bracketed by passing blanks.	Analyst / Supervisor	
	CCV	At beginning and end of each run sequence and every 10 samples.	The %R must be within 80-120% of the true value.	If analyte exceeds with a positive bias and is non-detect, results will be qualified. Detected analytes and analytes with negative bias will be requested for qualification/narration with client. If client approval is not received, correct problem, then rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since last acceptable CCV. If reanalysis cannot be performed, data must be qualified and explained in the case narrative.Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	CCV (undistilled)	CCV (undistilled) at the beginning and end of each run sequence and after every 10 samples.	The %R must be within 90-100% of true value.	If the CCV (undistilled) fails high, report samples that are <LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst / Supervisor	
	Tune Verification - BFB	Every 24 hours, or every 12 hours if project requires.	SW – 846 tune criteria.	Correct problem then repeat tune.	Analyst / Supervisor	
GC/MS – VOCs Low Level (soil gas)	ICAL – Minimum of a 5-point calibration curve is prepared.	Prior to sample analysis.	% RSD must be ≤ 30 with 4 compounds allowed out to ≤ 40% RSD.	Correct problem then repeat ICAL.	Analyst / Supervisor	Air Toxics Ltd. SOP-83
	(ICV) – Second Source	After each ICAL, and daily, prior to sample analysis.	%Rs for 90% of "Standard" compounds must be within 70-130%; for 80% of "Non-standard" compounds, %Rs must be within 60-140%. No %R may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Recalibrate the instrument if the criteria cannot be met.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	CCV	At the start of each day and, if required by a specific project, every 12 hours.	%Rs must be within 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than four compounds (Continued on next page) from the standard list recover outside of 70-130%, then corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%R.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5-point ICAL.	Analyst / Supervisor	
	Tune Verification - BFB	Every 24 hours, or every 12 hours if project requires.	SW-846 tune criteria.	Correct problem then repeat tune.	Analyst / Supervisor	
GC/MS – VOCs SIM (soil gas)	ICAL – Minimum of a 5-point calibration curve is prepared.	Prior to sample analysis.	%RSD must be ≤ 30% for standard compounds with 10% of the compound list allowed out to ≤ 40% %RSD.	Correct problem then repeat ICAL.	Analyst / Supervisor	Air Toxics Ltd. SOP-38
	ICV – Second Source	After each ICAL, and daily prior to sample analysis.	%Rs for 90% of "Standard" compounds must be within 70-130%; for 80% of "Non-standard" compounds, %Rs must be within 60-140%. No %R may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	CCV	At the start of each day and, if required by a specific project, every 12 hours.	%Rs must be within 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than 10% of compounds from the standard list recover outside of 70-130%, CA will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any Compound exceeds 60-140%R.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new calibration curve.	Analyst / Supervisor	

WS #28.1 -- Laboratory QC Samples Table
WS #28.6 -- Laboratory QC Samples Table
WS #28.12 -- Laboratory QC Samples Table

WS #28.1 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: VOCs
Analytical Method/SOP Reference: SW-846 8260B / **Empirical SOP-202**

Quality Control (QC) Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch of 20 or fewer samples	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater. Common contaminants must not be detected > LOQ.	Investigate source of contamination and rerun method blank prior to analysis of samples, if possible. Evaluate the samples and associated QC, if blank results are above LOQ, then report sample results that are <LOQ or >10X the blank concentration. Re-prepare and reanalyze blank and those samples that were >LOQ and <10X the blank.	Analyst, Supervisor	Bias/ Contamination	Same as QC Acceptance Limits
Laboratory Control Sample (LCS)	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.2 limits as per Appendix G of the DoD QSM.	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12-hour clock and is acceptable, then narrate. If the LCS recoveries are high, but the sample results are <LOQ, then narrate. Otherwise, reprepare and reanalyze the LCS and associated samples.	Analyst, Supervisor	Accuracy/Bias	Same as QC Acceptance Limits

Quality Control (QC) Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD Quality Systems Manual (QSM) Version 4.2 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met unless RPDs indicate obvious extraction/ analysis difficulties, then re-prepare and reanalyze MS/MSD.	Analyst, Supervisor	Accuracy/Bias / Precision	Same as QC Acceptance Limit
Internal Standards (ISs)	Every field sample, standard, and QC sample - three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the ICAL midpoint standard or daily CCV for each IS.	Inspect mass spectrometer and gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Supervisor	Accuracy	Same as QC Acceptance Limits
Surrogates	All field and QC samples - four per sample- Dibromofluoromethane 1,2-dichloroethane-d4 Toluene-d8 BFB	%Rs must meet the DoD QSM Version 4.2 limits as per Appendix G of the DoD QSM.	If sample volume is available, then reprepare and reanalyze sample for confirmation of matrix interference when appropriate.	Analyst, Supervisor	Accuracy/Bias	Same as QC Acceptance Limits
Results between DL and LOQ	NA	Apply “J” qualifier to results detected between DL and LOQ.	None	Analyst, Supervisor	Accuracy	Same as QC Acceptance Limits

SOP – standard operating procedure

WS #28.6 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: Metals (Including Mercury)
Analytical Method/SOP Reference: SW-846 6010C, 7470A, & 7471A/ Empirical SOP103/105

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	No analytes detected > 1/2 LOQ or > 1/10 the amount measured in any sample or 1/10 the regulatory limit, whichever is greater.	Re-analyze to confirm the positive value. Notify the PM for further action. Re-prepare the samples associated with the Blank. Noncompliance report will be required for data reported	Analyst, Supervisor	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%R must be within 80-120%.	Evaluate and reanalyze, if possible. If the LCS recoveries are high, but the sample results are < LOQ, then narrate. Otherwise, redigest and reanalyze all associated samples for failed target analyte(s).	Analyst, Supervisor	Accuracy/Bias	Same as QC Acceptance Limits
MS	One per preparatory batch of 20 or fewer samples of similar matrix	%R should be within 80-120% (if sample is < 4x spike added).	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor	Accuracy/Bias	Same as QC Acceptance Limits
Sample Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix	The RPD should be ≤ 20% for duplicate samples for both water and soils.	Narrate any results that are outside control limits.	Analyst, Supervisor	Precision	Same as QC Acceptance Limits

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Serial Dilution	One per preparatory batch with sample concentration(s) >50x LOD	The 5-fold dilution result must agree within ±10%D of the original sample result if result is >50x LOD.	Perform post-spike addition.	Analyst, Supervisor	Accuracy/Bias	Same as QC Acceptance Limits
Post Digestion Spike (does not apply to mercury)	One is performed when serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD	The %R must be within 75-125% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results for affected analytes for all associated samples with “J”.	Analyst, Supervisor	Accuracy/Bias	Same as QC Acceptance Limits
Results between DL and LOQ	Not known at this time	Apply “J” qualifier to results between DL and LOQ.	None	Analyst, Supervisor	Accuracy	Same as QC Acceptance Limits

WS #28.12 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil Gas
Analytical Group: VOCs
Analytical Method/SOP Reference: TO-15/Eurofins - Air Toxics SOP-83 and 38

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One after each CCV and Laboratory Control Sample (LCS).	Results must be less than the laboratory LOQ.	Inspect the system and re-analyze the blank.	Analyst, Supervisor	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	As each standard, blank, and sample is being loaded. Three per sample: 1,2-DCE-d4 toluene-d8 4-BFB	%Rs must be 70-130%.	For blanks: Inspect the system and reanalyze the blank. For samples: Re-analyze the sample unless obvious matrix interference is documented. If the %R is within limits in the reanalysis, report the second analysis. If %R is out-of-limits a second time, then narrate results.	Analyst, Supervisor	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	After each ICAL, and daily, prior to sample analysis.	%Rs for 90% of the "Standard" compounds must have %Rs between 70-130%; for 80% of "Non-standard" compounds, %Rs must be between 60-140%. No %R may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Recalibrate the instrument if the criteria cannot be met.	Analyst, Supervisor	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
IS	As each standard, blank, and sample is being loaded. Three per sample: bromochloromethane 1,4-difluorobenzene chlorobenzene-d5	RTs for blanks and samples must be within ±0.33 minutes of the RT in the CCV and within ±40% of the area counts of the daily CCV ISs.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.	Analyst, Supervisor	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

WS #30 -- Analytical Services Table

WS #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization	Backup Laboratory/ Organization
Soil, Groundwater, and Aqueous QC Blanks	VOC (including low level for aqueous samples)	See **WS #18**	SW-846 8260B	21 calendar days	Brian Richard brichard@empirlabs.com Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 (615) 345-1115	N/A
	Explosives including nitroglycerin		SW-846 8330B			
	Metals (total and dissolved for groundwater) including mercury		SW-846 6010C, 7470A, and 7471A			
	Cyanide		SW-846 9012B			
	Nitroguanidine		SW-846 8330 modified			
	Perchlorate		SW-846 6850			
	TOC		SW-846 9060 and (Lloyd Kahn for soil)			
Groundwater	Anions		EPA 300			
	Sulfide		SM 4500S-2CF			
	Dissolved Gases		RSK 175			
	Alkalinity		SM 2320			
Soil gas	VOCs		Modified TO-15 Hi/Lo		Ausha Scott Air Toxics Ltd. 180B Blue Ravine Road Folsom CA 95630 916-985-1000 ext 1027 ascott@airtoxics.com	

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization	Backup Laboratory/ Organization
Soil	Permeability		ASTM D5084		Mark Dobday GeoTesting Express, Inc. 1145 Massachusetts Ave. Boxborough, MA 01719 Toll Free 800-434-1062 Fax 978 635 0266	
	Effective Porosity		ASTM D854, D2216			
	Bulk Density		ATI RP40			
	Grain size		ASTM D422			

Empirical will also analyze aqueous and solid IDW samples but details of these analyses are not provided as the data are used for disposal purposed only.

WS #31 -- Planned Project Assessments Table

WS #31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Action CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Laboratory System Audit **(1)**	Every 2 years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Empirical and Air Toxics	Laboratory QAM or Laboratory Manager, Empirical and Air Toxics	Laboratory QAM or Laboratory Manager, Empirical and Air Toxics

1. Empirical and Air Toxics have successfully completed the laboratory assessment process required as part of the DoD QSM Version 4.1 under the DoD ELAP by a recognized Accrediting Body. The DoD ELAP accreditation letters are included in **Appendix B**. Geotesting express is not DoD ELAP-accredited, but due to the nature of these analyses accreditation is not required.

APPENDIX C

Final UFP SAP (March 2011)

Final

Remedial Investigation Sampling and Analysis Plan

Site 43 – Toluene Disposal Area

Naval Support Facility Indian Head
Indian Head, Maryland

Naval Facilities Engineering Command
Washington

Contract Number N62472-03-D-0057
Contract Task Order 114

March 2011

TETRA TECH

SAP Worksheet (WS) #1 – Title and Approval Page

(UFP-QAPP Manual Section 2.1)

FINAL
REMEDIAL INVESTIGATION
SAMPLING AND ANALYSIS PLAN
(FIELD SAMPLING PLAN AND QUALITY ASSURANCE PROJECT PLAN)

SITE 43 – TOLUENE DISPOSAL

NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

March 2011

Prepared for:
Naval Facilities Engineering Command Washington
1314 Harwood Street, S.E.
Washington Navy Yard, D.C. 20374-5018

Prepared by:
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, Pennsylvania 19406

Prepared under:
Contract No. N62472-03-D-0057
Contract Task Order 114

Approval Signatures:

Rob Sok, P.G. – PM, Tetra Tech	3/21/11 Date
Tom Johnston, Ph.D. – CLEAN QAM, Tetra Tech	3/21/11 Date
Joe Rail, P.E. – RPM, NAVFAC Washington	3/21/11 Date

*Government Chemist signature is attached

SAP Worksheet (WS) #1 -- Title and Approval Page
(UFP-QAPP Manual Section 2.1)

DRAFT
REMEDIAL INVESTIGATION
SAMPLING AND ANALYSIS PLAN
(FIELD SAMPLING PLAN AND QUALITY ASSURANCE PROJECT PLAN)

SITE 43 – TOLUENE DISPOSAL

NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

October 2010

Prepared for:
Naval Facilities Engineering Command Washington
1314 Harwood Street, S.E.
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234 Mall Boulevard, Suite 260
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Prepared under:
Contract No. N62472-03-D-0057
Contract Task Order 114

Review Signatures:

Rob Sok, P.G. – PM, Tetra Tech — Date: 10-22-10

T.E. Johnston

Tom Johnston, Ph.D. – CLEAN QAM, Tetra Tech — Date: 10-22-10

Approval Signatures:

Joseph Rail

Joe Rail, P.E. – RPM, NAVFAC Washington — Date: 10-22-10

Jonathan Tucker

Ken Bowers – Navy QAO, NAVFAC Atlantic — Date: 10/26/10

EXECUTIVE SUMMARY

Tetra Tech NUS, Inc. (Tetra Tech) has prepared this Sampling and Analysis Plan (SAP) for a Remedial Investigation (RI) at Naval Support Facility (NSF) Indian Head (IH), Site 43 – Toluene Disposal Area (Site) in Indian Head, Maryland for Contract Task Order (CTO) No. 114, under the Comprehensive Long-Term Environmental Action Navy (CLEAN), Contract N62472-03-D-0057,

Site 43 includes two areas, separated by approximately 700 feet, along Gallery Road in the southwestern portion of the Main Area of Naval Support Facility Indian Head (NSF-IH). The first area is near the northern corner of Building 1040 near the intersection of Gallery and Schuyler Roads. The second area is near a utility pole approximately 30 feet northwest of and across Gallery Road from Building 1041. Reportedly, for a period of more than 2 years during parts-cleaning operations, unknown quantities of spent organic solvents were improperly disposed in the drainage swale outside the door of Building 1040 (acetone) and at the base of the pole near Building 1041 (acetone and toluene). Contaminants present in the waste could have migrated to downstream areas of the drainage swales present at both areas, and/or could have migrated to shallow groundwater.

Prior to the 2005 Site Screening Process (SSP), no environmental sampling had been conducted at the Building 1040 area. A Site Inspection was performed in 1992 in the Building 1041 area (E/A&H, 1994). Four surface soil samples were collected near the utility pole, and acetone was detected at one location. Ten soil-gas borings were completed, and soil-gas samples were collected. Toluene and chlorinated solvents were detected at 3 of the 10 soil-gas locations. According to the Federal Facility Agreement (FFA), a remedial investigation/feasibility study is required at Site 43. However, the Indian Head Installation Restoration Team decided to conduct the SSP to verify that a remedial investigation was necessary.

As part of the SSP, surface soil, subsurface soil, and shallow groundwater samples were collected at each area. In addition, a membrane interface probe (MIP) was used to characterize subsurface conditions as well. Quality control samples (duplicates and blanks) were also collected. The samples were analyzed for Target Compound List volatile organic compounds (VOCs), explosives, nitrocellulose, nitroglycerin, nitroguanidine, Target Analyte List metals, and/or cyanide.

Based on the SSP at Site 43, the Navy concluded that:

- Prior activities have resulted in the release of hazardous substances, or other potentially hazardous constituents at concentrations of potential environmental concern.
- No unacceptable risks exist to ecological receptors based on exposure to surface soil at Site 43.
- Unacceptable risk exists to human health based on a residential use scenario.

- Unacceptable non-cancer risk is driven by the elevated cobalt concentration detected in Site groundwater near Building 1041.
- Unacceptable cancer and non-cancer risks are also driven by the high concentration (36,000 µg/L) of trichloroethene detected in shallow groundwater near Building 1040.
- Although migration of soil contaminants to groundwater is not considered to be problematic, the source of the trichloroethene detected in shallow groundwater was not found during the SSP investigation in the area of Building 1040.

Based on these conclusions, further investigation is warranted at Site 43. This SAP details the RI activities designed to further characterize the nature and extent of contamination, assess human health risks, including the potential for subsurface vapors to intrude into buildings, and help determine further corrective action at Site 43.

The RI will include soil and groundwater sampling to investigate potential source areas and delineate contamination encountered during the Site screening investigations. Approximately 15 soil borings, 7 temporary wells, and 4 permanent monitoring wells will be installed during the investigation. To investigate the potential vapor intrusion pathway near Buildings 1040 and 720, 4 soil gas probes will be installed and sampled.

This SAP was generated for, and complies with, applicable United States Navy, Maryland Department of Environment (MDE), and United States Environmental Protection Agency (EPA) Region 3 requirements, regulations, guidance, and technical standards, as appropriate. This includes the Department of Defense (DoD), Department of Energy (DoE), and EPA Interagency Data Quality Task Force (IDQTF) environmental requirements regarding federal facilities. The SAP is presented in the format of standard worksheets as specified in the Uniform Federal Policy Quality Assurance Project Plan (UFP-QAPP) guidance document (IDQTF, 2005).

This SAP outlines the organization, project management, objectives, planned activities, measurement, data acquisition, assessment, oversight, and data review procedures associated with the planned investigations at Site 43.

Protocols for sample collection, handling, and storage, chain of custody, laboratory and field analyses, data validation, and reporting are also addressed in this SAP. The investigation procedures used will comply with Tetra Tech Standard Operating Procedures (SOPs). The field work and sampling are scheduled to begin in May 2011. A complete project schedule is detailed in **Worksheet No. 16**.

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ACRONYMS AND ABBREVIATIONS

#	number
%	percent
%RSD	percent relative standard deviation
%R	percent recovery
%D	percent drift or percent difference
°C	degrees Celsius
°F	degrees Fahrenheit
a.k.a.	also known as
amu	atomic mass unit
BFB	bromofluoro-benzene
CA	Corrective Action
$CaCO_3$	calcium carbonate
CAS	Chemical Abstracts Service [number]
CCB	continuing calibration blank
CCC	Calibration Check Compound
CCV	Continuing Calibration Verification
CERCLA	Comprehensive Environmental Response, Compensation, and Liability Act
CFR	Code of Federal Regulations
CLEAN	Comprehensive Long-Term Environmental Action Navy
CT	carbon tetrachloride
CTO	Contract Task Order
DCA	dichloroethane
DCB	dichlorobenzene
DNB	dinitrobenzene
DCE	dichloroethene or dichloroethylene
DNAPL	dense non-aqueous phase liquid
DNT	dinitrotoluene
DO	dissolved oxygen
DoD	Department of Defense
DPT	direct push technology
DQI	data quality indicator
DQO	Data Quality Objective
DV	data validation
DVM	Data Validation Manager
ELAP	[DoD] Environmental Laboratory Accreditation Program
EPA	U.S. Environmental Protection Agency
EPC	exposure point concentration
ERP	Environmental Restoration Program
FID	Flame Ionization Detector
FOL	Field Operations Leader
FS	Feasibility Study
FSP	Field Sampling Plan
FTMR	Field Task Modification Request
g	gram
GC/MS	Gas Chromatograph / Mass Spectrometer
GC-FID	Gas Chromatograph – Flame Ionization Detector
H&S	health and safety

HASP	Health and Safety Plan
HI	Hazard Index
HMX	His/Her Majesty's Explosive (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine)
HPLC	High Pressure Liquid Chromatograph
HSA	hollow-stem auger
HSM	Health and Safety Manager
IC	Ion Chromatograph
ICB	initial calibration blank
ICAL	initial calibration
ILCR	Incremental Lifetime Cancer Risk
ICP	Inductively Coupled Plasma
ICP-AES	Inductively Coupled Plasma - Atomic Emission Spectroscopy
ICP/MS	Inductively Coupled Plasma / Mass Spectrometer
ICS	interference check standard (ICSA and ICSB)
ICV	initial calibration verification
ID	identification
IDW	investigation-derived waste
IRCDQM	Installation Restoration Chemical Data Quality Manual
IRP	Installation Restoration Program
IS	internal standard
L	liter
LCS	laboratory control sample
LCSD	laboratory control sample duplicate
LC/MS	Liquid Chromatograph / Mass Spectrometer
LOD	Limit of Detection
LOQ	Limit of Quantification
MCL	Maximum Contaminant Level
MDE	Maryland Department of the Environment
MDL	method detection limit
MIBK	methyl isobutyl ketone (a.k.a. 4-methyl-2-pentanone)
µg	microgram(s)
µg/kg	microgram(s) per kilogram
µg/L	microgram(s) per liter
mg	milligram(s)
mg/kg	milligram(s) per kilogram
mg/L	milligram(s) per liter
mL	milliliter
MPC	measurement performance criteria
MRP	Munitions Response Program
MS	matrix spike
MSD	matrix spike duplicate
MS/MSD	matrix spike and matrix spike duplicate
N/A	not applicable
NAVFAC	Naval Facilities Engineering Command
NDA	non-disclosure agreement
NFESC	Naval Facilities Engineering Service Center
No.	number
NSF-IH	Naval Support Facility Indian Head
ORP	oxidation-reduction potential
OSHA	Occupational Safety and Health Administration
oz	ounce

PAL	project action limit
PCA	tetrachloroethane
PCE	tetrachloroethene or tetrachloroethylene
PID	photoionization detector
PIL	project indicator limit
PM	Project Manager
POC	Point of Contact
PPE	personal protective equipment
PQL	project quantitation limit
PQO	Project Quality Objective
PSL	Project Screening Limit
PT	Proficiency testing (previously known as performance evaluation [PE] sample)
QA	quality assurance
QAM	Quality Assurance Manager
QAO	Quality Assurance Officer
QAPP	Quality Assurance Project Plan
QC	quality control
QL	quantitation limit
QSM	Quality Systems Manual
r	linear regression correlation coefficient
r^2	non-linear regression coefficient of determination
RAB	Restoration Advisory Board
RCRA	Resource Conservation and Recovery Act
RF	Response Factor
RDX	Royal Demolition Explosive (Hexahydro-1,3,5-trinitro-1,3,5-triazine)
RI	Remedial Investigation
RI/FS	Remedial Investigation and Feasibility Study
RPD	relative percent difference
RPM	Remedial Project Manager
RSL	[EPA] Regional Screening Level
RT	retention time
SAP	Sampling and Analysis Plan
SD	serial dilution
SDG	Sample Delivery Group
SIM	Selective Ion Monitoring
SOP	Standard Operating Procedure
SOW	scope of work
SPCC	System Performance Check Compound
SQL	Structured Query Language
SSO	Site Safety Officer
SSP	Site Screening Process
tbd	to be determined
TCA	trichloroethane
TCB	trichlorobenzene
TCLP	Toxicity Characteristic Leachate Procedure
Tetra Tech	Tetra Tech NUS, Inc.
TNB	trinitrobenzene
TNT	trinitrotoluene
TOC	total organic carbon
UCL	upper confidence limit
UFP	Uniform Federal Policy

VC	vinyl chloride
V.I.	vapor intrusion
VOA	volatile organic analysis [vial]
VOC	volatile organic compounds
WS	Worksheet (or SAP Worksheet)

WS #2 -- SAP Identifying Information

(UFP-QAPP Manual Section 2.2.4)

Site Name/Number:	Naval Support Facility Indian Head (NSF-IH) Site 43 – Toluene Disposal
Operable Unit:	Not applicable (N/A)
Contractor Name:	Tetra Tech NUS, Inc. (Tetra Tech)
Contract Number:	N62472-03-D-0057
Contract Title:	Comprehensive Long-Term Environmental Action Navy (CLEAN)
Work Assignment Number:	CTO 114

1. This Sampling and Analysis Plan (SAP) was prepared in accordance with the requirements of the *Uniform Federal Policy for Quality Assurance Plans (*UFP-QAPP*)* (U.S. Environmental Protection Agency [EPA], 2005) and *EPA Guidance for Quality Assurance Project Plans, EPA QA/G-5, QAMS* (EPA, 2002).

2. Identify regulatory program: Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)

3. This SAP is a project-specific SAP.

4. List dates of scoping sessions that were held:

Scoping Session	Date
October 2009 Tier I Partnering Team Meeting	October 22, 2009
April 2010 Tier I Partnering Team Meeting	April 1, 2010

5. List dates and titles of any SAP documents written for previous Site work that are relevant to the current investigation.

Title	Date
Master UFP-SAP for NSF-IH	May 2009
NSF-IH Background Investigation	October 2002
Work Plan for Phase I Site Screening Process (SSP)	February 2005
Work Plan for Phase I Supplemental SSP	September 2007
Work Plan for Phase IA SSP	September 2008
Site 43 SSP Report	October 2009

6. List organizational partners (stakeholders) and connection with lead organization: NSF-IH (facility), EPA Region 3 (federal regulatory stakeholder), Maryland Department of the Environment (MDE) (state regulatory stakeholder)

7. Lead organization: Navy / NAVFAC Washington

8. If any required SAP elements or required information are not applicable to the project or are provided elsewhere, then note the omitted SAP elements and provide an explanation for their exclusion below: There are no exclusions.

Crosswalk Table: A crosswalk table is not necessary. All 37 WSs are included herein as directed by the Navy. Therefore, no crosswalk table is provided on this worksheet.

WS #3 -- Distribution List

(UFP-QAPP Manual Section 2.3.1)

Each person in this table will be responsible for distributing copies of the SAP to appropriate personnel within their organization. For example, the Tetra Tech PM will be responsible for distributing copies of the SAP to all Tetra Tech personnel listed in **WS #4** (*Project Personnel Sign-Off Sheet*).

Name of SAP Recipients	Title / Role	Organization	Telephone Number (Optional)	Email Address or Mailing Address	Document Control Number (Optional)
Joe Rail	Navy Remedial Project Manager (RPM)	NAVFAC Washington	202-685-3105	joseph.rail@navy.mil	N/A
Nicholas Carros	NSF-IH ERP Lead / Facility Point of Contact (POC)	NAVFAC Washington	301-744-2263	nicholas.carros@navy.mil	N/A
Dennis Orenshaw	EPA Region 3 RPM	EPA Region 3	215-814-3361	orenshaw.dennis@epa.gov	N/A
Curtis DeTore	MDE RPM	MDE	410-537-3791	cdetore@mde.state.md.us	N/A
John Tucker (electronic only)	Navy Quality Assurance Officer (QAO)	NAVFAC Atlantic	757-322-8288	jonathan.tucker@navy.mil	N/A
Bonnie Capito (copy of final cover letter)	Librarian, Records Manager, Administrative Record Manager	NAVFAC Atlantic	757-322-4785	bonnie.capito@navy.mil	N/A
Scott Nesbit	Tetra Tech Activity Coordinator	Tetra Tech	412-921-8945	scott.nesbit@tetratech.com	N/A
Rob Sok	Tetra Tech Project Manager (PM)	Tetra Tech	757-466-4904	rob.sok@tetratech.com	N/A
tbd	Field Operations Leader (FOL) and Site Safety Officer (SSO)	Tetra Tech	tbd	tbd	N/A
Kelly Carper (electronic only)	Project Chemist	Tetra Tech	412-921-7273	kelly.carper@tetratech.com	N/A
Lee Leck (electronic only)	Data Manager and Database Manager	Tetra Tech	412-921-8856	lee.leck@tetratech.com	N/A
Joe Samchuck (electronic copy only)	Data Validation (DV) Manager (DVM)	Tetra Tech	412-921-8510	joseph.samchuck@tetratech.com	N/A
Glenn Wagner	NAVFAC Atlantic Administrative Record PM	Tetra Tech	412-921-8386	glenn.wagner@tetratech.com	N/A

Name of SAP Recipients	Title / Role	Organization	Telephone Number (Optional)	Email Address or Mailing Address	Document Control Number (Optional)
Tom Johnston (cover letter)	Navy CLEAN Quality Assurance Manager (QAM)	Tetra Tech	412-921-8615	tom.johnston@tetratech.com	N/A
John Trepanowski (cover letter)	Navy CLEAN Program Manager	Tetra Tech	610-491-9688	john.trepanowski@tetratech.com	N/A
Matt Soltis (electronic only)	Health and Safety (H&S) Manager (HSM)	Tetra Tech	412-921-8912	matt.soltis@tetratech.com	N/A
RAB Members (Final, electronic version only)	NSF-IH Restoration Advisory Board (RAB) Members	Indian Head local residential community	Various	Various	N/A
Karen Lopez (electronic only)	Laboratory Subcontractor PM	Air Toxics Ltd.	916-605-3410	klopez@airtoxics.com	N/A
Mark Dobday (electronic only)	Laboratory Subcontractor PM	Geotesting Express	978-635-0424	mdobday@geotesting.com	N/A
Kim Kostzer (electronic copy only)	Empirical Laboratories, LLC (Empirical) PM	Empirical	615- 345-1115	kkostzer@empirilabs.com	N/A
tbd (electronic only)	Utility Location Subcontractor	tbd	tbd	tbd	N/A
tbd (electronic only)	Drilling Subcontractor	tbd	tbd	tbd	N/A
tbd (electronic only)	Direct Push Technology (DPT) / Membrane Interface Probe (MIP) Subcontractor	tbd	tbd	tbd	N/A
tbd (electronic only)	Survey Subcontractor	tbd	tbd	tbd	N/A
tbd (electronic only)	Investigation-Derived Waste (IDW) Management Subcontractor	tbd	tbd	tbd	N/A

WS #4 -- Project Personnel Sign-Off Sheet

(UFP-QAPP Manual Section 2.3.2)

Certification that project personnel have read the text will be obtained by one of the following methods as applicable:

- In the case of regulatory agency personnel with oversight authority, approval letters or emails will constitute verification that applicable sections of the SAP have been reviewed. Copies of regulatory agency approval letters / emails will be retained in the project files as project records (see **WS #29**).
- Emails will be sent to the listed Navy, Tetra Tech, and subcontractor project personnel who will be requested to verify by email that they have read the applicable SAP sections and the date on which they were reviewed. Copies of the verification email will be included in the project files (see **WS #29**).
- A copy of this [signed] WS will be retained in the project files and identified as a project document in **WS #29**.

Key personnel will be instructed to read the SAP prior to attending an internal Site-specific kick-off meeting for field activities. The Tetra Tech PM will track when the reviews have been completed, obtain signatures, and ensure that the completed sign-off sheet is included in the central project file.

Name [1]	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Navy and Regulatory Project Team					
Joe Rail	NAVFAC Washington / Navy RPM/Manages project	202-685-3105	*See **WS #1** for signature*	All	
Nicholas Carros	NAVFAC Washington / Facility POC/Manages project	301-744-2263		All	
Dennis Orenshaw	EPA Region 3 / Federal Regulatory RPM/ Manages project	215-814-3361		All	
Curtis DeTore	MDE / State Regulatory RPM/Manages project	410-537-3791		All	
Tetra Tech Project Team					
Scott Nesbit	Tetra Tech Activity Coordinator/Coordinates Tetra Tech activities at this facility	412-921-8945		All	
Rob Sok	Tetra Tech PM/ Manages project	757-466-4904	*See **WS #1** for signature*	All	
tbd	Tetra Tech / FOL and SSO/As FOL, manages field work; as SSO, oversees safety operations in the field	tbd		All	

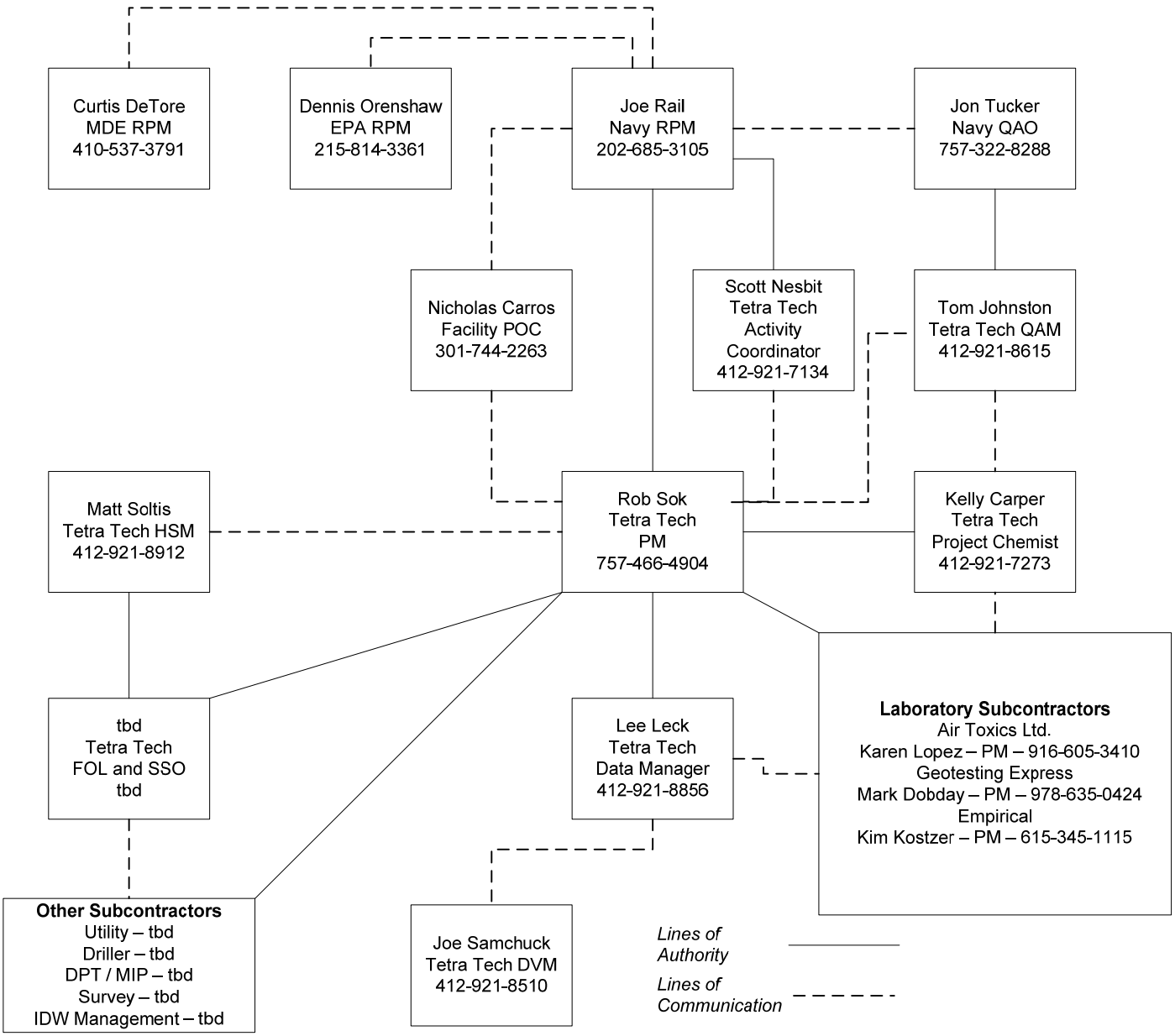
Name [1]	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
Kelly Carper	Tetra Tech Project Chemist/oversees chemistry-related activities	412-921-7273		All	
Joe Samchuck	Tetra Tech / DVM/manages data validation activities	412-921-8510		**WS #12, 14, 15, 19, 20, 23-28, 30, and 34-37**	
Lee Leck	Tetra Tech / Data Manager and Database Manager/ Manages database operations	412-921-8856		**WS #12, 14, 15, 19, 20, 23-28, 30, and 34-37**	
Matt Soltis	Tetra Tech / HSM/Manages Tetra Tech corporate Health and Safety Program	412-921-8912	*See H&S Plan (HASP) in* **Appendix A** *for signature* [2]	All	
Tom Johnston	Tetra Tech / Navy CLEAN QAM/Manages quality assurance activities for Tetra Tech CLEAN program	412-921-8615	*See* ***WS #1*** *for signature*	All	
Subcontractors					
Karen Lopez	Air Toxics Ltd. / PM/Manages daily project activities	916-605-3410		**WS #6, 12, 14, 15, 19, 20, 23-28, 30, 34-36**	
Mark Dobday	Geotesting Express / PM/ Manages daily project activities	978-635-0424			
Kim Kostzer	Laboratory PM/Manages project for Empirical	615-345-1115			
tbd	tbd / Utility Location Subcontractor/ Manages daily project activities	tbd		**WS #6, 14, 17,** and **Figures**	
tbd	tbd / Drilling Subcontractor/ Manages daily project activities	tbd		**WS #6, 14, 17**, and **Figures**	

Name (1)	Organization/Title/Role	Telephone Number (optional)	Signature/email receipt	SAP Section Reviewed	Date SAP Read
tbd	tbd / DPT / MIP Subcontractor/ Manages daily project activities	tbd			
tbd	tbd / Survey Subcontractor/ Manages daily project activities	tbd		**WS #14** and **Figures**	
tbd	tbd / IDW Management Subcontractor/ Manages daily project activities	tbd		**WS #14**	

1. Persons listed on this worksheet will be responsible for distributing the SAP to the appropriate people within their organization.
2. The HSM will be responsible for developing the Site-specific HASP based on field activities presented in this SAP.

WS #5 -- Project Organizational Chart

(UFP-QAPP Manual Section 2.4.1)



WS #6 -- Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or email	Procedure
SAP amendments	Tetra Tech FOL	tbd	tbd	Tetra Tech FOL will verbally inform Tetra Tech PM within 24 hours of realizing a need for an amendment.
Changes in schedule	Tetra Tech PM	Rob Sok	757-466-4904	Tetra Tech PM will document the changes within 5 days and send the Navy RPM a concurrence letter within 7 days of identifying the need for change.
	NAVFAC RPM	Joe Rail	202-685-3105	NAVFAC RPM will send scope change to Tetra Tech Program office before work is started.
	Tetra Tech FOL	tbd	tbd	Tetra Tech PM will verbally inform Navy RPM and Facility POC on the day that schedule change is known and document via schedule impact letter within 1 business-day of when impact is realized.
Issues in the field that result in changes in scope of field work	Tetra Tech PM	Rob Sok	757-466-4904	Tetra Tech FOL will verbally inform Tetra Tech PM within 1 business-day of when the issue is discovered.
	NAVFAC RPM	Joe Rail	202-685-3105	
	Facility POC	Nicholas Carros	301-744-2263	
	Tetra Tech FOL	tbd	tbd	
	Tetra Tech PM	Rob Sok	757-466-4904	Tetra Tech PM will inform Navy RPM (verbally or via email) by close of the next working day.
	NAVFAC RPM	Joe Rail	202-685-3105	NAVFAC RPM will issue scope change approval (verbally or via email at RPM discretion). If warranted (as determined by the NAVFAC RPM), scope change will be documented before work is executed. The Tetra Tech FOL will document the changes on a FTMR form within 2 days of identifying the need for change and obtain required approvals within five days of initiating the form.

Communication Drivers	Responsible Affiliation	Name	Phone Number and/or email	Procedure
Recommendations to stop work and initiate work upon corrective action (CA)	Tetra Tech FOL and SSO	tbd	tbd	If Tetra Tech is the responsible party for a stop work command, the Tetra Tech FOL will inform onSite personnel, subcontractor(s), the Navy RPM, the Facility POC, and the identified Project Team members within 1 hour (verbally or by email). If a subcontractor is the responsible party, the subcontractor PM must inform the Tetra Tech FOL within 15 minutes, and the Tetra Tech FOL will then follow the procedure listed above.
	Tetra Tech PM	Rob Sok	757-466-4904	
	Tetra Tech QAM	Tom Johnston	412-921-8615	
	Tetra Tech HSM	Matt Soltis	412-921-8912	
	NAVFAC RPM	Joe Rail	202-685-3105	
	Facility POC	Nicholas Carros	301-744-2263	
Corrective Action for field program	Tetra Tech QAM	Tom Johnston	412-921-8615	Tetra Tech QAM will notify Tetra Tech PM within one business day that the Corrective Action has been completed. Tetra Tech PM will then notify the Navy RPM within one business day
	Tetra Tech PM	Rob Sok	757-466-4904	
Field data quality issues	Tetra Tech FOL and SSO	tbd	tbd	Tetra Tech FOL will inform Tetra Tech PM verbally or by email (at FOL discretion) on the same day that a field data quality issue is discovered.
	Tetra Tech PM	Rob Sok	757-466-4904	
Analytical laboratory data quality issues	Laboratory PM	Karen Lopez Mark Dobday Kim Kostzer	916-605-3410 978-635-0424 615-345-1115	Laboratory PM will notify (verbally or via email as PM discretion) Tetra Tech Project Chemist within one business day of when an issue related to laboratory data is discovered.
	Project Chemist	Kelly Carper	412-921-7273	Tetra Tech Project Chemist will notify (verbally or via email at project chemist discretion) DV staff and Tetra Tech PM within one business day

WS #7 -- Personnel Responsibilities Table

(UFP-QAPP Manual Section 2.4.3)

Name	Title / Role	Organizational Affiliation	Responsibilities
Joe Rail	NAVFAC Washington Remedial Project Manager/ Manages project f	Navy	Oversees project implementation, including scoping, data review, and evaluation.
Nicholas Carros	NAVFAC Washington / Serves as point of contact for fieldwork	Navy	Supports Navy RPM role and responsibilities; is Facility Point of Contact (POC) for scheduling and coordinating fieldwork.
Dennis Orenshaw	Remedial Project Manager / Manages project	EPA Region 3	Participates in scoping, data review, and evaluation.
Curtis DeTore	Remedial Project Manager / Manages project	MDE	Participates in scoping, data review, evaluation, and approves the SAP.
Scott Nesbit	Tetra Tech Activity Coordinator/Coordinates all facility work for Tetra Tech	Tetra Tech	Oversees all Tetra Tech project related work at the facility.
Tom Johnston	QAM / Oversees program and project quality assurance activities	Tetra Tech	Ensures quality aspects of the CLEAN program are implemented, documented, and maintained.
Kelly Carper	Project Chemist / Conducts data validation and reporting	Tetra Tech	Participates in project scoping, prepares laboratory scope of work (SOW), and coordinates laboratory-related functions with laboratory. Oversees data quality reviews and quality assurance of data validation deliverables.
Rob Sok	PM / Manages project on a daily basis	Tetra Tech	Oversees project, financial, schedule, and technical day to day management of the project
tbd	FOL / Manages field operations	Tetra Tech	Supervises, coordinates, and performs field sampling activities.
	SSO / Oversees Site activities to ensure safety requirements are met		Responsible for on-Site project specific health and safety training and monitoring Site conditions. Details of these responsibilities are presented in the HASP.
Joseph Samchuck	DVM / Oversees data validation activities	Tetra Tech	Manages data validation activities within Tetra Tech, including: • Ensure quality assurance of data validation deliverables. • Provide technical advice on data usability. • Coordinate and maintain data validation review schedule.
Lee Leck	Data Manager / Manages analytical and applicable field data	Tetra Tech	Manages Tetra Tech databases and ensures correct input of data.
Matt Soltis	HSM / Oversees H&S activities	Tetra Tech	Oversees Tetra Tech's Navy CLEAN Program H&S

Name	Title / Role	Organizational Affiliation	Responsibilities
Karen Lopez	Laboratory PM / Manages project	Air Toxics Ltd.	Coordinates analyses with laboratory chemists, lower tier subcontract laboratories, ensures laboratory SOW is followed, provides QA of data packages, and communicates with Tetra Tech project staff
Mark Dobday		Geotesting Express	
Kim Kostzer		Empirical	
tbd	Utility Subcontractor / Performs utility mark-out	tbd	Performs third-party utility clearance and mark-out (in addition to Miss Utility) at and near intrusive work according to SOW.
tbd	Drilling Subcontractor / Performs drilling activities	tbd	Performs drilling techniques to advance soil borings and installs monitoring wells according to SOW.
tbd	DPT / MIP Subcontractor / Performs DPT and MIP activities	tbd	Performs soil boring, macrocoring / soil sampling, air sampling preparation, and MIP data collection techniques according to SOW.
tbd	IDW Subcontractor / Handles IDW	tbd	Responsible for transport and disposal of IDW according to SOW.
tbd	Survey Subcontractor / Surveys locations	tbd	Determines location coordinates and elevation data for monitoring wells according to SOW.

In some cases, one person may be designated responsibilities for more than one position. For example, the FOL may also be responsible for SSO duties. This action will be performed only as credentials, experience, and availability permits.

WS #8 -- Special Personnel Training Requirements Table

(UFP-QAPP Manual Section 2.4.4)

Each site worker will be required to have completed appropriate Hazardous Waste Operations and Emergency Response (HAZWOPER) training specified in Occupational Safety and Health Administration (OSHA) 29 Code of Federal Regulations (CFR) 1910.120 (e). Project-specific safety requirements are addressed in greater detail in the site-specific HASP (**Appendix A**).

WS #9.1 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 43 Remedial Investigation (RI) **Projected Date(s) of Sampling**: Summer/Fall 2010 **Project Manager**: Ed Corack	**Site Name**: Site 43 – Toluene Disposal **Site Location**: NSF-IH, Indian Head, Maryland. See **Figure 2**.

Date of Session: October 22, 2009

Scoping Session Purpose: Review SSP results and recommendations, and scope RI fieldwork

Name	Title	Affiliation	Phone #	Email Address	Project Role
Joe Rail	RPM	NAVFAC Washington	202-685-3105	joseph.rail@navy.mil	RPM
Nate Delong	RPM	NAVFAC Washington	202-685-3279	nathan.delong@navy.mil	Alternate RPM
Nicholas Carros	IRPM	NAVFAC Washington and NSF-IH	301-744-2263	nicholas.carros@navy.mil	IRPM
Dennis Orenshaw	RPM	EPA Region 3	215-814-3361	orenshaw.dennis@epa.gov	RPM
Curtis DeTore	RPM	MDE	410-537-3791	cdetore@mde.state.md.us	RPM
Ed Corack	PM	Tetra Tech	757-466-4908	ed.corack@tetratech.com	PM; SAP lead author
Scott Nesbit	Activity Coordinator	Tetra Tech	412-921-8945	scott.nesbit@tetratech.com	Oversees NSF-IH activities for Tetra Tech.

Comments/Decisions: Discussed results of the SSP results. Initial results were discussed with the Team in June 2009. The SSP Investigation at Site 43 included a multi-phased SSP, which began in 2004. The investigation concluded that prior activities have resulted in the release of hazardous substances of potential environmental concern. The Site screening risk assessment concluded: (1) there were no ecological risks and (2) there is potentially unacceptable human health risk from exposure to groundwater. The vapor intrusion pathway was not assessed

The Team discussed the proposed analytical requirements for groundwater and soil samples. Analyzing for the full suite of parameters is no longer required for RIs. The three rounds of groundwater sampling and MIP investigation indicate halogenated volatile organic compounds (VOCs) are present at the Site (i.e., chlorinated VOCs [CVOCs] are the Site-related CERCLA contaminants).

Action Items: Tetra Tech will proceed with finalizing the SSP Report and preparation of the RI UFP-SAP. Proposed sample locations, etc. will be discussed at the next scoping session.

Consensus Decisions:

- Soil gas sampling to assess the vapor intrusion pathway near Building 720 will be included in the RI.
- Groundwater and soil will be analyzed for target compound list VOCs. Perchlorate will be analyzed if needed (as determined through further DQO development) and will be included in the SAP if appropriate.
- DPT and MIP will be used in the center of Site.
- A Triad decision criteria tree will be created to identify the contamination source

Further, the following will be performed for the RI:

- Soil borings for lithology.
- Conduct organic vapor field screening and soil sample collection for VOC detection.
- Install permanent monitoring wells at each boring location.
- Obtain groundwater samples from new and existing wells and analyze for the full suite of parameters. Check for dense non-aqueous phase liquid using the Oil Red O dye field test.
- Measure the hydraulic conductivity of the aquifer (slug tests) and gauge for groundwater flow.
- Collect soil samples at locations of elevated MIP measurements in the vadose zone.

Please note that the Triad approach and MIP was later omitted during the planning and production of this SAP.

WS #9.2 -- Project Scoping Session Participants Sheet

(UFP-QAPP Manual Section 2.5.1)

Project Name: Site 43 RI **Projected Date(s) of Sampling**: Fall 2010 **Project Manager**: Ed Corack	**Site Name**: Site 43 – Toluene Disposal **Site Location**: NSF-IH, Indian Head, Maryland. See **Figure 2.**

Date of Session: April 1, 2010

Scoping Session Purpose: Review proposed approach and sample locations to finalize UFP-SAP.

Name	Title	Affiliation	Phone #	Email Address	Project Role
Joe Rail	RPM	NAVFAC Washington	202-685-3105	joseph.rail@navy.mil	RPM
Nate Delong	RPM	NAVFAC Washington	202-685-3279	nathan.delong@navy.mil	Alternate RPM
Nicholas Carros	IRPM	NAVFAC Washington	301-744-2263	nicholas.carros@navy.mil	IRPM
Dennis Orenshaw	RPM	EPA Region 3	215-814-3361	orenshaw.dennis@epa.gov	RPM
Curtis DeTore	RPM	MDE	410-537-3791	cdetore@mde.state.md.us	RPM
Ed Corack	PM	Tetra Tech	757-466-4908	ed.corack@tetratech.com	PM; SAP lead author
Scott Nesbit	Activity Coordinator	Tetra Tech	412-921-8945	scott.nesbit@tetratech.com	Oversees NSF-IH activities for Tetra Tech.

Comments/Decisions: Reviewed Site figures with planned sample and well locations (see **Figures 8 and 9** for finalized locations). The following will be performed for the RI.

- Collect soil borings at four locations (to be permanent monitoring wells) for lithology and CSM development.
- Conduct organic field vapor screening and soil sample collection for VOCs.
- Install and sample four monitoring wells around Building 1040 (also re-sample well S43MW001).
  - Check for dense non-aqueous phase liquid using Oil Red O dye field test.
  - Gauge for groundwater flow analysis.
  - Conduct slug testing for hydraulic conductivity estimation.
- Conduct additional membrane interface probe/direct-push technology investigations around Building 1040.
  - Collect soil samples at elevated membrane interface probe measurements in the vadose zone.
- Collect soil gas samples at Building 1040 slab perimeter.
- Re-sample monitoring well S43MW002 near Building 1041 to verify cobalt result.

Action Items: Tetra Tech will finalize the UFP-SAP.

Consensus Decisions: The Team approved the sampling approach and SOW proposed for Site 43. The UFP-SAP will be finalized (pre-draft) and submitted for Navy review (followed by Draft submittal for Team review).

WS #10 -- Conceptual Site Model

(UFP-QAPP Manual Section 2.5.2)

This WS summarizes the current Conceptual Site Model (CSM) for Site 43 – Toluene Disposal (see **WS #11** for the problem statement and DQO process). Background information, including Site location and description, Site history, and a brief summary of the Site geology and hydrogeology are included. Further, a summary of environmental investigations and the limitations of previously collected data are provided.

10.1 SITE LOCATION AND DESCRIPTION

NSF-IH is located in northwestern Charles County, Maryland and is approximately 25 miles southwest of Washington, D.C. (**Figure 1**). NSF-IH is a military facility consisting of the Main Area on the Cornwallis Neck Peninsula and the Annex on Stump Neck. As shown on **Figure 2**, the Main Area is bounded by the Potomac River on the northwest, west, and south, Mattawoman Creek to the south and east, and the Town of Indian Head to the northeast. Stump Neck Annex is located across Mattawoman Creek and is not contiguous with the Main Area. The location of Site 43 is shown on **Figure 2**.

The Indian Head peninsula is located in the Atlantic Coastal Plain Physiographic Province, approximately 8 to 10 miles east of the Fall Line that marks the western extent of the physiographic province. Indian Head has gently rolling to undulating topography with elevations ranging from sea level to more than 100 feet (ft) above mean sea level (msl). The higher elevations are on the eastern portion of the facility, and the land surface generally slopes to the southwest and southeast. The portion of NSF-IH along the Potomac River is characterized by 20- to 100-ft bluffs. The portion along Mattawoman Creek is more gently sloping.

The primary mission of NSF-IH consists of the following:

- To provide services in energetics for all warfare centers through engineering, fleet and operation support, manufacturing technology, limited production, and industrial base support.
- To provide research, development, testing, and evaluation of energetic materials, ordnance devices and components, and other related ordnance engineering standards including chemicals, propellants and their propulsion systems, explosives, pyrotechnics, warheads, and simulators.
- To provide support to all warfare centers, military departments, and the ordnance industry for special weapons, explosive safety, and ordnance environmental issues.
- To execute other responsibilities as assigned by the Commander of the NSF-IH.

NSF-IH is generally surrounded by commercial, residential, and State Park land to the east and south of the Main Area and Stump Neck Annex. The town of Indian Head (not shown on figures) is located just east of the facility where mostly residential neighborhoods are located. The Indian Head Highway (Route 210) extends eastward from NSF-IH main gate, attracting businesses and providing access to residential areas off the main highway. The Potomac River borders the main installation to the north and west, and Stump Neck area to the west. Mason Neck National Wildlife Refuge (not shown) is located across the Potomac River, north of the Main Area. The Mattawoman Natural Environment Area (not shown) is state-owned property located along the southern edge of Mattawoman Creek east of the Main Area.

Site 43 - Toluene Disposal includes two separate areas in the southwestern portion of the Main Area. The first is near a utility pole approximately 30 feet northwest of and across the road from Building 1041 (**Figures 3 and 4**), and the second is the northern corner of Building 1040 (**Figures 3 and 5**). It is estimated that, for a period of more than 2 years during parts-cleaning operations, unknown quantities of

spent solvents were improperly disposed in the drainage ditch outside the door (northeast) of Building 1040 (acetone) and at the base of the pole by Building 1041 (acetone and toluene). The Site is an industrial area where the production of energetics still occurs in Building 720.

A relatively steep earthen, grass-covered bunker is located immediately northwest of the utility pole near Building 1041. The area northeast of this vicinity is relatively flat and covered with mowed grasses and a few trees. The area near Building 1040 also is relatively flat. The area surrounding the building southwest of Schuyler Road is mostly covered with mowed grasses. The area northeast of Schuyler Road is mostly covered with unmowed grasses and trees. Refer to the Tetra Tech (2010) SSP Report for additional information and Site photographs.

10.2 SITE HISTORY

Site 43 was reportedly active between the late 1950s and 1989 when solvents, used in the Cast Plant (Buildings 1040 and 1041) to remove propellants and oily residues from various metal parts, were disposed of directly on the ground outside Building 1040 at the drainage ditch and at the utility pole across from Building 1041. This cleaning activity took place two or three times per week in Building 1041. After the parts were cleaned, the spent solvents were to be combined or 'slummed' with sawdust and placed in 55-gallon drums for disposal at the Cast Plant burn point (Site 19). On many occasions, however, the spent solvents were carried across Galley Road and poured on the ground at the base of the telephone pole (NEESA, 1992). At Building 1040, acetone was reportedly disposed of in the drainage ditch just outside the door. The total quantity of solvents disposed immediately outside Buildings 1040 and 1041 was estimated to be between 23,000 and 30,000 gallons according to the NEESA (1992) *Preliminary Assessment (PA) Report*. No signs of stressed vegetation or solvent dumping stains were observed at the Site during Site inspections in 1995 and 1996 (nor more recently in 2009).

A review of the operational history and/or functions of nearby buildings was conducted to evaluate potential sources of groundwater contamination (Tetra Tech, 2007). According to the E/A&H (1994) *Site Inspection (SI) Report*, acetone was known to have been used in Building 1040 (Parts Cleaning House). Based on information provided by NSF-IH, trichloroethene (TCE) was reportedly used for production processes in Building 720 (Grain Manufacture and Motor Load Building), which is adjacent to Building 1040 (**Figure 3**). The duration of use and quantities of TCE used are not known and have not been documented. Other buildings near Building 1040 include Building 1360 - Scrap Powder Shed (approximately 80 ft south), Building 1257 - Transformer Station (approximately 110 feet north-northwest), and Building 821 - Toilet Building (approximately 150 west-southwest). It is not likely that these latter buildings are the source of TCE detected in shallow groundwater.

A review of sewer lines was conducted to determine whether a leaking sewer could be a source of TCE detected in shallow groundwater (Tetra Tech, 2007). There are no sanitary sewers in the immediate vicinity of Building 1040 (**Figure 3**). The nearest sanitary sewer begins at Building 821 (Toilet Building), located across Gallery Road from Buildings 720 and 1040, and runs approximately 300 ft northwest and north to the former sewage treatment plant. Another sanitary sewer is located approximately 200 ft northwest of and parallel to Gallery Road. There are also sanitary sewers associated with Building 713 (offices – not shown on figures), Building 714 (change house, laundry, and offices), and Buildings 812 and 813 (lunch houses), all located north to north-northwest of Building 1040 near the former sewage treatment plant. The distance between the former sewage treatment plant and Building 1040 is approximately 400 ft. These observations indicate that these sewers are not likely to be sources for the localized shallow TCE contamination near Building 1040.

There is a short length of storm sewer emanating from Building 720 that empties into a drainage swale or ditch adjacent to Building 1040 and Schuyler Road (**Figure 3**). Based on Site topography and facility

maps, water in the ditch would flow to the southeast. Most of the storm drainage in the area is provided by ditches/swales along the sides of roads within piping/culverts under the roads.

The building descriptions above are from Dodohara (1976) *Water Pollution Study at the Naval Ordnance Station*. Although the building uses may have changed since the original report, this older report provides a historical perspective of nearby activities and sewer lines that are a potential source of groundwater contamination. The uses of the buildings served by nearby sanitary sewers do not appear to be sources of the contaminants detected in shallow groundwater. However, this RI will provide information necessary to evaluate the extent of contamination.

10.3 SITE PHYSIOGRAPHY

10.3.1 Soils and Geology

NSF-IH is located in the Atlantic Coastal Plain Physiographic Province and is underlain by unconsolidated sand, gravel, and clay from the Pleistocene and Cretaceous Periods. The soil in this area consists of silty and sandy loams, with minor amounts of gravel. The soils tend to have low permeability and low shrink-swell potential. Four dominant soil associations are found at Indian Head (U.S. Department of Agriculture, Soil Conservation Service, 1974; CH2M HILL, 2003):

- Beltsville-Gravelly Land-Bourne Association—The soils within this association are level to moderately sloping, moderately well-drained and loamy, and moderately deep. They also include dense, root-inhibiting fragipans and steep, gravelly soil materials.

- Beltsville-Exum-Wickham Association—This association is characterized by level to moderately sloping, moderately well-drained and well-drained loamy soils. Soils within this association are moderately deep, and include dense, root-inhibiting fragipans and steep, gravelly soil materials.

- Evesboro-Keyport-Elkton Association—This association is characterized by level to moderately sloping, excessively drained, sandy soils and moderately well-drained and poorly drained, level to gently sloping, loamy soils with clayey subsoil.

- Bibb-Tidal Marsh-Swamp Association—This association is characterized by level or nearly level, poorly drained, and generally located on floodplains and in miscellaneous unclassified wetlands.

Logs from soil borings installed near Building 1040 indicate that shallow geologic conditions consist primarily of silty clay, clayey sand, and sandy clay overlain by silt to silty sand. Some gravel is present. The shallow geologic conditions near Building 1041 consist primarily of clayey sand, silty sand, sandy clay, and clayey silt overlain by clayey silt, silty clay, and silty sand. Some gravel is present, especially at locations near Gallery Road. Soil borings are provided in Appendices A and B of the Tetra Tech (2010) SSP Report.

As noted previously, the facility and Site are in the Atlantic Coastal Plain Physiographic Province. The province consists of an eastward-thickening wedge of interbedded sand and clay units depoSited in fluvial and marine environments. The deposits range in thickness from 650 ft to 900 ft and in age from Cretaceous (the Potomac Group) to Quaternary (Upper Lowland Deposits) (Vroblesky and Fleck, 1991).

NSF-IH is immediately underlain by Quaternary deposits approximately 100 ft thick (Hiortdahl, 1997). They are of fluvial and estuarine origin as cut-and-fill deposits in paleochannels of the early Potomac River system. They generally consist of medium- to coarse-grained sand and gravel grading upward to silt and clay. Isolated cobbles and boulders may be found near the base of the deposits.

The Patapsco Formation, the uppermost unit of the Cretaceous Potomac Group, immediately underlies the Quaternary deposits (Vroblesky and Fleck, 1991). The top of the Patapsco is about 45 ft below mean sea level (msl). The Patapsco is characterized by layers of fine- to medium-grained sand and silt separated by thick layers of clay. Typically, the deposits within the Patapsco Formation grade from coarse-grained at the bottom to finer-grained at the top.

The Patapsco is immediately underlain by the tough, massive clay of the Arundel Formation, which is then underlain by the medium- to coarse-grained sand of the Patuxent Formation. The Patuxent is subsequently underlain by gneissic, schistosic, and gabbroic bedrock. The depth to bedrock beneath Indian Head is approximately 650 to 900 ft below ground surface (bgs) (CH2M HILL, 2003).

10.3.2 Hydrogeology, Hydrology, and Drainage

The shallow groundwater beneath Site 43 is unconfined. The water table at Site 43 was consistently encountered between 6 and 10 ft bgs during previous investigations (see below). The water table is recharged by precipitation that infiltrates the ground surface. The shallow groundwater flow direction could not be determined with the limited data collected during the SSP investigations (see below); however, the potentiometric surface is higher near Building 1040 than Building 1041. (CH2M HILL, 2003):

> Hiortdahl (1990) states that, although there are numerous localized water-bearing systems within the Lowland Deposits, these water-bearing units are not used as a potable water source by the facility or on the Indian Head Peninsula. The main aquifer is a series of units within the Potomac Group; the Patapsco unit is nearest the ground surface. The facility is the largest user of groundwater in the area and withdraws an average of 1 to 2 million gallons per day. Most of the production wells are screened in the Patapsco Formation.

Major water bodies at the Main Area of NSF-IH include the Potomac River and Mattawoman Creek (Chicamuxen Creek lies southeast of the Stump Neck Annex). The Potomac River flows almost 400 miles from its headwaters in the Allegheny Mountains of West Virginia. Near NSF-IH, the Potomac broadens and becomes saltier from the increasing influence of the Chesapeake Bay. Mattawoman and Chicamuxen Creeks are tidal tributaries to the lower Potomac River. The Potomac River bounds the Main Area to the north and northwest. Due to the topography of the peninsula, most of the surface water drainage flows into Mattawoman Creek, which forms its southeastern boundary.

The Building 1041 utility pole is located in a drainage ditch adjacent to Gallery Road (**Figure 4**). A drainage ditch is located between Building 1040 and Schuyler Road (**Figure 5**). These are mostly dry until large precipitation events. Runoff either infiltrates into the soil or is combined with other runoff from nearby areas. A drainage and groundwater divide (exact location unknown) extending along the NSF-IH Main Area is located west of Site 43 nearer the shore of the Potomac River. Based on the expected presence of this divide (and Site topography, including drainage swales), it is assumed that all groundwater at Site 43 flows to the south or southeast toward Mattawoman Creek (to be determined/confirmed during the RI). Brown and Root Environmental (1997) reported that combined runoff at the Site flows toward and eventually discharges to Mattawoman Creek through a series of drainage ditches and storm water pipes. The discharge point is more than 2,000 feet south of the Site.

10.4 SUMMARY OF ENVIRONMENTAL WORK CONDUCTED

A Preliminary Assessment (PA) was performed in 1991 (NEESA, 1992). The PA recommended a Site Inspection (SI) for the area near the utility pole across the road from Building 1041. Sampling was not recommended for Building 1040 because acetone would readily volatilize under ambient conditions rather than migrate through the soil to groundwater. **Figure 6** below presents a timeline for historical environmental work conducted at Site 43.

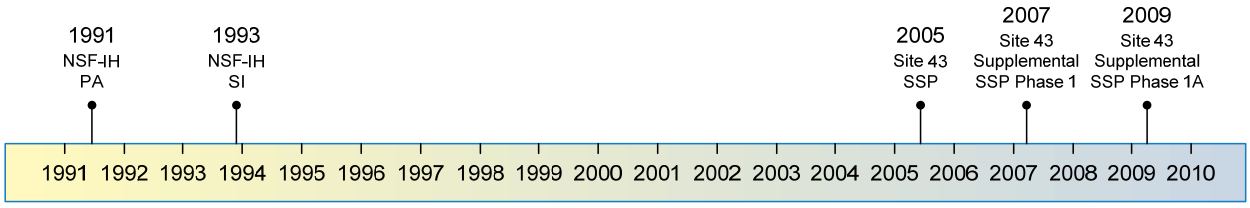


Figure 6 – Timeline of Historical Environmental work at Site 43

An SI was performed in the Building 1041 area in 1993 (E/A&H, 1994). Ten soil-gas borings were completed to 12 ft bgs, and soil-gas samples collected from these borings were analyzed for EPA Target Compound List (TCL) volatile organic compounds (VOCs) (**Figure 4**). Toluene and chlorinated solvents were detected at three of the soil-gas locations near the utility pole. Four surface soil samples (0 to 1 ft bgs) were collected near the utility pole and analyzed for TCL VOCs and TCL semivolatile organic compounds (SVOCs). Soil at the base of the utility pole was reported as sandy silt with some clays and small gravel. There was no visible indication of stained soil, stressed vegetation, or elevated organic vapor field readings. The VOC, acetone was detected at one location. No other VOCs or SVOCs were detected. The SI recommended additional sampling to determine whether VOCs were present in subsurface soil over a larger area.

10.4.1 2005 SSP Investigation

A Site Screening Process (SSP) Investigation began in 2005 to determine whether residual contamination in soil or groundwater at Site 43 poses potential risks to human health or the environment (Tetra Tech Tech 2005). The sampling results would be used to determine whether the risks exist at levels that require the initiation of a removal action (unacceptable), RI or further evaluation (potentially unacceptable), or no further action (acceptable).

Building 1041. Four soil borings were installed during the 2005 investigation in the Building 1041 vicinity (**Figure 4**). Soil borings S43SB003 through S43SB005 were installed to 8 ft bgs. Soil boring S43SB006 was installed to 23 ft bgs and converted into shallow groundwater monitoring well S43MW002 (screened 12 to 22 ft bgs). The soil borings did not encounter the underlying aquitard clay layer (expected at 20 to 30 ft bgs). Four surface and four subsurface soil samples (see **Tables 1 and 2**) were collected from the soil borings down to only 3 feet bgs, and a groundwater sample was collected from the monitoring well installed near Building 1041. All samples were analyzed for TCL VOCs, nitroaromatics, metals, and cyanide. Several VOCs (2-butanone, acetone, carbon disulfide, methylene chloride, and toluene), one explosive (3-nitrotoluene), and several metals were detected near Building 1041 in surface soil (**Table 1**). No organic vapors were detected during field screening of soil cores. VOCs (2 butanone, acetone, and methylene chloride) and metals were detected near Building 1041 in subsurface soil (**Table 2**). Explosives were not detected in any subsurface soil sample. TCE was detected in shallow groundwater at 1.3 micrograms per liter (µg/L) (**Table 3**). A risk screening evaluation was performed based on the

limited surface and subsurface soil samples, there were no unacceptable risks to human health or the environment from chemicals detected in soil and shallow groundwater under a residential use scenario at Building 1041. Therefore, no additional investigation was proposed in this vicinity except to verify the groundwater screening results.

Building 1040. During the 2005 SSP Investigation in the Building 1040 vicinity, two soil borings were installed (S43SB001 to 8 ft bgs and S43SB002 to 20 ft bgs) (**Figure 5**). SB002 was converted into shallow groundwater monitoring well S43MW001 (screened 9 to 19 ft bgs). The depth to groundwater was approximately 9 ft bgs. Two surface and two subsurface soil samples (see **Tables 1 and 2**) were collected from the soil borings down to only 2 feet bgs, and a groundwater sample was collected from the monitoring well. All samples were analyzed for VOCs, nitroaromatics, metals, and cyanide. One VOC (methylene chloride), one explosive constituent (nitrocellulose), and several metals were detected in soils near Building 1040 (**Table 1**). No organic vapors were detected during field screening of the soil cores, but TCE and several metals were detected in subsurface soil near Building 1040 (**Table 2**). A risk screening evaluation based on the limited soil sampling was performed and there were no unacceptable risks to human health or the environment from chemicals detected in soil. However, TCE (54,000 µg/L) and cis-1,2-DCE (530 µg/L) were detected in shallow groundwater in monitoring well MW01 (**Table 3**). Based on these results, the shallow groundwater is contaminated and poses potential risks to human health under a residential use scenario; however, a contaminant source area was not found. Therefore, additional investigation was warranted to determine the source and extent of shallow groundwater contamination detected near Building 1040. The presence of 1,2-cis-dichloroethene in the same well as TCE is an indication that TCE is degrading biochemically, although geochemical conditions are not ideal for this type of degradation.

Geochemical and field parameter data from 2005 are presented below for Site groundwater.

Location	Screen Depth	pH	Conductivity (mS/m)	Temperature (°C)	Turbidity (NTU)	DO (mg/L)	ORP (mV)
MW01	9-19 ft bgs	5.3	0.226	19	2.4	0	106
MW02	12-22 ft bgs	4.57	0.368	21	0.1	0.3	352

10.4.2 2007 SSP Supplemental Investigation (Phase 1)

Supplemental SSP efforts continued at the Site in 2007 (Tetra Tech, 2007). The scope of the 2007 SSP Supplemental Investigation (termed Supplemental Phase 1 SSP) included a desktop audit of historical information related to Site and nearby operations, direct push technology (DPT) groundwater sampling, monitoring well sampling, and membrane interface probe (MIP) field screening for VOCs in groundwater (and to collect more Site lithological information).

The MIP probe was used to detect VOCs in the subsurface at 10 locations (S43MIP01 through S43MIP10) as shown on **Figure 7**. MIP responses indicating the presence of chlorinated (or brominated) VOCs occurred at MIP01 and MIP05 at depths of approximately 19 to 27 ft bgs. The soil conductivity sensor data suggested a basal clay layer beginning at approximately 25 ft bgs. New soil boring S43SB007 was installed at MIP10's location to confirm the soil lithology. MIP logs are provided as Appendix B in the Tetra Tech (2010) SSP Report.

DPT grab groundwater samples were collected at select locations to confirm the MIP results and from existing wells MW01 and MW02 to confirm the results of the 2005 investigation (**Table 3**). All groundwater samples were analyzed for VOCs and were evaluated for the presence of dense non-aqueous phase liquid (DNAPL) using the Oil Red O dye field test (there was no indication of DNAPL).

Several VOCs were detected near Building 1040, however, TCE was the only constituent detected at a significant concentration (>36,000 µg/L; **Table 3**). TCE was detected again at Building 1041 in well MW02 (3.2 µg/L). Geochemical and field parameter data from 2007 are presented below for Site groundwater.

Location	Screen Depth	pH	Conductivity (mS/m)	Temperature (°C)	Turbidity (NTU)	DO (mg/L)	ORP (mV)
TW001	21-25 ft bgs	5.44	39	15.8	turbid	not measured	
TW002	23-27 ft bgs	6.46	33	15.7	turbid	not measured	
TW003	10-20 ft bgs	5.5	14	16.8	turbid	not measured	
TW004	20-24 ft bgs	5.4	22	16.2	turbid	not measured	
TW005	20-24 ft bgs	6.1	20	16.1	turbid	not measured	
MW01	9-19 ft bgs	5.4	22	18.1	0	1.6	not measured
MW02	12-22 ft bgs	4.39	40	19.6	0	0	not measured

10.4.3 2009 SSP Supplemental Investigation (Phase 1A)

The next phase of the SSP supplemental work (termed Supplemental Phase 1A SSP) was recommended to further delineate VOCs in shallow groundwater at the Site. MIP field screening was utilized again during the 2009 effort (Tetra Tech, 2008): nine locations at Building 1040 (MIP16 through MIP24) and five locations at Building 1041 (MIP11 through MIP15) (**Figure 7**). MIP logs are provided in Appendix C of the Tetra Tech (2009) SSP Report.

At Building 1040, the probes were advanced to depths ranging from approximately 25 to 29 ft bgs. A confining unit was identified from approximately 23 to 25 ft bgs. Elevated MIP readings indicated the presence of halogenated compounds at MIP17 (12 ft bgs), MIP19 (18 ft bgs), and MIP20 (12 ft bgs). All three locations were located within 45 ft of monitoring well MW001, the location of the elevated TCE concentrations in 2005 and 2007. No other significant readings were recorded adjacent to Building 1040.

At Building 1041, the probes were advanced to depths ranging from approximately 19 to 25 ft bgs, with a confining unit identified from approximately 16 to 23 ft bgs. No significant readings were made at any of the locations; however a PID reading in the shallow subsurface at S43MIP13 was observed at this location near Building 1041. The result was indicative of a surface release of aromatic hydrocarbons, possibly fuel from lawn maintenance activities.

10.4.4 2009 SSP Report

The Tetra Tech (2009) SSP Report summarized the 2005 through 2009 SSP activities, presented the results, and provided a human health risk screening evaluation based on the limited samples collected during Site sceening investigations (previous screenings determined that ecological risks were not potentially unacceptable, thus limiting current concerns to human health risk only). The risk screening evaluations were performed separately for Buildings 1040 and 1041. The vapor intrusion pathway was not assessed in the human health risk screening evaluation.

For Building 1040, chromium was identified as a chemical of potential concern (COPC) for surface soil; manganese was identified as a COPC for subsurface soil; and bromoform, TCE, antimony, cobalt, iron, and manganese were identified as COPCs for shallow groundwater. The risk characterization resulted in a total Incremental Lifetime Cancer Risk (ILCR) of 2×10^{-2}, two orders of magnitude greater than the EPA acceptable risk range of 10^{-4} to 10^{-6}. The Hazard Index (HI) was 5, which exceeds the EPA threshold of

unity. The unacceptable cancer-risk is driven primarily by the high detection of TCE (36,000 µg/L) in shallow groundwater. The non-cancer risk is driven by cobalt in shallow groundwater. Following additional evaluation of the limited soil sampling, it was determined that there are no unacceptable risks to hypothetical future human residents associated with exposure to COPCs in soil to a depth of 3 ft bgs. There are no surface water bodies near enough to raise concerns about exposure to surface water or sediment at Site 43.

The TCE concentrations observed in groundwater (at approximately 15 feet bgs) indicate the potential for migration of TCE from soil to groundwater; however, a source of this contamination was not found in soil during the SSP. Additional soil sampling is needed near Building 1040 to target deeper soils in the vadose zone to further investigate the potential soil source for TCE.

For Building 1041, iron was identified as a COPC for subsurface soil and 1,1-dichloroethane, TCE, antimony, cobalt, manganese, and nickel were identified as COPCs for shallow groundwater. There were no COPCs for surface soil. The risk screening resulted in a total ILCR of 1×10^{-5}, which is within the EPA acceptable risk range. The HI for some target organs exceeded the EPA threshold of unity, caused by the concentration of cobalt in groundwater. Following additional evaluation, there are no unacceptable risks to hypothetical future human residents associated with exposure to COPCs in soils to a depth of 3 feet bgs.

The potential for migration of soil contaminants to groundwater is not considered to be problematic.

Considering the potential unacceptable human health risk from exposure to VOCs in groundwater at the Site (Building 1040), the Site was recommended to proceed to an RI and Feasibility Study (FS) (RI/FS). Additional groundwater data are needed to confirm and assess the potential risk from exposure to cobalt in groundwater at Buildings 1040 and 1041.

10.5 LIMITATIONS OF PREVIOUS DATA

Data collected during historical investigations at Site 43 have partially delineated the horizontal and vertical extent of VOC-contaminated groundwater. These historical investigations did not define the source area of TCE. DPT groundwater samples cannot be used to obtain an accurate estimate of actual risk. Therefore, additional monitoring wells must be installed and sampled to provide more usable data for risk calculations. These monitoring wells are also needed to determine localized groundwater flow and refine the conceptual site model. Additional sampling of groundwater is also needed to assess the potential risk from exposure to cobalt in groundwater.

During the screening investigations, soil samples were collected down to a depth of only 2 to 3 feet bgs and deeper vadose zone soils were not sampled. Due to the limited depth of the soil sampling and limited number of soil samples collected in the Site screening investigations, additional soil sampling is needed to refine the CSM and provide the necessary analytical data needed to complete a HHRA for Site 43 during this RI.

10.6 CONCEPTUAL SITE MODEL (CSM) SUMMARY

The basic components of the conceptual model that represent an understanding of the Site are presented below. An interpretive conceptual Site model (CSM) is presented as **Figure 8**. This CSM will be updated in the RI Report with new data and information collected during the RI field work.

Contaminant Sources

Based on the historical investigations conducted at Site 43, VOCs represent the primary contaminants of concern. It is assumed that the contamination resulted from releases to surface soil during historical dumping of solvents outside of Buildings 1040 and 1041. Ancillary metals contamination could have resulted from the cleaning of metal parts with the solvents. Based on data collected to date, no continuing contaminant source has been identified in the soil, and this is interpreted to indicate that a source is likely to be a localized area of soil contamination near Building 1040 that has leached or continues to leach into groundwater.

Contaminant Migration Pathways

Contaminant migration pathways potentially affecting current conditions at Site 43 consist of the following:

- Migration of VOCs via leaching of soils to groundwater

- Migration of dissolved VOCs in groundwater and potential migration of free phase DNAPL in the direction of migration being influenced by groundwater flow and, if present, by preferential flow pathways

  Following the initial infiltration of the VOCs (dumping and subsequent flushing from precipitation), the VOC transport migration pathway is in accordance with the predominant flow direction of groundwater (assumed southeast). Groundwater eventually discharges into Mattawoman Creek. Typically, the chemical properties of VOCs prevent them from migrating very far at significant concentrations, primarily due to biodegradation rates. The rates of horizontal advective flow in the groundwater system will be evaluated after additional Site data (hydraulic conductivity, calculated groundwater seepage velocity, etc.) are collected/determined during the RI field effort. The rate of migration for TCE is affected very little by retardation in the aquifer (retardation coefficients are just over 1), but is significantly affected by natural attenuation mechanisms (degradation, dispersion, dilution, etc.); however, aquifer geochemical conditions at the Site do not support ideal biodegradation of VOCs.

- Migration of VOCs via vaporization / volatilization. Volatile VOCs (e.g., TCE) in the groundwater may volatilize and migrate through building foundations as vapor into indoor air.

Exposure Pathways

Based on findings to date (1994 SI and the 2005-2009 SSP Investigation), there are no human exposures under current Site conditions, not considering the unevaluated potential for vapor intrusion issues. Exposure to indoor air vapor is a potential concern. The primary medium at the Site through which future potential residential receptors could be exposed to VOCs is groundwater (which would also be the source of any soil gas / indoor air vapor). See **Figure 8** for graphically representation of CSM. Potential exposure routes under a hypothetical future exposure scenario (potable water use of groundwater or during construction) include dermal contact, ingestion, and inhalation, as well as inhalation of indoor air vapor.

Potential Receptors

The potential receptors that may be exposed to contaminated media at Site 43 include Site workers and hypothetical future residents via ingestion, inhalation, or dermal contact with contaminated soils or groundwater. Future Site use is uncertain, but is anticipated to be industrial considering the base mission.

Summary

The vertical and horizontal extent of VOCs in groundwater has not been fully delineated and actual groundwater flow direction has not been established with the limited monitoring well network in place at the Site. A continuing source of VOCs from Site soils has not been identified, but rather a localized area in groundwater above which historical dumping occurred. Site lithological data suggests a basal clay unit at approximately 25 ft bgs may prevent downward migration of VOCs into lower aquifers. VOCs appear to have limited migration at the Site. There are no current potential receptors or exposure routes, except for the potential vapor intrusion pathway.

WS #11 -- Project Quality Objectives/Systematic Planning Process Statements

(UFP-QAPP Manual Section 2.6.1)

11.1 PROBLEM DEFINITION

1. Historical dumping activities at NSF-IH Site 43 – Toluene Disposal have impacted soil and surficial aquifer groundwater. Risk screening conducted on the limited historical sampling at the Site has shown these contaminants could result in an unacceptable level of human health risk. Based on the current CSM and consensus of the project team, further characterization of the nature and extent of VOC, energetic/explosives, and metals contamination in soil and groundwater near Building 1040 is needed to refine the CSM and evaluate the need for future corrective action at Site 43. This characterization includes a determination of:
    - horizontal and vertical extent of explosives-, solvent-, and/or metals-contaminated soils in the Building 1040 source area.
    - whether there is a continuing source of VOC contamination in soils near Building 1040.
    - extent of the TCE groundwater contamination near Building 1040.
    - localized groundwater flow direction near Building 1040
2. Determine if there is potential for a complete VOC vapor intrusion pathway near Building 1040 and if further assessment is needed for vapor intrusion.
3. A baseline human health risk assessment (HHRA) is needed to evaluate human health risks and determine the need for further corrective action.
4. Verification of cobalt detections in groundwater near Building 1041 to assess the need for further investigation and/or evaluation into the source of cobalt in groundwater.

11.2 INFORMATION INPUTS

Soil and groundwater data are needed for VOCs, energetics/explosives, and metals. The data to be collected will be used to conduct a risk assessment in support of an FS. These data also include geotechnical and wet chemistry parameters.

The following inputs are needed to attain project objectives:

1. Field Screening Data:

    a. Oil Red O dye DNAPL-screening in groundwater to determine if DNAPL is present at the Site. Previous shallow screening found no DNAPL.

    b. Visual observations and photoionization detector (PID) measurements to help identify contamination, and to assist in the selecting depths for investigating soil. Visual and PID data inputs are considered qualitative because they are not necessarily an accurate measure of VOC concentrations in the soil. These qualitative data are of sufficient quality to facilitate consideration of actual sample locations selected for laboratory analysis. The tendency will be to collect samples for VOC analysis at locations within a soil interval where the FOL decides is a location of greatest contaminant levels based on the highest PID reading and visible staining.

    c. Field water quality data to estimate geochemical conditions in the surficial aquifer (pH, dissolved oxygen [DO], turbidity, iron II, and oxidation-reduction potential [ORP]).

2. Chemical Data: Subsurface soil and groundwater concentration data for VOCs, energetics, and metals, and soil gas VOC target analytes. These analytical fractions are included on the basis of the CSM to cover the known potential contaminants that may have impacted soil and groundwater (and

potentially soil gas) because these chemicals have not been evaluated at soil depths greater than 3 feet bgs. Groundwater and soil must also be analyzed at representative locations for wet chemistry parameters [(nitrate, nitrite, sulfate, sulfide, chloride, alkalinity, and total organic carbon (TOC)], dissolved gases (methane, ethane, and ethene). See **WS #15** for the full list of analytes. Sample collection procedures for various media are listed in **WSs #14, 18**, and **21**. The geotechnical and wet chemistry data will be collected for later evaluation (as part of the FS) in accordance with *Guidance for Conducting Remedial Investigations and Feasibility Studies Under CERCLA, Interim Final* (EPA, 1988) to determine whether natural attenuation could serve as a viable remediation mechanism.

RCRA characteristic analyses must be conducted on soil and aqueous IDW in accordance with 40 CFR 261. The data will be used to determine proper disposal procedures for IDW.

3. Physical Data: The physical data to help characterize groundwater at the Site.

    a. Groundwater stabilization parameters (mentioned above in Field Screening Data, and collected in accordance with SOP SA-1.2) to include pH, specific conductance, turbidity, temperature, DO, and ORP, and will be measured before groundwater sampling to ensure representative samples are collected.

    b. Groundwater levels measured at Site monitoring wells prior to the collection of samples to determine groundwater depths and flow direction.

    c. Samples collected for geotechnical parameters (bulk density, hydraulic conductivity, and effective porosity) to support the RI and FS reports.

    d. Slug test data to be used to estimate the hydraulic conductivity of the aquifer, which will allow for groundwater velocity and contaminant migration estimates.

4. Project Screening Limits (PSLs): PSLs were used to ensure that analytical methods were selected with enough sensitivity to support the initial risk screening and subsequent risk assessment governed by this SAP. Subsurface soil, groundwater, and soil gas data must be compared to the PSLs presented in **WS #15**. Primary PSLs/criteria are the "Selected PSLs," which are lowest, most conservative values for each medium. These primary PSLs will be used to screen concentrations and determine which compounds must be included in the HHRA. Other PSL candidates will serve as secondary PSLs/criteria in many cases and will include background criteria for inorganics in soil (Tetra Tech, 2002) as presented in **WS #15**. In this case, background criteria will be used to screen out concentrations of inorganics that are naturally occurring after the initial risk assessment is conducted. Statistics used to represent exposure point concentrations are identified in Section 11.4. Project Action Levels will be used to determine whether additional investigation or corrective action is needed to mitigate risks. For non-carcinogens the PSL is associated with HI=1 (same target organ effect); for carcinogens it is associated with an exceedance of 1×10^{-5} incremental lifetime cancer risk (ILCR).

Worksheet No. 15 lists the PSLs and PALs as well as the laboratory quantitation limits of quantitation (QLs) and statistically-derived method detection limits (MDLs). The laboratories are able to achieve QLs that are below the PSLs for most analytes. For purposes of selecting COPCs in the HHRA, one-half the LOD will be used when the LOD is above the PSL.

11.3 STUDY BOUNDARIES

The primary populations of interest for evaluating the extent of contamination are subsurface soil and groundwater within and near the known contaminant source areas at Site 43 that may be acting directly or indirectly as a source of unacceptable exposure for potential receptors. Also of interest are soil and groundwater in areas that are not contaminated so that a boundary indicating the extent of contamination can be drawn between these two populations for each medium. The horizontal boundary for this investigation is limited to the vicinity of Building 1040 (approximately a 200 foot radius from Building 1040) and the localized area at MW002 near Building 1041. The vertical boundaries for soil are the ground surface to the basal clay aquitard.

For a typical risk assessment the soil population of interest is soil within the 200-foot radius described above to a depth of 10 feet bgs. This constitutes the soil exposure unit (EU). The groundwater population of interest is groundwater in the most contaminated portion of the contaminant plumes within the exposure unit for soil.

For evaluating the potential for a complete vapor migration pathway from soil or groundwater to indoor air, the population of interest is soil and groundwater within and near the known contaminant source areas at Site 43 that may be acting as a source of indoor air contamination.

To establish/confirm the vertical boundaries, lithological data must be collected at each designated location. Groundwater upper vertical boundaries are established by the water table (to be measured) and the basal clay aquitard. Soil gas vertical boundaries are established from the ground surface to the water table. Subsurface soil data represents the subsurface soil population contained within the study area boundaries described above.

For confirming the presence of cobalt contamination in groundwater, the population of interest is groundwater in the immediate vicinity of Building 1041 as represented by well MW02.

11.4 ANALYTIC APPROACH

No decision rules were necessary to govern the fieldwork and a Triad approach was not developed. However, to resolve the problem statement of Section 11.1, multiple decision rules were developed and are presented below.

For the HHRA, if any Site-related contaminant in any soil, groundwater, or soil gas sample exceeds the COPC screening value (primary PSL), the target chemical will be selected as a COPC for inclusion in the risk assessment. Except for lead, the 95 percent upper confidence limit (UCL) of the mean of the results from each analyte/medium will be used as an estimate of the exposure point concentration (EPC) within the exposure unit for each COPC. For lead, the mean lead concentration will be used for comparison to the relevant action level and incorporation into the lead exposure model (i.e., IEBK model). Chemical-specific UCLs will be calculated using EPA's latest ProUCL software. If the UCL is greater than the maximum detected concentration for that medium, the maximum detected concentration will represent the EPC. The mean lead concentration will represent the EPC. EPCs established in this manner represent a reasonable maximum exposure (RME).

To determine whether the vapor intrusion pathway is potentially complete, VOC concentrations in soil gas and groundwater will be used to calculate expected indoor air concentrations. The Johnson and Ettinger model will be used to calculate representative indoor air concentrations from groundwater and soil gas results.

The following decision rules are based on evaluation of the subsurface soil population which represents the subsurface soil decision unit. Similarly, groundwater data and soil gas data represent the groundwater and soil gas population contained within the study area boundaries; and the groundwater and soil gas populations represent their respective decision units.

Decision Rule 1: If the measured concentration of any Site-related contaminant in the sample collected from any new plume periphery well or peripheral soil sample location exceeds the MCL (or PSL in cases where no MCL exists), then re-evaluate the CSM and DQOs to determine whether additional sampling is necessary to complete the delineation of contamination. Factors to be considered by the project team when re-evaluating the CSM, if necessary, include the extent of delineation achieved from the initial sampling versus the cost of additional sampling as well as the consequences of incomplete delineation. If all Site-related contaminant concentrations on the perimeter are less than criteria, then it will be concluded that the Site-related contamination is delineated and an FS will be conducted.

Note: The project team may decide that perfect bounding of contamination in all directions is not necessary but this decision must be deferred until data are collected and spatial patterns of contamination are evident.

Decision Rule 2: If the modeled concentration of any VOC from soil gas or groundwater exceed the applicable PSL for indoor air, then recommend additional evaluation to determine if mitigation for vapor intrusion is needed, otherwise recommend no further action. DQOs for additional sampling and/or investigation, if necessary, would be developed and documented in an FTMR.

Decision Rule 3: For all target analytes except lead, if the ILCR for the RME scenario for exposure to Site-related contaminants is $>1\times10^{-5}$ for carcinogens or if the HI is greater than unity for noncarcinogens, and following potential Team risk management decisions, then conduct an FS to evaluate actions that can be taken to mitigate the unacceptable risk. If the Integrated Exposure Uptake Biokinetic (IEUBK) model for lead indicates that greater than 5 percent of a population's blood-lead concentrations are >10 micrograms per deciliter, and following potential Team risk management decisions, then conduct an FS to evaluate actions that can be taken to mitigate unacceptable risk. If these risk criteria for non-lead contaminant and lead are not exceeded, recommend no further action.

Note: Cobalt and TCE data will be collected from MW02 for information purposes to be used in future planning in support of an FS.

11.5 PERFORMANCE CRITERIA

Sample locations were selected to target the source areas delineated in previous investigations at the Site. Biased sampling is appropriate to establish the lateral and vertical extent of contamination at each Building. The number of biased samples required to achieve full delineation was estimated by the project team based on visual patterns of known contamination and known behavior of released contaminants in the environment. After new data have been collected, the Project Team will use concentration patterns in the new data combined with previously collected data to determine whether the contamination is satisfactorily delineated. The sampling design (see **WS #17**) requires sufficient sampling to establish perimeter samples with target analyte concentrations less than criteria, and interior samples with target analyte concentrations greater than criteria Delineation with the planned number of samples will be considered satisfactory unless samples on the three-dimensional perimeter of the sampling pattern exceed criteria and the exceedances are sufficiently large in magnitude and number to render additional delineation cost-effective.

The need to also conduct a risk assessment requires selecting sampling locations that are spatially representative of the areas anticipated to be contaminated. This biased approach is conservative and represents a "worst case" estimate of risks. If risks are acceptable under this scenario, the Project Team can confidently conclude that risks would be no worse if a different sampling pattern or strategy were used. If risks are unacceptable, the Project Team will recommend that an FS be conducted and any additional delineation required to support the FS can be conducted in a supplemental investigation.

11.6 PLANS FOR OBTAINING DATA

See **WS #17** for a detailed sampling design.

WS #12 -- Measurement Performance Criteria Table

(UFP-QAPP Manual Section 2.6.2)

Field Quality Control (QC) Samples

QC Sample	Analytical Group	Frequency	Data Quality Indicators (DQIs)	Measurement Performance Criteria (MPC)	QC Sample Assesses Error for Sampling (S), Analytical (A) or both (S&A)
Aqueous Equipment Rinsate Blanks [(1)]	VOCs, energetics, metals	One per 20 field samples per matrix per sampling equipment. [(1)]	Accuracy/Bias/ Contamination	No analytes ≥½ Limit of Quantitation (LOQ), except common laboratory contaminants, which must be <LOQ.	S&A
Aqueous Trip Blanks	VOCs	One per cooler containing VOC samples.	Accuracy/Bias/ Contamination	No analytes ≥½ LOQ, except common laboratory contaminants, which must be <LOQ.	S&A
Field Blanks	VOCs	One per site visit	Accuracy/Bias/ Contamination	No analytes ≥½ LOQ, except common laboratory contaminants, which must be <LOQ.	S&A
Solid and Aqueous Field Duplicate	All Fractions	One per 10 field samples collected.	Precision	Values >5 times LOQ: Relative Percent Difference (RPD) ≤30% [(2)(3)] (aqueous); ≤50% (solid). [(2)(3)]	S
Aqueous Cooler Temperature Indicator	All Fractions	One per cooler.	Representativeness	Temperature must be between 0 and 6 Degrees Celsius (°C).	S

1. Equipment rinsate blanks will be collected if non-dedicated submersible pumps or other equipment are used.
2. If duplicate values for non-metals are < 5x LOQ, the absolute difference should be < 2x LOQ.
3. If duplicate values for metals are < 5x LOQ, the absolute difference should be < 4x LOQ.

WS #13 -- Secondary Data Criteria and Limitations Table

(UFP-QAPP Manual Section 2.7)

Secondary Data	Data Source	Data Generator(s)	How Data Will Be Used	Limitations on Data Use
SSP results	Tetra Tech, *SSP Investigation Report for Site 43*, May 2009	Tetra Tech (with agreement from the Navy, EPA and MDE) collected soil, groundwater, and MIP data in 2005, 2007, and 2009.	Data was used to place new sample locations for the RI. Groundwater data from monitoring wells will be compared to new groundwater data at Site 43.	Historical VOC data from 2005 to 2009 may not be indicative of current Site conditions
Background Data	Tetra Tech, Background Investigation Report, October 2002	Tetra Tech (with agreement from the Navy, EPA, and MDE) collected soil and groundwater data throughout the facility to establish background values for soil and groundwater at NSF-IH in 1997.	Data was previously used during the SSP for comparison to Site concentrations. Background data will be used again for comparison / data analysis in the forthcoming RI.	Potentially limited background groundwater dataset, because it is based on data from five monitoring wells.

WS #14 -- Summary of Project Tasks

(UFP-QAPP Manual Section 2.8.1)

14.1 FIELD INVESTIGATION TASK PLAN

The field tasks are summarized below. A short description of these tasks is also provided.

- Mobilization/Demobilization
- Utility Clearance
- Field Monitoring Equipment Calibration
- Soil Borings and Subsurface Soil Sampling
- Groundwater Grab Sampling and DNAPL Testing
- Monitoring Well Installation
- Monitoring Well Sampling
- Water Level Measurements
- Slug Tests and Soil Gas Sampling
- Investigation-Derived Waste Management
- Surveying
- Field Equipment Decontamination Procedures
- Field Documentation Procedures
- Sample Custody and Shipment Tasks

Additional project-related tasks include:

- Analytical Tasks
- Data Management
- Assessment and Oversight
- Data Review
- Project Reports

14.2 FIELD TASKS

Also see the following appendices in the NSF-IH Master UFP-SAP (Tetra Tech, 2009):

- Appendix B – Field and General Sampling Operations
- Appendix C – Example Forms
- Appendix D – Facility Standard Operating Procedures (SOPs)

14.2.1 Mobilization/Demobilization

Mobilization shall consist of the delivery of all equipment, materials, and supplies to the Site, the complete assembly in satisfactory working order of all such equipment at the Site, and the satisfactory storage at the Site of all such materials and supplies. Tetra Tech will coordinate with the facility to identify locations for the storage of equipment and supplies. Site-specific H&S training will be provided to all Tetra Tech subcontractors as part of the Site mobilization.

Demobilization shall consist of the prompt and timely removal of all equipment, materials, and supplies from the Site following completion of the work. Demobilization includes the cleanup and removal of IDW generated during the investigation.

14.2.2 Utility Clearance

Prior to the commencement of any intrusive activities, the Tetra Tech FOL will coordinate with the utility subcontractor to identify and mark-out utilities that may be present within the proposed drilling areas. Subsurface utilities will also be cleared by the drilling subcontractor by notifying the utility clearing service. See Facility SOP HS-1.0 for conducting subsurface soil investigations for further information.

14.2.3 Field/Monitoring Equipment Calibration

These procedures are described in **WS #22**.

14.2.4 Soil Borings and Subsurface Soil Sampling

Subsurface soil samples will be obtained from four soil borings during new monitoring well installations and from seven locations using DPT methods. The proposed soil boring and soil sample locations are presented on **Figures 8 and 9**. With a truck-mounted DPT, continuous soil cores will be obtained to the target depth at each location by advancing a macrocore (4 or 5 feet) to the basal clay layer/aquitard (expected at 25 to 30 ft bgs). The core barrel assembly will be withdrawn and the soils will be screened, described, and sampled.

The soil will be described by the Tetra Tech Project Geologist and a boring log will be constructed. Soil cores will be screened along their entire with a photoionization detector (PID) for evidence of potential contamination. Any visual signs of potential contamination (such as soil staining) will be noted and samples will be collected for VOCs.

A Shelby tube sample will be collected (from SB11 during installation of monitoring well MW06) from the clay layer for offSite laboratory conductivity testing. One soil sample will be tested for effective porosity and bulk density. Three soil samples will be tested for grain size analysis. See **WSs #17** and **18**.

Soil samples will be collected from the macrocores as described herein and outlined in **WS #17**. Soil sampling, soil logging, sample handling, and DPT work procedures are discussed in Facility SOPs GH-1.3, GH-1.4, GH-1.5, SAQ-1.3, and SA-2.5. The use of the PID is described in the manufacturer's instructions.

14.2.5 Groundwater Grab Sampling

Groundwater grab samples will be collected from the seven DPT locations (**Figures 8 and 9**, and **WSs #17** and **18**). Groundwater grab samples will be collected from just above the clay aquitard (expected at 25 to 30 ft bgs; to be determined by the Project Geologist in the field with lithological data) from approximately 15 to 25 ft bgs. Groundwater grab samples will be collected via the DPT sampling sleeve using a peristaltic pump, per Facility SOPs SA-1.1 and SA-2.4.

14.2.6 DNAPL Testing

All of the groundwater samples will be field tested for the presence of DNAPL. The test will consist of collecting a small amount of groundwater and dropping a drop of Oil Red O dye into the sample. Presence of DNAPL will be indicated almost immediately with the appearance of a distinctive red color.

14.2.7 Monitoring Well Installation

Four monitoring wells will be installed to bound the currently understood VOC extent in groundwater (based on groundwater and MIP data from the SSP investigations) and evaluate groundwater conditions in the study area. Proposed locations are shown on **Figures 8 and 9**.

Soil borings will be drilled at the proposed monitoring well locations to confirm the subsurface lithology and depths to ensure proper depths for well installation (to be screened just above the clay layer). The soil cores will be screened visually and along their entire length with a PID in accordance with the PID manufacturer's instructions. The boring information will be recorded in accordance with Facility SOP SA-6.3. The monitoring wells will be installed using hollow-stem auger (HSA) drilling methods.

Monitoring well installation procedures are discussed in Facility SOP GH-2.8 and each of the new monitoring wells will be developed in accordance with this SOP.

A Shelby tube sample will be collected at SB11 (during installation of monitoring well MW06) from the clay layer for offSite laboratory conductivity testing.

14.2.8 Groundwater Sampling

Groundwater sampling will be conducted at each monitoring well using low-flow sampling procedures, per Facility SOP SA-1.1. A 2-inch diameter, variable speed submersible pump and controller (e.g., Grundfos) and/or peristaltic pump will be used for groundwater sample purging and collection activities, in combination with a continuous flow-through cell suitable for taking water quality measurements. Turbidity measurements will be made using a separate field turbidity meter that is specifically designed to measure turbidity only. Groundwater samples collected for perchlorate will be field filtered.

14.2.9 Water Level Measurements

Prior to the start of sampling, a synoptic round of groundwater level measurements will be made. Depth to groundwater will be measured at each monitoring well, per Facility SOP GH-1.2. Along with the subsequent survey effort, this will provide for generation of groundwater elevation contour maps and provide information on groundwater flow patterns and gradients. Water-level measurements will be completed within the shortest time possible on the same day, and no sooner than 24 hours after a significant precipitation event to minimize the precipitation effects on the data. Water level measurements will be recorded to the nearest 0.01 foot and referenced to a top of casing notch or north side of the well casing. The measurement instrument will be decontaminated prior to conducting the measurement and between each monitoring well.

14.2.10 Slug Testing

In situ hydraulic conductivity testing, or slug testing, will be conducted in accordance with Facility SOP GH-2.4. Slug tests will be performed on three monitoring wells: existing monitoring well MW01 and to-be-installed monitoring wells MW04 and MW06.

14.2.11 Soil Gas Sampling

Soil gas samples will be collected from the four locations shown on **Figures 8 and 9**, per Facility SOP SA-2.4. The locations are near each of Buildings 1040 and 720, both in the vicinity of the assumed source area. Near sub-slab soil vapor samples will be collected from a temporary sampling probe

installed with DPT methods. The samples will be collected using 6-L SUMMA™ canisters equipped with flow controllers set to collect samples over a 30-minute time period.

14.2.12 Investigation-Derived Waste (IDW) Management

Waste soils will be generated during the installation of the soil borings and monitoring wells. The soil IDW consists of the excess soil cuttings from the soil borings that were not collected for laboratory analyses, and the soils produced during the drilling of the boreholes for monitoring well installations. The waste soil will be collected and placed in 55-gallon drums for waste characterization sampling and analysis. Drums will be moved inside the diked area next to Building 289 that has secondary containment. Based upon the results of the waste characterization, and upon Navy approval, the waste soil will be transported and appropriately disposed off-Site at a Navy-approved disposal facility by the IDW subcontractor.

Waste water will be generated during DPT grab groundwater sampling, well development, sampling of the monitoring wells, and during decontamination procedures. All aqueous IDW will be containerized pending waste characterization analyses. Drums will be stored on pallets inside the diked area next to Building 289. Based on the results of the waste characterization, and upon Navy approval, the water will be transported and appropriately disposed offSite at a Navy-approved disposal facility by the IDW subcontractor.

14.2.13 Surveying

A surveyor licensed in the state of Maryland will be subcontracted by Tetra Tech to survey the horizontal location and vertical elevation of each of the monitoring wells (existing and to-be-installed). The horizontal measurements shall be accurate to 0.1 ft. The vertical elevation measurements shall be accurate to 0.01 ft at the top-of-riser at each monitoring well. Each of the locations will be surveyed in the North American Datum (NAD) of 1983, State Plane Coordinate System of Virginia (feet) relative to the coordinates of established Site benchmarks or the nearest United States Geological Survey (USGS) benchmark.

14.2.14 Field Equipment Decontamination

Decontamination of equipment will be conducted in accordance with Facility SOP SA-7.1. Decontamination fluids will be containerized and characterized for appropriate disposal with other IDW.

14.2.15 Field Documentation

Field documentation will be performed in accordance with Facility SOP SA-6.3. A summary of all field activities will be properly recorded in a bound logbook with consecutively numbered pages that cannot be removed. Logbooks will be assigned to field personnel and will be stored in a secured area when not in use. At a minimum, the following information will be recorded in the Site logbook:

- Name of the person to whom the logbook is assigned.
- Project name.
- Project start date.
- Names and responsibilities of on-Site project personnel including subcontractor personnel.
- Arrival/departure of Site visitors.
- Arrival/departure of equipment.
- Sampling activities and sample log sheet references.
- Description of subcontractor activities.

- Sample pick-up information, including chain-of-custody numbers, air bill numbers, carrier, time, and date.
- Description of borehole or monitoring well installation activities and operations.
- H&S issues.

All entries will be written in ink and no erasures will be made. If an incorrect entry is made, striking a single line through the incorrect information will make the correction; the person making the correction will initial and date the change. Boring logs, sampling forms, and other field forms will be used to document field activities.

14.2.16 Sample Custody and Shipment

Data management and sample tracking tasks are described in **WS #27** and in Facility SOP CT-05.

14.3 ADDITIONAL PROJECT-RELATED TASKS

14.3.1 Analytical Tasks

Chemical analysis will be performed at the following laboratory's: Empirical, Air Toxics Ltd., and Geotesting Express. Analyses will be performed in accordance with the analytical methods identified in **Worksheet No. 30**. PSLs and PALs for the target analytes specified in **Worksheet No. 15** will be met, with the exceptions noted. The laboratory (ies) will perform the chemical analyses following laboratory-specific SOPs (see **WSs #19** and **23**) that were developed based on the methods listed in **WSs #19** and **30**. Copies of laboratory SOPs that will be used on this project are included in **Appendix B.**

All soil results will be reported by the laboratory on a dry-weight basis. Results of percent moisture will be reported in each analytical data package and electronic data files. This information will also be captured in the project database which will eventually be uploaded to NIRIS. Percent moisture information will also be captured in the Site Investigation report.

14.3.2 Data Management

Project documentation and records:

- Field sample collection and field measurement records are described in **WSs #27** and **29**.
- Laboratory data package deliverables are described in the analytical specifications.
- Data assessment documents and records are listed in **WS #29**.
- Data recording formats are described in **WS #27**.

After the field investigation is completed, the field sampling log sheets will be organized by date and media and filed in the project files. The field logbooks for this project will be used only for these Sites and will also be categorized and maintained in the project files after the completion of the field program. Project personnel completing concurrent field sampling activities may maintain multiple field logbooks. When possible, logbooks will be segregated by sampling activity. The field logbooks will be titled on the basis of date and activity.

The data-handling procedures to be followed by the laboratories will meet the requirements of the laboratory technical specification. The laboratory electronic data will be automatically uploaded into the Tetra Tech database in accordance with proprietary Tetra Tech processes.

The Tetra Tech PM (or designee) is responsible for the overall tracking and control of data generated for the project.

- Data Tracking – Data are tracked from generation to archiving in the Tetra Tech project-specific files. The Tetra Tech Project Chemist (or designee) is responsible for tracking the samples collected and shipped to the laboratories. Upon receipt of the data packages from the laboratories, the Tetra Tech Project Chemist oversees the data validation effort, which includes verifying that the data packages are complete and that results for all samples have been delivered by the laboratories.

- Data Storage, Archiving, and Retrieval – The data packages received from the laboratories are tracked in the data validation logbook. After the data are validated, the data packages are entered into the Tetra Tech CLEAN file system and archived in secure files. The field records including field logbooks, sample logs, chain-of-custody records, and field calibration logs are submitted by the Tetra Tech FOL to be entered into the CLEAN file system prior to archiving in secure project files. The project files are audited for accuracy and completeness. At the completion of the Navy contract the records are stored by Tetra Tech and ultimately sent to NAVFAC.

- Data Security – The Tetra Tech project files are restricted to designated personnel only. Records can only be borrowed temporarily from the project file using a sign-out system. The Tetra Tech Data Manager maintains the electronic data files. Access to the data files is restricted to qualified personnel only. File and data backup procedures are routinely performed.

14.3.3 Assessment and Oversight

Refer to **WS #32** for assessment findings and Corrective Actions and to **WS #33** for QA management reports.

14.3.4 Data Review

Data verification is described in **WS #34**; data validation is described in **WSs #35** and **36**; and data usability assessment is described in **WS #37**.

14.3.5 Project Report

A Draft RI Report will be prepared and submitted to the Navy and regulators (i.e., the Partnering Team) for review. The report will include a summary of the work performed in the approved UFP-SAP, field modifications as documented by the Tetra Tech FOL, summary and analysis of the analytical results, updated CSM, baseline HHRA, and conclusions and/or recommendations for the Site.

Tetra Tech will respond to comments received on the draft report. The final version of the report will be submitted in hardcopy and electronic format to the project stakeholders.

WS #15.1 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

PSLs for all matrices:

- Carcinogenic Risk PSL = Incremental Lifetime Cancer Risk > 1×10^{-5}
- Non-carcinogenic Risk (for chemicals affecting the same target organ) PSL = Hazard index > 1

Matrix: Soil

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)		Selected PAL (3)(7)	PAL Reference (4)	PQL Goal (5)	Laboratory-Specific QLs		
		EPA RSL Residential Soil (1)	MDE Standard Residential Soil (2)				LOQ (6)(7)	LOD (6)(7)	MDL (6)
VOCs (mg/kg)									
Acetone	67-64-1	6,100	7,000	6,100	EPA RSL	2,033	0.02	0.01	0.005
Benzene	71-43-2	1.1	12	1.1	EPA RSL	0.4	0.005	0.0025	0.00125
Bromodichloromethane	75-27-4	0.27	10	0.27	EPA RSL	0.1	0.005	0.0025	0.00125
Bromoform	75-25-2	61	81	61	EPA RSL	20	0.005	0.0025	0.00125
Bromomethane	74-83-9	0.73	11	0.73	EPA RSL	0.2	0.01	0.005	0.0025
Butanone, 2- (Methyl Ethyl Ketone [MEK])	78-93-3	2,800	4,700	2,800	EPA RSL	933	0.01	0.005	0.0025
Carbon Disulfide	75-15-0	82	780	82	EPA RSL	27	0.005	0.0025	0.00125
Carbon Tetrachloride (CT)	56-23-5	0.61	4.9	0.61	EPA RSL	0.2	0.005	0.0025	0.00125
Chlorobenzene	108-90-7	29	160	29	EPA RSL	10	0.005	0.0025	0.00125
Chloroethane (Ethyl Chloride)	75-00-3	1,500	220	220	MDE	73	0.01	0.005	0.0025
Chloroform	67-66-3	0.29	78	0.29	EPA RSL	0.1	0.005	0.0025	0.00125
Chloromethane	74-87-3	12	*none*	12	EPA RSL	4	0.01	0.005	0.0025
Cyclohexane	110-82-7	700	*none*	700	EPA RSL	233	0.005	0.0025	0.00125
Dibromochloromethane (Chlorodibromomethane)	124-48-1	0.68	7.6	0.68	EPA RSL	0.2	0.005	0.0025	0.00125

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)		Selected PAL [3][7]	PAL Reference [4]	PQL Goal [5]	Laboratory-Specific QLs		
		EPA RSL Residential Soil [1]	MDE Standard Residential Soil [2]				LOQ [6][7]	LOD [6][7]	MDL [6]
Dibromochloropropane (Dibromo-3-Chloropropane, 1,2-)	96-12-8	0.0054	0.2	0.0054	EPA RSL	0.002	0.01	0.005	0.0025
Dibromoethane, 1,2- (a.k.a. Ethylene dibromide)	106-93-4	0.034	0.32	0.034	EPA RSL	0.01	0.005	0.0025	0.00125
Dichlorobenzene (DCB), 1,2-	95-50-1	190	700	190	EPA RSL	63	0.005	0.0025	0.00125
Dichlorobenzene (DCB), 1,4-	106-46-7	2.4	27	2.4	EPA RSL	0.8	0.005	0.0025	0.00125
Dichlorodifluoromethane	75-71-8	18	*none*	18	EPA RSL	6	0.01	0.005	0.0025
Dichloroethane (DCA), 1,1-	75-34-3	3.3	1,600	3.3	EPA RSL	1	0.005	0.0025	0.00125
Dichloroethane (DCA), 1,2-	107-06-2	0.43	7	0.43	EPA RSL	0.1	0.005	0.0025	0.00125
Dichloroethene (DCE), 1,1-	75-35-4	24	390	24	EPA RSL	8	0.005	0.0025	0.00125
Dichloroethene (DCE), cis-1,2-	156-59-2	16	78	16	EPA RSL	5	0.005	0.0025	0.00125
Dichloroethene (DCE), trans-1,2-	156-60-5	15	160	15	EPA RSL	5	0.005	0.0025	0.00125
Dichloropropane, 1,2-	78-87-5	0.89	9.4	0.89	EPA RSL	0.3	0.005	0.0025	0.00125
Dichloropropene, 1,3-	10061-02-6	1.7	6.4	1.7	EPA RSL	0.6	0.005	0.0025	0.00125
Ethylbenzene	100-41-4	5.4	780	5.4	EPA RSL	2	0.005	0.0025	0.00125
Hexanone, 2-	591-78-6	21	*none*	21	EPA RSL	7	0.005	0.0025	0.00125
Isopropylbenzene (Cumene)	98-82-8	210	780	210	EPA RSL	70	0.005	0.0025	0.00125
Methyl Acetate	79-20-9	7,800	*none*	7800	EPA RSL	2,600	0.01	0.005	0.0025
Methyl Isobutyl Ketone (MIBK) (4-Methyl-2-Pentanone)	108-10-1	530	*none*	530	EPA RSL	177	0.005	0.0025	0.00125
Methyl tert-Butyl Ether (MTBE)	1634-04-4	43	160	43	EPA RSL	14	0.005	0.0025	0.00125
Methylene Chloride (Dichloromethane)	75-09-2	11	85	11	EPA RSL	4	0.01	0.005	0.0025
Styrene	100-42-5	630	1,600	630	EPA RSL	210	0.005	0.0025	0.00125
Tetrachloroethane (PCA), 1,1,2,2-	79-34-5	0.56	3.2	0.56	EPA RSL	0.2	0.005	0.0025	0.00125

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)		Selected PAL (3)(7)	PAL Reference (4)	PQL Goal (5)	Laboratory-Specific QLs		
		EPA RSL Residential Soil (1)	MDE Standard Residential Soil (2)				LOQ (6)(7)	LOD (6)(7)	MDL (6)
Tetrachloroethene (PCE)	127-18-4	0.55	1.2	0.55	EPA RSL	0.2	0.005	0.0025	0.00125
Toluene	108-88-3	500	630	500	EPA RSL	167	0.005	0.0025	0.00125
Trichloro-1,2,2-trifluoroethane, 1,1,2-	76-13-1	4,300	*none*	4,300	EPA RSL	1,433	0.01	0.005	0.0025
Trichlorobenzene (TCB), 1,2,3-	120-82-1	4.9	*none*	4.9	EPA RSL	2	0.005	0.0025	0.00125
Trichlorobenzene (TCB), 1,2,4-	87-61-6	6.2	78	6.2	EPA RSL	2	0.005	0.0025	0.00125
Trichloroethane (TCA), 1,1,1-	71-55-6	870	16,000	870	EPA RSL	290	0.005	0.0025	0.00125
Trichloroethane (TCA), 1,1,2-	79-00-5	1.1	11	1.1	EPA RSL	0.4	0.005	0.0025	0.00125
Trichloroethene (TCE)	79-01-6	2.8	1.6	1.6	MDE	0.5	0.005	0.0025	0.00125
Trichlorofluoromethane	75-69-4	79	*none*	79	EPA RSL	26	0.01	0.005	0.0025
Vinyl Chloride (VC)	75-01-4	0.06	0.09	0.06	EPA RSL	0.02	0.005	0.0025	0.00125
Xylenes, Total	1330-20-7	63	1,600	63	EPA RSL	21	0.01	0.005	0.0025
Dioxane, 1,4-	123-91-1	4.9	*none*	4.9	EPA RSL	15	0.200	0.100	0.050
Explosives, Energetics, and Oxidizers (mg/kg)									
Dinitrobenzene (DNB), 1,3-	99-65-0	0.61	*none*	0.61	EPA RSL	0.2	0.400	0.200	0.100
Dinitrotoluene (DNT), 2,4-	121-14-2	1.6	16	1.6	EPA RSL	0.5	0.400	0.200	0.100
Dinitrotoluene (DNT), 2,6-	606-20-2	6.1	7.8	6.1	EPA RSL	2	0.400	0.200	0.100
Dinitrotoluene (DNT), 2-Amino-4,6-	35572-78-2	15	*none*	15	EPA RSL	5	0.400	0.200	0.100
Dinitrotoluene (DNT), 4-Amino-2,6-	19406-51-0	15	*none*	15	EPA RSL	5	0.400	0.200	0.100
HMX	2691-41-0	380	*none*	380	EPA RSL	127	0.400	0.200	0.100
Nitrobenzene	98-95-3	4.8	3.9	3.9	MDE	1	0.400	0.200	0.100
Nitrotoluene, 2- (o-Nitrotoluene)	88-72-2	2.9	*none*	2.9	EPA RSL	1	0.400	0.200	0.100
Nitrotoluene, 3- (m-Nitrotoluene)	99-08-1	0.61	*none*	0.61	EPA RSL	0.2	0.400	0.200	0.100

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)		Selected PAL [3][7]	PAL Reference [4]	PQL Goal [5]	Laboratory-Specific QLs		
		EPA RSL Residential Soil [1]	MDE Standard Residential Soil [2]				LOQ [6][7]	LOD [6][7]	MDL [6]
Nitrotoluene, 4- (p-Nitrotoluene)	99-99-0	30	*none*	30	EPA RSL	10	0.400	0.200	0.100
RDX	121-82-4	5.5	*none*	5.5	EPA RSL	2	0.400	0.200	0.100
Tetryl (Trinitrophenylmethylnitramine)	479-45-8	24	*none*	24	EPA RSL	8	0.400	0.200	0.100
Trinitrobenzene (TNB), 1,3,5-	99-35-4	220	*none*	220	EPA RSL	73	0.400	0.200	0.100
Trinitrotoluene (TNT), 2,4,6-	118-96-7	19	*none*	19	EPA RSL	6.3	0.400	0.200	0.100
Nitrocellulose	9004-70-0	18,000,000	*none*	18,000,000	EPA RSL	6,000,000	4.0	2.0	1.0
Nitroglycerin	55-63-0	0.61	*none*	0.61	EPA RSL	0.2	1.00	0.500	0.250
Nitroguanidine	556-88-7	610	*none*	610	EPA RSL	203	0.100	0.0500	0.0250
Perchlorate	14797-73-0	5.5	5.5	5.5	EPA RSL	2	0.0050	0.0020	0.00060
Inorganics (mg/kg)									
Aluminum	7429-90-5	7,700	7,800	7,700	EPA RSL	2,567	40.0	20.0	10.0
Antimony	7440-36-0	3.1	3.1	3.1	EPA RSL	1	2.00	1.60	1.00
Arsenic [8]	7440-38-2	0.39	0.43	0.39	EPA RSL	0.1	0.500	0.300	0.150
Barium	7440-39-3	1,500	1,600	1,500	EPA RSL	500	8.00	4.00	1.00
Beryllium	7440-41-7	16	16	16	EPA RSL	5.3	1.00	0.400	0.200
Cadmium	7440-43-9	7	3.9	3.9	MDE	1.3	1.00	0.400	0.200
Chromium, Total [8]	7440-47-3	0.29	23	0.29	EPA RSL	0.1	0.500	0.200	0.100
Cobalt	7440-48-4	2.3	*none*	2.3	EPA RSL	0.8	0.75	0.50	0.25
Copper	7440-50-8	310	310	310	EPA RSL	103	2.00	1.60	0.800
Cyanide	57-12-5	160	160	160	EPA RSL	53	0.75	0.25	0.125
Iron	7439-89-6	5,500	5,500	5,500	EPA RSL	1,833	20.0	12.0	6.00
Lead	7439-92-1	400	400	400	EPA RSL	133	1.00	0.600	0.300
Manganese	7439-96-5	180	160	160	MDE	53	3.00	1.20	0.600

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)		Selected PAL (3)(7)	PAL Reference (4)	PQL Goal (5)	Laboratory-Specific QLs		
		EPA RSL Residential Soil (1)	MDE Standard Residential Soil (2)				LOQ (6)(7)	LOD (6)(7)	MDL (6)
Mercury	7439-97-6	0.56	0.78	0.56	EPA RSL	0.2	0.033	0.033	0.0130
Nickel	7440-02-0	150	160	150	EPA RSL	50	2.00	1.20	0.600
Selenium	7782-49-2	39	39	39	EPA RSL	13	2.00	1.00	0.600
Silver	7440-22-4	39	39	39	EPA RSL	13	2.00	0.400	0.200
Thallium (8)	7440-28-0	*none*	0.55	0.55	MDE	0.2	0.400	0.200	0.150
Vanadium (8)	7440-62-2	39	7.8	7.8	EPA RSL	2.6	0.625	0.500	0.250
Zinc	7440-66-6	2,300	2,300	2,300	EPA RSL	767	4.00	2.00	1.00

mg/kg – milligrams per kilogram CAS – Chemical Abstracts Service [number] LOQ – Limit of Quantitation LOD – Limit of Detection MDL – Method Detection Limit
HMX - His/Her Majesty's Explosive (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) RDX - Royal Demolition Explosive (Hexahydro-1,3,5-trinitro-1,3,5-triazine)

Note: In most cases where the LOQ exceeds the PAL, the DL is less than PSL. Therefore data usability is not expected to be affected. The target analytes will be detectable if not quantifiable. By including the analytes on the target compound list and striving to quantify as low a concentration as possible with conventional analytical methods, the project team ensures that gross contamination will not be overlooked even though the PSL is less than the LOQ.

1. U.S. Environmental Protection Agency (EPA) (November 2010) Regional Screening Level (RSL) for soil under residential scenario. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for chromium VI used as surrogate for [total] chromium. RSL for methylmercury used as surrogate for mercury.
2. Maryland Department of Environment (MDE) (2008) Cleanup Standards for residential soil. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.
3. Selected PAL is the lowest (most conservative) of the evaluated PSLs.
4. PAL References consist of EPA RSLs or MDE Cleanup Standards (see Notes 1 and 2 above).
5. Project Quantitation Limit (PQL) Goal is set at 1/3 the PSL.
6. The Laboratory QLs and MDLs from the subcontracted laboratory(ies) are presented and are current as of June 2010. The QLs and MDLs are subject to change. The actual limits will be evaluated during the data usability assessment to ensure that the actual QLs and MDLs are satisfactory to support the data evaluations.
7. All PALs are above QLs, except as noted: Shading indicates the Laboratory-Specific LOQ(s) is(are) greater than the Selected PAL. For purposes of selecting COPCs in the baseline human health risk assessment, ½ the LOQ will be used.
8. Laboratory will perfom a 4x concentration on these metals.

WS #15.2 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Note: Refer to Section 11.2, Item 4 for an explanation of these secondary screening values.

Matrix: Soil (Secondary/Candidate Criteria)
Analytical Group: Inorganics

Chemical / Analyte	CAS Number	Carcinogenic (c) or Non-Carcinogenic (n)	NSF Indian Head (NSF-IH) Background Values	
			NSF-IH Background Surface Soil (mg/kg)	NSF-IH Background Subsurface Soil (mg/kg)
Inorganics				
Aluminum	7429-90-5	n	19,700	21,400
Antimony	7440-36-0	n	not detected	not detected
Arsenic	7440-38-2	c	14.9	28.7
Barium	7440-39-3	n	80.4	66.5
Beryllium	7440-41-7	n	1.1	1.5
Cadmium	7440-43-9	n	2.5	0.61
Calcium	7440-70-2		2,060	1,270
Chromium, Total	7440-47-3	c	33.4	59.1
Chromium (III)	16065-83-1	c	*none*	*none*
Chromium (VI)	18540-29-9	c	*none*	*none*
Cobalt	7440-48-4	n	22.3	14.7
Copper	7440-50-8	n	20.3	47.6
Cyanide	57-12-5	n	not analyzed	not analyzed
Iron	7439-89-6	n	38,500	35,200
Lead	7439-92-1	n	62.5	38.6
Magnesium	7439-95-4		1,620	2,940
Manganese	7439-96-5	n	1,390	155
Mercury (elemental)	7439-97-6	n	0.16	0.14
Mercury, Methyl-	22967-92-6	n	*none*	*none*
Nickel (soluble salts)	7440-02-0	n	15.4	15.9
Potassium	2023695		1,470	3,440
Selenium	7782-49-2	n	1.2	3.8

Chemical / Analyte	CAS Number	Carcinogenic (c) or Non-Carcinogenic (n)	NSF Indian Head (NSF-IH) Background Values	
			NSF-IH Background Surface Soil (mg/kg)	NSF-IH Background Subsurface Soil (mg/kg)
Silver	7440-22-4	n	0.84	1.1
Sodium	7440-23-5		120	461
Thallium	7440-28-0		2.3	4.1
Vanadium	7440-62-2	n	53.3	102
Zinc	7440-66-6	n	37.5	49.7

WS #15.3 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Groundwater

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)				Selected PAL [5][9]	PAL Reference [6]	Project Quantitation Limit Goal [7]	Laboratory-Specific QLs		
		EPA RSL Tapwater [1]	MDE Standard Groundwater [2]	Federal MCL [3]	Residential RSL-Based V.I. Screening Value [4]				LOQ [8][9]	LOD [8][9]	MDL [8]
VOCs (µg/L)											
Acetone	67-64-1	2,200	550	*none*	22,857,143	550	MDE	183	10.0	5.00	2.50
Benzene	71-43-2	0.41	5	5	1.3	0.41	EPA RSL	0.1	1.00	0.50	0.25
Bromodichloromethane	75-27-4	0.12	80	*none*	0.8	0.12	EPA RSL	0.04	1.00	0.50	0.12
Bromoform	75-25-2	8.5	80	*none*	100	8.5	EPA RSL	2.8	1.00	0.50	0.25
Bromomethane	74-83-9	0.87	0.85	*none*	17.3	0.85	MDE	0.3	2.00	1.00	0.50
Butanone, 2- (Methyl Ethyl Ketone [MEK])	78-93-3	710	700	*none*	2,260,870	700	MDE	233	10.0	5.00	2.50
Carbon Disulfide	75-15-0	100	100	*none*	1,237	100	EPA RSL	33	1.00	0.50	0.25
Carbon Tetrachloride (CT)	56-23-5	0.44	5	5	0.15	0.15	V.I.	0.05	1.00	0.50	0.11
Chlorobenzene	108-90-7	9.1	100	100	400	9.1	EPA RSL	3	1.00	0.50	0.25
Chloroethane (Ethyl Chloride)	75-00-3	2,100	3.6	*none*	22,222	3.6	MDE	1	2.00	1.00	0.50
Chloroform	67-66-3	0.19	80	*none*	0.7	0.19	EPA RSL	0.06	1.00	0.50	0.13
Chloromethane	74-87-3	19	19	*none*	261	19	EPA RSL	6	1.00	0.50	0.25
Cyclohexane	110-82-7	1300	*none*	*none*	1,033	1,033	V.I.	344	1.00	0.50	0.25
Dibromochloromethane (Chlorodibromomethane)	124-48-1	0.15	80	*none*	2.8	0.15	EPA RSL	0.05	1.00	0.50	0.14
Dibromochloropropane (Dibromo-3-Chloropropane, 1,2-)	96-12-8	0.00032	0.2	0.2	0.03	0.00032	EPA RSL	0.0001	1.00	0.50	0.25
Dibromoethane, 1,2- (Ethylene dibromide)	106-93-4	0.0065	0.05	0.05	0.2	0.0065	EPA RSL	0.002	1.00	0.50	0.25
Dichlorobenzene (DCB), 1,2-	95-50-1	37	600	600	2,692	37	EPA RSL	12	1.00	0.50	0.25
Dichlorobenzene (DCB), 1,4-	106-46-7	0.43	0.28	75	2.2	0.28	MDE	0.09	1.00	0.50	0.25
Dichlorodifluoromethane	75-71-8	39	*none*	*none*	15	15	V.I.	5	2.00	1.00	0.50

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)				Selected PAL [5][9]	PAL Reference [6]	Project Quantitation Limit Goal [7]	Laboratory-Specific QLs		
		EPA RSL Tapwater [1]	MDE Standard Groundwater [2]	Federal MCL [3]	Residential RSL-Based V.I. Screening Value [4]				LOQ [8][9]	LOD [8][9]	MDL [8]
Dichloroethane (DCA), 1,1-	75-34-3	2.4	90	*none*	6.5	2.4	EPA RSL	0.8	1.00	0.50	0.25
Dichloroethane (DCA), 1,2-	107-06-2	0.15	5	5	2	0.15	EPA RSL	0.05	1.00	0.50	0.13
Dichloroethene (DCE), 1,1-	75-35-4	34	7	7	191	7	MDE	2	1.00	0.50	0.25
Dichloroethene (DCE), cis-1,2-	156-59-2	7.3	70	70	371	7.3	EPA RSL	2	1.00	0.50	0.25
Dichloroethene (DCE), trans-1,2-	156-60-5	11	100	100	371	11	EPA RSL	4	1.00	0.50	0.25
Dichloropropane, 1,2-	78-87-5	0.39	5	5	2	0.39	EPA RSL	0.13	1.00	0.50	0.25
Dichloropropene, 1,3-	10061-02-6	0.43	0.44	*none*	4	0.43	EPA RSL	0.14	1.00	0.50	0.25
Ethylbenzene	100-41-4	1.5	700	700	3	1.5	EPA RSL	0.5	1.00	0.50	0.25
Hexanone, 2-	591-78-6	4.7	*none*	*none*	8,158	4.7	EPA RSL	1.6	5.00	2.50	1.25
Isopropylbenzene (Cumene)	98-82-8	68	66	*none*	894	66	MDE	22	1.00	0.50	0.25
Methyl Acetate	79-20-9	3,700	*none*	*none*	*none*	3,700	EPA RSL	1,233	2.00	1.00	0.50
Methyl Isobutyl Ketone (MIBK) (4-Methyl-2-Pentanone)	108-10-1	200	630	*none*	553,571	200	EPA RSL	67	5.00	2.50	1.25
Methyl tert-Butyl Ether (MTBE)	1634-04-4	12	20	*none*	392	12	EPA RSL	4	1.00	0.50	0.25
Methylene Chloride (Dichloromethane)	75-09-2	4.8	5	5	40	4.8	EPA RSL	1.6	2.00	1.00	0.50
Styrene	100-42-5	160	100	100	9,091	100	MDE	33	1.00	0.50	0.25
Tetrachloroethane (PCA), 1,1,2,2-	79-34-5	0.067	0.053	*none*	2.8	0.053	MDE	0.02	1.00	0.50	0.13
Tetrachloroethene (PCE)	127-18-4	0.11	5	5	0.6	0.11	EPA RSL	0.04	1.00	0.50	0.10
Toluene	108-88-3	230	1,000	1,000	19,259	230	EPA RSL	77	1.00	0.50	0.25
Trichloro-1,2,2-trifluoroethane, 1,1,2-	76-13-1	5,900	*none*	*none*	1,409	1,409	V.I.	470	1.00	0.50	0.25
Trichlorobenzene (TCB), 1,2,3-	120-82-1	2.9	*none*	*none*	36	2.9	EPA RSL	1	1.00	0.50	0.25

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)				Selected PAL [5][9]	PAL Reference [6]	Project Quantitation Limit Goal [7]	Laboratory-Specific QLs		
		EPA RSL Tapwater [1]	MDE Standard Groundwater [2]	Federal MCL [3]	Residential RSL-Based V.I. Screening Value [4]				LOQ [8][9]	LOD [8][9]	MDL [8]
Trichlorobenzene (TCB), 1,2,4-	87-61-6	2.9	70	70	36	2.9	EPA RSL	1	1.00	0.50	0.25
Trichloroethane (TCA), 1,1,1-	71-55-6	910	200	200	7,429	200	MDE	67	1.00	0.50	0.25
Trichloroethane (TCA), 1,1,2-	79-00-5	0.24	5	5	4.4	0.24	EPA RSL	0.08	1.00	0.50	0.10
Trichloroethene (TCE)	79-01-6	2	5	5	3	2	EPA RSL	0.7	1.00	0.50	0.25
Trichlorofluoromethane	75-69-4	130	*none*	*none*	183	130	EPA RSL	43	2.00	1.00	0.50
Vinyl Chloride (VC)	75-01-4	0.016	2	2	0.15	0.016	EPA RSL	0.005	1.00	0.50	0.25
Xylenes, Total	1330-20-7	20	10,000	10,000	476	20	EPA RSL	7	2.00	1.00	0.50
Dioxane, 1,4-	123-91-1	0.67	*none*	*none*	1,600	0.67	EPA RSL	0.2	20.0	10.0	3.33
Explosives, Energetics, and Oxidizers (µg/L)											
Dinitrobenzene (DNB), 1,3-	99-65-0	0.37	*none*	*none*	*none*	0.37	EPA RSL	0.1	0.320	0.160	0.0800
Dinitrotoluene (DNT), 2,4-	121-14-2	0.22	7.3	*none*	*none*	0.22	EPA RSL	0.1	0.320	0.160	0.0800
Dinitrotoluene (DNT), 2,6-	606-20-2	3.7	3.7	*none*	*none*	3.7	EPA RSL	1	0.320	0.160	0.0800
Dinitrotoluene (DNT), 2-Amino-4,6-	35572-78-2	7.3	*none*	*none*	*none*	7.3	EPA RSL	2	0.320	0.160	0.0800
Dinitrotoluene (DNT), 4-Amino-2,6-	19406-51-0	7.3	*none*	*none*	*none*	7.3	EPA RSL	2	0.320	0.160	0.0800
HMX	2691-41-0	180	*none*	*none*	*none*	180	EPA RSL	60	0.320	0.160	0.0800
Nitrobenzene	98-95-3	0.12	0.35	*none*	*none*	0.12	EPA RSL	0.04	0.320	0.160	0.0800
Nitrotoluene, 2- (o-Nitrotoluene)	88-72-2	0.31	*none*	*none*	*none*	0.31	EPA RSL	0.1	0.320	0.160	0.0800
Nitrotoluene, 3- (m-Nitrotoluene)	99-08-1	0.37	*none*	*none*	*none*	0.37	EPA RSL	0.12	0.320	0.160	0.0800
Nitrotoluene, 4- (p-Nitrotoluene)	99-99-0	4.2	*none*	*none*	*none*	4.2	EPA RSL	1.4	0.320	0.160	0.0800
RDX	121-82-4	0.61	*none*	*none*	*none*	0.61	EPA RSL	0.2	0.320	0.160	0.0800
Tetryl (Trinitrophenylmethylnitramine)	479-45-8	15	*none*	*none*	*none*	15	EPA RSL	5	0.320	0.160	0.0800

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)				Selected PAL [5][9]	PAL Reference [6]	Project Quantitation Limit Goal [7]	Laboratory-Specific QLs		
		EPA RSL Tapwater [1]	MDE Standard Groundwater [2]	Federal MCL [3]	Residential RSL-Based V.I. Screening Value [4]				LOQ [8][9]	LOD [8][9]	MDL [8]
Trinitrobenzene (TNB), 1,3,5-	99-35-4	110	*none*	*none*	*none*	110	EPA RSL	37	0.320	0.160	0.0800
Trinitrotoluene (TNT), 2,4,6-	118-96-7	2.2	*none*	*none*	*none*	2.2	EPA RSL	0.7	0.320	0.160	0.0800
Nitrocellulose	9004-70-0	11×10^6	*none*	*none*	*none*	11×10^6	EPA RSL	3,666,667	1200	800	400
Nitroglycerin	55-63-0	0.37	*none*	*none*	*none*	0.37	EPA RSL	0.1	0.800	0.400	0.200
Nitroguanidine	556-88-7	370	*none*	*none*	*none*	370	EPA RSL	123	10.0	5.00	2.50
Perchlorate	14797-73-0	2.6	2.6	15	*none*	2.6	EPA RSL	0.9	0.5	0.2	0.0660
Inorganics (µg/L)											
Aluminum	7429-90-5	3,700	3700	none	*none*	3,700	EPA RSL	1233	200	100	50.0
Antimony [10]	7440-36-0	1.5	6	6	*none*	1.5	EPA RSL	0.5	2.5	2.00	1.25
Arsenic[10]	7440-38-2	0.045	10	10	*none*	0.045	EPA RSL	0.02	2.5	1.50	0.75
Barium	7440-39-3	730	2,000	2,000	*none*	730	EPA RSL	243	40.0	10.0	5.00
Beryllium	7440-41-7	7.3	4	4	*none*	4	MDE	1	1.25	0.50	0.25
Cadmium	7440-43-9	1.8	5	5	*none*	1.8	EPA RSL	0.6	1.25	0.50	0.25
Chromium [10]	7440-47-3	0.043	100	100	*none*	0.043	EPA RSL	33	2.5	1.0	0.50
Cobalt[10]	7440-48-4	1.1	*none*	*none*	*none*	1.1	EPA RSL	0.4	3.125	2.50	1.25
Copper	7440-50-8	150	1,300	1,300	*none*	150	EPA RSL	50	10.0	8.00	4.00
Cyanide	57-12-5	73	200	200	*none*	73	EPA RSL	24	20.0	10.0	5.00
Iron	7439-89-6	2,600	2,600	*none*	*none*	2,600	EPA RSL	867	100	60.0	30.0
Lead	7439-92-1	*none*	15	15	*none*	15	MDE	5	3.00	3.00	1.50
Manganese	7439-96-5	88	73	*none*	*none*	73	MDE	24	15.0	6.00	3.00
Mercury	7439-97-6	0.057	0.37	2	0.66	0.057	EPA RSL	0.02	0.20	0.20	0.080
Nickel	7440-02-0	73	73	*none*	*none*	73	EPA RSL	24	10.0	6.00	3.00
Selenium	7782-49-2	18	50	50	*none*	18	EPA RSL	6	10	5.00	3.00
Silver	7440-22-4	18	18	*none*	*none*	18	EPA RSL	6	10.0	2.00	1.00
Thallium	7440-28-0	*none*	2	2	*none*	2	MDE	0.7	2.00	1.00	0.75
Vanadium [10]	7440-62-2	18	3.7	*none*	*none*	3.7	MDE	1.2	3.125	2.50	1.25

Chemical / Analyte	CAS Number	Evaluated Project Screening Limits (PSLs)				Selected PAL [5][9]	PAL Reference [6]	Project Quantitation Limit Goal [7]	Laboratory-Specific QLs		
		EPA RSL Tapwater [1]	MDE Standard Groundwater [2]	Federal MCL [3]	Residential RSL-Based V.I. Screening Value [4]				LOQ [8][9]	LOD [8][9]	MDL [8]
Zinc	7440-66-6	1,100	1,100	*none*	*none*	1,100	EPA RSL	367	20.0	10.0	5.00
OTHER / DISSOLVED GASES (µg/L)											
Methane	74-82-8	*none*	*none*	*none*	*none*	2	PIL	1	4.00	2.00	1.00
Ethane	74-84-0	*none*	*none*	*none*	*none*	1	PIL	1	4.00	2.00	1.00
Ethene	74-85-1	*none*	*none*	*none*	*none*	1	PIL	1	4.00	2.00	1.00
OTHER / WET CHEMISTRY (µg/L)											
Alkalinity (Total, as $CaCO_3$)	ALKALINITY	*none*	*none*	*none*	*none*	50,000	PIL	20,000	1000	1000	1000
Chloride	16887-00-6	*none*	*none*	*none*	*none*	10,000	PIL	2,000	500	330	170
Nitrate (Nitrogen, as Nitrate)	14797-55-8	5,800	*none*	10,000	*none*	1,000	PIL	200	200	100	33.0
Nitrite (Nitrogen, as Nitrite)	14797-65-0	*none*	*none*	*none*	*none*	1,000	PIL	200	200	100	33.0
Sulfate	14808-79-8	*none*	*none*	*none*	*none*	20,000	PIL	5,000	2000	1000	500
Sulfide	18496-25-8	*none*	*none*	*none*	*none*	100	PIL	30	4000	2000	800
TOC	TOC	*none*	*none*	*none*	*none*	20,000	PIL	500	2000	500	250

µg/L – micrograms per liter CAS – Chemical Abstracts Service [number] LOQ – Limit of Quantitation LOD – Limit of Detection MDL – Method Detection Limit
HMX - His/Her Majesty's Explosive (Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine) RDX - Royal Demolition Explosive (Hexahydro-1,3,5-trinitro-1,3,5-triazine)

Note: In most cases where the LOQ exceeds the PSL, the DL is less than PSL. Therefore data usability is not expected to be affected. The target analytes will be detectable if not quantifiable. By including the analytes on the target compound list and striving to quantify as low a concentration as possible with conventional analytical methods, the project team ensures that gross contamination will not be overlooked even though the PSL is less than the LOQ.

1. U.S. Environmental Protection Agency (EPA) (November 2010) Regional Screening Level (RSL) for tap water. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for Chromium VI used for chromium. RSL for methylmercury used for mercury.
2. Maryland Department of Environment (MDE) (2008) Cleanup Standards for Type I and II Aquifers. Standards based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents.
3. Federal Maximum Contaminant Level (MCL) per National Primary Drinking Water Regulations.
4. Vapor Intrusion (V.I.) screening values for groundwater calculated using the EPA (December 2009) RSLs for indoor air and methodology detailed in Appendix D of the EPA (2002) Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils.
5. Selected PAL is the lowest (most conservative) of the evaluated PSLs.
6. PAL References indicate the source of the selected PSL from the evaluated PSL list (see Notes 1 through 4 above), or if the PAL is selected as a Performance Indicator Level (PIL) determined for chemicals and analytes in the "OTHER" category. The OTHER chemicals and analytes provide information about the aquifer conditions and have no inherent risk (except for Nitrate, which has an EPA RSL of 5,800 µg/L and MCL of 10,000 µg/L; however, the preferred PSL/PIL for nitrate is 1,000 µg/L to evaluate natural biodegradation potential in the aquifer). (Aluminum, chloride, copper, iron, manganese, silver, sulfate, and zinc have Federal Secondary Drinking Water Standards, which are not evaluated herein).
7. Project Quantitation Limit (PQL) Goal is set at 1/3 the PSL.

8. The Laboratory LOQs and MDLs from the subcontracted laboratory(ies) are presented and are current as of June 2010. The QLs and MDLs are subject to change. The actual limits will be evaluated during the data usability assessment to ensure that the actual LOQs and MDLs are satisfactory to support the data evaluations.
9. All PALs are above QLs, except as noted: Shading indicates the Laboratory-Specific LOQ(s) is(are) greater than the Selected PAL. For purposes of selecting COPCs in the baseline human health risk assessment, ½ the LOQ will be used.
10. Laboratory will perfom a 4x concentration on these metals.

WS #15.4 -- Reference Limits and Evaluation Table

(UFP-QAPP Manual Section 2.8.1)

Matrix: Soil Gas

Chemical / Analyte	CAS Number	Selected PAL [1][4]	PQL Goal [2]	Laboratory-Specific QLs		
		EPA RSL Residential Indoor Air [1]		LOQ [3][4]	LOD [3][4]	MDL [3]
VOCs (µg/m^3)						
Acetone	67-64-1	3,200	1,067	1.2	0.154	0.154
Benzene	71-43-2	0.31	0.1	0.16	0.003	0.003
Bromodichloromethane	75-27-4	0.066	0.02	0.67	0.087	0.087
Bromoform	75-25-2	2.2	0.7	1	0.165	0.165
Bromomethane	74-83-9	0.52	0.2	0.39	0.039	0.039
Butanone, 2- (a.k.a. Methyl Ethyl Ketone (MEK))	78-93-3	520	173.3	0.29	0.091	0.091
Carbon Disulfide	75-15-0	73	24.3	1.6	0.084	0.084
Carbon Tetrachloride (CT)	56-23-5	0.16	0.05	0.63	0.346	0.346
Chlorobenzene	108-90-7	5.2	1.7	0.46	0.051	0.051
Chloroethane (a.k.a. Ethyl Chloride)	75-00-3	1,000	333	0.26	0.04	0.04
Chloroform	67-66-3	0.11	0.04	0.49	0.068	0.068
Chloromethane	74-87-3	9.4	3.1	0.21	0.033	0.033
Cyclohexane	110-82-7	630	210	0.34	0.052	0.052
Dibromochloromethane (Chlorodibromomethane)	124-48-1	0.09	0.03	0.85	0.094	0.094
Dibromoethane, 1,2- (Ethylene dibromide)	106-93-4	0.0041	0.001	0.77	0.061	0.061
Dichlorobenzene (DCB), 1,2-	95-50-1	21	7	0.6	0.174	0.174
Dichlorobenzene (DCB), 1,4-	106-46-7	0.22	0.07	0.6	0.126	0.126
Dichlorodifluoromethane (Freon 12)	75-71-8	21	7	0.49	0.074	0.074
Dichloroethane (DCA), 1,1-	75-34-3	1.5	0.5	0.081	0.004	0.004

Chemical / Analyte	CAS Number	Selected PAL [1][4]	PQL Goal [2]	Laboratory-Specific QLs		
		EPA RSL Residential Indoor Air [1]		LOQ [3][4]	LOD [3][4]	MDL [3]
Dichloroethane (DCA), 1,2-	107-06-2	0.094	0.03	0.081	0.024	0.024
Dichloroethene (DCE), 1,1-	75-35-4	21	7	0.04	0.008	0.008
Dichloroethene (DCE), cis-1,2-	156-59-2	6.3	2.1	0.079	0.008	0.008
Dichloroethene (DCE), trans-1,2-	156-60-5	6.3	2.1	0.4	0.004	0.004
Dichloropropane, 1,2-	78-87-5	0.24	0.1	0.46	0.055	0.055
Ethylbenzene	100-41-4	0.97	0.3	0.087	0.009	0.009
Hexanone, 2-	591-78-6	3.1	1.0	2	0.07	0.07
Isopropylbenzene (Cumene)	98-82-8	42	14	0.49	0.049	0.049
Methyl Isobutyl Ketone (MIBK) (4-Methyl-2-Pentanone)	108-10-1	310	103	0.41	0.066	0.066
Methyl tert-Butyl Ether (MTBE)	1634-04-4	9.4	3	0.36	0.004	0.004
Methylene Chloride (Dichloromethane)	75-09-2	5.2	2	0.69	0.076	0.076
Styrene	100-42-5	100	33	0.42	0.047	0.047
Tetrachloroethane (PCA), 1,1,2,2-	79-34-5	0.042	0.01	0.14	0.014	0.014
Tetrachloroethene (PCE)	127-18-4	0.41	0.1	0.14	0.007	0.007
Toluene	108-88-3	520	173	0.075	0.004	0.004
Trichloro-1,2,2-trifluoroethane, 1,1,2- (Freon 13)	76-13-1	3,100	1,033	0.77	0.115	0.115
Trichlorobenzene (TCB), 1,2,4-	87-61-6	0.21	0.1	3.7	1.729	1.729
Trichloroethane (TCA), 1,1,1-	71-55-6	520	173	0.11	0.005	0.005
Trichloroethane (TCA), 1,1,2-	79-00-5	0.15	0.05	0.11	0.011	0.011
Trichloroethene (TCE)	79-01-6	1.2	0.4	0.11	0.005	0.005
Trichlorofluoromethane (Freon 11)	75-69-4	73	24	0.56	0.083	0.083
Vinyl Chloride (VC)	75-01-4	0.16	0.05	0.026	0.005	0.005
m,p-Xylene	106-42-3	10	3	0.17	0.009	0.009
o-Xylene	95-47-6	10	3	0.087	0.009	0.009

Chemical / Analyte	CAS Number	Selected PAL [1][4]	PQL Goal [2]	Laboratory-Specific QLs		
		EPA RSL Residential Indoor Air [1]		LOQ [3][4]	LOD [3][4]	MDL [3]
Dioxane, 1,4-	123-91-1	0.32	0.1	0.36	0.09	0.09

µg/m^3 – micrograms per cubic meter
PAL – Project Action Limit LOQ – Limit of Quantitation LOD – Limit of Detection MDL – Method Detection Limit PQL – Project Quantitation Limit
EPA RSL – U.S. Environmental Protection Agency Regional Screening Level

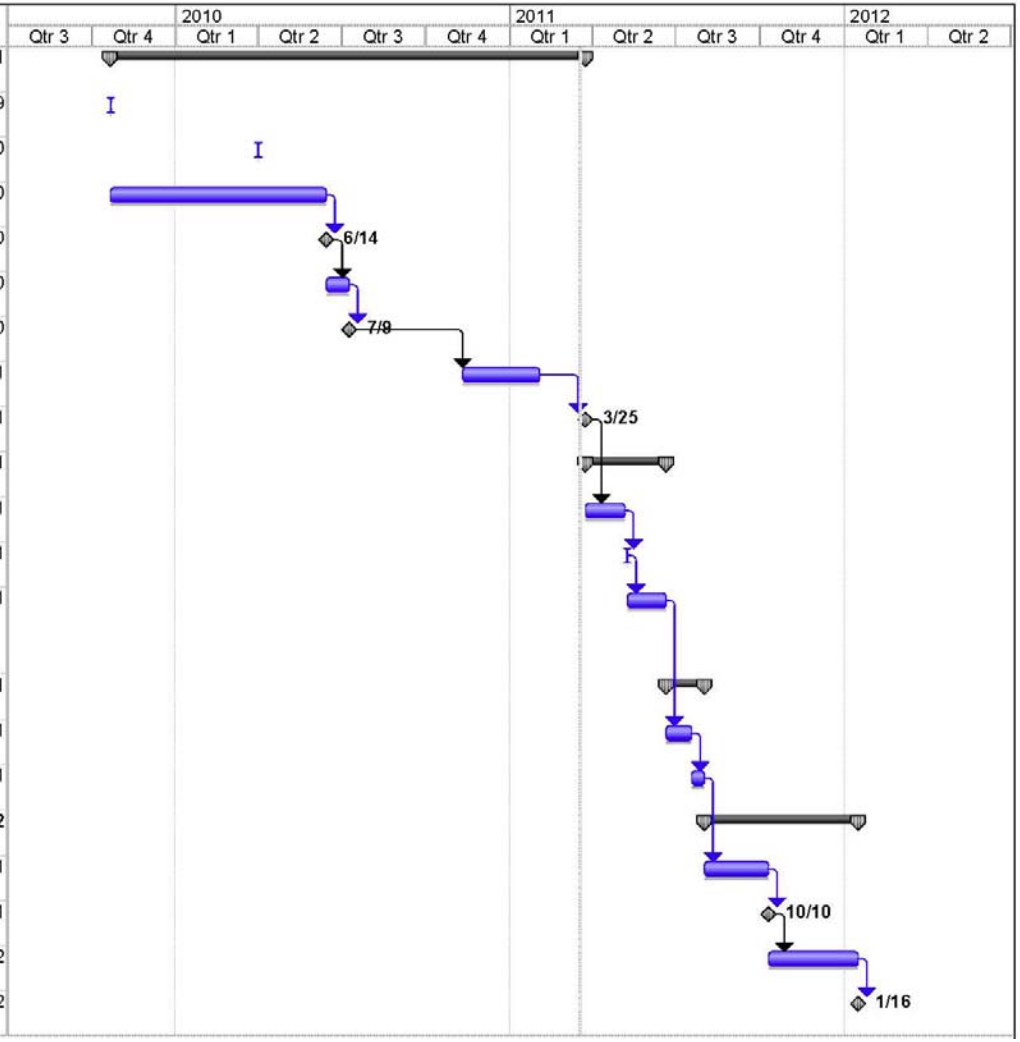
Note: In most cases where the LOQ exceeds the PAL, the DL is less than PAL. Therefore data usability is not expected to be affected. The target analytes will be detectable if not quantifiable. By including the analytes on the target compound list and striving to quantify as low a concentration as possible with conventional analytical methods, the project team ensures that gross contamination will not be overlooked even though the PAL is less than the LOQ.

1. Selected Project Action Limit (PAL) is the U.S. Environmental Protection Agency (EPA) (December 2009) Regional Screening Level (RSL) for indoor air. RSLs based on non-carcinogenic effects have been divided by 10 to account for exposure to multiple constituents. RSL for trans-1,2-DCE used as surrogate for cis-1,2-DCE.
2. Project Quantitation Limit (PQL) Goal is set at 1/3 the PAL.
3. The Laboratory QLs and MDLs from the subcontracted laboratory(ies) are presented and are current as of June 2010. The QLs and MDLs are subject to change. The actual limits will be evaluated during the data usability assessment to ensure that the actual QLs and MDLs are satisfactory to support the data evaluations.
4. All PALs are above QLs, except as noted: Shading indicates the Laboratory-Specific LOQ(s) is(are) greater than the Selected PAL. For purposes of selecting COPCs in the baseline human health risk assessment, ½ the LOQ will be used.

WS #16 -- Project Schedule

(UFP-QAPP Manual Section 2.8.2)

ID	Task Name	Start	Finish
1	**Work Plan (UFP-SAP)**	**Thu 10/22/09**	**Fri 3/25/11**
2	Scoping Session #1	Thu 10/22/09	Thu 10/22/09
3	Scoping Session #2	Thu 4/1/10	Thu 4/1/10
4	Prepare Work Plan	Thu 10/22/09	Mon 6/14/10
5	Submit Pre-Draft	Mon 6/14/10	Mon 6/14/10
6	Comment Resolution	Tue 6/15/10	Fri 7/9/10
7	Submit Draft	Fri 7/9/10	Fri 7/9/10
8	Comment Resolution	Thu 11/11/10	Wed 2/2/11
9	Submit Final	Fri 3/25/11	Fri 3/25/11
10	**Field Work**	**Fri 3/25/11**	**Mon 6/20/11**
11	Preparation	Fri 3/25/11	Fri 5/6/11
12	Utility Clearance	Mon 5/9/11	Mon 5/9/11
13	Drilling / DPT / MIP / Sampling (weekends only)	Tue 5/10/11	Mon 6/20/11
14	**Analytical Data**	**Tue 6/21/11**	**Mon 8/1/11**
15	Laboratory	Tue 6/21/11	Mon 7/18/11
16	Data Validation and Loading	Tue 7/19/11	Mon 8/1/11
17	**Report**	**Tue 8/2/11**	**Mon 1/16/12**
18	Prepare Report	Tue 8/2/11	Mon 10/10/11
19	Submit Draft	Mon 10/10/11	Mon 10/10/11
20	Comment Resolution	Tue 10/11/11	Mon 1/16/12
21	Submit Final	Mon 1/16/12	Mon 1/16/12



WS #17 -- Sampling Design and Rationale

(UFP-QAPP Manual Section 3.1.1)

The sampling design presented in the following table includes the collection of soil, groundwater, and soil gas samples to further characterize Site 43. The sampling will provide the data needed to evaluate risks to human health, determine if the potential vapor intrusion pathway is a concern and needs further evaluation, and support an FS.

Location	Matrix	Depth of Samples	Analysis [(1)]	Method	Number of Samples	Rationale	Sampling Strategy
Building 1041	Monitoring well groundwater	9-19 ft bgs	TCE, cobalt	See **WS #19**	1	Sample will be collected from MW02 to evaluate concentrations of Site-related contaminants only (i.e., to confirm results from 2005 and 2007).	See **WSs #11** and **14**
			Wet Chemistry		1	These parameters detail the aquifer conditions to evaluate the nature and fate and transport of contaminants, and to provide for thorough development of remedial alternatives in the FS.	
Building 1040	Soil	Two-ft interval just above water table (~8 ft bgs)	VOCs, Metals, Energetics		11	Samples will be collected to evaluate potential contaminant sources in soil. Soil data will be used in the baseline HHRA to evaluate potential risks. Soil samples are located in, upgradient of, and downgradient of the assumed source area (based on previous SSP investigation data).	
		Clay layer and below water table.	Laboratory hydraulic conductivity (Shelby tube), effective porosity, and bulk density		1	Sample will be collected to provide Site-specific data for use during the FS alternatives' development. Shelby tube will be collected from clay layer at SB11 during installation of MW06. Effective porosity and bulk density will be tested on soil below the water table at SB08 during installation of MW03.	

Location	Matrix	Depth of Samples	Analysis [(1)]	Method	Number of Samples	Rationale	Sampling Strategy
		Below water table.	Grain size		3	Samples will be collected to provide Site-specific data for use during the FS alternatives' development. Grain size testing will be performed on soil samples below the water table at SB08, SB09, and SB10 during installation of MW03, MW04, and MW05, respectively.	
	DPT Grab Groundwater	~15-25 ft bgs	VOCs, Metals, Energetics		7	Samples will be collected to refine the known limits of a potential VOC hot spot in groundwater. Samples are placed in, upgradient of, and downgradient of the assumed source area (based on previous SSP investigation data).	
			Wet Chemistry		4	Wet chemistry parameters will be analyzed at TW06 (upgradient of), TW07 and TW09 (in), and TW11 (downgradient of the source area). These parameters detail the aquifer conditions to evaluate the nature and fate and transport of contaminants, and provide for thorough development of remedial alternatives in the FS.	
	Monitoring well groundwater	12-22 ft bgs and four wells to-be-installed ~15-25 ft bgs	VOCs, Metals, Energetics (field filter for perchlorate samples)		5	Samples will be collected from MW01 to add to the temporal dataset and from the four new wells to bound the VOC plume. Data will be used in the baseline HHRA and to characterize the Site.	
			Wet Chemistry		1	These parameters detail the aquifer conditions to evaluate the nature of contaminants and fate and transport of contaminants, and provide for thorough development of remedial alternatives in the FS.	

Location	Matrix	Depth of Samples	Analysis [1]	Method	Number of Samples	Rationale	Sampling Strategy
	Soil Gas	Samples will be collected in the vadose zone approximately 1 to 2 feet above the water table (~6-8 feet bgs.)	VOCs		4	Samples will be collected to evaluate the potential vapor intrusion pathway into occupied Site buildings above and near the groundwater VOC plume.	

1. Groundwater "Wet Chemistry" Analytical group includes chloride, nitrate, nitrite, sulfate; methane, ethane, and ethene; sulfide; alkalinity; and dissolved TOC. Dissolved TOC samples will be field-filtered. Groundwater "Metals" Analytical Group samples will include both field-filtered (dissolved metals) and non-field-filtered (total metals) samples. VOCs for all media include 1,4-Dioxane. The "Energetics" Analytical Group for groundwater and soil includes nitroaromatics, nitrocellulose, nitroguanidine, nitroglycerin, and perchlorate.

WS #18 -- Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Sampling Location / ID Number [1]	Depth	Analytical Group	Number of Samples (identify field duplicates [2])	Sampling SOP Reference [3]
SOIL SAMPLES [4]				
SB08 (MW03) / S43SB008*TDBD*	Above the water table	VOCs, Metals, Energetics	1 (plus 1 duplicate)	GH-1.5, SA-1.3, SA-2.5
		Grain size, bulk density, effective porosity	1	GH-1.5, SA-1.3, SA-2.5
SB09 (MW04)/ S43SB009*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
		Grain size	1	GH-1.5, SA-1.3, SA-2.5
SB10 (MW05)/ S43SB010*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
		Grain size	1	GH-1.5, SA-1.3, SA-2.5
SB11 (MW06)/ S43SB011*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
	Clay Layer (~25 ft bgs)	Shelby Tube [5]	1	GH-1.5, SA-1.3, SA-2.5
SB12 (TW06)/ S43SB012*TDBD*	Above the water table	VOCs, Metals, Energetics	1 (plus 1 duplicate)	GH-1.5, SA-1.3, SA-2.5
SB13 (TW07)/ S43SB013*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
SB14 (TW08)/ S43SB014*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
SB15 (TW09)/ S43SB015*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
SB16 (TW10)/ S43SB016*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
SB17 (TW11)/ S43SB017*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
SB18 (TW12)/ S43SB018*TDBD*	Above the water table	VOCs, Metals, Energetics	1	GH-1.5, SA-1.3, SA-2.5
MONITORING WELL AND DPT GRAB GROUNDWATER SAMPLES [6]				
MW01 / S43MW00103	9-19 ft bgs	VOCs, Metals (Total and Dissolved), Energetics, Wet Chemistry	1 (plus 1 duplicate)	SA-1.1

Sampling Location / ID Number [1]	Depth	Analytical Group	Number of Samples (identify field duplicates [2])	Sampling SOP Reference [3]
MW02 / S43MW00203	12-22 ft bgs	TCE, Cobalt (Total and Dissolved), Wet Chemistry	1	SA-1.1
MW03 / S43MW00301	On top of basal unit ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics	1	SA-1.1
MW04 / S43MW00401	On top of basal unit. ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics	1	SA-1.1
MW05 / S43MW00501	On top of basal unit. ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics	1	SA-1.1
MW06 / S43MW00601	On top of basal unit . ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics	1	SA-1.1
TW06 / S43TW006*TDBD*	On top of basal unit . ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics, Wet Chemistry	1 (plus 1 duplicate)	SA-1.1, SA-2.5
TW07 / S43TW007*TDBD*	On top of basal unit . ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics, Wet Chemistry	1	SA-1.1, SA-2.5
TW08 / S43TW008*TDBD*	On top of basal unit . ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics,	1	SA-1.1, SA-2.5
TW09 / S43TW009*TDBD*	On top of basal unit . ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics, Wet Chemistry	1	SA-1.1, SA-2.5
TW10 / S43TW010*TDBD*	On top of basal unit . ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics	1	SA-1.1, SA-2.5
TW11 / S43TW011*TDBD*	On top of basal unit . ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics, Wet Chemistry	1	SA-1.1, SA-2.5
TW12 / S43TW012*TDBD*	On top of basal unit . ~15-25 ft bgs.	VOCs, Metals (Total and Dissolved), Energetics	1	SA-1.1, SA-2.5
SOIL GAS SAMPLES				
SG01 / S43SG01*TDBD*	Near Building 1040. ~6-8 ft bgs.	VOCs	1 (plus 1 duplicate)	SA-2.4
SG02 / S43SG02*TDBD*	Near Building 1040. ~6-8 ft bgs.	VOCs	1	SA-2.4
SG03 / S43SG03*TDBD*	Near Building 720. ~6-8 ft bgs.	VOCs	1	SA-2.4

Sampling Location / ID Number [1]	Depth	Analytical Group	Number of Samples (identify field duplicates [2])	Sampling SOP Reference [3]
SG04 / S43SG04*TDBD*	Near Building 720. ~6-8 ft bgs.	VOCs	1	SA-2.4

1. Refer to SOP CT-04 for sample numbering system.
2. Field duplicate locations may change in the field based sampling order and samples per day. Duplicate samples will have "-D" added to the parent sample ID (this is a deviation from Facility SOP CT-04).
3. Facility SOPs are provided in Appendix D of the NSF-IH Master UFP-SAP.
4. "TDBD" refers to the two-digit top depth and two-digit bottom depth from which the soil sample is collected. Samples will be collected just above the water table (determined in the field).
5. Shelby tube will be collected in the basal clay layer for offSite laboratory testing of hydraulic conductivity.
6. Groundwater samples from monitoring wells MW01 and MW02 are being collected for the third time (Round 3), while the to-be-installed monitoring wells will be collected for the first time (Round 1). Groundwater "Wet Chemistry" Analytical group includes chloride, nitrate, nitrite, sulfate; methane, ethane, and ethene; sulfide; alkalinity; and dissolved TOC. Dissolved TOC samples will be field-filtered. Groundwater "Metals" Analytical Group samples will include both field-filtered (dissolved metals) and non-field-filtered (total metals) samples. Filtered samples will have a "-F" added to the parent sample ID (consistent with the Facility SOP CT-04). For DPT grab groundwater sample IDs, the "TDBD" refers to the two-digit top depth and two-digit bottom depth of the screened interval from which the sample is collected.

VOCs for all media include 1,4-Dioxane.

Energetics for groundwater and soil include nitroaromatics, nitrocellulose, nitroguanidine, nitroglycerin, and perchlorate (samples will be field filtered for perchlorate).

WS #19 -- Analytical SOP Requirements Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	Analytical and Preparation Method / SOP Reference[1]	Sample Size	Containers (number, size, and type)[2]	Preservation Requirements	Maximum Holding Time[(3)] (preparation / analysis)
Soil	VOCs	SW-846 5035/8260B, **Empirical SOP-202/225**	5 grams (g) (each)	Three terra cores sample vials (5g size)	Methanol/sodium bisulfite solution Cool to 0 to 6 °C	14 days from sampled date to analyze
Groundwater and aqueous QC samples	VOCs	SW-846 5030/8260B, **Empirical SOP-202**	Three 40-milliliter (mL) glass vials	5 mL	Hydrochloric acid (HCl) to pH<2; Cool to ≤ 6 °C; no headspace	14 days to analysis
Soil	Explosives (including nitroglycerin)	SW-846 8330B **Empirical SOP-327**	One 4-oz glass jar	2 to 3 pounds	Cool to 0 to 6 °C	14 days until extraction, 40 days to analysis
Groundwater	Explosives (including nitroglycerin)	SW-846 8330B **Empirical SOP-327**	Two 1 – L glass amber bottles	1000 mL	Cool to 0 to 6 °C	7 days until extraction, 40 days to analysis
Soil	Nitroguanidine	SW846 8330 modified / **Empirical SOP-233**	30 g	One 8-oz wide-mouth glass jar	Cool to 0 to 6 °C	14 days from sampled date to extract / 40 days from extraction to analysis
Groundwater	Nitroguanidine	SW846 8330 modified / **Empirical SOP-233**	1-L	One 1-L amber glass bottle	Cool to 0 to 6 °C	28 days from sampled date to analysis
Soil	Perchlorate	SW-846 6850 **Empirical SOP-239**	One 4-oz glass jar	5 g	Cool to ≤ 6 °C	28 days to analysis
Groundwater	Perchlorate	SW-846 6850 **Empirical SOP-239**	500 mL plastic	100 mL	Cool to ≤ 6 °C	28 days to analysis
Soil	Metals	SW-846 3050B/ 6010C, **Empirical SOP-100/105**	One 4-oz glass jar	1 to 2 g	Cool to ≤ 6 °C	180 days to analysis except mercury, 28 days for mercury
Groundwater	Metals	SW-846 3010A/ 6010C, **Empirical SOP-100/105**	One 500-mL plastic bottle	50 mL	Nitric acid to pH <2; Cool to ≤ 6 °C	180 days to analysis except mercury, 28 days for mercury
Soil	Cyanide	SW-846 9012A, **Empirical SOP-164/175**	One 4-oz glass jar	5 g	Cool to ≤ 6 °C	14 days to analysis
Groundwater	Cyanide	SW-846 9012A, **Empirical SOP-164/175**	One 250-mL plastic bottle	100 mL	Sodium hydroxide to a pH > 12; Cool to ≤ 6 °C	14 days to analysis
Groundwater	Anions (nitrate, nitrite, chloride and sulfate)	USEPA 300.0, **Empirical SOP-145**	One 500-mL plastic bottle	5 mL for each analyte	Cool to ≤ 6 °C	Nitrate & Nitrite – 48 hours from sampled time to analysis. Chloride & Sulfate – 28 days from sampled date to analysis
Groundwater	Dissolved sulfide	SM4500S F, **Empirical SOP-153**	One 500-mL plastic bottle	200 mL	1 mL 2 N zinc acetate with NaOH to a pH >9; Cool to 4 (± 2) °C	7 days to analysis
Soil	TOC	Lloyd Kahn, **Empirical SOP-221**	One 4-oz glass jar	30 g	Cool to ≤6 °C	14 days to analysis

Groundwater	TOC	SW-846 9060/9060A, **Empirical SOP-221**	One 500-mL plastic bottle	250 mL	Sulfuric acid to pH <2; Cool to 4 (± 2) °C	28 days
Groundwater	Dissolved Gases	RSK SOP 175, **Empirical SOP-236**	Three 40-mL glass vials	15 mL	HCl to pH <2; Cool to 4 (± 2) °C	14 days from sampled date to analyze
Groundwater	Alkalinity	SM 2320B, **Empirical SOP-154**	One 500-mL plastic bottle	25 mL	Cool to ≤6 °C	14 days from sampled date to analyze
Solid IDW	TCLP – Volatiles	SW846 1311 & 8260B	25 g	One 8-oz wide-mouth glass jar	Cool to ≤6 °C	14 days from sampled date to leaching, 14 days from leaching to analysis
Aqueous IDW	TCLP – Volatiles	SW846 1311 & 8260B	500 mL	One 1-L amber glass bottle	Cool to ≤6 °C	14 days from sampled date to leaching, 14 days from leaching to analysis
Solid IDW	TCLP – Metals	SW846 1311, 6010C & 7470A	100 g	One 8-oz wide-mouth glass jar	Cool to ≤6 °C	28 days from sampled date to leaching, 28 days from leaching to analysis
Aqueous IDW	TCLP – Metals	SW846 1311, 6010C & 7470A	500 mL	One 1-L High Density Polyethylene (HDPE) bottle	Cool to ≤6 °C	28 days from sampled date to leaching, 28 days from leaching to analysis
Solid IDW	Corrosivity	SW846 9045D	20 g	One 4-oz wide-mouth glass jar	Cool to ≤6 °C	As soon as possible after laboratory receipt
Aqueous IDW	Corrosivity	SW846 9040C	20 mL	One 100mL High Density Polyethylene (HDPE) bottle	Cool to ≤6 °C	As soon as possible after laboratory receipt
Solid IDW	Ignitibility	SW846 1010	20 g	One 4-oz wide-mouth glass jar	Cool to ≤6 °C	As soon as possible after laboratory receipt
Aqueous IDW	Ignitibility	SW846 1010	100 mL	One 100mL High Density Polyethylene (HDPE) bottle	Cool to ≤6 °C	As soon as possible after laboratory receipt
Soil gas	VOCs	Modified EPA TO-15/ Air Toxics SOPs-83 and 38 (simultaneous low level and SIM analysis)4	6 Liter Summa	500 milliliter (mL) maximum	None required	30 days to analysis
Geotechnical Soil[5]	Permeability	ASTM D5084	Shelby tube	Shelby tube	None required	As soon as possible after laboratory receipt
	Effective Porosity	ASTM D854, D2216	Shelby tube	Shelby tube	None required	As soon as possible after laboratory receipt
	Bulk Density	ATI RP40	Shelby tube	Shelby tube	None required	As soon as possible after laboratory receipt
	Grain size	ASTM D422	Shelby tube	Shelby tube	None required	As soon as possible after laboratory receipt

[1] Laboratory SOPs are subject to revision and updates during duration of the project, the laboratory will use the most current revision of the SOP at the time of analysis.

[2] Sample size is a minimum; the containers listed will be filled to compensate for any required re-analysis or re-extractions. For samples requiring Matrix Spike (MS)/Matrix Spike Duplicate (MSD), containers listed should be tripled.

[3] Maximum holding time is calculated from the time the sample is collected to the time the sample is prepared/extracted.

[4] Soil Gas samples will undergo SIM and low level VOC analyses, The analytes marked with an asterisk on Worksheets 15 will be reported from the SIM run all others from the low level scan. Both analyses can be run from the same Summa canister.

[5] IDW sample analyses and geotechnical sample analyses are presented on this worksheet for the utilization of field personnel. Quality control information is not presented in any of the remaining worksheets as these samples.

WS #20 -- Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1)

Matrix	Analytical Group	No. of Sampling Locations	No. of Field Duplicates (1)	No. of MS/MSDs (2)	No. of Source Water Blanks (3)	No. of Equip. Blanks (4)	No. of VOA Trip Blanks (5)	No. of PT Samples (6)	Total No. of Samples to Lab
Soil	VOCs	11	2	1/1	1	4	4	N/A	24
	Metals	11	2	1/1	0	4	0	N/A	19
	Energetics	11	2	1/1	0	4	0	N/A	19
	TOC	11	0	1/1	0	0	0	N/A	13
Groundwater	VOCs	13	2	1/1	1	4	4	N/A	26
	Metals (7)	13	2	1/1	0	4	0	N/A	21
	Energetics (7)	13	2	1/1	0	4	0	N/A	21
	Wet Chemistry (7)	6	0	1/1	0	0	0	N/A	8
Soil Gas	VOCs	4	1	0	1	0	0	N/A	6

1. One field duplicate for every 10 or fewer investigative samples.
2. Although the matrix spike (MS)/matrix spike duplicate (MSD) is not typically considered a field QC sample, it is included here because location determination is often established in the field. One MS/MSD sample for every 20 or fewer investigative samples per sample matrix (e.g., groundwater, soil).
3. One source water blank for VOC analysis per water source. It is not anticipated that mutipule sources of water will be utilized.
4. For non-disposable equipment, one equipment blank per type of non-dedicated, decontaminated equipment, with a minimum of one per week per sampling device/instrument. For pre-cleaned, dedicated, and/or disposal sampling equipment, equipment blank samples will not be collected.
5. One trip blank per cooler containing samples for VOCs analysis (not including methane, ethane, and ethene [dissolved gases]).
6. The number of batch or project-specific proficiency testing (PT) samples is optional but highly recommended. N/A to this project.
7. Groundwater “Wet Chemistry” Analytical group includes chloride, nitrate, nitrite, sulfate; methane, ethane, and ethene; sulfide; alkalinity; and dissolved TOC. Dissolved TOC samples will be field-filtered. Groundwater “Metals” Analytical Group samples will include both field-filtered (dissolved metals) and non-field-filtered (total metals) samples. Energetics for groundwater and soil include nitroaromatics, nitrocellulose, nitroguanidine, nitroglycerin, and perchlorate.

WS #21 -- Project Sampling SOP References Table

(UFP-QAPP Manual Section 3.1.2)

This worksheet lists the SOPs to be used/referenced during the RI effort. The Facility SOPs are provided in Appendix D of the NSF-IH Master UFP-SAP.

SOP Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
Sample Management					
CT-04	Sample Nomenclature, 02/04, Rev. 0	NSF-IH	N/A	N	None.
Geology					
GH-1.1	Site Reconnaissance, 02/04, Rev. 0	NSF-IH	N/A	N	None.
GH-1.2	Evaluation of Existing Monitoring Wells and Water Level Measurement, 02/04, Rev. 0	NSF-IH	Water level indicator	N	None.
GH-1.3	Soil and Rock Drilling Methods, 02/04, Rev. 0	NSF-IH	Drilling rig and accessories	N	None.
GH-1.5	Borehole and Sample Logging, 02/04, Rev. 0	NSF-IH	NA	N	None.
Hydrogeology					
GH-2.4	In-Situ Hydraulic Conductivity Testing, 02/04, Rev. 0	NSF-IH	Pump, bailer, or slug and water level indicator	N	None.
GH-2.8	Groundwater Monitoring Well Installation, 02/04, Rev. 0	NSF-IH	Drilling rig, accessories, and well supplies	N	None.
GH-2.9	Well Abandonment, 02/04, Rev. 0	NSF-IH	Well sealing materials	N	None.
Field Analytical Parameters					
SA-1.6	Natural Attenuation Parameter Collection, 03/08, Rev. 0	NSF-IH	Field test kits	N	None.
Water/Soil Sampling					
SA-1.1	Groundwater Sample Acquisition and OnSite Water Quality Testing, 03/08, Rev. 1	NSF-IH	Pump, tubing, water quality meter, and accessories	N	None.

SOP Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
SA-1.3	Soil Sampling, 03/08, Rev. 1	NSF-IH	EnCore sampler, trowel, shovel, hand auger, and/or split-barrel sampler	N	None.
SA-2.5	Direct Push Technology (Geoprobe/Hydropunch), 02/04, Rev. 0	NSF-IH	Drilling equipment and accessories	N	None.
Air Sampling					
SA-2.2	Air Monitoring and Sampling, 02.04, Rev. 0	NSF-IH	Air sampling pump and accessories, photoionization detector (PID), and/or flame ionization detector (FID)	N	None.
SA-2.4	Soil Gas Sampling, 02/04, Rev. 0	NSF-IH	Drilling equipment and accessories, air sampling pump and accessories, and PID or FID	N	None.
Documentation					
SA-6.1	Non-Radiological Sample Handling, 02/04, Rev. 0	NSF-IH	Sample bottleware, packaging material, shipping materials, field filtration equipment	N	None.
SA-6.3	Field Documentation, 02/04, Rev. 0	NSF-IH	Field logbook, field sample forms, boring logs	N	None.

SOP Reference Number	Title, Revision Date and / or Number	Originating Organization of Sampling SOP	Equipment Type	Modified for Project Work? (Y/N)	Comments
Decontamination					
SA-7.1	Decontamination of Field Equipment, 03/08, Rev. 1	NSF-IH	Decontamination equipment, phosphate-free detergent, deionized water	N	None.
Health and Safety					
HS-1.0	Utility Locating and Excavation Clearance, 02/04, Rev. 0	NSF-IH	Remote subsurface sensing equipment, magnetometer, GPR	N	None.

WS #22 -- Field Equipment Calibration, Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.1.2.4)

Field Equipment	Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	Facility SOP Reference (1)	Comments
PID	Visual Inspection, Calibration	Daily, before use	Manufacturer's Guidance	Replace	Tetra Tech FOL or designee	SA-2.2, Manufacturer's Guidance	None.
DPT/Drill Rig	Inspection	Daily	Equipment inspection sheet criteria	Replace	Tetra Tech FOL or designee	GH-1.3, GH-1.5, GH-2.8, SA-2.5	None.
Disposable Hand Trowel	Inspection	Per use	N/A	Replace	Tetra Tech FOL or designee	SA-1.3	None.
Water Level Meter	Visual Inspection	Daily	Manufacturer's Guidance	Replace	Tetra Tech FOL or designee	Manufacturer's Guidance	None.
Multi-Parameter Water Quality Meter (pH, Temp., Sp. Cond., D.O., ORP)	Visual Inspection, Calibration	Daily, before use	Manufacturer's Guidance	Replace	Tetra Tech FOL or designee	SA-1.1, Manufacturer's Guidance	None.
Turbidity Meter	Visual Inspection, Calibration	Daily, before use	Manufacturer's Guidance; Calibrations must bracket expected values. Initial Calibration Verification (ICV) must be + or -10 Nephelometric Turbidity Units (NTUs) of target value.	Replace	Tetra Tech FOL or designee	SA-1.1, Manufacturer's Guidance	None.
Groundwater sampling pumps and tubing	Inspect pumps, tubing and air/sample line quick-connects	Regularly	Maintained in good working order per manufacturer's recommendations	Replace	Tetra Tech FOL or designee	SA-1.1, Manufacturer's Guidance	None.

Activities may include: calibration, verification, testing, maintenance, and/or inspection.
1. Facility SOPs are provided in Appendix D of the NSF-IH Master UFP-SAP. Also see Appendix B (Field and General Sampling Operations) and Appendix C (Example Forms) in the Master UFP-SAP.

WS #23 -- Analytical SOP References Table

(UFP-QAPP Manual Section 3.2.1)

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (1)
Empirical SOP-100	Metals Digestion/ Preparation, Methods 3005A/ USEPA CLP ILMO 4.1 Aqueous, 3010A, 3030C, 3050B, USEPA CLP ILMO 4.1 (Soil/Sediment), 200.7, Standard Methods 3030C (Revision 20, 04/27/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Metals Digestion	None/Preparation	Empirical	N
Empirical SOP-103	Mercury Analysis in Water by Manual Cold Vapor Technique, Methods USEPA SW846 7470A and 245.1, CLP-M 4.1 (Revision 18, 04/11/10)	Definitive	Groundwater, and aqueous QC samples/ Mercury	Flow Injection Mercury Analyzer	Empirical	N
Empirical SOP-104	Mercury Analysis in Soil/Sediment by Manual Cold Vapor Technique, Methods SW846 7471A, 7471B, 245.5, and CLP-ILM 4.1 (Revision 19, 04/11/10)	Definitive	Soil / Mercury	Flow Injection Mercury Analyzer	Empirical	N
Empirical SOP-105	Metals by Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES) Technique, SW-846 Methods 6010B, 6010C, USEPA Method 200.7, Standard Methods 19th Edition 2340B, USEPA CLP ILMO 4.1 (Revision 16, 04/11/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Metals	ICP-AES	Empirical	N
Empirical SOP-145	Determination of Inorganic Anions in Water by Ion Chromatography using Dionex DX-500 Ion Chromatographs with Hydroxide Eluent and Dionex Column AS18, Method 300.0 Guidance (Revision 7, 03/25/10)	Definitive	Groundwater and aqueous QC samples / anions	Dionex Ion Chromatography	Empirical	N
Empirical SOP-153	Sulfide Method 376.1 and Standard Methods SM4500S-F(19th Edition) Titrametric, Iodine) with Sample Pretreatment to Remove Interfering Substances or to Concentrate the Sulfide (Revision 4, 09/07/10)	Definitive	Groundwater and aqueous QC samples/sulfide	Buret	Empirical	N
Empirical SOP-154	Alkalinity by USEPA Method 310.1, SM2320B (Revision 15, 09/07/10)	Definitive	Groundwater and aqueous QC samples / alkalinity	Buret/pH meter	Empirical	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (1)
Empirical SOP-164	Distillation of Aqueous/Solid Samples for Cyanide, Total and Amenable, SW846 Method 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM4500-CN C, G, 18th and 19th ED/ USEPA CLP ILMO 4.1 (Revision 15, 03/25/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Cyanide preparation	None/ Distillation	Empirical	N
Empirical SOP-175	Post-Distillation Analysis for Cyanide by Lachat Flow Injection Analyzer, Methods 335.4, SW846 9012A, USEPA-CLP 4.1; Addendum for USEPA CLP ILM 05.2 Aqueous/Soil/ Sediment (Revision 11, 09/07/10)	Definitive	Soil, groundwater, and aqueous QC samples/ Cyanide	Automated Ion Analyzer	Empirical	N
Empirical SOP-202	GC/MS Volatiles using USEPA Method 624 and SW846 Method 8260B, Including Appendix IX Compounds (Revision 23, 09/09/10)	Definitive	Soil, groundwater, and aqueous QC samples/ VOCs	Gas Chromatography/Mass Spectroscopy (GC/MS)	Empirical	N
Empirical SOP-221	TOC SM5310C, SW846 Method 9060/9060A and Lloyd Kahn Method (Revision 9, 07/12/10)	Definitive	Soil/ TOC	TOC Analyzer	Empirical	N
Empirical SOP-225	GC/MS Volatile Non-Aqueous Matrix Extraction using SW-846 Method 5035 for 8260B Analysis (Revision 9, 9/07/10)	Definitive	Soil / VOCs Extraction	Purge and Trap instrumentation coupled with GC/MS	Empirical	N
Empirical SOP-233	Nitroguanidine in soil and water by High Performance Liquid Chromatography (Revision 3, 02/23/10).	Definitive	Soil, groundwater, and aqueous QC samples / Perchlorate	High Performance Liquid Chromatography (HPLC)	Empirical	N
Empirical SOP-239	Perchlorate in Water, Soil, and Solid Waste Using High Performance Liquid Chromatography/ Electrospray Ionization/ Mass Spectrometry by SW846 Method 6850 (Revision 04, 09/07/10)	Definitive	Soil, groundwater, and aqueous QC samples / Perchlorate	High Performance Liquid Chromatography/ Electrospray Ionization/ Mass Spectrometry (HPLC/ESI/MS)	Empirical	N
Empirical SOP-327	Nitroaromatics and Nitramines by High Performance Liquid Chromatography (HPLC) Method 8330, 8330A and 8332 (Revision 18, 09/07/10)	Definitive	Soil, groundwater, and Aqueous QC Blanks /Explosives and Nitroglycerin	HPLC	Empirical	N

Lab SOP Number	Title, Revision Date, and / or Number	Definitive or Screening Data	Matrix and Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (1)
SOP-83[1]	Analysis of VOCs in Summa Polished Canisters by GC/MS Low Level, Revision 17.1, December 2009.	Definitive	Soil Gas, Indoor Air, and Outdoor Air/ VOCs	Agilent/HP GC/MS	Air Toxics Ltd.	No
SOP-38[1]	Analysis of VOCs in Summa Polished Canisters by GC/MS SIM, Revision 17.1, December 2009.	Definitive	Soil Gas, Indoor Air, and Outdoor Air/ VOCs	Agilent/HP GC/MS	Air Toxics Ltd.	No

SOP – standard operating procedure

1. Air Toxics Ltd. SOPs are proprietary and cannot be provided as part of this submittal but can be reviewed via the internet after the party wishing to review the SOP signs a nondisclosure agreement (NDA). The review process is as follows:
   - Air Toxics Ltd. sends the NDA in PDF to the party wishing to view the SOP.
   - The potential viewer signs and returns the NDA to Air Toxics Ltd. via email.
   - Air Toxics Ltd. notifies the potential viewer that they will be allowed to view the SOP and provides a user name, password, web Site address and a notification that viewing will be supported for a very limited time (e.g., 0.5 hrs).
   - The viewer accesses the web Site and views the SOP. Downloading, copying, and printing of the SOP are prevented.

WS #24 -- Analytical Instrument Calibration Table

(UFP-QAPP Manual Section 3.2.2)

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
GC/MS VOCs	Bromofluoro-benzene (BFB) Tune	Prior to each Initial Calibration (ICAL) and at the beginning of each 12-hour period.	Must meet the ion abundance criteria required by the method (SW8260B; Section 7.3.1; Table 4).	Retune and/or clean or replace source. No samples may be accepted without a valid tune.	Analyst/ Supervisor	Empirical SOP-202
	ICAL – a minimum of a 5-point calibration is prepared for all target analytes	Upon instrument receipt, for major instrument changes, or when continuing calibration verification (CCV) does not meet criteria.	The average response factor (RF) for System Performance Check Compound (SPCCs) must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent relative standard deviation (%RSD) for RFs for calibration check compounds (CCCs) must be ≤ 30%; and %RSD for each target analyte must be ≤ 15%, or the linear regression correlation coefficient (r) must be ≥ 0.995; or the coefficient of determination (r^2) must be ≥ 0.99 (6 points are required for second order).	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	Retention Time (RT) Window Position Establishment	Once per ICAL for each analyte and surrogate.	Position shall be set using the midpoint standard of the ICAL curve when ICAL is performed. On days when ICAL is not performed, the initial CCV is used.	NA.	Analyst / Supervisor	
	Evaluation of Relative Retention Times (RRTs)	With each sample.	RRT of each target analyte must be within ± 0.006 RRT units.	Correct problem, then rerun ICAL.	Analyst / Supervisor	
	Initial Calibration Verification (ICV) – Second Source	Once after each ICAL, prior to beginning a sample run.	The percent recovery (%R) for all target analytes must be within 80-120% of true values.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst/ Supervisor	
	CCV	Perform one per 12-hour analysis period after tune and before sample analysis.	The minimum RF for SPCCs must be ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachloroethane, ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. The percent difference or percent drift (%D) for all target analytes and surrogates must be ≤ 20%.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	
HPLC Nitroguanidine	ICAL	Prior to analyzing any samples. A minimum of 5-points for linear fits and a minimum of 6-points for quadratic Fits. Low standard at the LOQ level.	Average RF if %RSD < 20%. Linear fit with correlation coefficient of 0.995 or better. Quadratic fit with squared correlation coefficient of 0.99 or better.	Samples cannot be analyzed until there is a passing calibration. Re-evaluate curve mix and makeup. Re-run curve, Check instrument for maintenance needs. Re-prepare the curve standards	Analyst/ Supervisor	**Empirical SOP-233**
	Retention Time Established	Once per ICAL and at the beginning of the analytical shift.	Set using the midpoint of the ICAL or the initial CCV when ICAL is not performed.	NA.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	ICV	Alternate source standard to be analyzed after every calibration curve	%D must be < 15%.	Samples cannot be analyzed until there is a passing ICV. Re-analyze an ICV from a different source. Re-prepare and re-analyze the ICV. Re-calibrate and verify standard preparations and Sources.	Analyst/ Supervisor	
	CCV	At the beginning of every sequence. For every 10-client samples. The concentration must be varied from low to mid range. At the end of every sequence	%D must be < 15%.	Re-prepare and re-analyze the CCV. Re-calibrate and verify standard preparations and sources. Re-analyze all samples analyzed since the last passing CCV.	Analyst/ Supervisor	
HPLC Explosives (including Nitrglycerin)	ICAL – A minimum of a 5-point calibration curve is analyzed.	After major instrument maintenance; upon failure of CCV.	The %RSD for RFs for each analyte must be ≤ 20%; or the linear regression correlation coefficient (r) must be ≥ 0.995.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	**Empirical SOP-327**
	ICV - Second source	Once after each ICAL, prior to beginning a sample run.	All project analytes must be within established RT windows. The %Rs for all analytes must be within 80-120% of true value.	Correct problem, re-prepare and re-analyze ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst/ Supervisor	
	CCV	After each 10 field samples and at the end of the analytical sequence.	%D for all target compounds must be ≤ 20%;	Correct problem and rerun CCV and all samples analyzed before and after the failing CCV. If that fails, repeat ICAL and re-analyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
LCMS Perchlorate	Tune	Prior to ICAL and after any mass calibration or maintenance is performed.	Tuning standards must contain the analytes of interest and meet acceptance criteria outlined in the laboratory SOP.	Re-tune instrument. If the tuning will not meet acceptable criteria, an instrument mass calibration must be performed and the tuning redone. Sample analysis shall not proceed without acceptable tuning.	Analyst/ Supervisor	**Empirical SOP-239**
	ICAL – A minimum of a five-point calibration is prepared.	Upon instrument receipt, major instrument change, or when the CCV does not meet criteria.	The %RSD for RFs must be ≤ 20% or r must be ≥ 0.995. The concentration corresponding to the absolute value of the Y-intercept of the calibration curve must be ≤ LOD.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	
	ICV – Second Source	Once after each ICAL, prior to the analysis of samples.	The %R must be within 85-115%.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples can be analyzed until ICV has been verified.	Analyst/ Supervisor	
	CCV	Before sample analysis and after every 10 field samples, and at the end of the sequence.	Low-range standard: Perchlorate must be within 50-150 %R. Mid-range standard: Perchlorate must be within 85-115 %R.	Correct problem and rerun CCV. If that fails, repeat ICAL and re-analyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	
	Limit of Detection Verification (LODV) (per batch)	Prior to sample analysis and at the end of the sequence. It can be analyzed after every 10 samples to reduce the re-analysis rate.	The %R must be within 70-130%. Per the DoD Quality Systems Manual (QSM) for Environmental Laboratories, Version 4.1, the perchlorate spike concentration must be approximately 2 times the LOD.	Correct problem, rerun LODV and all samples since last successful LODV. If a sample with perchlorate concentration at or between the LOD and LOQ is bracketed by a failing LODV, it must be re-analyzed. A sample with a concentration above the LOQ can be reported. Results cannot be reported without a valid LODV.	Analyst/ Supervisor	
IC Anions	ICAL – A minimum of 8 points and establish linear calibration range.	Perform after major instrument maintenance and upon failure of second consecutive CCV.	Must be < 15% RSD over linear range, or r ≥0.995.	Correct the problem, then repeat ICAL.	Analyst/ Supervisor	**Empirical SOP-145**
	ICV – Second Source	After ICAL and each analytical sequence.	The %R must be within 90-110% of the true value.	Recalibrate.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	CCV	Every 10 samples and at the end of the analytical sequence.	The %R must be within 90-110% of the true value.	Recalibrate.	Analyst/ Supervisor	
Buret Sulfide	Standardization	Daily prior to sample analysis.	Standardized using 0.25 N sodium thiosulfate.	An acceptable titrant is compared against an independent source identified as an LCS/ICV.	Analyst/ Supervisor	**Empirical SOP-154**
	ICV – Second Source	After ICAL and each analytical sequence.	The %R must be within 80-120% of the true value.	Recalibrate.	Analyst/ Supervisor	
	CCV	At beginning and end of each run sequence and every 10 samples.	The %R must be within 80-120 % of the true value.	If the CCV fails high, report samples that are less than the LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst/ Supervisor	
TOC Analyzer	ICAL – a minimum of a 5-point calibration is prepared	Upon instrument receipt, major instrument change, or when the CCV does not meet criteria.	The RSD for RFs for the target analyte must be ≤ 20%, or r must be ≥ 0.995.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	**Empirical SOP-221**
	ICV – Second Source	Once after each ICAL prior to sample analysis.	The %R must be within 90-110% of true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst/ Supervisor	
	CCV	Analyze standard at the beginning and end of sequence and after every 10 samples.	The %R must be within 90-110% of true value.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst/ Supervisor	
ICP-AES Metals	ICAL - a 1-point calibration per manufacturer's guidelines is prepared for all target analytes	At the beginning of each day, or if the QC is out of criteria, prior to sample analysis.	None; only one high standard and a calibration blank must be analyzed. If more than one calibration standard is used, r must be ≥ 0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst/ Supervisor	**Empirical SOP-100/105**
	ICV – Second Source	Following ICAL, prior to the analysis of samples.	The %R of all target analytes must be within 90-110% of true value.	Investigate reasons for failure, reanalyze once. If still unacceptable, correct problem and repeat ICAL.	Analyst/ Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	CCV	At the beginning and end of the sequence and after every 10 samples.	The %R of all target analytes must be within 90-110% of true value.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze all affected samples.	Analyst/ Supervisor	
	Initial Calibration Blank (ICB)	Before beginning a sample sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze.	Analyst / Supervisor	
	Continuing Calibration Blank (CCB)	After the initial CCV, after every 10 samples, and at the end of the sequence.	No target analytes detected > LOD.	Correct the problem, then re-prepare and reanalyze calibration blank and all affected samples.	Analyst / Supervisor	
	Low-Level Check Standard (if using 1-point ICAL)	Daily after 1-point ICAL and before samples.	The %R of all target analytes must be within 80-120% of true value.	Investigate and perform necessary equipment maintenance. Recalibrate and reanalyze all affected samples.	Analyst / Supervisor	
	Interference Check Standards (ICS – ICS A and ICS B)	At the beginning of an analytical run.	The absolute value of ICS A recoveries for non-spiked analytes must be < LOD; and ICS B recoveries must be within 80-120 %R of true value.	Terminate analysis; locate and correct problem; reanalyze ICS.	Analyst / Supervisor	
Cold Vapor Mercury Analyzer	ICAL – A minimum of a 5-point calibration is prepared	Perform daily prior to sample analysis.	The %RSD for RFs must be ≤ 20%, or r must be ≥ 0.995.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst/ Supervisor	**Empirical SOP-103/104**
	ICB	Before beginning a sample sequence.	No mercury detected > LOD.	Correct problem, then re-prepare and reanalyze.	Analyst / Supervisor	
	ICV - Second Source	Once after each ICAL and prior to sample analysis	The %R must be within 90-110% of the true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst / Supervisor	
	CCB	After each CCV, after every 10 samples, and at the end of the sequence	No mercury detected > LOD.	Investigate source of contamination, rerun any samples not bracketed by passing blanks.	Analyst / Supervisor	
	CCV	At beginning and end of each run sequence and every 10 samples.	The %R must be within 80-120% of the true value.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	CCV (undistilled)	CCV (undistilled) at the beginning and end of each run sequence and after every 10 samples.	The %R must be within 90-100% of true value.	If the CCV (undistilled) fails high, report samples that are <LOQ. Recalibrate and/or reanalyze samples back to last acceptable CCV.	Analyst / Supervisor	
Lachat Cyanide	ICAL– A minimum of a 6-point calibration is prepared	Perform daily prior to sample analysis.	r must be ≥ 0.995. All calibration standards must be distilled if samples are expected to contain sulfides.	Correct problem then repeat ICAL. No samples may be run until ICAL has passed.	Analyst, Supervisor	**Empirical SOP-164/175**
	ICV – Second Source	At the start of every sequence prepared fresh daily (undistilled), prior to sample analysis.	The %R must be within 85-115% of true value.	Correct problem and verify ICV. If that fails, correct problem and repeat ICAL. No samples may be run until ICV has been verified.	Analyst, Supervisor	
	ICB	After the ICV (undistilled).	Must be < LOQ for the target analyte.	Determine source of contamination, correct problem, and restart sequence.	Analyst, Supervisor	
	CCV	Every 10 samples (undistilled).	The %R must be within 85-115% of true value.	Correct problem and rerun CCV. If that fails, repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst, Supervisor	
	ICV (distilled, high and low)	Once per ICAL.	The %R must be within 85-115% of true value.	Determine cause for failure, correct problem, and redistill standards.	Analyst, Supervisor	
GC/FID Dissolved Gases	ICAL – Minimum of a 5-point calibration curve is prepared.	Perform after major instrument maintenance and upon failure of second consecutive CCV.	RSD for each analyte must be ≤ 20%, or r must be ≥ 0.995.	Recalibrate and/or perform the necessary equipment maintenance. Check the calibration standards. Reanalyze the affected data.	Analyst / Supervisor	**Empirical SOP-236**
	ICV – Second Source	Once after each initial calibration.	The %R of all analytes must be within 75-125% of the true value.	Correct problem and verify second source standard. Reanalyze ICAL.	Analyst / Supervisor	
	CCV	Perform one every 10 samples and at the end of analysis period.	The %R of all analytes must be within 80-120% of the true value.	Repeat ICAL and reanalyze all samples analyzed since the last successful CCV.	Analyst / Supervisor	
pH meter Alkalinity	Standardization	Daily prior to sample analysis.	Standardize using pH 7 and pH 4, adjust as needed, and reread. Must be within ± 0.05 pH units to proceed.	Re-standardize.	Analyst / Supervisor	**Empirical SOP-154**

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
	Buffer check	Check every 3 hours.	Must be within ± 0.20 pH units.	Re-standardize and rerun samples.	Analyst / Supervisor	
GC/MS – VOCs Low Level (soil gas)	Tune Verification - BFB	Every 24 hours, or every 12 hours if project requires.	SW – 846 tune criteria.	Correct problem then repeat tune.	Analyst / Supervisor	Air Toxics Ltd. SOP-83
	ICAL – Minimum of a 5-point calibration curve is prepared.	Prior to sample analysis.	% RSD must be ≤ 30 with 4 compounds allowed out to ≤ 40% RSD.	Correct problem then repeat ICAL.	Analyst / Supervisor	
	(ICV) – Second Source	After each ICAL, and daily, prior to sample analysis.	%Rs for 90% of "Standard" compounds must be within 70-130%; for 80% of "Non-standard" compounds, %Rs must be within 60-140%. No %R may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Recalibrate the instrument if the criteria cannot be met.	Analyst / Supervisor	
	CCV	At the start of each day and, if required by a specific project, every 12 hours.	%Rs must be within 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than four compounds (Continued on next page) from the standard list recover outside of 70-130%, then corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%R.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5-point ICAL.	Analyst / Supervisor	

Instrument	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference (1)
GC/MS – VOCs SIM (soil gas)	Tune Verification - BFB	Every 24 hours, or every 12 hours if project requires.	SW-846 tune criteria.	Correct problem then repeat tune.	Analyst / Supervisor	Air Toxics Ltd. SOP-38
	ICAL – Minimum of a 5-point calibration curve is prepared.	Prior to sample analysis.	%RSD must be ≤ 30% for standard compounds with 10% of the compound list allowed out to ≤ 40% %RSD.	Correct problem then repeat ICAL.	Analyst / Supervisor	
	ICV – Second Source	After each ICAL, and daily prior to sample analysis.	%Rs for 90% of "Standard" compounds must be within 70-130%; for 80% of "Non-standard" compounds, %Rs must be within 60-140%. No %R may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.	Analyst / Supervisor	
	CCV	At the start of each day and, if required by a specific project, every 12 hours.	%Rs must be within 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than 10% of compounds from the standard list recover outside of 70-130%, CA will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any Compound exceeds 60-140%R.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new calibration curve.	Analyst / Supervisor	

1. Laboratory SOPs are subject to revision and updates during duration of the project, the laboratory will use the most current revision of the SOP at the time of analysis.
SOP – standard operating procedure

WS #25 -- Analytical Instrument and Equipment Maintenance, Testing, and Inspection Table

(UFP-QAPP Manual Section 3.2.3)

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action (CA)	Responsible Person	SOP Reference
GC/MS	Check pressure and gas supply daily. Bake out trap and column, manual tune if BFB not in criteria, change septa as needed, cut column as needed, change trap as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	VOCs	Ion source, injector liner, column, column flow, purge lines, purge flow, trap.	Prior to ICAL and/or as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	**Empirical SOP-202/225**
HPLC	Check pressure and gas supply daily - change when <200 pounds per square inch (psi), change analytical column as needed, change mobile phase when insufficient for run or contamination, change inlet filters as needed for contamination.	Nitroglycerin	Check pump pressure, check for leaks, check for adequate mobile phase.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	**Empirical SOP-327**
HPLC/ESI/MS	Clean the source and capillary needle. Change analytical column as needed, change mobile phase when insufficient for run or contamination, change inlet filters as needed for contamination.	Perchlorate	Check pump pressure, check for leaks, check for adequate mobile phase.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	**Empirical SOP-239**
IC Dionex	Check and clean segments weekly, clean reagent tubes monthly. Change syringes , eluent, and dispensing needle, all as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Anions	Check tubing. Verify that Chromatography is acceptable.	Prior to initial calibration or as necessary.	Must meet ICAL and continuing calibration criteria.	Correct the problem and repeat ICAL or CCV.	Analyst/ Supervisor	**Empirical SOP-145**

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action (CA)	Responsible Person	SOP Reference
Lachat	Check and clean segments weekly, clean reagent tubes monthly. Change lamp, change diluent and wash tubes, change mixing paddles and syringes, and change dispensing needle, all as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Cyanide	Tubing and rollers.	Prior to ICAL or as necessary.	Acceptable ICAL and CCV.	Recalibrate and/or perform necessary equipment maintenance. Reanalyze samples not bracketed by passing CCV.	Analyst/ Supervisor	**Empirical SOP-164/175**
TOC Analyzer	Replace sample tubing, clean sample boat, replace syringe.	TOC	Tubing, sample boat, syringe	As needed.	Must meet ICAL and continuing calibration criteria.	Repeat maintenance activity of remove from service.	Analyst/ Laboratory Area Supervisor	**Empirical SOP-221**
pH Meter	Keep probe wet at all times and inspect prior to use. Rinse thoroughly between uses.	Alkalinity	Visual inspection of probe.	Each use.	NA.	Remove from service.	Analyst, Department Manager	**Empirical SOP-154**
GC/FID	Check pressure and gas supply daily. Change septa and/or liner as needed, replace or cut column as needed. Other maintenance specified in lab Equipment Maintenance SOP.	Dissolved Gases	Injector liner, septa, column, column flow.	Daily and prior to each use.	Must meet ICAL and continuing calibration criteria.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	**Empirical SOP-236**

Instrument / Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action (CA)	Responsible Person	SOP Reference
ICP-AES	Clean torch assembly and spray chamber when discolored or when degradation in data quality is observed. Clean nebulizer, check argon, and replace peristaltic pump tubing as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Metals	Torch, nebulizer chamber, pump, pump tubing.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	**Empirical SOP-100/105**
Mercury Analyzer	Replace peristaltic pump tubing, replace mercury lamp, replace drying tube, clean optical cell and/or clean liquid/gas separator as needed. Other maintenance specified in laboratory Equipment Maintenance SOP.	Mercury	Tubing, sample probe, optical cell.	Prior to ICAL and as necessary.	Acceptable ICAL and CCV.	Correct the problem and repeat ICAL or CCV.	Analyst, Department Manager	**Empirical SOP-103/104**
GC/MS, Low Level and SIM (soil gas)	Check the vacuum ion gauge, clean the ion source and replace the filaments, replace the injection port liner, replace pump oil (if necessary). Replace the seal and septum. Clip off 3 feet of the column. Perform a sensitivity check.	VOCs	Check the gas supply. Visually inspect the seal, liner, septum, and column.	Cleaning of ion source on quarterly basis or as needed. Replace internal components as needed. Perform a sensitivity check after an ICAL.	All analytes / surrogates recover within the specified parameters under the associated SOP.	Recalibrate and/or perform the necessary equipment maintenance.	Analyst / Supervisor	Air Toxics Ltd. SOPs-83 and 38
Buret	Check Buret for any cracks or chips. Rinse buret prior to each use and at the end of each day.	Sulfide	Visual inspection for cracks or chips.	Each use.	NA.	Remove from service.	Analyst, Department Manager	**Empirical SOP-153**

SOP – standard operating procedure

WS #26 -- Sample Handling System

(UFP-QAPP Manual Appendix A)

SAMPLE COLLECTION, PACKAGING, AND SHIPMENT
Sample Collection (Personnel/Organization): FOL or designee/TtNUS
Sample Packaging (Personnel/Organization): FOL or designee/TtNUS
Coordination of Shipment (Personnel/Organization): FOL or designee/TtNUS
Type of Shipment/Carrier: Federal Express
SAMPLE RECEIPT AND ANALYSIS
Sample Receipt (Personnel/Organization): Sample Custodians/Empirical and Air Toxics
Sample Custody and Storage (Personnel/Organization): Sample Custodians/Empirical and APPL
Sample Preparation (Personnel/Organization): Extraction Laboratory, Metals Preparation Laboratory, Dioxins Preparation Laboratory /Empirical and Air Toxics
Sample Determinative Analysis (Personnel/Organization): GC Laboratory, GC/MS Laboratory, Metals Laboratory, Dioxins Laboratory/Empirical and Air Toxics
SAMPLE ARCHIVING
Field Sample Storage (Number of days from sample collection): 60 days from receipt
Sample Extract/Digestate Storage (number of days from extraction/digestion): 3 months from sample digestion/extraction
Biological Sample Storage (Number of days from sample collection): NA
SAMPLE DISPOSAL
Personnel/Organization: Sample Custodians/Empirical and Air Toxics

WS #27 -- Sample Custody Requirements Table

(UFP-QAPP Manual Section 3.3.3)

27.1 SAMPLE NOMENCLATURE, SAMPLE COLLECTION DOCUMENTATION, HANDLING, AND TRACKING PROCEDURES

The following sections outline the procedures that will be used to document project activities and sample collection, handling, tracking, and custody procedures during the investigation.

27.1.1 Sample Nomenclature

Refer to **WS #18** for how the samples will be labeled. Also, refer to **WS #20** for how the field QA/QC samples will be generated.

Sample nomenclature will be conducted in general accordance with the procedures outlined in Facility SOP CT-04 (Sample Nomenclature). Sample nomenclature put forth for this field event has been selected based on historical usage. The sample nomenclature for each tracking number includes the Site being investigated, sample media identifier, and sample location number. The standard sample matrix and type codes used for this field event are as follows: Duplicate samples will be submitted to the laboratory as blind duplicates. The QA/QC type codes used for this field event are as follows: TB for Trip Blanks and RB for rinsate blanks. Field QC blanks will be labeled sequentially followed by the date (i.e., TB-20101213, FB-20101214, etc.). Samples to be used for matrix spikes and matrix spike duplicates will be labeled MS/MSD on the container label and noted on the chain-of-custody, as required in the laboratory QA Plan; however, “MS/MSD” will not be part of the unique sample identifier in order to maintain consistency with the project database. Additional information regarding protocol for sample labeling is contained in Facility SOP SA-6.3 in the NSF-IH Master UFP-SAP.

27.1.2 Sample Collection Documentation

Documentation of field observations will be recorded in a field logbook and/or field log sheets including sample collection logs, boring logs, VOC screening logs, and monitoring well construction logs. Field logbooks utilized on this project will consist of a bound, water-resistant logbook. All pages of the logbook will be numbered sequentially and observations will be recorded with indelible ink.

Field sample log sheets will be used to document sample collection details, and other observations and activities will be recorded in the field logbook. Instrument calibration logs will be used to record the daily instrument calibration. Example field forms are included in Appendix C of the Master UFP-SAP.

For sampling and field activities, the following types of information will be recorded in the field log as appropriate:

- Site name and location
- Date and time of logbook entries
- Personnel and their affiliations
- Weather conditions
- Activities involved with the sampling
- Subcontractor activity summary
- Site observations including Site entry and exit times

- Site sketches made onSite
- Visitor names, affiliations, arrival and departure times
- Health and safety issues including PPE

27.1.3 Sample Handling and Tracking System

Following sample collection into the appropriate bottle ware, all samples will be immediately placed on ice in a cooler. The glass sample containers will be enclosed in bubble-wrap in order to protect the bottle ware during shipment. The cooler will be secured using strapping tape along with a signed custody seal. Sample coolers will be delivered to a local courier location for priority overnight delivery to the selected laboratory for analysis. Samples will be preserved as appropriate based on the analytical method. The laboratories will provide pre-preserved sample containers for sample collection. Samples will be maintained at 4°C (±2°C) until delivery to the laboratory. Proper custody procedures will be followed throughout all phases of sample collection and handling.

After collection, each sample will be maintained in the sampler's custody until formally transferred to another party (e.g., Federal Express). For all samples collected, chain-of-custody forms will document the date and time of sample collection, the sampler's name, and the names of all others who subsequently held custody of the sample. Specifications for chemical analyses will also be documented on the chain-of-custody form. Facility SOP SA-6.3 (Field Documentation) provides further details on the chain-of-custody procedure, which is provided in Appendix D of the Master UFP-SAP.

These subsections outline the procedures that will be used by field and laboratory personnel to document project activities and sample collection procedures during this SI. All forms must be filled in as completely as possible.

Sample handling requirements are described in **WS #26**. Tetra Tech personnel will collect the samples. The samplers will take care not to contaminate samples through improper handling. Samples will be sealed in appropriate containers, packaged by Tetra Tech personnel and placed into sealed coolers under chain-of-custody in accordance with the applicable SOP (see **WS #21**). Samples to be analyzed for VOCs will be accompanied by a VOC trip blank. All coolers will contain a temperature blank. Samples will be transferred under chain-of-custody to a courier as described below. Once received by the laboratory, receipt will be documented on the chain-of-custody form and the samples will be checked in. The samples will remain under chain-of-custody throughout the analysis period to ensure their integrity is preserved. Details are provided below.

Samples to be delivered to the laboratory(s) will be made by a public courier (i.e., Federal Express). After samples have been collected, they will be sent to the laboratory(s) within 24 hours. Under no circumstances will sample holding times be exceeded.

27.2 FIELD SAMPLE CUSTODY PROCEDURES

Chain-of-custody protocols will be used throughout sample handling to establish the evidentiary integrity of sample containers. These protocols will be used to demonstrate that the samples were handled and transferred in a manner that would eliminate possible tampering. Samples for the laboratory will be packaged and shipped in accordance with Facility SOP SA-6.1.

A sample is under custody if:

- The sample is in the physical possession of an authorized person.
- The sample is in view of an authorized person after being in his/her possession.

- The sample is placed in a secure area by an authorized person after being in his/her possession.
- The sample is in a secure area, restricted to authorized personnel only.

Custody documentation is designed to provide documentation of preparation, handling, storage, and shipping of all samples collected. A multi-part form is used with each page of the form signed and dated by the recipient of a sample or portion of sample. The person releasing the sample and the person receiving the sample each will retain a copy of the form each time a sample transfer occurs.

Integrity of the samples collected during the Site investigation will be the responsibility of identified persons from the time the samples are collected until the samples, or their derived data, are incorporated into the final report.

The Tetra Tech FOL is responsible for the care and custody of the samples collected until they are delivered to the laboratory or are entrusted to a carrier. When transferring samples, the individuals relinquishing and receiving them will sign, date, and note the time on the chain-of-custody form. This record documents the sample custody transfer from the sampler to the laboratory, often through another person or agency (common carrier). Upon arrival at the laboratory, internal sample custody procedures will be followed as defined in the Laboratory SOPs included in **Appendix B**.

27.3 LABORATORY SAMPLE CUSTODY PROCEDURES

Each shipment of samples arriving by commercial carrier must be accompanied by an airbill, airbill sticker, or manifest. The sample receiving coordinator shall sign the airbill/manifest and record the date and time the shipment was received on the document. Samples are received and checked for breakage, preservation, and agreement between bottles and chain of custody.

All samples are kept for at least 90 days from the time the samples are received. After 90 days, the samples are disposed of unless otherwise specified by the client. Disposal of all samples must be recorded in the sample disposal logbook.

27.3.1 Laboratory Sample Identification Procedures

The LIMS program for sample log-in procedure automatically assigns unique lab identifiers to the samples and produce forms used by managers to track every sample from log-in to invoicing. Once the samples have been logged-in, the sample labels will be printed which contain the laboratory sample IDs for each bottle, as well as the tests to be performed on the samples.

27.3.2 Laboratory Chain-of-Custody Procedures

An internal chain of custody is created and maintained from cooler receipt, as samples are taken from the controlled storage area to the preparation laboratories, and when the extracts/digestates are transferred to the instrumentation labs. The internal chain of custody is continued through sample disposal.

When it becomes necessary to subcontract sample analyses to another laboratory, custody of the samples must be transferred to that laboratory in the form of a chain of custody for subcontracted analysis. The sample coordinator will complete the information on the chain of custody regarding the name and address of the lab and the sample information. The sample coordinator must sign and date the form to release custody of the samples and retain a copy of the form in the chain of custody records. The chain of custody will accompany the samples in the sealed container being shipped to the subcontracted lab. The returned chain-of-custody shall be placed in the data package.

Air Toxics Ltd. The laboratory sample custody procedures (receipt of samples, archiving, and disposal) documented in Air Toxics Ltd. SOPs will be followed. It is important to include the serial number of the canister on the chain-of-custody. A sample receipt form will be completed to note conditions and any

discrepancies. The chain-of-custody form will be compared against the sample containers received for correctness. Samples will be logged into the LIMS and assigned a unique log number that can be tracked through processing. Tetra Tech will be notified of any problems.

WS #28.1 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: VOCs
Analytical Method/SOP Reference: SW-846 8260B / Empirical SOP-202

Quality Control (QC) Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
Method Blank	One per preparatory batch of 20 or fewer samples	All target analytes must be ≤ ½ LOQ, except common lab contaminants, which must be < LOQ.	Investigate source of contamination and rerun method blank prior to analysis of samples, if possible. Evaluate the samples and associated QC, if blank results are above LOQ, then report sample results that are <LOQ or >10X the blank concentration. Re-prepare and reanalyze blank and those samples that were >LOQ and <10X the blank.	Analyst, Supervisor, Data Validator	Bias/Contamination	Same as QC Acceptance Limits
Laboratory Control Sample (LCS)	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12-hour clock and is acceptable, then narrate. If the LCS recoveries are high, but the sample results are <LOQ, then narrate. Otherwise, reprepare and reanalyze the LCS and associated samples.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits

Quality Control (QC) Sample	Frequency/Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Data Quality Indicator (DQI)	Measurement Performance Criteria (MPC)
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD Quality Systems Manual (QSM) Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	CA will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met unless RPDs indicate obvious extraction/ analysis difficulties, then re-prepare and reanalyze MS/MSD.	Analyst, Supervisor, Data Validator	Accuracy/Bias/ Precision	Same as QC Acceptance Limit
Internal Standards (ISs)	Every field sample, standard, and QC sample - three per sample- Fluorobenzene Chlorobenzene-d5 1,4-dichlorobezene-d4	RTs must be within ± 30 seconds and the response areas must be within -50% to +100% of the ICAL midpoint standard for each IS.	Inspect mass spectrometer and gas chromatograph for malfunctions; mandatory reanalysis of samples analyzed while system was malfunctioning.	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits
Surrogates	All field and QC samples - four per sample- Dibromofluoromethane 1,2-dichloroethane-d4 Toluene-d8 BFB	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If sample volume is available, then reprepare and reanalyze sample for confirmation of matrix interference when appropriate.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Results between DL and LOQ	NA	Apply “J” qualifier to results detected between DL and LOQ.	None	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

SOP – standard operating procedure

WS #28.2 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: Explosives including Nitroglycerin
Analytical Method/SOP Reference: SW-846 8330B / Empirical SOP-327

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	All target analytes must be ≤ ½ LOQ.	(1) Investigate source of contamination (2) Re-prepare and analyze method blank and all samples processed with the contaminated blank. (3) Qualify results if re-extraction/re-analysis not feasible.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch of 20 or fewer samples of similar matrix.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and is acceptable, then narrate. If the LCS recoveries are high, but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze the LCS and associated samples.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix.	%Rs must meet the DoD Quality Systems Manual (QSM) Version 4.1 limits as per Appendix G of the DoD QSM. The RPD between MS and MSD should be ≤ 30%.	Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met, unless RPDs indicate obvious extraction/ analysis difficulties, then re-prepare and reanalyze MS/MSD.	Analyst, Supervisor, Data Validator	Accuracy / Precision	Same as Method/SOP QC Acceptance Limits.
Surrogate Spikes	All field and QC samples - one per sample. One surrogate added: 1-chloro-3-nitrobenzene.	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	If surrogate recovery falls outside acceptance criteria, the sample should be re-extracted and re-analyzed.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Second Column Confirmation	All positive results must be confirmed.	RPD must be <40% from primary concentration.	None. Apply flag if RPD >40% and discuss in the case narrative.	Analyst, Supervisor	Presence/ precision	Same as Method/SOP QC Acceptance Limits.
Results between DL and LOQ	NA.	Apply "J" qualifier to results detected between DL and LOQ.	NA	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits.

WS #28.3 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: Nitroguanidne
Analytical Method/SOP Reference: SW-846 8330 modified / Empirical SOP-233

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparation batch of 20 or fewer samples of similar matrix.	All target analytes must be ≤ ½ LOQ.	(1) Investigate source of contamination (2) Re-prepare and analyze method blank and all samples processed with the contaminated blank. (3) Qualify results if re-extraction/re-analysis not feasible.	Analyst, Supervisor, Data Validator	Contamination / Bias	Same as Method/SOP QC Acceptance Limits.
LCS	One per preparatory batch of 20 or fewer samples of similar matrix.	%Rs must be between 70-130%.	Evaluate and reanalyze if possible. If an MS/MSD was performed in the same 12 hour clock and is acceptable, then narrate. If the LCS recoveries are high, but the sample results are <LOQ, then narrate. Otherwise, re-prepare and reanalyze the LCS and associated samples.	Analyst, Supervisor, Data Validator	Accuracy / Bias	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch of 20 or fewer samples of similar matrix.	%Rs must be between 70-130%. The RPD between MS and MSD should be ≤ 30%.	Corrective action will not be taken for samples when recoveries are outside limits and surrogate and LCS criteria are met, unless RPDs indicate obvious extraction/ analysis difficulties, then re-prepare and reanalyze MS/MSD.	Analyst, Supervisor, Data Validator	Accuracy / Precision	Same as Method/SOP QC Acceptance Limits.

WS #28.4 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: Perchlorate
Analytical Method/SOP Reference: SW846 6850 / **Empirical SOP-239**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Isotope Ratio $^{35}Cl/^{37}Cl$	Every sample, batch QC sample, and standard.	Monitor for the daughter ion at masses 83/85. Theoretical ratio ~ 3.06. Ratio must be within 2.3 to 3.8.	If criteria are not met, the sample must be rerun. If the sample was not pretreated, the sample should be re-extracted using cleanup procedures. If, after cleanup, the ratio still fails, use alternative techniques to confirm presence of perchlorate.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
IS	Every sample, batch QC sample, standard, instrument blank, and method blank.	Measured ^{18}O IS area must be within 50-150% of the average IS area counts of the ICAL. RRT of the perchlorate ion must be within 0.98-1.02.	Rerun the sample at increasing dilutions until the 50-150% acceptance criteria is met. If criteria cannot be met with dilution, interference is suspected - use additional pretreatment steps.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Interference Check Sample (ICS)	One per batch to verify method performance at the matrix conductivity threshold (MCT). At least one ICS must be analyzed daily.	The %R must be within 70-130% of true value.	Correct problem and re-analyze all samples in that batch. Replace cleanup filters or column if necessary. No samples may be reported that are associated with a failing ICS.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.
Reagent Blank	Prior to calibration, after over-range samples, and at the end of the analytical sequence.	No perchlorate detected > ½ LOQ.	Re-analyze reagent blank (until no carryover is observed) and all samples processed since the contaminated blank. Results may not be reported without a valid reagent blank.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.

Method Blank	One per preparatory batch of 20 or fewer samples.	Perchlorate must be ≤ ½ LOQ.	Investigate source of contamination and evaluate the samples and associated QC: i.e. If the blank results are above ½ LOQ, then report sample results which are non-detect. Otherwise, re-prepare blank and associated samples.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
LCS (LCSD not required)	One per preparatory batch of 20 or fewer samples. LCS must be spiked at the LOQ.	%Rs must be between 80-120. RPD should be ≤ 15%, if LCSD is analyzed.	If the %R of a target analyte in the LCS is greater than the upper control limit, and there are no positive findings for that compound, no further action is taken. Otherwise, re-analyze the LCS and affected samples or flag the results. If the %R of any target analyte is below the lower control limit, re-analyze the LCS and affected samples.	Analyst, Supervisor, Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.
MS/MSD	One per preparatory batch of 20 or fewer samples per matrix. The MS and MSD must be spiked at the LOQ.	%Rs should be between 80-120. RPD should be ≤ 15%	Flag the parent sample for failed analytes, which exceed the acceptance criteria.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as Method/SOP QC Acceptance Limits.

WS #28.5 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater, and Aqueous QC Blanks
Analytical Group: Anions
Analytical Method/SOP Reference: EPA 300.0 / Empirical SOP-145

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per batch of up to 20 samples	Analyte concentration must be <½ LOQ.	Correct problem, reprepare and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
LCS	One per batch of up to 20 samples	%R must be within 90-110%.	Correct problem, reprepare, and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One set is performed for each batch of up to 10 samples of the same matrix.	%R must be between 80-120%, MS/MSD %RPD must be <20%.	Failure to meet the control limits shall be discussed in the case narrative. If both the LCS and MS are unacceptable, all associated samples must be re-analyzed.	Analyst, Supervisor, Data Validator	Precision / Accuracy	Same as Method/SOP QC Acceptance Limits.

WS #28.6 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: Metals (Including Mercury)
Analytical Method/SOP Reference: SW-846 6010C, 7470A, & 7471A/ Empirical SOP103/105

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	All target analytes must be ≤ ½ LOQ.	Re-analyze to confirm the positive value. Notify the PM for further action. Re-prepare the samples associated with the Blank. Noncompliance report will be required for data reported	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%R must be within 80-120%.	Evaluate and reanalyze, if possible. If the LCS recoveries are high, but the sample results are < LOQ, then narrate. Otherwise, redigest and reanalyze all associated samples for failed target analyte(s).	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
MS	One per preparatory batch of 20 or fewer samples of similar matrix	%R should be within 80-120% (if sample is < 4x spike added).	Flag results for affected analytes for all associated samples with "N".	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Sample Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix	The RPD should be ≤ 20% for duplicate samples for both water and soils.	Narrate any results that are outside control limits.	Analyst, Supervisor, and Data Validator	Precision	Same as QC Acceptance Limits
Serial Dilution	One per preparatory batch with sample concentration(s) >50x LOD	The 5-fold dilution result must agree within ±10%D of the original sample result if result is >50x LOD.	Perform post-spike addition.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Post Digestion Spike (does not apply to mercury)	One is performed when serial dilution fails or target analyte concentration(s) in all samples are < 50x LOD	The %R must be within 75-125% of expected value to verify the absence of an interference. Spike addition should produce a concentration of 10-100x LOQ.	Flag results for affected analytes for all associated samples with "J".	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Results between DL and LOQ	Not known at this time	Apply "J" qualifier to results between DL and LOQ.	None	Analyst, Supervisor, Data Validator	Accuracy	Same as QC Acceptance Limits

WS #28.7 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: Total Cyanide
Analytical Method/SOP Reference: **SW-946 9012A/ Empirical SOP-164, 175**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparatory batch of 20 or fewer samples of similar matrix	Cyanide must be ≤ ½ LOQ.	Stop analysis, correct problem, and recalibrate.	Analyst, Supervisor, Data Validator	Bias/Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Re-prepare and reanalyze the LCS and all associated samples.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
MS	One per preparatory batch of 20 or fewer samples of similar matrix	%Rs must meet the DoD QSM Version 4.1 limits as per Appendix G of the DoD QSM.	Flag data with an "N", unless recovery is > 4x the spike added; If the sample results exceed 4x the spike added, then spike the un-spiked aliquot of the sample at 2x the indigenous level or 2x the LOQ.	Analyst, Supervisor, Data Validator	Accuracy/Bias	Same as QC Acceptance Limits
Sample Duplicate	One per preparatory batch of 20 or fewer samples of similar matrix	RPD should be ≤ 20%, if concentration is > 5x LOQ; or within ± the LOQ, if the concentration is < 5x LOQ.	Flag data for associated samples with an "*."	Analyst, Supervisor, and Data Validator	Precision	Same as QC Acceptance Limits

WS #28.8 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater, and Aqueous QC Blanks
Analytical Group: Sulfide
Analytical Method/SOP Reference: Standard Methods 4500 S2- F / Empirical SOP-153

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparation batch	No target analytes ≥ LOD.	Correct problem. If required, re-prepare and reanalyze method blank and all samples processed with the contaminated blank.	Analyst, Laboratory Department Manager, and Data Validator	Bias/Contamination	Same as QC Acceptance Limits.
LCS	One per sample preparation batch	%R must be between 80-120%.	Correct problem, then reprepare and reanalyze the LCS and all samples in the associated preparatory batch for failed analytes, if sufficient sample material is available.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One MS/MSD per analytical/preparation batch	%R must be between 75-125%. RPD ≤ 20%	Identify problem; if not related to matrix interference, re-extract and reanalyze MS/MSD and all associated batch samples in accordance with DoD QSM requirements.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias Precision	Same as QC Acceptance Limits.

WS #28.9 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil, Groundwater, and Aqueous QC Blanks
Analytical Group: TOC
Analytical Method/SOP Reference: Lloyd Kahn and SW846 Method 9060 / Empirical **SOP-221**

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per preparatory batch of 20 or fewer samples	The target analyte must be ≤ ½ LOQ.	Correct problem, re-prepare and reanalyze along with associated samples.	Analyst, Supervisor, Data Validator	Bias/ Contamination	Same as QC Acceptance Limits
LCS	One per preparatory batch of 20 or fewer samples	%R must be within 80-120% of true value.	Correct problem, re-prepare, and reanalyze along with associated samples.	Analyst, Supervisor, Data Validator	Accuracy/ Bias	Same as QC Acceptance Limits
MS/MSD	One per preparatory batch of 20 or fewer samples per matrix	%R should be within 80-120% of true value. RPD should be ≤ 20%.	Contact client for guidance.	Analyst, Supervisor, Data Validator	Accuracy/ Bias/ Precision	Same as QC Acceptance Limits

WS #28.10 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater, and Aqueous QC Blanks
Analytical Group: Dissolved Gases
Analytical Method/SOP Reference: RSK SOP 175 / Empirical SOP-236

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per batch of up to 20 samples	Analyte concentration must be <½ LOQ.	Correct problem, re-prepare and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
LCS	One per batch of up to 20 samples	%R must be within 80-120% of the expected value.	Correct problem, re-prepare, and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples	%R should be within 75-125% of the expected value. RPD ≤ 20%	Contact client for guidance.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias/ Precision	Same as QC Acceptance Limits.

WS #28.11 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Groundwater, and Aqueous QC Blanks
Analytical Group: Wet Chemistry
Analytical Method/SOP Reference: Alkalinity / SM2320B Empirical SOP-154

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One per batch of up to 20 samples	Analyte concentration must be <½ LOQ.	Correct problem, reprepare and reanalyze along will associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Contamination /Bias	Same as QC Acceptance Limits.
LCS	One per batch of up to 20 samples	%R must be within 90-110% of the expected value.	Correct problem, reprepare, and reanalyze along with associated samples.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy/Bias	Same as QC Acceptance Limits.
MS/MSD	One per batch of up to 20 samples	%R should be within 75-125% of the expected value. RPD ≤ 20%	Contact client for guidance.	Analyst, Laboratory Department Manager, and Data Validator	Accuracy.Bias/Precision	Same as QC Acceptance Limits.

WS #28.12 -- Laboratory QC Samples Table

(UFP-QAPP Manual Section 3.4)

Matrix: Soil Gas
Analytical Group: VOCs
Analytical Method/SOP Reference: TO-15/Air Toxics Ltd. SOP-83 and 38

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
Method Blank	One after each CCV and Laboratory Control Sample (LCS).	Results must be less than the laboratory LOQ.	Inspect the system and re-analyze the blank.	Analyst or Laboratory Department Manager, Data Validator	Bias/ Contamination	Same as Method/SOP QC Acceptance Limits.
Surrogate	As each standard, blank, and sample is being loaded. Three per sample: 1,2-DCE-d4 toluene-d8 4-BFB	%Rs must be 70-130%.	For blanks: Inspect the system and reanalyze the blank. For samples: Re-analyze the sample unless obvious matrix interference is documented. If the %R is within limits in the reanalysis, report the second analysis. If %R is out-of-limits a second time, then narrate results.	Analyst or Laboratory Department Manager, Data Validator	Accuracy/Bias	Same as Method/SOP QC Acceptance Limits.
LCS	After each ICAL, and daily, prior to sample analysis.	%Rs for 90% of the "Standard" compounds must have %Rs between 70-130%; for 80% of "Non-standard" compounds, %Rs must be between 60-140%. No %R may be <50%.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Recalibrate the instrument if the criteria cannot be met.	Analyst or Laboratory Department Manager, Data Validator	Accuracy/ Bias Precision also, if LCSD is analyzed	Same as Method/SOP QC Acceptance Limits.

QC Sample	Frequency / Number	Method / SOP QC Acceptance Limits	CA	Person(s) Responsible for CA	DQI	MPC
IS	As each standard, blank, and sample is being loaded. Three per sample: bromochloromethane 1,4-difluorobenzene chlorobenzene-d5	RTs for blanks and samples must be within ±0.33 minutes of the RT in the CCV and within ±40% of the area counts of the daily CCV ISs.	**For blanks:** Inspect the system and reanalyze the blank. **For samples:** Re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.	Analyst or Laboratory Department Manager, Data Validator	Accuracy/ Bias	Same as Method/SOP QC Acceptance Limits.

WS #29 -- Project Documents and Records Table

(UFP-QAPP Manual Section 3.5.1)

Document	Where Maintained
Field Documents Field Logbook Field Sample Forms Chain of Custody Records Air Bills Sampling Instrument Calibration Logs Sampling Notes Photographs FTMR Forms This SAP HASP	Field documents will be maintained in the project file located in the Tetra Tech Pittsburgh office.
Laboratory Documents Sample receipt, custody, and tracking record Equipment calibration logs Sample preparation logs Analysis Run logs CA forms Reported field sample results Reported results for standards, QC checks, and QC samples Extraction/clean-up records Raw data	Laboratory documents will be included in the hardcopy and portable documents format deliverables from the laboratory. Laboratory data deliverables will be maintained in the Tetra Tech Pittsburgh project file and in long-term data package storage at a third-party professional document storage firm. Electronic data results will be maintained in a database on a password protected SQL server.
Assessment Findings Field Sampling Audit Checklist (if conducted) Analytical Audit Checklist (if conducted) Data Validation Memoranda (includes tabulated data summary forms)	All assessment documents will be maintained in the Tetra Tech Pittsburgh office.
Reports RI/FS Report	All reports will be stored in hardcopy in the Tetra Tech Pittsburgh project file and electronically in the server library.

WS #30 -- Analytical Services Table

(UFP-QAPP Manual Section 3.5.2.3)

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization	Backup Laboratory/ Organization
Soil, Groundwater, and Aqueous QC Blanks	VOC (including low level for aqueous samples)	See **WS #18**	SW-846 8260B	21 calendar days	Kim Kostzer kkostzer@empirlabs.com Empirical Laboratories, LLC 621 Mainstream Drive, Suite 270 Nashville, TN 37228 (615) 345-1115	N/A
	Explosives including nitroglycerin		SW-846 8330B			
	Metals (total and dissolved for groundwater) including mercury		SW-846 6010C, 7470A, and 7471A			
	Cyanide		SW-846 9012B			
	Nitroguanidine		SW-846 8330 modified			
	Perchlorate		SW-846 6850			
	TOC		SW-846 9060 and (Lloyd Kahn for soil)			
Groundwater	Anions		EPA 300			
	Sulfide		SM 4500S-2CF			
	Dissolved Gases		RSK 175			
	Alkalinity		SM 2320			
Soil gas	VOCs		Modified TO-15 Hi/Lo		Ausha Scott Air Toxics Ltd. 180B Blue Ravine Road Folsom CA 95630 916-985-1000 ext 1027 ascott@airtoxics.com	
Soil	Permeability		ASTM D5084		Mark Dobday GeoTesting Express, Inc. 1145 Massachusetts Ave.	
	Effective Porosity		ASTM D854, D2216			

Matrix	Analytical Group	Sample Locations/ID Numbers	Analytical Method	Data Package Turnaround Time	Laboratory / Organization	Backup Laboratory/ Organization
	Bulk Density		ATI RP40		Boxborough, MA 01719 Toll Free 800-434-1062 Fax 978 635 0266	
	Grain size		ASTM D422			

Empirical will also analyze aqueous and solid IDW samples but details of these analyses are not provided as the data are used for disposal purposed only.

WS #31 -- Planned Project Assessments Table

(UFP-QAPP Manual Section 4.1.1)

Assessment Type	**Frequency**	**Internal or External**	**Organization Performing Assessment**	**Person(s) Responsible for Performing Assessment** (title and organizational affiliation)	**Person(s) Responsible for Responding to Assessment Findings** (title and organizational affiliation)	**Person(s) Responsible for Identifying and Implementing Corrective Action CA)** (title and organizational affiliation)	**Person(s) Responsible for Monitoring Effectiveness of CA** (title and organizational affiliation)
Laboratory System Audit (1)	Every 2 years	External	DoD ELAP Accrediting Body	DoD ELAP Accrediting Body Auditor	Laboratory QAM or Laboratory Manager, Centauri, Microbac and Air Toxics, and Accutest	Laboratory QAM or Laboratory Manager, Centauri, Microbac and Air Toxics, and Accutest	Laboratory QAM or Laboratory Manager, Centauri, Microbac and Air Toxics, and Accutest

1. Empirical and Air Toxics have successfully completed the laboratory assessment process required as part of the DoD QSM Version 4.1 under the DoD ELAP by a recognized Accrediting Body. The DoD ELAP accreditation letters are included in **Appendix B**. Geotesting express is not DoD ELAP-accredited, but due to the nature of these analyses accreditation is not required.

WS #32 -- Assessment Findings and Corrective Action Responses

(UFP-QAPP Manual Section 4.1.2)

Assessment Type	Nature of Deficiencies Documentation	Individual(s) Notified of Findings (name, title, organization)	Timeframe of Notification	Nature of Corrective Action Response Documentation	Individual(s) Receiving Corrective Action Response (name, title, organization)	Timeframe for Response
Laboratory System Audit	Written audit report	Marcia McGinnity, Empirical, Melanie Levesque, Air Toxics	Specified by DoD ELAP Accrediting Body	Letter	DoD ELAP Accrediting Body	Specified by DoD ELAP Accrediting Body

WS #33 -- QA Management Reports Table

(UFP QAPP Manual Section 4.2)

Type of Report	Frequency	Projected Delivery Date(s)	Person(s) Responsible for Report Preparation	Report Recipient(s)
DV Report	Per Sample Delivery Group (SDG)	Within 3 weeks of receipt of laboratory data package	Tetra Tech DVM or designee	Tetra Tech PM and project file
Major Analysis Problem Identification (Internal Tetra Tech Memorandum)	When persistent analysis problems are detected by Tetra Tech that may impact data usability	Immediately upon detection of problem (on the same day)	Tetra Tech QAM	Tetra Tech PM, QAM, Program Manager, and project file
Project Monthly Progress Report	Monthly for duration of project	Monthly	Tetra Tech PM	Navy RPM; Tetra Tech QAM, Program Manager, and project file
Laboratory QA Report	When significant plan deviations result from unanticipated circumstances	Immediately upon detection of problem (on the same day)	Laboratory PM, Centauri, Microbac and Air Toxics, and Accutest	Tetra Tech PM and project file

WS #34 -- Verification (Step I) Process Table

(UFP-QAPP Manual Section 5.2.1)

Verification Input	Description	Internal / External	Responsible for Verification (name, organization)
Chain-of-custody forms	The Tetra Tech FOL or designee will review and sign the chain-of-custody form to verify that all samples listed are included in the shipment to the laboratory and the sample information is accurate. The forms will be signed by the sampler and a copy will be retained for the project file, the Tetra Tech PM, and the Tetra Tech Data Validators. See Facility SOP SA-6.3 in the Master UFP-SAP.	Internal	Tetra Tech Sampler and FOL
	The Laboratory Sample Custodian will review the sample shipment for completeness, integrity, and sign accepting the shipment. The Tetra Tech Data Validators will check that the chain-of-custody form was signed and dated by the Tetra Tech FOL or designee relinquishing the samples and also by the Laboratory Sample Custodian receiving the samples for analyses.	Internal and External	1 - Laboratory Sample Custodian, Empirical and Air Toxics 2 – Tetra Tech Data Validators
SAP Sample Tables/ Chain-of-Custody Forms	Verify that all proposed samples listed in the SAP tables have been collected.	Internal	Tetra Tech FOL or designee
Sample Log Sheets	Verify that information recorded in the log sheets is accurate and complete.	Internal	Tetra Tech FOL or designee
Sample Coordinates	Verify that actual sample locations are correct and in accordance with the SAP proposed locations. Document any discrepancies in the final report.	Internal	Tetra Tech PM, FOL, or designee
SAP/ Field Logs/ Analytical Data Packages	Ensure that all sampling SOPs were followed. Verify that deviations have been documented and MPCs have been achieved. Particular attention should be given to verify that samples were correctly identified, that sampling location coordinates are accurate, and that documentation establishes an unbroken trail of documented chain-of-custody from sample collection to report generation. Verify that the correct sampling and analytical methods/SOPs were applied. Verify that the sampling plan was implemented and carried out as written and that any deviations are documented.	Internal	Tetra Tech PM or designee
SAP/ Laboratory SOPs/ Raw Data/ Applicable Control Limits Tables	Ensure that all laboratory SOPs were followed. Verify that the correct analytical methods/SOPs were applied. Establish that all method QC samples were analyzed and in control as listed in the analytical SOPs. If method QA is not in control, the Laboratory QAM will contact the Tetra Tech PM via telephone or email for guidance prior to report preparation.	Internal	Laboratory QAM, Centaur Empirical and Air Toxics
SAP/ Chain-of-Custody Forms	Check that field QC samples listed in **WS #20** were collected as required.	Internal	Tetra Tech FOL or designee

Verification Input	**Description**	**Internal / External**	**Responsible for Verification** (name, organization)
Analytical Data Packages	All analytical data packages will be verified internally for completeness by the laboratory performing the work. The Laboratory QAM will sign the case narrative for each data package.	Internal	Laboratory QAM, Empirical and Air Toxics
Audit reports	Audit checklists and narratives noting any deficiencies (if an audit is performed).	Internal	Tetra Tech PM, QAM, and FOL

WS #35 -- Validation (Steps IIa and IIb) Process Table

(UFP-QAPP Manual Section 5.2.2) (Figure 37 UFP-QAPP Manual) (Table 9 UFP-QAPP Manual)

Step IIa / IIb	Validation Input	Description	Responsible for Validation
IIa	Chain-of-Custody Forms	Custody - Ensure that the custody and integrity of the samples was maintained from collection to analysis and the custody records are complete and any deviations are recorded. Review that the samples were shipped and stored at the required temperature and sample pH for chemically-preserved samples meet the requirements listed in **WS #19**. Ensure that the analyses were performed within the holding times listed in **WS #19**.	Tetra Tech Project Chemist or Data Validators
IIa/IIb	SAP/ Laboratory Data Packages/ EDDs	Accuracy - Ensure that the laboratory QC samples listed in **WS #28** were analyzed and that the MPCs listed in **WS #12** were met for all field samples and QC analyses. Check that specified field QC samples were collected and analyzed and that the analytical QC criteria set up for this project were met.	Tetra Tech Project Chemist or Data Validators
		Precision - Check the field sampling precision by calculating the RPD for field duplicate samples. Check the laboratory precision by reviewing the RPD or percent difference values from laboratory duplicate analyses; MS/MSDs; and laboratory control sample (LCS) / LCS duplicate (LCSD), if available. Ensure compliance with the methods and project MPCs accuracy goals listed in **WS #12**.	
		Representativeness - Check that the laboratory recorded the temperature at sample receipt and the pH of the chemically preserved samples to ensure sample integrity from sample collection to analysis.	
		Completeness - Review the chain-of-custody forms generated in the field to ensure that the required analytical samples have been collected, appropriate sample identifications have been used, and correct analytical methods have been applied. The Tetra Tech Data Validator will verify that elements of the data package required for validation are present, and if not, the laboratory will be contacted and the missing information will be requested. Validation will be performed as per **WS #36**. Check that all data have been transferred correctly and completely to the final Structured Query Language (SQL) database.	
IIb	SAP/ Laboratory Data Packages/ EDDs	Sensitivity - Ensure that the project LOQs listed in **WS #15** were achieved.	Tetra Tech Project Chemist or Data Validators
		PSLs - Discuss the impact on reported DLs due to matrix interferences or sample dilutions performed because of the high concentration of one or more other contaminants, on the other target compounds reported as non-detected. Document this usability issue and inform the Tetra Tech PM. Review and add PSLs to the laboratory EDDs. Flag samples and notify the Tetra Tech PM of samples that exceed PSLs listed in **WS #15**.	
		QA/QC - Ensure that all QC samples specified in the SAP were collected and analyzed and that the associated results were within prescribed SAP acceptance limits. Ensure that QC samples and standards prescribed in analytical SOPs were analyzed and within the prescribed control limits. If any significant QC deviations occur, the Laboratory QAM shall have contacted the Tetra Tech PM.	

Step IIa / IIb	Validation Input	Description	Responsible for Validation
		Deviations - Summarize deviations from methods, procedures, or contracts in the Data Validation Report. Determine the impact of any deviation from sampling or analytical methods and SOPs requirements and matrix interferences effect on the analytical results. Qualify data results based on method or QC deviation and explain all the data qualifications. Print a copy of the project database qualified data depicting data qualifiers and data qualifiers codes that summarize the reason for data qualifications. Determine if the data met the MPCs and determine the impact of any deviations on the technical usability of the data.	

WS #36 -- Analytical Data Validation (Steps IIa and IIb) Summary Table

(UFP-QAPP Manual Section 5.2.2.1)

Step IIa / IIb	Matrix	Analytical Group	Validation Criteria	Data Validator
IIa and IIb	Soil, Groundwater, and Aqueous QC Blanks	VOCs, Dissolved Gases, Explosives, Nitroguanidine and Perchlorate	Validation will be performed using SW-846 8260B, RSK SOP 175, SW-846 8330B, and SW-846 6850 method specific criteria, and those criteria listed in **WS #12, #15, #24**, and **#28**. If not included in the aforementioned the logic outlined in U.S. EPA Region 3 Modifications to the Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (2004) should be used to apply qualifiers to data to the extent possible.	Tetra Tech Data Validation Specialist
IIa and IIb	Soil gas	VOCs	Validation will be performed using TO-15 method specific criteria, and those criteria listed in **WS #12, #15, #24**, and **#28**. If not included in the aforementioned the logic outlined in U.S. EPA Region 3 Modifications to the Laboratory Data Validation Functional Guidelines for Evaluating Organic Analyses (2004) should be used to apply qualifiers to data to the extent possible.	Tetra Tech Data Validation Specialist
IIa and IIb	Soil, Groundwater, and Aqueous QC Blanks	Metals (including mercury and cyanide)	Validation will be performed using SW-846 6010C, 9012B, 7470A, and 7471A method specific criteria, and those criteria listed in **WS #12, #15, #24**, and **#28**. If not included in the aforementioned the logic outlined in U.S. EPA Region 3 Modifications to the Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses (1993) should be used to apply qualifiers to data to the extent possible.	Tetra Tech Data Validation Specialist
IIa and IIb	Groundwater and (Soil TOC)	Anions, sulfide, alkalinity, and TOC	Validation will be performed using EPA 300, EPA 376.1, EPA 310.1, and SW-846 9060 method specific criteria, and those criteria listed in **WS #12, #15, #24**, and **#28**.	Tetra Tech Data Validation Specialist

IDW and geotechnical analyses are not validated.

WS #37 -- Usability Assessment

(UFP-QAPP Manual Section 5.2.3)

Summarize the usability assessment process and all procedures, including interim steps and any statistics, equations, and computer algorithms that will be used.

The usability of the data directly affects whether project objectives can be achieved. The following characteristics will be evaluated at a minimum. The results of these evaluations will be included in the project report. The characteristics will be evaluated for multiple concentration levels if the evaluator determines that this is necessary. To the extent required by the type of data being reviewed, the assessors will consult with other experienced individuals to render sound technical assessments of these DQI characteristics:

Completeness

For each matrix that was scheduled to be sampled, the Tetra Tech FOL acting on behalf of the project team will prepare a table comparing planned samples/analyses to collected samples/analyses. If deviations from the scheduled sample collection or analyses are identified, the Tetra Tech PM and technical staff will determine whether the deviations compromise the ability to meet project objectives. If they do, the Tetra Tech PM will consult with the Navy RPM and other project team members, as necessary (determined by the Navy RPM), to develop appropriate corrective actions. The completeness goal for this project is 90%.

Precision

The Tetra Tech Project Chemist acting on behalf of the project team will determine whether precision goals for field duplicates and laboratory duplicates were met. This will be accomplished by comparing duplicate results to precision goals identified in **Worksheets 12 and 28** for 25% of the data. This will also include a comparison of field and laboratory precision with the expectation that field duplicate results will be no less precise than laboratory duplicate results. If the goals are not met, or data has been flagged as estimated (J qualifier), limitations on the use of the data will be described in the project report.

Accuracy

The Tetra Tech Project Chemist acting on behalf of the project team will determine whether the accuracy/bias goals were met for project data. This will be accomplished by comparing percent recoveries of LCS, LCSD, MS, MSD, and surrogate compounds to accuracy goals identified in **Worksheet 28** for 25% of the data. This assessment will include an evaluation of field and laboratory contamination, instrument calibration variability, and analyte recoveries for surrogates, matrix spike, and laboratory control samples. If the goals are not met, limitations on the use of the data will be described in the project report. Bias of the qualified results and a description of the impact of identified non-compliances on a specific data package or on the overall project data will be described in the project report.

Representativeness

A Tetra Tech Project Scientist identified by the Tetra Tech PM and acting on behalf of the project team will determine whether the data are adequately representative of intended populations, both spatially and temporally. This will be accomplished by verifying that samples were collected and processed for analysis in accordance with the SAP, by reviewing spatial and temporal data variations, and by comparing these characteristics to expectations. The usability report will describe the representativeness of the data for each matrix and analytical fraction. This will not require quantitative comparisons unless the Tetra Tech Project Scientist determines that a quantitative analysis is required.

Comparability

The Tetra Tech Project Chemist acting on behalf of the project team will determine whether the data generated under this project are sufficiently comparable to historical Site data generated by different methods and for samples collected using different procedures and under different Site conditions. This will be accomplished by comparing overall precision and bias among data sets for each matrix and analytical fraction. This will not require quantitative comparisons unless the Tetra Tech Project Chemist determines that such quantitative analysis is required.

Sensitivity

The Tetra Tech Project Chemist acting on behalf of the project team will determine whether project sensitivity goals listed in **Worksheet No. 15** are achieved. The overall sensitivity and quantitation limits from multiple data sets for each matrix and analysis will be compared. If sensitivity goals are not achieved, the limitations on the data will be described. The Tetra Tech Project Chemist will enlist the help of the Project Risk Assessor to evaluate deviations from planned sensitivity goals.

Project Assumptions and Data Outliers

The Tetra Tech PM and designated team members will evaluate whether project assumptions are valid. This will typically be a qualitative evaluation but may be supported by quantitative evaluations. The type of evaluation depends on the assumption being tested. Quantitative assumptions include assumptions related to data distributions (e.g., normal versus log-normal) and estimates of data variability. If potential outliers exist, the data set will be evaluated statistically and outliers will be removed if a review of the associated statistic indicates that the results have an assignable cause that renders them inconsistent with the rest of the data. During this evaluation, the team will consider whether outliers could be indications of unanticipated Site conditions.

Describe the evaluative procedures used to assess overall measurement error associated with the project.

After completion of the data validation, the data and data quality will be reviewed to determine whether sufficient data of acceptable quality are available for decision making. A statistical evaluation of the data will not be performed because a biased sampling design was employed that does not support the use of statistics in assessing whether DQOs were attained.

Identify the personnel responsible for performing the usability assessment.

The Tetra Tech PM, Project Chemist, FOL, and Project Scientist will be responsible for conducting the listed data usability assessments. The data usability assessment will be reviewed by the Navy RPM during the report review. If deficiencies affecting the attainment of project objectives are identified, the review will take place either in a face to face meeting or a teleconference among the partnering team depending on the extent of identified deficiencies. If no significant deficiencies are identified, the data usability assessment will simply be documented in the project report and reviewed during the normal document review cycle.

Describe the documentation that will be generated during usability assessment and how usability assessment results will be presented so that they identify trends, relationships (correlations), and anomalies.

The data will be presented in tabular format, including data qualifications such as estimation (J, UJ) or rejection (R). Written documentation will support the non-compliant estimated or rejected data results. The project report will identify and describe the data usability limitations and suggest re-sampling or other corrective actions, if necessary.

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TABLES

TABLE 1

SUMMARY OF POSITIVE DETECTIONS - SURFACE SOIL
SITE 43 - TOLUENE DISPOSAL
NSF-IH, INDIAN HEAD, MARYLAND

LOCATION	S43SB001	S43SB002	S43SB003	S43SB003	S43SB003	S43SB004	S43SB005	S43SB006
SAMPLE NUMBER	S43SS0010001	S43SS0020001	S43SS0030001	S43SS0030001-AVG	S43SS0030001-D	S43SS0040001	S43SS0050001	S43SS0060001
DEPTH RANGE (FEET)	0 - 1	0 - 1	0 - 1	0 - 1	0 - 1	0 - 1	1 - 2	1 - 2
SAMPLE DATE	07/14/05	07/14/05	07/14/05	07/14/05	07/14/05	07/14/05	07/14/05	07/14/05
Volatile Organics (µg/kg)								
2-BUTANONE	15 U	20 U	8 J	8 J	13 U	14 U	10 U	7 U
ACETONE	15 U	20 U	31 J	39.5 J	48 J	12 J	25 J	16 J
CARBON DISULFIDE	15 U	20 U	13 U	13 U	13 U	14 U	10 U	0.8 J
METHYLENE CHLORIDE	8 B	210 J	130 J	66.5 J	6 B	130 J	6 B	9 B
TOLUENE	15 U	20 U	13 U	13 U	13 U	14 U	10 U	0.8 J
Explosives (mg/kg)								
3-NITROTOLUENE	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.25 U	0.043 J	0.25 U
NITROCELLULOSE	8 J	9 J	1.2 B	1.25 B	1.3 B	1.4 B	1.4 B	1 B
Metals (mg/kg)								
ALUMINUM	4410 J	7230 J	17000 J	17650 J	18300 J	18400 J	7190 J	3370 J
ARSENIC	4.3	5.3 K	5.7 K	4.8 K	3.9	3.8	5.2 K	4.6
BARIUM	25.9 J	64.0 J	165 J	311 J	457 J	112 J	37.8 J	15.1 J
BERYLLIUM	0.27	0.40	1.2	1.1	0.95	0.63	0.37	0.19
CADMIUM	0.95	0.82 K	0.35 K	0.24 K	0.13	0.22	0.20 K	0.16 K
CALCIUM	1610	1170	279	267.5	256	350	1140	20000
CHROMIUM	19.9 J	37.5 J	20.2 J	18.65 J	17.1 J	20.2 J	27.5 J	23.5 J
COBALT	7.0	19.3	1.7 L	2.05 L	2.4	3.2	5.7	4.1
COPPER	22.8 J	25.7 J	20.2 J	17.1 J	14.0 J	13.9 J	13.0 J	17.1 J
IRON	13500	23400	34600	25700	16800	16500	19700	12100
LEAD	91.0	42.3	22.9	21.3	19.7	15.8	9.5	5.5
MAGNESIUM	3860	765	469	502	535	675	369	11500
MANGANESE	228 J	813 J	9.9 J	9.9 J	9.9 J	15.9 J	156 J	102 J
MERCURY	0.05 U	0.05 U	0.062 K	0.062 K	0.063 U	0.34	0.051 U	0.043 U
NICKEL	38.8	12.6	5.1 L	6.25 L	7.4	7.1	6.6	7.7
POTASSIUM	299	441	366 L	416 L	466	530	351	366
SODIUM	281	465	99.9 K	105 K	111	102	173	133
VANADIUM	39.4	36.0	26.3	25.25	24.2	31.0	37.3	42.0
ZINC	70.8 K	58.4 K	14.5 K	15.35 K	16.2 K	19.4 K	13.2 K	9.7 K

B - Detected in blank; false positive.
J - Estimated.
K - Biased high.
L - Biased low.
U - Not detected.

TABLE 2

SUMMARY OF POSITIVE DETECTIONS - SUBSURFACE SOIL
SITE 43 - TOLUENE DISPOSAL
NSF-IH, INDIAN HEAD, MARYLAND

LOCATION	S43SB001	S43SB002	S43SB003	S43SB003	S43SB003	S43SB004	S43SB005	S43SB006
SAMPLE NUMBER	S43SB0010101	S43SB0020101	S43SB0030101	S43SB0030101-AVG	S43SB0030101-D	S43SB0040101	S43SB0050101	S43SB0060101
DEPTH RANGE (FEET)	1 - 2	1 - 2	1 - 2	1 - 2	1 - 2	1 - 2	2 - 3	2 - 3
SAMPLE DATE	07/14/05	07/14/05	07/14/05	07/14/05	07/14/05	07/14/05	07/14/05	07/14/05
Volatile Organics (µg/kg)								
2-BUTANONE	10 U	12 U	9 J	7.5 J	6 J	7 J	8 J	10 U
ACETONE	10 U	12 U	12 J	12.5 J	13 J	10 J	17 J	19 J
METHYLENE CHLORIDE	8 B	5 B	150 J	140 J	130 J	140 J	120 J	8 B
TRICHLOROETHENE	8 J	12 U	14 U	13.5 U	13 U	14 U	12 U	10 U
Metals (mg/kg)								
ALUMINUM	3490 J	3380 J	16200 J	16500 J	16800 J	16200 J	18300 J	18500 J
ARSENIC	3.6	2.7	11.3 K	12.4 K	13.5 K	8.5	2.6	3.0
BARIUM	20.4	29.5	93.4	92.9	92.4	106	133	134
BERYLLIUM	0.26	1.6	1.2	1.2	1.2	1.1	0.88	0.91
CADMIUM	0.16	0.40	0.41 K	0.405 K	0.40 K	0.16	0.058	0.10
CALCIUM	176	1280	143	168.5	194	116	296	131
CHROMIUM	18.2 J	17.3 J	18.5 J	20.3 J	22.1 J	16.7 J	17.1 J	17.0 J
COBALT	3.5	4.6	3.0	3.0	3.0	4.9	3.8	2.8
COPPER	9.7	21.9	16.8	17.25	17.7	14.8	12.0	11.6
IRON	14100	18700	41200	41350	41500	19200	8550	13400
LEAD	6.1	7.7	15.6	16.2	16.8	17.0	18.5	17.2
MAGNESIUM	196	777	586	611.5	637	607	629	579
MANGANESE	110 J	227 J	10.9 J	11.6 J	12.3 J	10.2 J	29.9 J	43.4 J
NICKEL	7.0	5.5	7.5 L	7.7 L	7.9 L	9.7	8.2	7.4
POTASSIUM	331	380	420 L	462 L	504 L	408	357	412
SODIUM	288	265	128 K	128.5 K	129 K	263	344	260
VANADIUM	16.2	25.3	49.2	52.5	55.8	38.7	18.3	22.2
ZINC	23.1 K	13.1 K	16.7 K	17.7 K	18.7 K	22.6 K	20.5 K	15.2 K
Miscellaneous (mg/kg)								
CYANIDE	1.4	0.097 U	0.13 U	0.125 U	0.12 U	0.12 U	0.11 U	0.21

B - Detected in blank; false positive.
J - Estimated.
K - Biased high.
L - Biased low.
U - Not detected.

TABLE 3

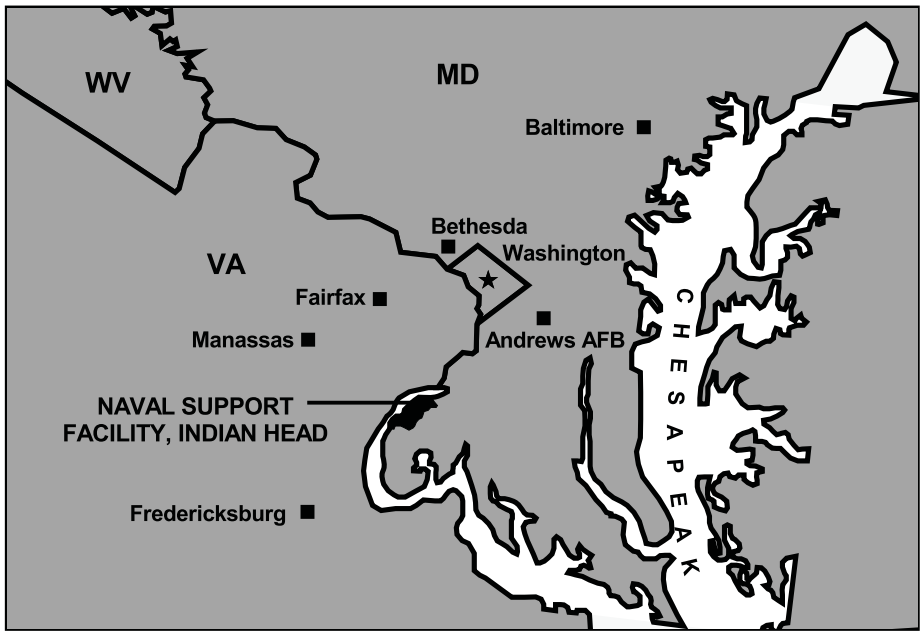
SUMMARY OF POSITIVE DETECTIONS - VOLATILE ORGANICS IN GROUNDWATER
SITE 43 - TOLUENE DISPOSAL
NSF-IH, INDIAN HEAD, MARYLAND

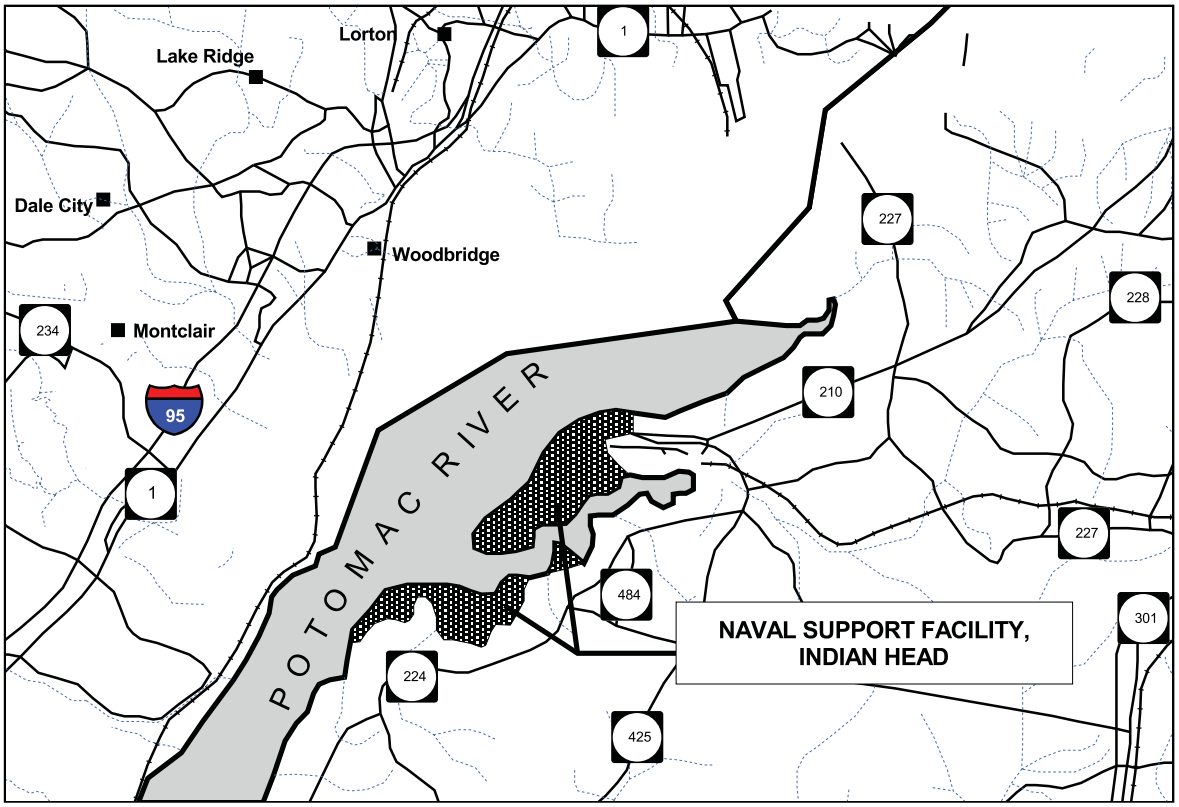
Parameter	MCL	RSL	S43MW001 2005	S43MW001-D (duplicate) 2005	S42MW002 2005	S43MW001 2007	S43MW001-D (duplicate) 2007	S42MW002 2007	S43TW001 2007	S43TW002 2007	S43TW003 2007	S43TW004 2007	S43TW005 2007
Volatile Organics (µg/L)													
2-Butanone	NA	7,100	5000 U	5000 U	5 U	8000 U	8000 U	5 U	5 U	5 U	4.2 J	5 U	5 U
Acetone	NA	22,000	5000 U	5000 U	5 U	8000 U	8000 U	5 U	5 U	5 U	17	5 U	5 U
Bromoform	80	8.5	500 U	500 U	0.5 U	800 U	**960**	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U	0.5 U
cis-1,2-Dichloroethene	70	370	**580**	**480 J**	0.5 U	800 U	800 U	0.5 U	0.36 J	0.5 U	0.5 U	0.5 U	0.5 U
Trichloroethene	5	1.7	**53000 J**	**55,000 J**	1.3 J	**36,000**	**36,000**	3.2	3.2	0.5 U	0.5 U	0.5 U	0.5 U

Shaded cell indicates exceedance of MCL.

µg/L	Micrograms per liter.
D	Field duplicate sample.
J	Estimated.
MCL	Maximum Contaminant Level.
MW	Monitoring well.
NA	Not available.
ORNL	Oak Ridge National Laboratory
RSL	Regional Screening Level (ORNL RSL for tap water, ORNL, April 2009).
TW	Temporary well.
U	Not detected above concentration noted.

FIGURES





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CHECKED BY: G. LATULIPPE — DATE: 12/03/07	FACILITY LOCATION MAP NAVAL SUPPORT FACILITY, INDIAN HEAD INDIAN HEAD, MARYLAND	APPROVED BY: G LATULIPPE — DATE: 12/03/07	
COST/SCHEDULE-AREA		APPROVED BY: — — DATE: —	
SCALE: AS NOTED		DRAWING NO.: FIGURE 1	REV: 0

P:\GIS\INDIANHEAD_NSWC\APR\SITE57_PILOTSTUDY_SITELOCATION.APR FACILITY LOCATION MAP 10/17/07 SS

P:\GIS\INDIANHEAD_NSWC\APR\COMMUNITYRELATIONSPLAN.APR SSP SITE 43 12/12/05 KMP

DRAWN BY K. PEILA DATE 6/2/04	Tetra Tech NUS, Inc.	CONTRACT NUMBER 2193	OWNER NUMBER 0006
CHECKED BY KCT DATE 6/2/04	SITE LOCATION MAP SITE 43 - TOLUENE DISPOSAL NAVAL SUPPORT FACILITY INDIAN HEAD INDIAN HEAD, MARYLAND	APPROVED BY KCT DATE 6/2/04	
COST/SCHEDULE-AREA		APPROVED BY — DATE —	
SCALE AS NOTED		DRAWING NO. FIGURE 2	REV 0

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N

812
Lunch House

714
Change House
Laundry

813
Lunch House

SCHUYLER ROAD

712
Auxiliary
Steam Plant

Former
Sewage Treatment Plant
Area

1482
Chlorinator

1531
Well Water
Treatment

1612

1257
Transformer
Station

Drainage Swale
near Building 1040

1963

1962

1040

SCHUYLER
ROAD

3022

721
Mold Filling

821
Toilet
Building

1916

1360
Scrap Powder
Shed

GALLERY ROAD

720
Grain Manufacturing
and Motor Loading

1193
Control House

1921

720A
Motor
Test Bay

722
Mold Filling

Telephone Pole
near Building 1041

1041

LEGEND

RAILROAD

STORM DRAIN

WATER LINE

STREAM/DRAINAGE DITCH

230 FOOT RESTRICTION

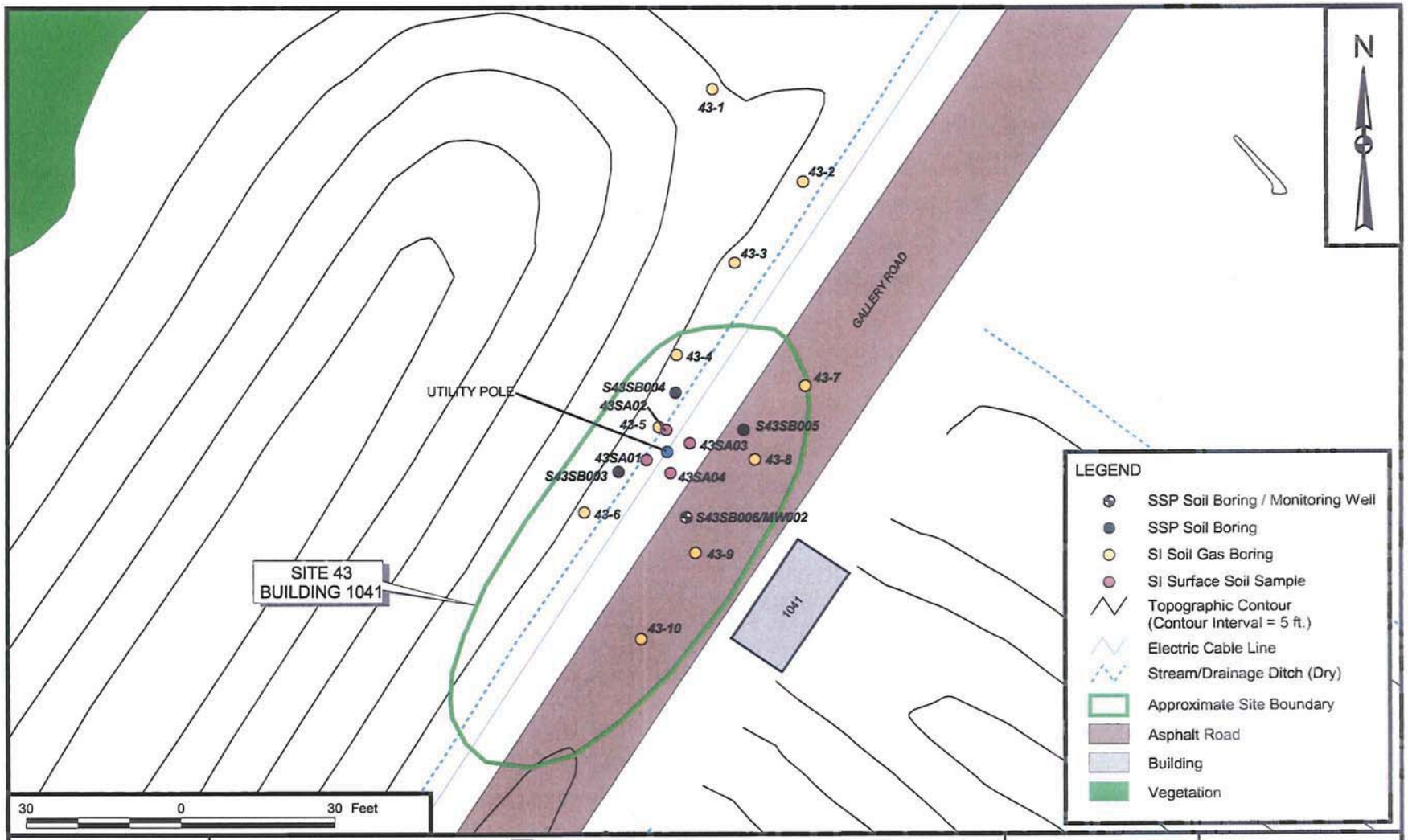
0 100 200

SCALE IN FEET

TETRA TECH NUS, INC.

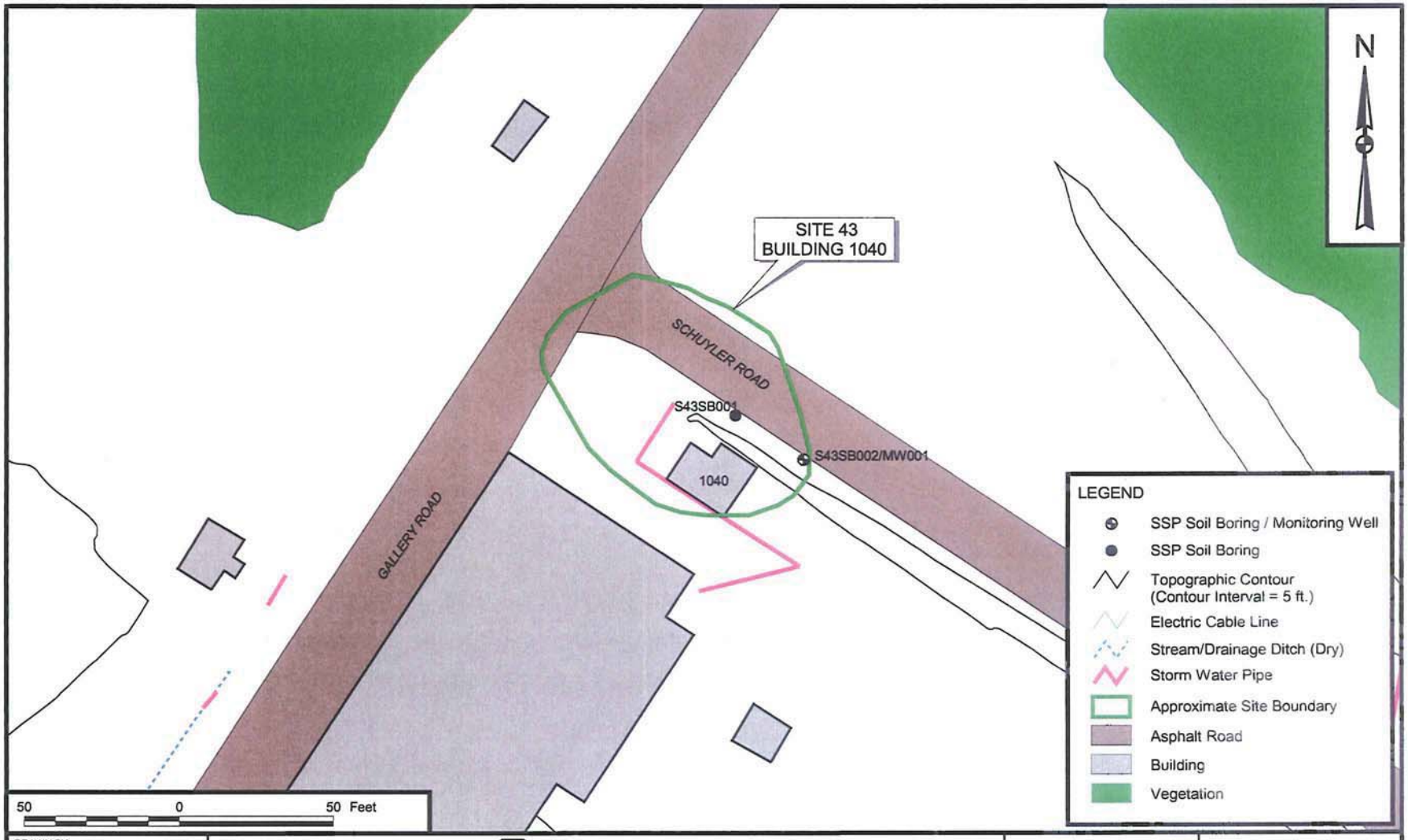
SITE LAYOUT
SITE 43 - TOLUENE DISPOSAL
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

FILE	SCALE
112G00771GM01.DWG	AS NOTED
FIGURE NUMBER: FIGURE 3	REV: 0 DATE: 06/02/10



DRAWN BY: K. PEILA — DATE: 06/03/04	Tetra Tech NUS, Inc.	CONTRACT NUMBER: 2193	OWNER NUMBER: CTO 114
CHECKED BY: S. NESBIT — DATE: 07/12/09	SAMPLE LOCATIONS SI (1994) AND SSP PHASE 1 (2005) SITE 43 - TOLUENE DISPOSAL NAVAL SUPPORT FACILITY INDIAN HEAD INDIAN HEAD, MARYLAND	APPROVED BY: KCT — DATE: 06/03/04	
COST/SCHEDULE-AREA		APPROVED BY: — — DATE: —	
SCALE: AS NOTED		FIGURE 4	REV: 0

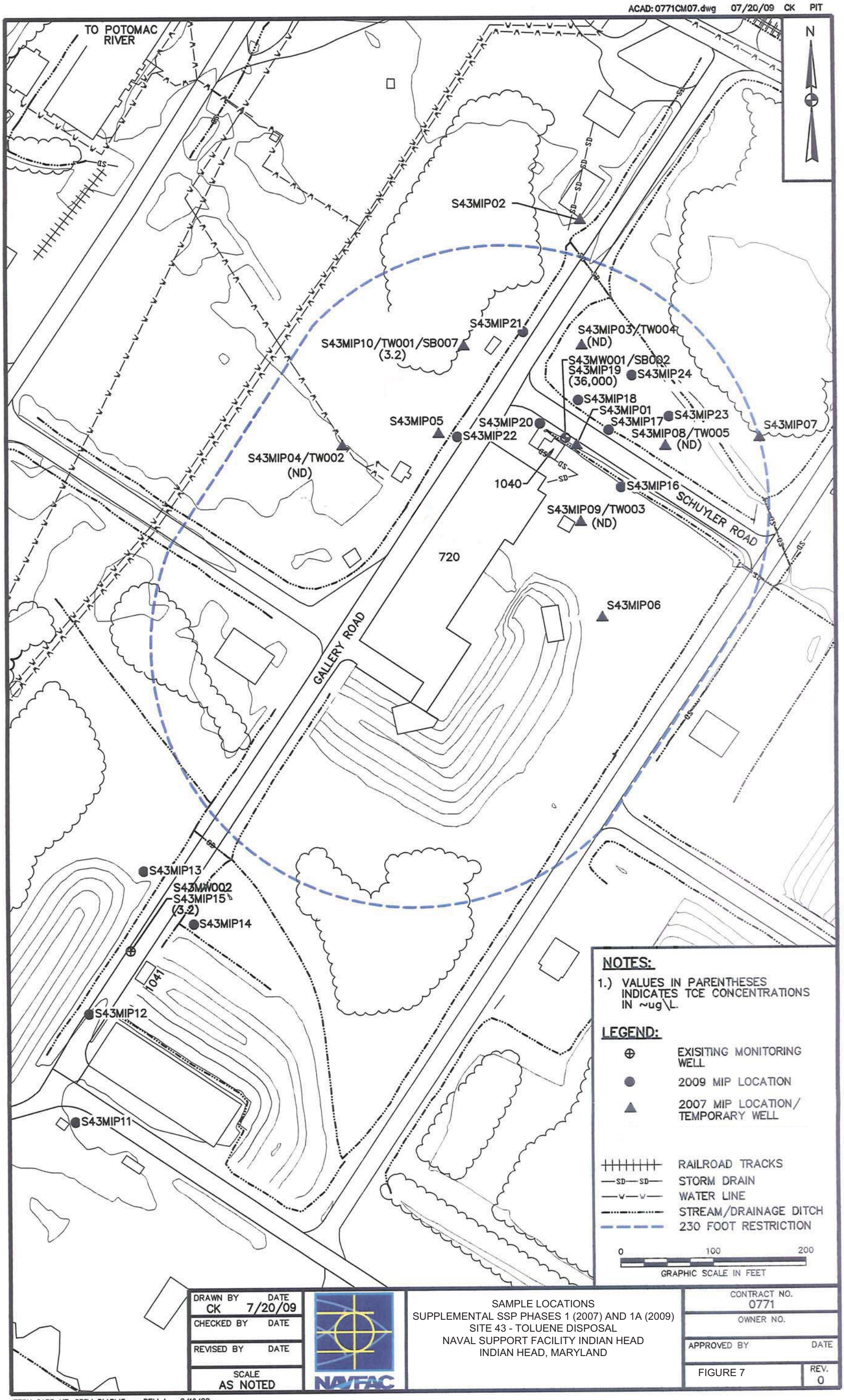
P:\GIS\INDIANHEAD_NSWC\APR\COMMUNITYRELATIONSPLAN.APR SITE 43 1041 SURVEYED WORK PLAN LAYOUT 07/12/09 JEE



DRAWN BY: K. PEILA — DATE: 06/03/04	Tetra Tech NUS, Inc.	CONTRACT NUMBER: 2193	OWNER NUMBER: CTO 114
CHECKED BY: S. NESBIT — DATE: 07/21/09	SAMPLE LOCATIONS SSP PHASE 1 (2005) SITE 43 - TOLUENE DISPOSAL NAVAL SUPPORT FACILITY INDIAN HEAD INDIAN HEAD, MARYLAND	APPROVED BY: KCT — DATE: 06/03/04	
COST/SCHEDULE-AREA		APPROVED BY: — — DATE: —	
SCALE: AS NOTED		FIGURE 5	REV: 0

P:\GIS\INDIANHEAD_NSWC\APR\COMMUNITYRELATIONSPLAN.APR SITE 43 1040 SSP LOCATIONS SURVEYED LAYOUT 07/21/09 JEE

ACAD: 0771CM07.dwg 07/20/09 CK PIT



DRAWN BY CK	DATE 7/20/09	SAMPLE LOCATIONS SUPPLEMENTAL SSP PHASES 1 (2007) AND 1A (2009) SITE 43 - TOLUENE DISPOSAL NAVAL SUPPORT FACILITY INDIAN HEAD INDIAN HEAD, MARYLAND
CHECKED BY	DATE	
REVISED BY	DATE	
SCALE AS NOTED		

NAVFAC

CONTRACT NO. 0771	
OWNER NO.	
APPROVED BY	DATE
FIGURE 7	REV. 0

FORM CADD NO. SDIV-BV.DWG - REV 1 - 9/10/98

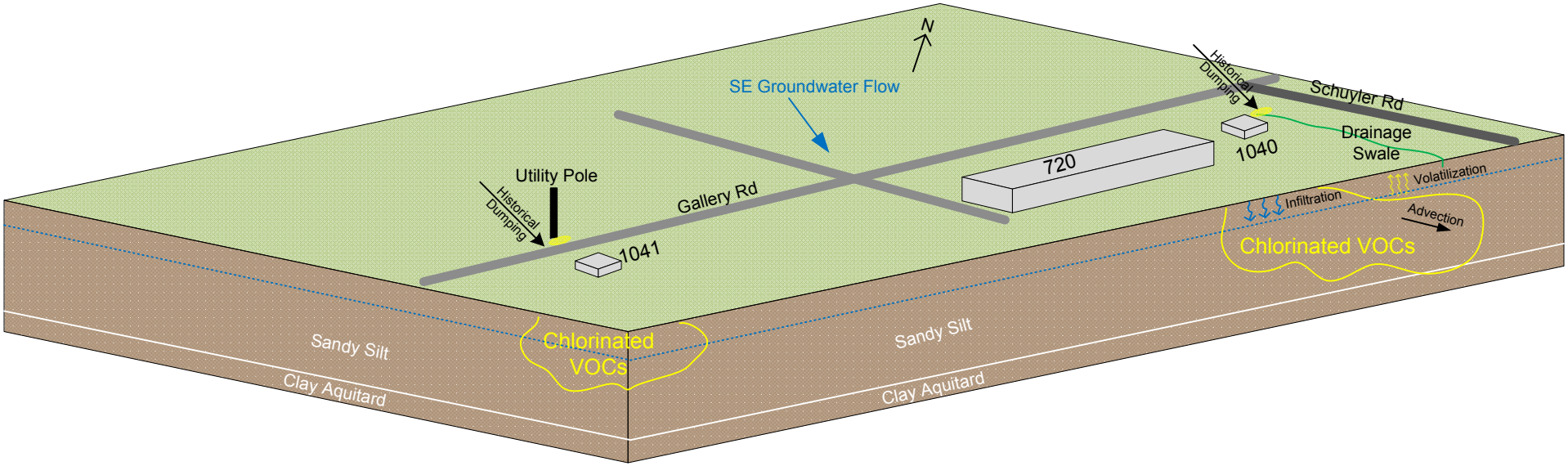


FIGURE 8
INTERPRETIVE CONCEPTUAL SITE MODEL
SITE 43 – TOLUENE DISPOSAL
NAVAL SUPPORT FACILITY INDIAN HEAD
INDIAN HEAD, MARYLAND

ACAD: F_11x17-BV.dwg 09/06/01 INL

N

SCHUYLER ROAD

GALLERY ROAD

1257

1040

720

1041

MW01
MW02
MW03
MW04
MW05
MW06
TW06
TW07
TW08
TW09
TW10
TW11
TW12
SG01
SG02
SG03
SG04

Note: Soil samples will be collected above the water table at each DPT grab groundwater sample location and soil gas sample location.

LEGEND

- RAILROAD
- STORM DRAIN
- WATER LINE
- STREAM/DRAINAGE DITCH
- 230 FOOT RESTRICTION
- Monitoring Well (MW03 through MW06 are to-be-installed)
- DPT grab groundwater sample
- Soil gas sample

0 100 200

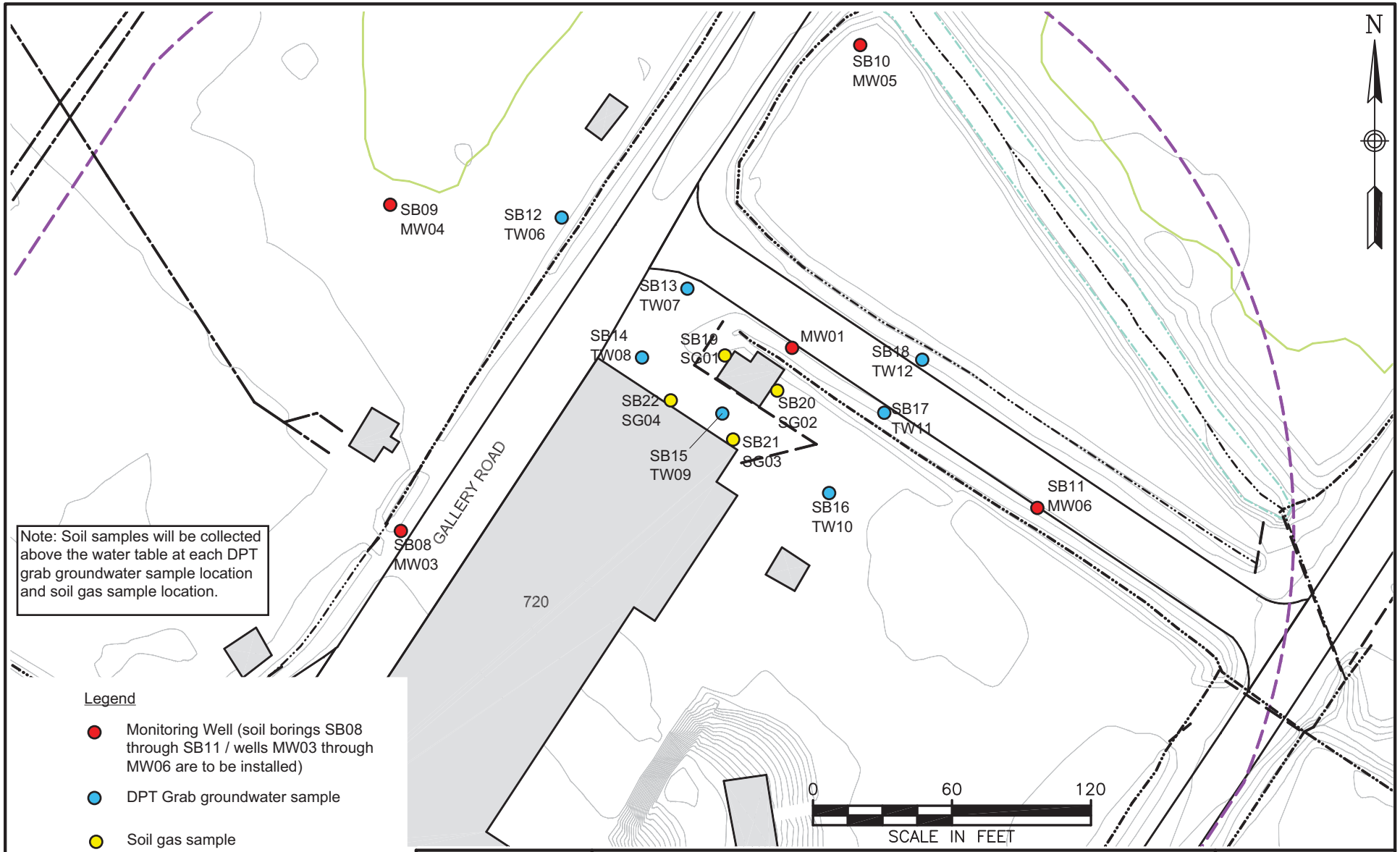
SCALE IN FEET

TETRA TECH NUS, INC.

PROPOSED LOCATIONS
SITE 43 – TOLUENE DISPOSAL
NAVAL SUPPORT FACILITY, INDIAN HEAD
INDIAN HEAD, MARYLAND

FILE	SCALE
112G00771GM01.DWG	AS NOTED
FIGURE 9	REV 0 / DATE 06/02/10

ACAD: TTNUS–8.5x11.dwg 08/03/01 INL



Legend

- Monitoring Well (soil borings SB08 through SB11 / wells MW03 through MW06 are to be installed)
- DPT Grab groundwater sample
- Soil gas sample
- STORM DRAIN
- WATER LINE
- STREAM/DRAINAGE DITCH
- 230 FOOT RESTRICTION

TETRA TECH NUS, INC.

PROPOSED LOCATIONS
SITE 43 – TOLUENE DISPOSAL
NAVAL SUPPORT FACILITY, INDIAN HEAD
INDIAN HEAD, MARYLAND

SCALE	
AS NOTED	
FILE	
FILE.DWG	
REV	DATE
0	7/23/01

FIGURE 10

Appendix A

Health and Safety Plan

Health and Safety Plan
for
Remedial Investigation
at
Site 43 – Toluene Spill

Naval Support Facility
Indian Head
Indian Head, Maryland

Naval Facilities Engineering Command Washington
NAVFAC Atlantic Contract No. N62472-03-D-0057
Contract Task Order 114

March 2011

HEALTH AND SAFETY PLAN
FOR

SITE 43 – TOLUENE SPILL

NAVAL SUPPORT FACILITY, INDIAN HEAD
INDIAN HEAD, MARYLAND

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY (CLEAN) CONTRACT

Submitted to:
Naval Facilities Engineering Command Washington
1314 Harwood Street, S.E.
Washington, D.C. 20374-5018

Submitted by:
Tetra Tech NUS, Inc.
234 Mall Boulevard, Suite 260
King of Prussia, PA 19406

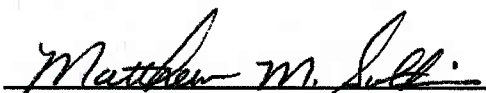
CONTRACT NO. N62472-03-D-0057
CONTRACT TASK ORDER 0114

MARCH 2011

PREPARED UNDER THE SUPERVISION OF:

ROBERT M. SOK, P.G.
PROJECT MANAGER
TETRA TECH NUS, INC.
NORFOLK, VIRGINIA

APPROVED FOR SUBMITTAL BY:



MATTHEW M. SOLTIS, CIH, CSP
CLEAN HEALTH & SAFETY MANAGER
TETRA TECH NUS, INC.
PITTSBURGH, PENNSYLVANIA

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1.0 INTRODUCTION

The objective of this Health and Safety Plan (HASP) is to provide the minimum safety practices and procedures for Tetra Tech NUS, Inc. (TtNUS) and subcontractor personnel engaged in the Site Screening Process Investigation that is to be conducted at the Site 43 Toluene Spill at the Naval Support Facility, Indian Head (NSF-IH), Indian Head, Maryland.

This HASP has been written using the latest available information regarding known or suspected chemical contaminants and potential and foreseeable physical hazards associated with the proposed work at Site 43. This HASP has been designed to be used in accordance with the TtNUS Health and Safety Guidance Manual (HSGM). The HSGM provides detailed information pertaining to procedures to be performed on site as directed by the HASP, as well as TtNUS standard operating procedures. Both the HASP and the HSGM must be present at the site to comply with the requirements stipulated in the Occupational Safety and Health Administration (OSHA) standard 29 CFR 1910.120.

This HASP has been written to support proposed tasks and techniques associated with the scope of work as presented in Section 4.0. Should the proposed work site conditions and/or suspected hazards change, or if new information becomes available, this document will be modified. Changes to the HASP will be made with the approval of the TtNUS CLEAN Health and Safety Manager (HSM) and the Project Manager (PM). The PM will notify the affected personnel of the changes.

1.1 AUTHORITY

This Contract Task Order (CTO) 0114 and the requirements set forth represent an integral part of an overall effort conducted under the Comprehensive Long - Term Environmental Action Navy (CLEAN) contract, administered through the U.S. Navy Naval Facilities Engineering Command Atlantic, as defined under Contract No. N62472-03-D-0057.

1.2 KEY PROJECT PERSONNEL AND ORGANIZATION

This section defines responsibility for site safety and health for TtNUS and subcontractor employees engaged in on site activities. Personnel assigned to these positions shall exercise the primary responsibility for the on site health and safety. These persons will be the primary point of contact for any questions regarding the safety and health procedures and the selected control measures.

- The TtNUS Project Manager (PM) is responsible for the overall direction and implementation of health and safety for this project.

- The TtNUS Field Operations Leader (FOL) is responsible for implementation of this HASP with the assistance of an appointed Site Safety Officer (SSO). The FOL manages field activities, executes the work plan, and enforces safety procedures, as applicable to the work plan.

- The SSO supports site activities by advising the FOL on the aspects of health and safety on site. These duties may include the following:

  - Coordinates health and safety activities with the FOL.
  - Selects, inspects, implements, and maintains personal protective equipment.
  - Establishes work zones and control points.
  - Directs and assists in the development of decontamination areas and procedures.
  - Implements air monitoring program in support of on site activities.
  - Verifies training and medical status of on site personnel status in relation to site activities.
  - Implements hazard communication, respiratory protection, and other safety and health programs, as necessary.
  - Provides site-specific training for on site personnel.

- Compliance with these requirements is monitored by the Project Health and Safety Officer (PHSO) and is coordinated through the Health and Safety Manager.

1.3 STOP WORK AUTHORIZATION

ALL employees are empowered, authorized, and responsible to STOP WORK at any time when an imminent and uncontrolled safety or health hazard is perceived. In a Stop Work event (immediately after the involved task has been shut down and the work area has been secured in a safe manner) the employee shall contact the Project Manager and the Corporate Health and Safety Manager. Through observations and communication, all parties involved shall then develop, communicate, and implement corrective actions necessary and appropriate to modify the task and to resume work.

1.4 SITE INFORMATION AND PERSONNEL ASSIGNMENTS

Site Name: Naval Support Facility Indian Head (NSF-IH) **Address:** Indian Head, Maryland

Site Point of Contact: N **Phone Number:** (301) 744-2263

Scheduled Activities: Remedial Investigation

Proposed Dates of Work: May 2011 until completion

Project Team:

TtNUS Management Personnel	Discipline/Tasks Assigned	Phone Number
Robert Sok	Project Manager (PM)	(757) 466-4904
Matthew M. Soltis, CIH, CSP	Health and Safety Manager (HSM)	(412) 921-8912
Fred Ramser	Field Operations Leader (FOL)	(412) 921-8838
James K. Laffey	Project Health and Safety Officer (PHSO)	(412) 921-8678

Non-TtNUS Personnel	Affiliation/Discipline/Tasks Assigned

Hazard Assessment (for purposes of 29 CFR 1010.132) for HASP preparation has been conducted by: James K. Laffey

2.0 EMERGENCY ACTION PLAN

2.1 INTRODUCTION

This section has been developed to direct and guide field personnel in the event of an emergency. Site activities will be coordinated with the client contact, Shawn Jorgensen. In the event of an emergency which cannot be mitigated using onsite resources, personnel will evacuate to a safe place of refuge and the appropriate emergency response agencies will be notified. It has been determined that the majority of potential emergency situations would be better supported by outside emergency responders. Based on this determination, TtNUS and subcontractor personnel will not provide emergency response support beyond the capabilities of onsite response. Workers who are ill or who have suffered a non-serious injury may be transported by site personnel to nearby medical facilities, provided that such transport does not aggravate or further endanger the welfare of the injured/ill person. The emergency response agencies listed in this plan are capable of providing the most effective response, and as such, will be designated as the primary responders. These agencies are located within a reasonable distance from the area of site operations, which ensures adequate emergency response time. Navy contact Shawn Jorgensen will be notified anytime outside response agencies are contacted. This Emergency Action Plan conforms to the requirements of 29 CFR 1910.38(a), as allowed in 29 CFR 1910.120(l)(1)(ii).

TtNUS will, through necessary services, provide the following emergency action measures:

- Initial stage fire fighting support and prevention
- Initial spill control and containment measures and prevention
- Removal of personnel from emergency situations such as confined space entry
- Initial medical support for injuries or illnesses requiring basic first-aid
- Site control and security measures as necessary

2.2 EMERGENCY PLANNING

Through the initial hazard/risk assessment effort, it is anticipated that emergencies resulting from chemical, physical, or fire hazards are unlikely given the nature of site activities. However, given the mission of NSF-IH, the possibility of encountering Unexploded Ordnance (UXO) must always be considered no matter how unlikely. To minimize and eliminate the potential for any emergency situations, emergency planning activities will include the following (which are the responsibility of the FOL):

- Coordinating with Base Fire Protection and Emergency Services to be notified of scheduled events and activities.

- Establishing and maintaining information at the project staging area (support zone) for easy access in the event of an emergency. This information will include the following:

  - Chemical Inventory (of chemicals used onsite), with Material Safety Data Sheets.
  - Onsite personnel medical records (Medical Data Sheets).
  - A log book identifying personnel onsite each day.
  - Hospital route maps with directions (these should also be placed in each site vehicle).
  - Emergency Notification - phone numbers.

The TtNUS FOL will be responsible for the following tasks:

- Identifying a chain of command for emergency action.

- Educating site workers to the hazards and control measures associated with planned activities at the site, and providing early recognition and prevention, where possible.

- Periodically performing practice drills to ensure site workers are familiar with incidental response measures.

- Providing the necessary equipment to safely accomplish identified tasks.

2.3 EMERGENCY RECOGNITION AND PREVENTION

2.3.1 Recognition

Emergency situations that may be encountered during site activities will generally be recognized by visual observation. To adequately recognize chemical exposures, site personnel must have a clear knowledge of signs and symptoms of exposure associated with site contaminants. This information is provided in Section 6.1 of this HASP. Tasks to be performed at the site, potential hazards associated with those tasks and the recommended control methods are discussed in detail in Sections 5.0 and 6.0. Additionally, early recognition of hazards will be supported by periodic site surveys to identify any situation predisposed to an emergency. The FOL will be responsible for performing surveys of work areas prior to initiating site operations and periodically while operations are being conducted. Survey findings will be documented by the FOL in the site logbook, however, site personnel will be responsible for reporting hazardous situations. Where potential hazards exist, TtNUS will initiate control measures to prevent adverse effects to human health and the environment.

The above actions will provide early recognition for potential emergency situations, and allow TtNUS to initiate necessary control measures. However, if the FOL determines that control measures are not sufficient to eliminate the hazard, TtNUS will withdraw from the site and notify the appropriate response agencies listed in Table 2-1.

2.3.2 Prevention

TtNUS and subcontractor personnel will minimize the potential for emergencies by following this HASP, the HSGM, and applicable OSHA regulations. Periodic site surveys of work areas and correction of any identified deficiencies prior to the commencement of that day's activities by the FOL will also assist in prevention of illness/injuries when hazards are recognized early and control measures initiated.

2.4 EVACUATION ROUTES, PROCEDURES, AND PLACES OF REFUGE

An evacuation will be initiated whenever recommended hazard controls are insufficient to protect the health, safety or welfare of site workers. Specific examples of conditions that may initiate an evacuation include, but are not limited to the following: severe weather conditions; fire or explosion; and evidence of personnel overexposure to potential site contaminants.

In the event of an emergency requiring evacuation, personnel will immediately stop activities and report to the designated safe place of refuge unless doing so would pose additional risks. When evacuation to the primary place of refuge is not possible, personnel will proceed to a designated alternate location and remain until further notification from the TtNUS FOL. Safe places of refuge will be identified prior to the commencement of site activities by the FOL and will be conveyed to personnel as part of the pre-activities briefing session. This information will be reiterated during daily safety meetings and indicated on the Safe Work Permits. Whenever possible, the safe place of refuge will also serve as the telephone communications point for that area. During an evacuation, personnel will remain at the refuge location until directed otherwise by the TtNUS FOL or the on-site Incident Commander of the Emergency Response Team. The FOL will perform a head count at this location to account for and to confirm the location of site personnel. Emergency response personnel will be immediately notified of any unaccounted personnel. The FOL will document the names of personnel onsite (on a daily basis) in the site Health and Safety Logbook. This information will be utilized to perform the head count in the event of an emergency.

Evacuation procedures will be discussed during the pre-activities training session, prior to the initiation of project tasks. Evacuation routes from the site and safe places of refuge are dependent upon the location at which work is being performed and the circumstances under which an evacuation is required. Additionally, site location and meteorological conditions (i.e., wind speed and direction) may dictate

evacuation routes. As a result, assembly points will be selected and communicated to the workers relative to the site location where work is being performed. Evacuation should always take place in an upwind direction from the site and away from water bodies.

2.5 EMERGENCY ALERTING AND ACTION/RESPONSE PROCEDURES

TtNUS personnel will likely be working in close proximity to each other during planned site activities. Site personnel will initiate emergency notification to onsite personnel by voice commands, hand signals, vehicle horns, or line of site communication to alert site personnel of an emergency. When project tasks are performed simultaneously on different sites, radios will be used to communicate emergency situations and request assistance. The Fire Department will provide rescue services, if needed, during confined space entry operations. The details for notification must be documented in the permit.

If an emergency warranting evacuation occurs, the following procedures are to be initiated:

- Initiate the evacuation via appropriate and/or available communication method (hand signals, voice commands, etc.).
- Report to the designated refuge point.
- Once non-essential personnel are evacuated, appropriate response procedures will be enacted to control the situation.
- Describe to the FOL (serving as the Incident Coordinator) pertinent incident details.

In the event that site personnel cannot mitigate the hazardous situation, the FOL will enact emergency notification procedures to secure additional assistance in the following manner:

Contact pertinent emergency contacts listed in Table 2-1 and report the incident. Give the emergency operator the location of the emergency, the type of emergency, the number of injured, and a brief description of the incident. Stay on the phone and follow the instructions given by the operator. The operator will then notify and dispatch the proper emergency response agencies.

2.6 EMERGENCY CONTACTS

Prior to initiating field activities, personnel will be thoroughly briefed on the emergency procedures to be followed in the event of an accident. Table 2-1 provides a list of emergency contacts and their associated

telephone numbers. This table must be posted where it is readily available to site personnel. Facility maps should also be posted showing potential evacuation routes and designated meeting areas.

TABLE 2-1
EMERGENCY REFERENCE
SITE 43 – INDIAN HEAD DIVISION - NSWC

AGENCY	TELEPHONE
EMERGENCY (fire, ambulance, rescue, police)	(301) 744-4333
Site Point of Contact Nicholas Carros	(301) 744-2263
Navy Remedial Project Manager Joseph Rail	(202) 685-3279
Hospital: Civista Medical Center	(301) 609-4000
Hospital: Fort Washington Hospital	(301) 292-7000
National Capital Poison Center	(800) 222-1222
Chemtrec	(800) 424-9300
National Response Center	(800) 424-8802
Utility One Call	8-1-1
TtNUS, Pittsburgh Office	(412) 921-7090
Health and Safety Manager Matthew M. Soltis, CIH, CSP	(412) 921-8912
Project Manager Robert Sok	(757) 466-4904
Project Health and Safety Officer James K. Laffey	(412) 921-8678

2.7 EMERGENCY ROUTES TO HOSPITALS

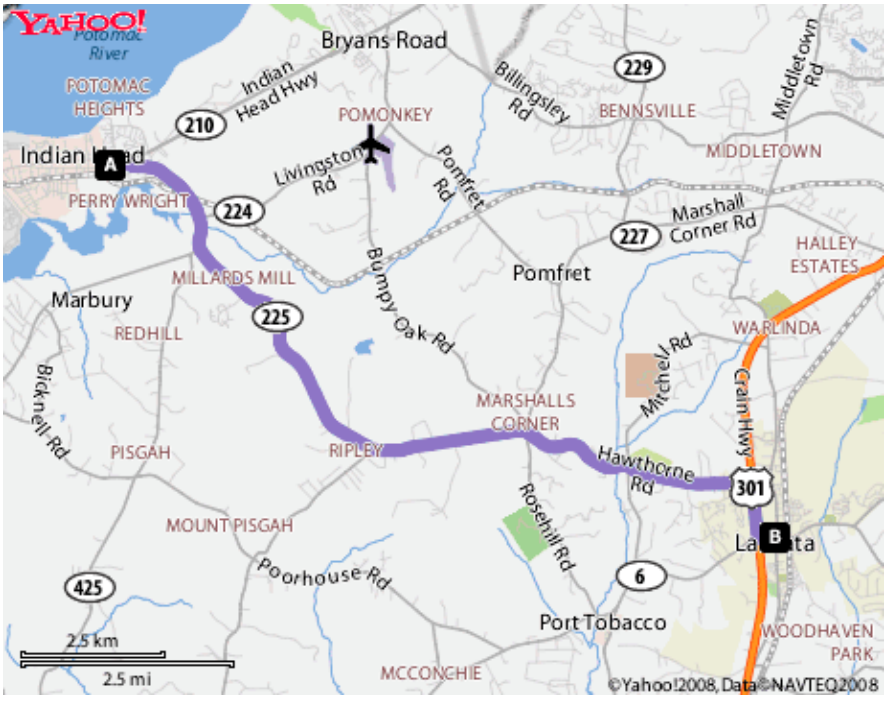
The closest hospital to the NSF-IH is the Civista Medical Center in La Plata, Maryland. The alternate hospital is Fort Washington Hospital in Fort Washington, Maryland. Maps showing the proximity of the NSF-IH to both of the hospitals are included as Figure 2-1 and 2-1.1.

Civista Medical Center
701 East Charles Street,
La Plata, MD 20646
(301) 609-4000

- Head northeast on Judy Lane toward Greenslade Rd
- Turn left at Greenslade Rd
- Turn right at Strauss Ave
- Turn left to stay on Strauss Ave to **MD Route 210**
- Exit the facility and proceed east on **MD Route 210** (Indian Head Highway) for 0.4 miles.
- Turn Right on Route 228 (Hawthorne Road) and follow for 10.4 miles.
- Turn Right on Route 301 South (Crane Highway) and follow for 0.7 mile.
- Turn Left on Route 6 (Charles Street) and follow for 0.6 mile.
- Turn Right into the hospital.

FIGURE 2-1

ROUTE TO CIVISTA MEDICAL CENTER

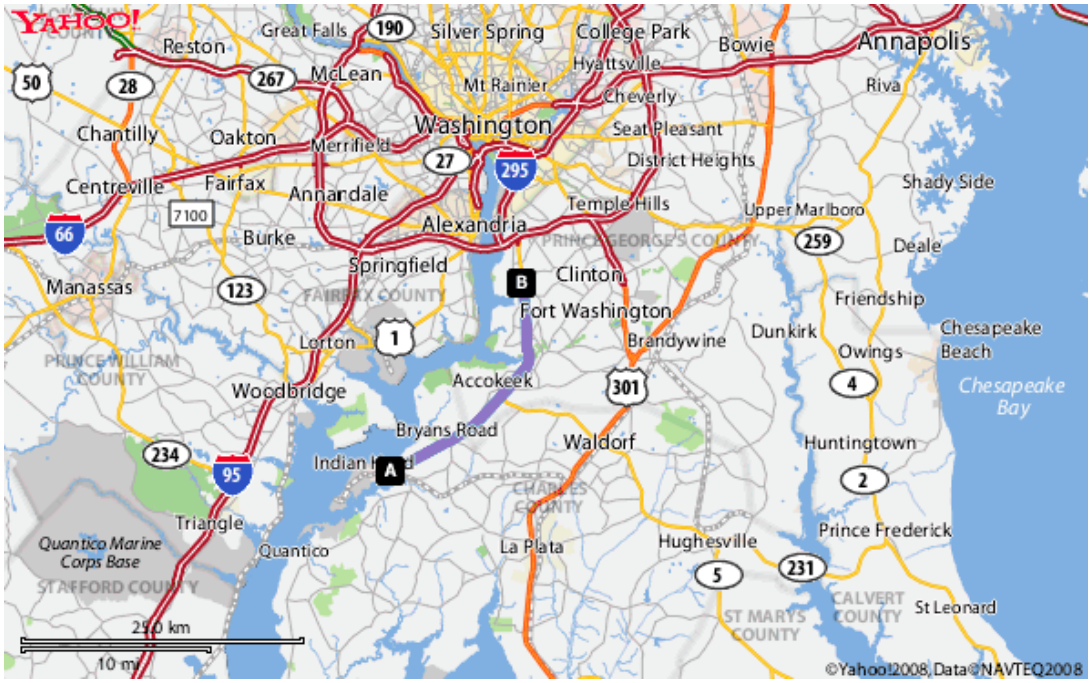


Fort Washington Hospital
11711 Livingston Road
Fort Washington, MD 20744
(301) 292-7000

- Head northeast on Judy Lane toward Greenslade Rd
- Turn left at Greenslade Rd
- Turn right at Strauss Ave
- Exit the facility and proceed east on **MD Route 210** (Indian Head Highway) for 14.5 miles.
- Turn Left on Swan Creek Road East (Hawthorne Road) and follow for less than 0.1 mile.
- Turn Right on Livingston Road and follow for 0.2 mile.
- Turn Left into the hospital.

FIGURE 2-1.1

ROUTE TO FORT WASHINGTON HOSPITAL CENTER



2.8 DECONTAMINATION PROCEDURES / EMERGENCY MEDICAL TREATMENT

During any site evacuation, decontamination procedures will be performed only if doing so does not further jeopardize the welfare of site workers. Decontamination will be postponed if the incident warrants immediate evacuation. However, it is unlikely that an evacuation would occur which would require workers to evacuate the site without first performing the necessary decontamination procedures.

TtNUS personnel will perform removal of personnel from emergency situations and may provide initial medical support for injury/illnesses requiring only first-aid level support. Medical attention above that level will require assistance and support from the designated emergency response agencies. Attachment I provides the procedure to follow when reporting an injury/illness, and the form to be used for this purpose. If the emergency involves personnel exposures to chemicals, follow the steps provided in Figure 2-2.

Because the distance to definitive medical support is greater than four minutes for life threatening injuries and greater than 15-minutes for non-life threatening injuries, First-Aid trained personnel will be onsite.

2.9 INJURY AND ILLNESS REPORTING

Any pertinent information regarding allergies to medications or other special conditions will be provided to medical service personnel. This information is listed on Medical Data Sheets filed onsite (see Attachment II) If an exposure to hazardous materials has occurred, provide hazard information in Section 6.0 to medical service personnel**.** As soon as possible, Navy contact Shawn Jorgensen must be informed of any incident or accident that requires medical attention.

2.10 PPE AND EMERGENCY EQUIPMENT

A first-aid kit, eye wash units (or bottles of disposable eyewash solution) and a fire extinguisher will be maintained onsite and shall be immediately available for use in the event of an emergency. This equipment will be located in the field office or site vehicle. Personnel identified within the field crew with bloodborne pathogen and first-aid training will be the only personnel permitted to offer first-aid assistance.

FIGURE 2-2

POTENTIAL RESPONSE PROTOCOL

The purpose of this protocol is to provide guidance for the medical management of injury situations.
In the event of a personnel injury or accident:

- Rescue, when necessary, employing proper equipment and methods.
- Give attention to emergency health problems -- breathing, cardiac function, bleeding, and shock.
- Transfer the victim to the medical facility designated in this HASP by suitable and appropriate conveyance (i.e. ambulance for serious events)
- Obtain as much exposure history as possible (a Potential Exposure report is attached).
- If the injured person is a Tetra Tech NUS employee, call the medical facility and advise them that the patient(s) is/are being sent and that they can anticipate a call from the WorkCare physician. WorkCare will contact the medical facility and request specific testing which may be appropriate. WorkCare physicians will monitor the care of the victim. Site officers and personnel should not attempt to get this information, as this activity leads to confusion and misunderstanding.
- Call WorkCare at 1-800-455-6155 and enter Extension 109 or follow the voice prompt for after hours and weekend notification and be prepared to provide the following:
  - Any known information about the nature of the injury.
  - As much of the exposure history as was feasible to determine in the time allowed.
  - Name and phone number of the medical facility to which the victim(s) has/have been taken.
  - Name(s) of the involved Tetra Tech NUS, Inc. employee(s).
  - Name and phone number of an informed site officer who will be responsible for further investigations.
  - Fax appropriate information to WorkCare at (714) 456-2154.
- Contact Corporate Health and Safety Department (Matt Soltis) and Human Resources Department (Marilyn Duffy) at (412) 921-7090.

As data is gathered and the scenario becomes more clearly defined, this information should be forwarded to WorkCare. WorkCare will compile the results of data and provide a summary report of the incident. A copy of this report will be placed in each victim's medical file in addition to being distributed to appropriately designated company officials.

Each involved worker will receive a letter describing the incident but deleting any personal or individual comments. A personalized letter describing the individual findings/results will accompany this generalized summary. A copy of the personal letter will be filed in the continuing medical file maintained by WorkCare.

FIGURE 2-2 (continued)
WORKCARE
POTENTIAL EXPOSURE REPORT

Name: _____ Date of Exposure: _____

Social Security No.: _____ Age: _____ Sex: _____

Client Contact: _____ Phone No.: _____

Company Name: _____

I. Exposing Agent

Name of Product or Chemicals (if known): _____

_____

Characteristics (if the name is not known)

Solid Liquid Gas Fume Mist Vapor

II. Dose Determinants

What was individual doing? _____

How long did individual work in area before signs/symptoms developed? _____

Was protective gear being used? If yes, what was the PPE? _____

Was their skin contact? _____

Was the exposing agent inhaled? _____

Were other persons exposed? If yes, did they experience symptoms? _____

III. Signs and Symptoms (check off appropriate symptoms)

Immediately With Exposure:

Burning of eyes, nose, or throat
Tearing
Headache
Cough
Shortness of Breath
Chest Tightness / Pressure
Nausea / Vomiting
Dizziness
Weakness

Delayed Symptoms:

Weakness
Nausea / Vomiting
Shortness of Breath
Cough
Loss of Appetite
Abdominal Pain
Headache
Numbness / Tingling

IV. Present Status of Symptoms (check off appropriate symptoms)

Burning of eyes, nose, or throat
Tearing
Headache
Cough
Shortness of Breath
Chest Tightness / Pressure
Cyanosis
Nausea / Vomiting
Dizziness
Weakness
Loss of Appetite
Abdominal Pain
Numbness / Tingling

Have symptoms: (please check off appropriate response and give duration of symptoms)

Improved: _____ Worsened: _____ Remained Unchanged: _____

V. Treatment of Symptoms (check off appropriate response)

None: _____ Self-Medicated: _____ Physician Treated: _____

3.0 SITE BACKGROUND

3.1 FACILITY HISTORY

The NSF-IH is located in northwestern Charles County, Maryland, approximately 25 miles southwest of Washington, DC. The NSF-IH is a military facility consisting of the main area on the Cornwallis Neck Peninsula and the Annex on Stump Neck. The main area is bounded by the Potomac River to the northwest, west, and south, Mattawoman Creek to the south and east, and the town of Indian Head to the northeast. Stump Neck Annex is located across Mattawoman Creek. The Stump Neck Annex is not contiguous with the main area. The primary mission of NSF-IH is to provide services in energetics, ordnance devices and components, and other related ordnance engineering standards, including chemicals, propellants, and their propulsion systems, explosives, pyrotechnics, warheads, and simulators.

3.2 SITE 43 - TOLUENE SPILL

Site 43 includes two separate areas in the southwestern portion of the Main Area. The first is located near a utility pole approximately 30 feet northwest of and across the road from Building 1041, and the second is the northern corner of Building 1040. It is estimated that, for a period of more than 2 years during parts-cleaning operations, unknown quantities of spent solvents were improperly disposed in the drainage ditch outside the door (northeast) of Building 1040 (acetone) and at the base of the pole by Building 1041 (acetone and toluene).

The majority of this current work lies within the Building 720 explosive arc (when site operations are active). Therefore, work can only occur on weekends when site operations are off-line.

4.0 SCOPE OF WORK

This section of the HASP addresses proposed site activities that are to be conducted while performing the soil boring, sampling and associated tasks. Specific tasks to be performed at Site 43:

- Mobilization/demobilization
- Soil Boring using Direct Push Technology (DPT) and Hollow Stem Auger (HSA)
  - Monitoring well installation and development
  - Membrane Interface Probe (MIP)
- Multi-media sampling
  - Soil
  - Groundwater
  - Soil gas with SUMMA Canisters
- Decontamination of sampling equipment and DPT Rig
- Investigation-Derived Waste (IDW) Handling
- Geographical land survey

No other activities are anticipated to be necessary. If it becomes apparent that additional or modified tasks must be performed beyond those listed above, the work is not to proceed until the FOL or SSO notifies the PM and the HSM, so that any appropriate modifications to this HASP can first be developed and communicated to the intended task participants.

5.0 TASKS/HAZARDS/ASSOCIATED CONTROL MEASURES SUMMARIZATION

The purpose of this section is to identify the anticipated hazards and appropriate hazard prevention/hazard control measures that are to be observed for each planned task or operation. These topics have been summarized for each planned task through the use of task-specific Safe Work Permits (SWPs), which are to be reviewed in the field by the SSO with all task participants prior to initiating any task. Additionally, potential hazard and hazard control matters that are relevant but are not necessarily task-specific are addressed it the following portions of this section.

Section 6.0 presents additional information on hazard anticipation, recognition, and control relevant to the planned field activities.

5.1 GENERAL SAFE WORK PRACTICES

In addition to the task-specific work practices identified on Table 5-1, follow these safe work practices when conducting work involving known and unknown site hazards. These safe work practices establish a pattern of general precautions and measures for reducing risks associated with hazardous site operations.

- Eating, drinking, chewing gum or tobacco, or taking medication, is permitted in the support zone only.

- Smoking is prohibited within the NSF-IH restricted area

- Wash hands and face thoroughly upon leaving a contaminated or suspected contaminated area. A thorough shower and washing must be conducted as soon as possible if excessive skin contamination occurs.

- Avoid contact with potentially contaminated substances by walking around puddles, pools, mud, or other such areas. Avoid, whenever possible, kneeling on the ground or leaning or sitting on equipment. Do not place monitoring equipment on potentially contaminated surfaces.

- Avoid contact with unknown or unidentified objects. Remember NSF-IH was and is a training facility for ordnance. UXO concerns can still be an issue at this site. Pin Flag and seek assistance in the identification of any articles of this nature that may impact the established work area. This situation is highly unlikely.
- Be familiar with and adhere to the instructions in the site-specific HASP.

- Be aware of the location of the nearest telephone and emergency telephone numbers.

- Only approved communication devices (including cell phones) are permitted within the NSF-IH restricted area.

- Attend briefings on anticipated hazards, equipment requirements, Safe Work Permits, emergency procedures, and communication methods before going on site.

- Rehearse unfamiliar operations prior to implementation.

- Maintain visual contact with each other and with other on-site team members by remaining in close proximity in order to assist each other in case of emergency.

- Establish appropriate Safety Zones including Support, Contamination Reduction, and Exclusion Zones.

- Minimize the number of personnel and equipment in contaminated areas (such as the Exclusion Zone). Non-essential vehicles and equipment should remain within the Support Zone.

- Establish appropriate decontamination procedures for leaving the site.

- Immediately report injuries, illnesses, and unsafe conditions, practices, and equipment to the Site Safety Officer (SSO).

- Observe coworkers for signs of toxic exposure and heat or cold stress.

- Inform co-workers of potential symptoms of illness, such as headaches, dizziness, nausea, or blurred vision.

5.2 SOIL BORING SAFE WORK PRACTICES

The following Safe Work Practices are to be followed when performing intrusive operations and when working in or around DPT Operations. Prior to intrusive investigation techniques check the SOP for Utility Locating and Excavation Clearance found in Attachment III of this HASP.

- Identify underground utilities and buried structures before performing intrusive activities

- A Competent Person (the SSO or designee) will inspect rigs, prior to the acceptance of the equipment at the site and prior to the use of the equipment.

- Repairs or deficiencies identified will be corrected prior to use.

- Document the inspection by using the Equipment Inspection Checklist provided in Attachment IV.

- Inspections will be conducted once every shift or following any repairs.

- Remove trip hazards around the point of operation hazards.

- The drill/DPT operator helper will establish an equipment staging and lay-down plan in order to keep the work area clear of clutter and slips, trips, and fall hazards.

- Potentially contaminated tool will be wrapped in polyethylene sheeting for storage and transport to the centrally located decontamination unit.

- Prior to the start of boring operations, one individual will be designated as the person responsible for immediate activation of the emergency stop device in the event of an emergency.

- This individual will be identified to the field crew and will be responsible for visually checking the work area and verbally alerting personnel in the vicinity of boring operations prior to engaging the equipment.

- Secure frayed or loose clothing, hair, and jewelry when working with drill/DPT equipment.

- Minimize contact to the extent possible with contaminated tools and environmental media.

- Support functions (sampling and screening stations) will be maintained a minimum distance from the rig of the height of the mast plus five feet to remove these activities from within physical hazard boundaries.

- Only qualified operators and knowledgeable ground crew personnel will participate in the operation of the drill/DPT rig.

- Minimize contact with potentially contaminated tools and media and to minimize lifting hazards, multiple personnel should move auger flights and other heavy tooling.

- Only personnel absolutely essential to the work activity will be allowed in the exclusion zone. Site visitors will be escorted.

- Equipment used within the exclusion zone will undergo a complete decontamination and evaluation by the SSO to determined cleanliness prior to moving to the next location, exiting the site, or prior to down time for maintenance.

- Motorized equipment will be fueled prior to the commencement of the day's activities. During fueling operations the equipment will be shutdown and bonded to the fuel provider.

- When not in use rigs will be shutdown, emergency brakes set, and wheels chocked.

- Areas will be restored to equal or better condition than original to remove any contamination brought to the surface and to remove any physical hazards.

6.0 HAZARD ASSESSMENT

This section provides information regarding the chemical and physical hazards associated with the proposed work sites including Site 43 of the NSF-IH and the activities that are to be conducted as part of the scope of work. Table 6-1 provides various information related to the chemical contaminants that may be present at the site.

6.1 CHEMICAL HAZARDS

Based on an evaluation of previous site data collected, in combination with historical information about the site, the primary contaminants of concern (COCs) at this site are VOCs, and from a worst-case scenario, could potentially reach concentrations that exceed the ACGIH TLV TWA_8 and the OSHA PEL TWA_8 and Ceiling. Although this is a possibility, it is very unlikely that they will approach airborne concentrations reaching current occupational exposure limits (OEL). Table 6-1 also shows these COCs and a comparison of potential worst case air concentrations with current OELs.

TABLE 6-1
COMPARISON OF VOLATILE ORGANIC CONCENTRATIONS
WITH CURRENT OCCUPATIONAL EXPOSURE LIMITS

VOCs			
Contaminant of Concern	Highest Concentration Previously Detected ug/L in water	Worst-Case Air Concentration That Could Be Encountered	Current OSHA PEL and ACGIH TLV
Bromoform	960	2.03 ppm	OSHA: 0.5 ppm, TWA_8 ACGIH TLV: 0.5 ppm
TCE	55,000	4,123 ppm	OSHA: 100 ppm, TWA_8 Ceiling: 200 ppm ACGIH TLV: 50 ppm

Table Notes:
TWA_8: Average air concentration over an 8-hour work period that is not to be exceeded
OSHA Ceiling: Maximum concentration in air that is not to be exceeded

As indicated in this table, from a worst-case scenario, TCE concentrations immediately above a captured air phase above contaminated soil (such as in the head space of a sample jar) could reach concentrations that exceed the ACGIH TLV TWA_8 and the OSHA PEL TWA_8 and Ceilings and STELs. In regarding the results of this data evaluation, it is important to recognize the following:

- the planned work area is outdoors, with ample natural ventilation that will reduce any airborne TCE through dilution and dispersion,

- the groundwater value used in this evaluation was the ***highest*** concentration previously detected (the Navy has since reduced COC concentrations in groundwater though source area treatments).

As a result of these factors, it is very unlikely that workers participating in this activity will encounter any airborne concentrations of these COCs that would represent an occupational exposure concern. To monitor this route, real-time direct reading monitoring instruments will be used (as described in Section 7.0).

6.1.1 Properties of COCs (VOCs)

Various VOCs have been previously detected at low concentrations in the initial screening samples collected during site investigatory activities. A majority VOCs are related to chlorinated solvents and associated degradation products, paint thinners, dry cleaning solvents, constituents of petroleum fuels (e.g., gasoline and natural gas), and crude oil tanking. Symptoms of exposure to 1,1,1-TCA, TCE, and vinyl chloride can include abdominal pain, irritiation of the skin, eyes, nose, and throat, dizziness, tremors, vomiting, gastro-intestinal (GI) bleeding, enlarged liver, pallor of the extremities, and frostbite like-symptoms.

Inhalation, Ingestion, and Skin Contact: Since VOCs can be present in the air, the greatest potential and most efficient route of exposure is through inhalation of airborne vapors. For this reason, monitoring equipment will be used to monitor work area concentrations, and personal protective equipment (PPE) and basic hygiene practices (washing face and hands before leaving the site) are required and will be extremely important. Exposure to VOCs through ingestion and direct skin contact is possible, however the likelihood of worker exposure through these routes is considered unlikely, provided that workers follow good personal hygiene and standard good sample collection/sample handling practices, and wear appropriate PPE as specified in this HASP.

6.1.2 On-Site Practices

The likelihood of worker exposure to the above COCs are considered very unlikely, provided that workers follow good personal hygiene and standard good sample collection/sample handling practices, and wear appropriate PPE as specified in this HASP. Examples of onsite practices that are to be observed that will protect workers from exposure via inhalation, ingestion or skin contact include the following:

- Avoid hand-to-mouth activities on site (eating, drinking, smoking, etc.)
- Washing hands upon leaving the work area and prior to performing any hand to mouth activities
- Wearing surgeon's-style gloves whenever handling potentially-contaminated media, including soils, hand tools, and sample containers.
- Proper respirator use in the event of visible dust during activities
- Proper use of monitoring instruments that will be used to detect the presence of VOCs.

6.2 PHYSICAL HAZARDS

In addition to the chemical hazards discussed above, the following physical hazards may be present during the performance of site activities.

- Contact with UXO
- Slips, trips, and falls.
- Heavy equipment hazards.
- Noise in excess of 85 decibels (dBA).
- Inclement weather.
- Natural hazards (contact with poisonous plants and disease carrying animals and insects).

These physical hazards and their applicability to each site task are discussed in detail in Table 5-1. Additionally, each of these physical hazards is discussed in detail in the TtNUS Health and Safety Manual.

6.2.1 Contact with UXO

Field personnel will practice UXO avoidance techniques at all times. However, given the mission and history of the sites, UXO may be encountered during operations. If UXO is encountered or there is any uncertainty about the presence of UXO, NSF-IH EOD personnel will be contacted to provide additional support during site activities.

6.2.2 Slip, Trip and Fall Hazards

Various potential slip, trip, and fall hazards may be encountered during the performance of planned site activities. These hazards are associated with working out doors where uneven or wet terrain may be encountered, or near the edge of bodies of water, as well as on boat decks and docks. To minimize the potential for worker injury from these hazards, the following requirements must be observed:

- Maintain proper housekeeping in work areas.

- Preview and inspect work areas to identify and eliminate slip, trip, or fall hazards. In outdoor locations, pay particular attention to sink holes or other depressions that may be encountered.

- Any work that is to be done on structures that are more than 6-feet above floor or ground level will require fall protection training and the use of 100% fall protection equipment.

- Cover, guard, barricade, and/or place warning postings over/at holes or openings that personnel may fall or step into.

- For traversing steep, slippery, or sloped terrain establish rope ladders to control ascent and descent to sampling areas or use alternative pathways.

- Maintain 5 foot distance from excavation.

6.2.3 Strains/Muscle Pulls

To prevent injuries due to improper lifting and carrying methods estimate the weight and configuration of the load (i.e., is it bulky or hard to safely grasp/lift/control). If it appears to be too heavy or bulky to safely handle alone, either use a mechanical lifting device or obtain help from another employee to lift the load. The use of mechanical lifting devises is always preferable over manual lifting. Use the following practices:

- Bend at the knees (not at the waist) when attempting a lift.
- Get a firm hold is obtained, and keep the load as close to the body as possible.
- Lift the load using your legs, and not the back.
- Avoid turning or twisting while holding a load.
- Preview the path of travel to identify and eliminate tripping hazards.
- Do not carry loads that obstruct the line of sight.

- When setting a load down use the leg muscles and do not bend at the waist.
- Divide heavier loads into smaller amounts.

6.3 NATURAL HAZARDS

Insect/animal bites and stings, poisonous plants, and inclement weather are natural hazards that may be present given the location of activities to be conducted. In general, avoidance of areas of known infestation or growth will be the preferred exposure control for insects/animals and poisonous plants. Specific discussion on principle hazards of concern follows:

6.3.1 Insect Bites and Stings

Insect bites and stings are difficult to control given the climate and environmental setting of NSF-IH. However, in an effort to minimize this hazard the following control measures will be implemented where possible.

- Commercially available bug sprays and repellents will be used whenever possible – Pesticides analytical screening includes chlordane, endrin, lindane, methoxychlor, toxaphene and heptachlor. Commercially available repellants may be used. Products such as DEET should not be applied directly to the skin due to potential irritation. This product, when permitted for use, should be applied over clothing articles.

- Where possible, loose-fitting and light-colored clothing with long sleeves should be worn. This will also aid in insect control by providing a barrier between the field person and the insects and to provide easy recognition of crawling insects against the lighter background. Pant legs should be secured to the work-boots using duct tape to prevent access by ticks. Mosquito nets are also recommended for use when commercially available repellents are not permitted.

- Clothing/limited body checks for ticks and other crawling insects should be conducted upon exiting heavily vegetated areas. Workers should perform a more detailed check of themselves when showering in the evening. Ticks prefer moist areas of the body (arm-pits, genitals, etc.) and will migrate to those locations.

- The FOL/SSO will preview access routes and work areas in an effort to identify physical hazards including nesting areas in and around the work sites. These areas will be flagged and communicated to site personnel.

- The FOL/SSO must determine if site personnel (through completion of Medical Data Sheets), suffer allergic reactions to bee and other insect stings and bites. Field crew members who are allergic to bites should have their emergency kit containing antihistamine and a preloaded syringe of epinephrine readily available.

Any allergies (insect bites, bee stings, etc.) must be reported on the Medical Data Sheet and to the SSO.

Tick-Borne Disease

During warm months (spring through early fall), tick-borne Lyme disease may pose a potential health hazard in Maryland which is listed as an area for Lyme disease. The longer a disease carrying tick remains attached to the body, the greater the potential for contracting the disease. Wearing long sleeved shirts and long pants (tucked into boots). Performing frequent body checks will prevent the tick from attaching for the long term. Site first aid kits should be equipped with medical forceps and rubbing alcohol to assist in tick removal. For information regarding tick removal procedures, and symptoms of exposure consult the HSGM.

Mosquito-Borne Illness

The Maryland Department of Health is urging people to take precautions to avoid potentially dangerous mosquitoes. Mosquitoes in Maryland may carry diseases including St. Louis encephalitis, Eastern Equine encephalitis, La Crosse encephalitis and West Nile virus.

The Maryland Department of Health, along with a variety of agencies, routinely conducts testing in mosquitoes and birds to monitor for possible mosquito-borne viruses. Mosquitoes become infected after biting infected birds. The symptoms for mosquito-borne illnesses may include headache, moderate to high fever, stiff neck and confusion. In serious cases coma, seizures or paralysis can result. Symptoms usually appear between 5 to 15 days after exposure to infected mosquitoes. Mosquito-borne illnesses may be mild or serious and can lead to death.

Precautions include:

- Limit outdoor activities during peak mosquito times – at dusk and dawn.
- Avoid standing water
- Wear long-sleeved shirts and long pants whenever you are outdoors.

- Apply insect repellent according to manufacturers instruction to exposed skin.
- Spray clothing with repellents containing permethrin or DEET
- Mosquitoes may bite through thin clothing.
- An effective repellent will contain 20% to 30% DEET (N,N-diethyl-meta-toluamide).
- Avoid products containing more than 30% DEET.

6.3.2 Snakes and Other Wild Animals

Indigenous animals including snakes (only two poisonous and approximately 27 non-poisonous varieties in Maryland), raccoons, and other animals native to the region may be present at the site. These animals may be encountered if work locations encroach on nesting or territories claimed by these animals.

There are two poisonous snakes indigenous to the State of Maryland, the Northern Copperhead and the Timber Rattlesnake. Only the Northern Copperhead is indigenous to this part of the state. It is important to remember that snakes are rarely aggressive towards humans. If you encounter a snake simply maintain a safe distance and move away from it, or allow it to move away.

See Section 4.0 of the HSGM for additional information (responding to a snake bite).

6.3.3 Poisonous Plants

Various plants which can cause allergic reactions may be encountered during field work. These include, poison ivy, poison oak, and poison sumac. Contact with these plants may occur when clearing vegetation for access to work areas, or as a result of movement through these plants. An irritating, allergic reaction can occur after direct contact with the plant or indirect contact through some piece of equipment or clothing article. Oils are transferred from the plant to exposed skin, clothing, or piece of equipment. The degree of the irritating, allergic reaction can vary significantly from one person to the next.

Protective measures to control and minimize the effects of this hazard may include, but not be limited to, the following:

- Identify plants for field personnel.
  - Poison Ivy - Characterized by climbing vines, three leaf configuration ovate to elliptical in shape, deep green leaves with a reddish tint, greenish flowers, and white berries.

- Poison Sumac - Characterized as a tall bush of the sumac family bearing compound leaves (7-13 entire leaflets), branched from a central axis, drooping, with auxiliary clusters of white fruit: However, these white fruits and berries may exist only during pubescent stages.

- Poison oak - Characterized as similar to poison ivy consisting of a shrub, stems erect, 0.3 to 2.0 meters tall, leaflets consist of broad thick lobes coarsely serrated configuration, denser at the base, less so than the top.

- Protective measures may include wearing disposable garments such as Tyvek when clearing brush. These may be carefully removed and disposed of along with any oils accumulated from the plants.

- Personal Hygiene - The oils obtained from the plants will only elicit an allergic response when the person's bare skin layer is contacted. This can be aggravated when skin pores are open (perspiring), or through breaks in the skin such as cuts, nicks, scratches, etc. This can also be accomplished when using excessively hot water for cleaning the skin, which also causes pores to open. Prior to break time, lunchtime, etc. personnel should wash with cool water and soap to remove as much of the oils as possible. In heavily vegetated areas of these plants, additional measures including barrier creams and blocks may be used to prevent the oils from accessing and penetrating the skin.

These plants present an airborne sensitization hazard when burned. This is not to occur as part of this scope of work and therefore will not be addressed.

6.3.4 Inclement Weather

Project tasks under this Scope of Work will be performed outdoors and near water. As a result, inclement weather may be encountered. In the event that adverse weather conditions arise (electrical storms, hurricanes, etc.), the FOL and/or the SSO will be responsible for temporarily suspending or terminating activities until hazardous conditions no longer exist.

6.3.5 Vehicular and Equipment Traffic

Hazards associated with vehicular and equipment traffic are sometimes unavoidable and exist during various site activities and whenever site personnel performed work on or near roadways. To minimize the potential for injuries associated with these hazards, traffic control measures will be instituted to protect site workers and the public.

7.0 AIR MONITORING

The primary COC has the potential to be present in concentrations that could present an inhalation hazard during planned site activities. To assure that such exposures are avoided and documented, a direct reading instrument will be used to monitor worker exposures to chemical hazards present at the site. A Photoionization Detector (PID) using a lamp energy of 10.6 eV will be used to monitor the air when working around open monitoring wells.

7.1 INSTRUMENTS AND USE

Instruments will be used primarily to monitor source points and worker breathing zone areas, while observing instrument action levels. The SSO shall obtain and document the daily background (BG) reading at an upwind, unaffected area and observe for readings above that BG level. The SSO shall monitor source areas (e.g., bore holes, environmental samples) for the presence of any reading above the daily-established BG level. If elevated readings are observed, the SSO shall monitor the workers breathing zone (BZ) areas with the FID. If the appropriate instrument Action Level is exceeded (see below), the following process will be followed:

- The SSO shall stop work and move site personnel upwind to a safe, unaffected area, where they will remain until further directed by the SSO.

- The SSO shall allow at least 5 minutes to pass so that the work area can ventilate, and will then re-approach the work area while continuously monitoring the BZ areas.

- Only when BG levels are regained in BZ areas will work be permitted to resume.

- If BG levels are not regained, the SSO will contact the HSM for additional direction.

Instrument Action Levels: The use of a FID will be acceptable, provided that the following action levels are observed.

- The maximum a worker can be exposed to is **30 ppm for 4 excursions of 5 minutes** in any one work day on a PID.

7.2 INSTRUMENT MAINTENANCE AND CALIBRATION

Hazard monitoring instruments will be maintained and pre-field calibrated by the TtNUS Equipment Manager and/or rental service employed. Operational checks and field calibration will be performed on the instruments each day prior to their use. Field calibration will be performed on instruments according to manufacturer's recommendations (for example, the FID must be field calibrated daily and an additional field calibration must be performed at the end of each day to determine any significant instrument drift). These operational checks and calibration efforts will be performed in a manner that complies with the employees health and safety training, the manufacturer's recommendations, and with the applicable manufacturer standard operating procedure. All calibration efforts must be documented. Figure 7-1 is provided for documenting these calibration activities. This information may instead be recorded in a field operations logbook, provided that the information specified in Figure 7-1 is recorded. This required information includes the following:

- Date calibration was performed
- Individual calibrating the instrument
- Instrument name, model, and serial number
- Any relevant instrument settings and resultant readings (before and after) calibration
- Identification of the calibration standard (lot no., source concentration, supplier)
- Any relevant comments or remarks

7.3 DOCUMENTING INSTRUMENT READINGS

The SSO is responsible for ensuring that air monitoring instruments are used in accordance with the specifications of this HASP and with manufacturer's specifications/recommendations. In addition, the SSO is also responsible for ensuring that the instrument use is documented. This requirement can be satisfied either by recording instrument readings on pre-printed sampling log sheets or in a field log book. **This includes the requirement for documenting instrument readings that indicate no elevated readings above noted daily background levels (i.e., no-exposure readings)**. At a minimum, the SSO must document the following information for each use of an air monitoring device:

- Date, time, and duration of the reading
- Site location where the reading was obtained
- Instrument used (e.g., FID, PID.)
- Personnel present at the area where the reading was noted
- Other conditions that are considered relevant to the SSO (such as weather conditions, possible instrument interferences, etc.)

FIGURE 7-1

DOCUMENTATION OF FIELD CALIBRATION

SITE NAME: _____ **PROJECT NO.:** _____

Date of Calibration	Instrument Name and Model	Instrument I.D. Number	Person Performing Calibration	Instrument Settings		Instrument Readings		Calibration Standard (Lot Number)	Remarks/ Comments
				Pre-Calibration	Post-Calibration	Pre-Calibration	Post-Calibration		

8.0 TRAINING/MEDICAL SURVEILLANCE REQUIREMENTS

8.1 INTRODUCTORY/REFRESHER/SUPERVISORY TRAINING

This section is included to specify health and safety training and medical surveillance requirements for both TtNUS and subcontractor personnel participating in site activities.

8.1.1 Requirements for TtNUS and Subcontractor Personnel

TtNUS and subcontractor personnel must complete the following training requirements:

- 40 hours of introductory hazardous waste site training prior to performing work at the NSF-IH facility.
- 8-Hour General Site Work Refresher Training for personnel who have had introductory training more than 12 months prior to site work.
- 8-hour General Site Worker Supervisory training in accordance with 29 CFR 1910.120 (e)(4) will be required for site supervisory personnel who will serve in a supervisory capacity.

Documentation of TtNUS introductory, supervisory, and refresher training as well as site-specific training will be maintained at the project. Copies of certificates or other official documentation will be used to fulfill this requirement.

8.2 SITE-SPECIFIC TRAINING

TtNUS will provide site-specific training to TtNUS employees and subcontractor personnel who will perform work on this project. Site-specific training will also be provided to other personnel (U.S. Department of Defense, EPA, etc.) who may enter the site to perform functions that may or may not be directly related to site operations. Site-Specific training will include:

- Names of designated personnel and alternates responsible for site safety and health
- Safety, health, and other hazards present on site
- Use of personal protective equipment
- Safe use of engineering controls and equipment
- Medical surveillance requirements
- Signs and symptoms of overexposure

- Contents of the Health and Safety Plan
- Emergency response procedures (evacuation and assembly points)
- Incipient response procedures
- Review of the contents of relevant Material Safety Data Sheets
- Review of the use of Safe Work Permits

Site-specific documentation will be established through the use of Figure 8-1. Site personnel and visitors must sign this document upon receiving site-specific training.

8.3 MEDICAL SURVEILLANCE

8.3.1 Medical Surveillance Requirements for TtNUS Personnel

TtNUS and subcontractor personnel participating in project field activities will have had a physical examination meeting the requirements of 29CFR 1910.120(f) and will be cleared by a licensed occupational physician to perform hazardous waste site work using respiratory protection.

Documentation for medical clearances will be maintained at the project location and be made available, as necessary.

8.3.2 Other Requirements for Field Personnel

Each field team member (including subcontractors) and visitors entering the Exclusion Zone(s) shall be required to complete and submit a copy of Medical Data Sheet found in the TtNUS HSGM and supplied as Attachment II. This shall be provided to the SSO, prior to participating in site activities. The purpose of this document is to provide site personnel and emergency responders with additional information that may be necessary in order to administer medical attention.

8.4 SUBCONTRACTOR EXCEPTIONS

Subcontractors who will not enter the Exclusion Zone during intrusive operations, and whose activities involve no potential for exposure to site contaminants, will not be required to meet the requirements for training/medical surveillance other than those stated for site-specific training (See Section 8.2).

FIGURE 8-1

SITE-SPECIFIC TRAINING DOCUMENTATION

My signature below indicates that I am aware of the potential hazardous nature of performing remedial investigation activities at Naval Support Facility, Indian Head and that I have received site-specific training which included the elements presented below:

- Names of designated personnel and alternates responsible for site safety and health
- Safety, health, and other hazards present on site
- Use of personal protective equipment
- Safe use of engineering controls and equipment
- Medical surveillance requirements
- Signs and symptoms of overexposure
- Emergency response procedures (evacuation and assembly points)
- Incipient response procedures
- Review of the contents of relevant Material Safety Data Sheets
- Review of the use of Safe Work Permits
- Stop Work Authorization and Procedure

I have been given the opportunity to ask questions and my questions have been answered to my satisfaction. The dates of my training and medical surveillance indicated below are accurate.

Name (Printed and Signature)	Site-Specific Training Date	40-Hour Training (Date)	8-Hour Refresher Training (Date)	8-Hour Supervisory Training (Date)	Medical Exam

9.0 SITE CONTROL

This section outlines the means by which TtNUS will delineate work zones and use these work zones in conjunction with decontamination procedures to prevent the spread of contaminants into previously unaffected areas of the site. It is anticipated that a three-zone approach will be used during work at this site, including an Exclusion Zone, a Contamination Reduction Zone, and a Support Zone. It is also anticipated that this control measure will be used to control access to site work areas. Use of such controls will restrict the general public, minimize potentials for the spread of contaminants and to protect individuals who are not cleared to enter the work areas.

9.1 EXCLUSION ZONE

The exclusion zone will be considered those areas of active operations plus an established safety zone, depending on the task. The following represent the exclusion zone boundaries for the identified tasks:

- Soil Borings using HSA or DPT/MIPs and monitoring well installation – The boundary perimeter will be established by determining the height of the mast, plus 5 feet or 25-foot whichever is more conservative.

- Groundwater Sampling – 10 feet surrounding the well head.

- Soil Gas Sampling - 10 feet surrounding the SUMMA Canister

- Decontamination (heavy equipment – steam/pressure washers) – 35 feet surrounding the point of operation. This operation will take place at a centralized location.

Where appropriate and necessary to direct facility personnel, this area will be delineated using barrier tape, cones and/or drive poles, and postings.

9.2 CONTAMINATION REDUCTION ZONE

The Contamination Reduction Zone (CRZ) will be a buffer area between the Exclusion Zone and any area of the site where contamination is not suspected. This area will also serve as a focal point in supporting Exclusion Zone activities. This area will be delineated using barrier tape, cones, and postings to inform and direct facility personnel. Decontamination will be conducted at a central location. Equipment potentially contaminated will be bagged and taken to that location for decontamination.

9.3 SUPPORT ZONE

The Support Zone for this project will include a staging area where site vehicles will be parked, equipment will be unloaded, and where food and drink containers will be maintained. The Support Zones will be established at areas of the site where exposure to site contaminants would not be expected during normal working conditions or foreseeable emergencies.

9.4 SAFE WORK PERMITS

Exclusion Zone work conducted in support of this project will be performed using Safe Work Permits to guide and direct field crews on a task by task basis. An example of the Safe Work Permit to be used is illustrated in Figure 9-1. Partially completed Permits for the work to be performed are included in Attachment V. The daily meetings conducted at the site will further support these work permits. This effort will ensure site-specific considerations and changing conditions are incorporated into the planning effort. Permits will require the signature of the FOL and SSHO. Use of these permits will provide the communication line for reviewing protective measures and hazards associated with each operation. This HASP will be used as the primary reference for selecting levels of protection and control measures. The work permit will take precedence over the HASP when more conservative measures are required based on specific site conditions.

9.5 SITE VISITORS

Site visitors for the purpose of this document are identified as representing the following groups of individuals:

- Personnel invited to observe or participate in operations by TtNUS
- Regulatory personnel (DOD, OSHA, MDE, EPA, etc.)
- NAVFAC Atlantic and NAVFAC Washington Navy Personnel
- Other authorized visitors

It is not anticipated that this operation will result in a large number of site visitors. However, as some visitors can reasonably be expected, the following requirements will be enforced:

- Site visitors will be routed to the FOL, who will sign them in to the field logbook. Information to be recorded in the logbook will include the individual's name (proper identification required), who they represent, and purpose for the visit.

FIGURE 9-1
SAFE WORK PERMIT

Permit No. _____ Date: _____ Time: From _____ to _____

I. Work limited to the following (description, area, equipment used): _____

II. Primary Hazards: Potential hazards associated with this task include _____

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____TtNUS
Equipment Inspection required ☐ Yes ☐ No Initials of Inspector _____TtNUS

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐

Respiratory equipment required
Yes ☐ Specify on the reverse
No ☒

Modifications/Exceptions: _____

VI. Chemicals of Concern	Hazard Monitoring	Action Level(s)	Response Measures

Primary Route(s) of Exposure/Hazard: _____

(Note to FOL and/or SHSO: Each item in Sections VII, VIII, and IX must be checked Yes, No, or NA)

VII. Additional Safety Equipment/Procedures

Hard-hat	☐ Yes	☐ No	Hearing Protection (Plugs/Muffs)	☐ Yes	☐ No
Safety Glasses	☐ Yes	☐ No	Safety belt/harness	☐ Yes	☐ No
Chemical/splash goggles	☐ Yes	☐ No	Radio/Cellular Phone	☐ Yes	☐ No
Splash Shield	☐ Yes	☐ No	Barricades	☐ Yes	☐ No
Splash suits/coveralls	☐ Yes	☐ No	Gloves (Type –)	☐ Yes	☐ No
Impermeable apron	☐ Yes	☐ No	Work/rest regimen	☐ Yes	☐ No
Steel toe Work shoes or boots	☐ Yes	☐ No	Chemical Resistant Boot Covers	☐ Yes	☐ No
High Visibility vest	☐ Yes	☐ No	Tape up/use insect repellent	☐ Yes	☐ No
First Aid Kit	☐ Yes	☐ No	Fire Extinguisher	☐ Yes	☐ No
Safety Shower/Eyewash	☐ Yes	☐ No	Other	☐ Yes	☐ No

Modifications/Exceptions: _____

VIII. Site Preparation

	Yes	No	NA
Utility Locating and Excavation Clearance completed	☐	☐	☐
Vehicle and Foot Traffic Routes Established/Traffic Control Barricades/Signs in Place	☐	☐	☐
Physical Hazards Identified and Isolated (Splash and containment barriers)	☐	☐	☐
Emergency Equipment Staged (Spill control, fire extinguishers, first aid kits, etc).	☐	☐	☐

IX. Additional Permits required (Hot work, confined space entry, excavation etc.). ☐ Yes ☐ No
If yes, SHSO to complete or contact Health Sciences, Pittsburgh Office (412) 921-7090

X. Special instructions, precautions: _____

Permit Issued by: _____ Permit Accepted by: _____

- Site visitors will be required to produce the necessary information supporting clearance onto the site. This includes information attesting to applicable training (40-hours of HAZWOPER training required for NAVFAC personnel) and medical surveillance, as stipulated in Section 8.0 of this document. In addition, to enter the site's operational zones during planned activities, visitors will be required to first go through site-specific training covering the topics stipulated in Section 8.2 of this document.

- Site visitors will be escorted while at the site.

Following this, the site visitor will be permitted to enter the site and applicable operational areas. Visitors are required to observe the protective equipment and site restrictions in effect at the area of their visit. Visitors not meeting the requirements as stipulated in this plan for site clearance will not be permitted to enter the site operational zones during planned activities. Any incidence of unauthorized site visitation will cause onsite activities to be terminated until that visitor can be removed. Removal of unauthorized visitors will be accomplished with support from the NSF-IH contact, if necessary.

9.6 SITE SECURITY

Site security will be accomplished using TtNUS field personnel. TtNUS will retain complete control over active operational areas. Exclusion Zone barriers, and any existing barriers at the site will be used to restrict the general public. The second line of security will take place at the work site referring interested parties to the FOL or designee. The FOL will serve as a focal point for non-project interested parties, and serve as the final line of security and the primary enforcement contact.

9.7 SITE MAP

Once the areas of contamination, access routes, topography, and dispersion routes are determined, a site map will be generated and adjusted as site conditions change. When possible, these maps will be posted to illustrate up-to-date collection of contaminants and adjustment of zones and access points.

9.8 BUDDY SYSTEM

Personnel engaged in on-site activities will practice the "buddy system" to ensure the safety of personnel involved in this operation.

9.9 MATERIAL SAFETY DATA SHEET (MSDS) REQUIREMENTS

TtNUS and subcontractor personnel will provide MSDSs for the chemicals brought on site. The contents of these documents will be reviewed by the SSO with the user(s) of the chemical substances prior to any

actual use or application of the substances on site. A chemical inventory of the chemicals used on site will be developed using the HSGM. The MSDSs will then be maintained in a central location (i.e., temporary office) and will be available for anyone to review upon request.

9.10 COMMUNICATION

As personnel will be working in proximity to one another during field activities, a supported means of communication between field crews members will not be necessary. External communication will be accomplished by using personal cell phones pre-determined and approved locations. External communication will primarily be used for the purpose of resource and emergency resource communications. Prior to the commencement of activities, the FOL will determine and arrange for telephone communications.

10.0 SPILL CONTAINMENT PROGRAM

10.1 SCOPE AND APPLICATION

It is not anticipated that bulk hazardous materials (over 55-gallons) will be handled at any given time. It is also not anticipated that such spillage of Investigative Derived Wastes (IDW) would constitute a danger to human health or the environment. However, as the job progresses, the potential may exist for accumulating (IDW) such as decontamination fluids and purge and well development waters, in a central staging area. Once these fluids and other materials have been characterized, they can be removed from this area and properly disposed.

10.2 POTENTIAL SPILL AREAS

The potential spill areas are:

- IDW Staging Area – These areas will be periodically monitored in an ongoing attempt to prevent and control further potential contamination of the environment.

- Drill/DPT Rig – These rigs are from time to time rupture hydraulic lines. In order to respond quickly spill pads or oil dry will be maintained at the DPT rig. If lines or connections are suspect plastic will be placed under the rig during operation to serve as a catch in the event of a release.

It is anticipated that the IDW generated as a result of this scope of work will be containerized, labeled, and staged to await further analyses. The results of these analyses will determine the method of disposal.

10.3 LEAK AND SPILL DETECTION

To establish an early detection of potential spills or leaks, a periodic walk-around by the personnel staging or disposing of drums or in the resource deployment area will be conducted during working hours to visually determine that storage vessels are not leaking. If a leak is detected, the contents will be transferred, using a hand pump, into a new vessel. The leak will be collected and contained using absorbents such as Oil pads, Oil-Dry, cat litter, or sand, which will be maintained at the storage location or within immediate access to the rig. Inspections will be documented in the project logbook.

10.4 PERSONNEL TRAINING AND SPILL PREVENTION

Personnel will be instructed in the procedures for incipient spill prevention, containment, and collection of hazardous materials in the site-specific training. The FOL and the SSO will serve as the Spill Response Coordinators for this operation, should the need arise.

10.5 SPILL PREVENTION AND CONTAINMENT EQUIPMENT

The following represents the minimum equipment that may be maintained (depending on anticipated need) at the staging areas for the purpose of supporting this Spill Prevention/Containment Program.

- Sand; clean fill, or other non-combustible absorbent (Oil-dry)
- Drums (55-gallon U.S. DOT 17-E or 17-H; UN1A2)
- Shovels, rakes, and brooms

10.5.1 PPE for Spill Control

Minimal PPE for spill control will be employed as needed. These materials may include:

- Nitrile work and inner gloves
- Tyvek coveralls
- Hard Hat
- Steel toed shoes with neoprene boot covers

10.6 SPILL CONTROL PLAN

This section describes the procedures the TtNUS field crewmembers will use upon the detection of a spill or leak.

- Notify the SSO or FOL immediately upon detection of a leak or spill. Activate emergency alerting procedures for that area to remove non-essential personnel.

- Employ the personal protective equipment stored at the staging area. Take immediate actions to stop the leak or spill by plugging or patching the container or raising the leak to the highest point in the vessel. Spread the absorbent material in the area of the spill, covering it completely.

- Transfer the material to a new vessel; collect and containerize the absorbent material. Label the new container appropriately. Await analyses for treatment and disposal options.

- Re-containerize spills, including top cover impacted by the spill. Await test results for treatment or disposal options.

It is not anticipated that a spill will occur that the field crew cannot handle. Should this occur, notification of the appropriate Emergency Response agencies will be carried out by the FOL or SSO in accordance with the procedures discussed in Section 2.0 of this HASP.

11.0 CONFINED-SPACE ENTRY

Personnel under the provisions of this HASP are not allowed, under any circumstances, to enter confined spaces.

A confined space is defined as a space that:

- Is large enough and so configured that an employee can bodily enter and perform assigned work.
- Has limited or restricted means for entry or exit (for example, tanks, manholes, sewers, vessels, silos, storage bins, hoppers, vaults, and pits are spaces that may have limited means of entry).
- Is not designed for continuous employee occupancy.

A Permit-Required Confined Space is defined as a confined space that has one or more of the following characteristics:

- Contains or has a potential to contain a hazardous atmosphere.
- Has an internal configuration such that an entrant could be trapped or asphyxiated by inwardly caving walls or by a floor that slopes downward and tapers to a smaller cross-section.
- Contains any other recognized, serious, safety or health hazard.

For further information on confined space operations, consult the HSGM or call the HSM. Any activity that may be considered a confined-space entry shall require modifications of this HASP and shall result in the immediate notification of the Project Health and Safety Officer. This determination shall be made by the FOL and SSHO.

12.0 MATERIALS AND DOCUMENTATION

The TtNUS FOL shall ensure the following materials/documents are taken to the project site and used when required.

- A complete copy of this HASP
- Health and Safety Guidance Manual
- Incident Reports
- Medical Data Sheets (multiple copies)
- Material Safety Data Sheets for the chemicals brought on site, including decon solutions, fuels, lime, sample preservatives, calibration gases, etc.
- A full-size OSHA Job Safety and Health Poster (See Attachment VI)
- Training/Medical Surveillance Documentation Form (Blank) (multiple copies)
- Emergency Reference Information (Section 2.0, extra copy for posting)

12.1 MATERIALS TO BE POSTED OR MAINTAINED AT THE SITE

The following documentation is to be posted or maintained at the site for quick reference purposes. In situations where posting of these documents is not feasible (such as no office trailer), these documents should be filed in a transportable file container and immediately accessible. The file should remain in the FOL's possession.

Chemical Inventory Listing (posted) - This list represents the chemicals brought on site, including decontamination solutions, sample preservatives, fuel, calibration gases, etc.. This list should be posted in a central area.

Material Safety Data Sheets (MSDSs) (maintained) - The MSDSs should also be in a central area accessible to site personnel. These documents should match the listings on the chemical inventory list for substances employed on site. It is acceptable to have these documents within a central folder and the chemical inventory as the table of contents.

The OSHA Job Safety & Health Protection Poster (posted) - This poster, as directed by 29 CFR 1903.2 (a)(1), should be conspicuously posted in places where notices to employees are normally posted. Each FOL shall ensure that this poster is not defaced, altered, or covered by other material. See Attachment VI of this HASP.

Site Clearance (maintained) - This is found within the training section of the HASP (See Figure 8-1). This list identifies site personnel, dates of training (including site-specific training), and medical surveillance and indicates not only clearance but also status. If personnel do not meet these requirements, they do not enter the site while site personnel are engaged in activities.

Emergency Phone Numbers and Directions to the Hospital(s) (maintained) - This list of emergency numbers and hospital directions will be maintained at phone communications points and in each site vehicle.

Medical Data Sheets/Cards (maintained) - Medical Data Sheets will be filled out by onsite personnel and filed in a central location. The Medical Data Sheet will accompany any injury or illness requiring medical attention to the medical facility. A copy of this sheet or a wallet card will be given to personnel to be carried on their person.

Investigative Derived Waste Inventory Log (maintained) – The FOL and/or the SSO shall log collected containers of IDW. An updated inventory will be submitted to the Base POC at the termination of each shift.

13.0 GLOSSARY

ACGIH	American Conference of Governmental Industrial Hygienists
CFR	Code of Federal Regulations
CIH	Certified Industrial Hygienist
CNS	Central Nervous System
CRZ	Contamination Reduction Zone
CSE	Confined Space Entry
CSP	Certified Safety Professional
CTO	Contract Task Order
dBA	Decibels average
DOD	Department of Defense
DOT	Department of Transportation
DPT	Direct Push Technology
EPA	Environmental Protection Agency
eV	electron Volts
FID	Flame Ionization Detector
FOL	Field Operations Leader
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HEPA	High Efficiency Particulate Air
HSA	Hollow Stem Auger
HSM	Health and Safety Manager
IDW	Investigative Derived Waste
LEL	Lower Explosive Limit
MDE	Maryland Department of the Environment
MSDS	Material Safety Data Sheet
N/A	Not Available
NSF-IH	Naval Support Facility, Indian Head
NIOSH	National Institute Occupational Safety and Health
OSHA	Occupational Safety and Health Administration (U.S. Department of Labor)
PE	Professional Engineer
PEL	Permissible Exposure Limit
PHSO	Project Health and Safety Officer
PID	Photo Ionization Detector
PM	Project Manager
PPE	Personal Protective Equipment

RAC	Remedial Action Contractor
SOP	Standard Operating Procedure
SSO	Site Safety Officer
STEL	Short Term Exposure Limit
TBD	To Be Determined
TtNUS	Tetra Tech NUS, Inc.
TWA	Time Weighted Average
UEL	Upper Explosive Limit
UST	Underground Storage Tank
UV	Ultraviolet

ATTACHMENT I

INCIDENT REPORT FORM

Report Date	Report Prepared By	Incident Report Number

INSTRUCTIONS:

All incidents (including those involving subcontractors under direct supervision of Tetra Tech personnel) must be documented on the IR Form.

Complete any additional parts to this form as indicated below for the type of incident selected.

TYPE OF INCIDENT (Check all that apply)	Additional Form(s) Required for this type of incident
Near Miss (No losses, but could have resulted in injury, illness, or damage)	☐ Complete IR Form Only
Injury or Illness	☐ Complete Form IR-A; Injury or Illness
Property or Equipment Damage, Fire, Spill or Release	☐ Complete Form IR-B; Damage, Fire, Spill or Release
Motor Vehicle	☐ Complete Form IR-C; Motor Vehicle

INFORMATION ABOUT THE INCIDENT

Description of Incident

_____

_____

_____

Date of Incident	Time of Incident
	_____ AM ☐ PM ☐ *OR* Cannot be determined ☐
Weather conditions at the time of the incident	**Was there adequate lighting?**
	_____ Yes ☐ No ☐

Location of Incident

_____ Was location of incident within the employer's work environment? Yes ☐ No ☐

Street Address	City, State, Zip Code and Country
Project Name	**Client:**
Tt Supervisor or Project Manager	**Was supervisor on the scene?**
	Yes ☐ No ☐

WITNESS INFORMATION (attach additional sheets if necessary)

Name	Company
Street Address	**City, State and Zip Code**
Telephone Number(s)	

CORRECTIVE ACTIONS

Corrective action(s) immediately taken by unit reporting the incident:

_____

_____

_____

Corrective action(s) still to be taken (by whom and when):

_____

_____

_____

ROOT CAUSE ANALYSIS LEVEL REQUIRED

Root Cause Analysis Level Required: Level - 1 ☐ Level - 2 ☐ None ☐

Root Cause Analysis Level Definitions

Level - 1	**Definition:** A Level 1 RCA is conducted by an individual(s) with experience or training in root cause analysis techniques and will conduct or direct documentation reviews, site investigation, witness and affected employee interviews, and identify corrective actions. Activating a Level 1 RCA and identifying RCA team members will be at the discretion of the Corporate Administration office. The following events may trigger a Level 1 RCA: ▪ Work related fatality ▪ Hospitalization of one or more employee where injuries result in total or partial permanent disability ▪ Property damage in excess of $75,000 ▪ When requested by senior management
Level - 2	**Definition:** A Level 2 RCA is self performed within the operating unit by supervisory personnel with assistance of the operating unit HSR. Level 2 RCA will utilize the 5 Why RCA methodology and document the findings on the tools provided. The following events will require a Level 2 RCA: ▪ OSHA recordable lost time incident ▪ Near miss incident that could have triggered a Level 1 RCA ▪ When requested by senior management

Complete the Root Cause Analysis Worksheet and Corrective Action form. Identify a corrective action(s) for each root cause identified within each area of inquiry.

NOTIFICATIONS

Title	Printed Name	Signature	Telephone Number	Date
Project Manager or Supervisor				
Site Safety Coordinator or Office H&S Representative				
Operating Unit H&S Representative				
Other: _____				

The signatures provided above indicate that appropriate personnel have been notified of the incident.

INSTRUCTIONS:

Complete all sections below for incidents involving injury or illness.
Do NOT leave any blanks.
Attach this form to the IR FORM completed for this incident.

Incident Report Number: (From the IR Form)

EMPLOYEE INFORMATION

Company Affiliation

Tetra Tech Employee? ☐ TetraTech subcontractor employee (directly supervised by Tt personnel)? ☐

Full Name	**Company (if not Tt employee)**

Street Address, City, State and Zip Code	**Address Type**
_____	Home address (for Tt employees) ☐
_____	Business address (for subcontractors) ☐

Telephone Numbers

Work: _____ Home: _____ Cell: _____

Occupation (regular job title)	**Department**

Was the individual performing regular job duties?	**Time individual began work**
Yes ☐ No ☐	_____ AM ☐ PM ☐ *OR* Cannot be determined ☐

Safety equipment

Provided? Yes ☐ No ☐

Used? Yes ☐ No ☐ If no, explain why

_____

_____

_____

Type(s) provided:

☐ Hard hat ☐ Protective clothing
☐ Gloves ☐ High visibility vest
☐ Eye protection ☐ Fall protection
☐ Safety shoes ☐ Machine guarding
☐ Respirator ☐ Other (list)

_____

NOTIFICATIONS

Name of Tt employee to whom the injury or illness was first reported	**Was H&S notified within one hour of injury or illness?**
	Yes ☐ No ☐

Date of report	**H&S Personnel Notified**

Time of report	**Time of Report**

If subcontractor injury, did subcontractor's firm perform their own incident investigation?

Yes ☐ No ☐ If yes, request a copy of their completed investigation form/report and attach it to this report.

INJURY / ILLNESS DETAILS

What was the individual doing just before the incident occurred? Describe the activity as well as the tools, equipment, or material the individual was using. Be specific. Examples: “Climbing a ladder while carrying roofing materials”; “Spraying chlorine from a hand sprayer”; “Daily computer key-entry”

What Happened? Describe how the injury occurred. Examples: “When ladder slipped on wet floor and worker fell 20 feet”; “Worker was sprayed with chlorine when gasket broke during replacement”; Worker developed soreness in wrist over time”

Describe the object or substance that directly harmed the individual: Examples: “Concrete floor”; “Chlorine”; “Radial Arm Saw”. If this question does not apply to the incident, write “Not Applicable”.

MEDICAL CARE PROVIDED

Was first aid provided at the site: Yes ☐ No ☐ If yes, describe the type of first aid administered and by whom?

Was treatment provided away from the site: Yes ☐ No ☐ If yes, provide the information below.

Name of physician or health care professional	**Facility Name**
Street Address, City State and Zip Code	**Type of Care?**
_____ _____	Was individual treated in emergency room? Yes ☐ No ☐ Was individual hospitalized overnight as an in-patient? Yes ☐ No ☐ Did the individual die? Yes ☐ No ☐ If yes, date: _____ Will a worker’s compensation claim be filed? Yes ☐ No ☐
Telephone Number	

NOTE: Attach any police reports or related diagrams to this report.

SIGNATURES

I have reviewed this report and agree that all the supplied information is accurate

Affected individual (print)	**Affected individual (signature)**	**Telephone Number**	**Date**

This form contains information relating to employee health and must be used in a manner that protects the confidentiality of the employee to the extent possible while the information is being used for occupational safety and health purposes.

INSTRUCTIONS:

Complete all sections below for incidents involving property/equipment damage, fire, spill or release.
Do NOT leave any blanks.
Attach this form to the IR FORM completed for this incident.

Incident Report Number: (From the IR Form)	

TYPE OF INCIDENT (Check all that apply)

Property Damage ☐ Equipment Damage ☐ Fire or Explosion ☐ Spill or Release ☐

INCIDENT DETAILS

Results of Incident: Fully describe damages, losses, etc.

_____

_____

_____

Response Actions Taken:

_____

_____

_____

Responding Agency(s) (i.e. police, fire department, etc.)	**Agency(s) Contact Name(s)**

DAMAGED ITEMS (List all damaged items, extent of damage and estimated repair cost)

Item:	**Extent of damage:**	**Estimated repair cost**

SPILLS / RELEASES (Provide information for spilled/released materials)

Substance	**Estimated quantity and duration**	**Specify Reportable Quantity (RQ)**
		_____ Exceeded? Yes ☐ No ☐ NA ☐

FIRES / EXPLOSIONS (Provide information related to fires/explosions)

Fire fighting equipment used? Yes ☐ No ☐ If yes, type of equipment: _____

NOTIFICATIONS

Required notifications	**Name of person notified**	**By whom**	**Date / Time**
Client: _____ Yes ☐ No ☐			
Agency: _____ Yes ☐ No ☐			
Other: _____ Yes ☐ No ☐			

Who is responsible for reporting incident to outside agency(s)? Tt ☐ Client ☐ Other ☐ Name: _____

Was an additional written report on this incident generated? Yes ☐ No ☐ If yes, place in project file.

INSTRUCTIONS:

Complete all sections below for incidents involving motor vehicle accidents. Do NOT leave any blanks. Attach this form to the IR FORM completed for this incident.

Incident Report Number: (From the IR Form)	

INCIDENT DETAILS

Name of road, street, highway or location where accident occurred	**Name of intersecting road, street or highway if applicable**

County	**City**	**State**

Did police respond to the accident?	**Did ambulance respond to the accident?**
Yes ☐ No ☐	Yes ☐ No ☐
Name and location of responding police department	**Ambulance company name and location**
Officer's name/badge #	

Did police complete an incident report? Yes ☐ No ☐ If yes, police report number: _____
Request a copy of completed investigation report and attach to this form.

VEHICLE INFORMATION

How many vehicles were involved in the accident? _____ (Attach additional sheets as applicable for accidents involving more than 2 vehicles.)

Vehicle Number 1 – Tetra Tech Vehicle		**Vehicle Number 2 – Other Vehicle**	
Vehicle Owner / Contact Information		**Vehicle Owner / Contact Information**	
Color		**Color**	
Make		**Make**	
Model		**Model**	
Year		**Year**	
License Plate #		**License Plate #**	
Identification #		**Identification #**	
Describe damage to vehicle number 1		**Describe damage to vehicle number 2**	
Insurance Company Name and Address		**Insurance Company Name and Address**	
Agent Name		**Agent Name**	
Agent Phone No.		**Agent Phone No.**	
Policy Number		**Policy Number**	

DRIVER INFORMATION

Vehicle Number 1 – Tetra Tech Vehicle		Vehicle Number 2 – Other Vehicle	
Driver's Name		**Driver's Name**	
Driver's Address		**Driver's Address**	
Phone Number		**Phone Number**	
Date of Birth		**Date of Birth**	
Driver's License #		**Driver's License #**	
Licensing State		**Licensing State**	
Gender	Male ☐ Female ☐	**Gender**	Male ☐ Female ☐
Was traffic citation issued to Tetra Tech driver? Yes ☐ No ☐		Was traffic citation issued to driver of other vehicle? Yes ☐ No ☐	
Citation #		**Citation #**	
Citation Description		**Citation Description**	

PASSENGERS IN VEHICLES (NON-INJURED)

List all non-injured passengers (excluding driver) in each vehicle.
Driver information is captured in the preceding section.
Information related to persons injured in the accident (non-Tt employees) is captured in the section below on this form.
Injured Tt employee information is captured on FORM IR-A

Vehicle Number 1 – Tetra Tech Vehicle		Vehicle Number 2 – Other Vehicle	
How many passengers (excluding driver) in the vehicle? ___		How many passengers (excluding driver) in the vehicle? ___	
Non-Injured Passenger Name and Address		**Non-Injured Passenger Name and Address**	
Non-Injured Passenger Name and Address		**Non-Injured Passenger Name and Address**	
Non-Injured Passenger Name and Address		**Non-Injured Passenger Name and Address**	

INJURIES TO NON-TETRATECH EMPLOYEES

Name of injured person 1				Address of injured person 1		
Age	**Gender**	**Car No.**	**Location in Car**	**Seat Belt Used?**	**Ejected from car?**	**Injury or Fatality?**
	Male ☐ Female ☐			Yes ☐ No ☐	Yes ☐ No ☐	Injured ☐ Died ☐
Name of injured person 2				**Address of injured person 2**		
Age	**Gender**	**Car No.**	**Location in Car**	**Seat Belt Used?**	**Ejected from car?**	**Injury or Fatality?**
	Male ☐ Female ☐			Yes ☐ No ☐	Yes ☐ No ☐	Injured ☐ Died ☐

OTHER PROPERTY DAMAGE

Describe damage to property other than motor vehicles	
Property Owner's Name	**Property Owner's Address**

COMPLETE AND SUBMIT DIAGRAM DEPICTING WHAT HAPPENED

ATTACHMENT II

MEDICAL DATA SHEET

MEDICAL DATA SHEET

This Medical Data Sheet must be completed by on-site personnel and kept in the command post during the conduct of site operations. This data sheet will accompany any personnel when medical assistance is needed or if transport to hospital facilities is required.

Project _____

Name _____ Home Telephone _____

Address _____

Age _____ Height _____ Weight _____

Person to notify in the event of an emergency: Name: _____

Phone: _____

Drug or other Allergies: _____

Particular Sensitivities : _____

Do You Wear Contacts?_____

What medications are you presently using? _____

_____

Name, Address, and Phone Number of personal physician: _____

_____

_____

Note: Health Insurance Portability and Accountability Act (HIPAA) Requirements

HIPAA took effect April 14, 2003. Loosely interpreted, HIPAA regulates the disclosure of Protected Health Information (PHI) by the entity collecting that information. PHI is any information about health status (such as that you may report on this Medical Data Sheet), provision of health care, or other information. HIPAA also requires TtNUS to ensure the confidentiality of PHI. This Act can affect the ability of the Medical Data Sheet to contain and convey information you would want a Doctor to know if you were incapacitated. So before you complete the Medical Data Sheet understand that this form will not be maintained in a secure location. It will be maintained in a file box or binder accessible to other members of the field crew so that they can accompany an injured party to the hospital.

DO NOT include information that you do not wish others to know, only information that may be pertinent in an emergency situation or treatment.

_____ _____ _____

Name (Print clearly) Signature Date

ATTACHMENT III

UTILITY LOCATING AND EXCAVATION CLEARANCE SOP

TETRA TECH NUS, INC.	STANDARD OPERATING PROCEDURES	Number HS-1.0	Page 1 of 15
		Effective Date 12/03	Revision 2
		Applicability Tetra Tech NUS, Inc.	
		Prepared Health & Safety	
Subject UTILITY LOCATING AND EXCAVATION CLEARANCE		Approved D. Senovich	

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1.0 PURPOSE

Utilities such as electric service lines, natural or propane gas lines, water and sewage lines, telecommunications, and steam lines are very often in the immediate vicinity of work locations. Contact with underground or overhead utilities can have serious consequences including employee injury/fatality, property and equipment damage, substantial financial impacts, and loss of utility service to users.

The purpose of this procedure is to provide minimum requirements and technical guidelines regarding the appropriate procedures to be followed when performing subsurface and overhead utility locating services. It is the policy of Tetra Tech NUS, Inc. (TtNUS) to provide a safe and healthful work environment for the protection of our employees. The purpose of this Standard Operating Procedure (SOP) is to aid in achieving the objectives of this policy, to present the acceptable procedures pertaining to utility locating and excavation clearance activities, and to present requirements and restrictions relevant to these types of activities. This SOP must be reviewed by any employee potentially involved with underground or overhead utility locating and avoidance activities.

2.0 SCOPE

This procedure applies to all TtNUS field activities where there may be potential contact with underground or overhead utilities. This procedure provides a description of the principles of operation, instrumentation, applicability, and implementability of typical methods used to determine the presence and avoidance of contact with utility services. This procedure is intended to assist with work planning and scheduling, resource planning, field implementation, and subcontractor procurement. Utility locating and excavation clearance requires site-specific information prior to the initiation of any such activities on a specific project. This SOP is not intended to provide a detailed description of methodology and instrument operation. Specialized expertise during both planning and execution of several of the methods presented may also be required.

3.0 GLOSSARY

Electromagnetic Induction (EMI) Survey - A geophysical exploration method whereby electromagnetic fields are induced in the ground and the resultant secondary electromagnetic fields are detected as a measure of ground conductivity.

Magnetometer – A device used for precise and sensitive measurements of magnetic fields.

Magnetic Survey – A geophysical survey method that depends on detection of magnetic anomalies caused by the presence of buried ferromagnetic objects.

Metal Detection – A geophysical survey method that is based on electromagnetic coupling caused by underground conductive objects.

Vertical Gradiometer – A magnetometer equipped with two sensors that are vertically separated by a fixed distance. It is best suited to map near surface features and is less susceptible to deep geologic features.

Ground Penetrating Radar – Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture.

4.0 RESPONSIBILITIES

Project Manager (PM)/Task Order Manager (TOM) - Responsible for ensuring that all field activities are conducted in accordance with this procedure.

Site Manager (SM)/Field Operations Leader (FOL) - Responsible for the onsite verification that all field activities are performed in compliance with approved SOPs or as otherwise directed by the approved project plan(s).

Site Health & Safety Officer (SHSO) – Responsible to provide technical assistance and verify full compliance with this SOP. The SHSO is also responsible for reporting any deficiencies to the Corporate Health and Safety Manager (HSM) and to the PM/TOM.

Health & Safety Manager (HSM) – Responsible for preparing, implementing, and modifying corporate health and safety policy and this SOP.

Site Personnel – Responsible for performing their work activities in accordance with this SOP and the TtNUS Health and Safety Policy.

5.0 PROCEDURES

This procedure addresses the requirements and technical procedures that must be performed to minimize the potential for contact with underground and overhead utility services. These procedures are addressed individually from a buried and overhead standpoint.

5.1 Buried Utilities

Buried utilities present a heightened concern because their location is not typically obvious by visual observation, and it is common that their presence and/or location is unknown or incorrectly known on client properties. This procedure must be followed prior to beginning any subsurface probing or excavation that might potentially be in the vicinity of underground utility services. In addition, the Utility Clearance Form (Attachment 3) must be completed for every location or cluster of locations where intrusive activities will occur.

Where the positive identification and de-energizing of underground utilities cannot be obtained and confirmed using the following steps, the PM/TOM is responsible for arranging for the procurement of a qualified, experienced, utility locating subcontractor who will accomplish the utility location and demarcation duties specified herein.

1. A comprehensive review must be made of any available property maps, blue lines, or as-builts prior to site activities. Interviews with local personnel familiar with the area should be performed to provide additional information concerning the location of potential underground utilities. Information regarding utility locations shall be added to project maps upon completion of this exercise.

2., A visual site inspection must be performed to compare the site plan information to actual field conditions. Any findings must be documented and the site plan/maps revised. The area(s) of proposed excavation or other subsurface activities must be marked at the site in white paint or pin flags to identify those locations of the proposed intrusive activities. The site inspection should focus on locating surface indications of potential underground utilities. Items of interest include the presence of nearby area lights, telephone service, drainage grates, fire hydrants, electrical service vaults/panels, asphalt/concrete scares and patches, and topographical depressions. Note the location of any emergency shut off switches. Any additional information regarding utility

locations shall be added to project maps upon completion of this exercise and returned to the PM/TOM.

3. If the planned work is to be conducted on private property (e.g., military installations, manufacturing facilities, etc.) the FOL must identify and contact appropriate facility personnel (e.g., public works or facility engineering) before any intrusive work begins to inquire about (and comply with) property owner requirements. It is important to note that private property owners may require several days to several weeks advance notice prior to locating utilities.

4. If the work location is on public property, the state agency that performs utility clearances must be notified (see Attachment 1). State "one-call" services must be notified prior to commencing fieldwork per their requirements. Most one-call services require, by law, 48- to 72-hour advance notice prior to beginning any excavation. Such services typically assign a "ticket" number to the particular site. This ticket number must be recorded for future reference and is valid for a specific period of time, but may be extended by contacting the service again. The utility service will notify utility representatives who then mark their respective lines within the specified time frame. It should be noted that most military installations own their own utilities but may lease service and maintenance from area providers. Given this situation, "one call" systems may still be required to provide location services on military installations.

5. Utilities must be identified and their locations plainly marked using pin flags, spray paint, or other accepted means. The location of all utilities must be noted on a field sketch for future inclusion on project maps. Utility locations are to be identified using the following industry-standard color code scheme, unless the property owner or utility locator service uses a different color code:

white	excavation/subsurface investigation location
red	electrical
yellow	gas, oil, steam
orange	telephone, communications
blue	water, irrigation, slurry
green	sewer, drain

6. Where utility locations are not confirmed with a high degree of confidence through drawings, schematics, location services, etc., the work area must be thoroughly investigated prior to beginning the excavation. In these situations, utilities must be identified using safe and effective methods such as passive and intrusive surveys, or the use of non-conductive hand tools. Also, in situations where such hand tools are used, they should always be used in conjunction with suitable detection equipment, such as the items described in Section 6.0 of this SOP. Each method has advantages and disadvantages including complexity, applicability, and price. It also should be noted that in some states, initial excavation is required by hand to a specified depth.

7. At each location where trenching or excavating will occur using a backhoe or other heavy equipment, and where utility identifications and locations cannot be confirmed prior to groundbreaking, the soil must be probed using a device such as a tile probe which is made of non-conductive material such as fiberglass. If these efforts are not successful in clearing the excavation area of suspect utilities, hand shoveling must be performed for the perimeter of the intended excavation.

8. All utilities uncovered or undermined during excavation must be structurally supported to prevent potential damage. Unless necessary as an emergency corrective measure, TtNUS shall not make any repairs or modifications to existing utility lines without prior permission of the utility owner, property owner, and Corporate HSM. All repairs require that the line be locked-out/tagged-out prior to work.

5.2 Overhead Power Lines

If it is necessary to work within the minimum clearance distance of an overhead power line, the overhead line must be de-energized and grounded, or re-routed by the utility company or a registered electrician. If protective measures such as guarding, isolating, or insulating are provided, these precautions must be adequate to prevent employees from contacting such lines directly with any part of their body or indirectly though conductive materials, tools, or equipment.

The following table provides the required minimum clearances for working in proximity to overhead power lines.

Nominal Voltage	Minimum Clearance
0 -50 kV	10 feet, or one mast length; whichever is greater
50+ kV	10 feet plus 4 inches for every 10 kV over 50 kV or 1.5 mast lengths; whichever is greater

6.0 UNDERGROUND LOCATING TECHNIQUES

A variety of supplemental utility locating approaches are available and can be applied when additional assurance is needed. The selection of the appropriate method(s) to employ is site-specific and should be tailored to the anticipated conditions, site and project constraints, and personnel capabilities.

6.1 Geophysical Methods

Geophysical methods include electromagnetic induction, magnetics, and ground penetrating radar. Additional details concerning the design and implementation of electromagnetic induction, magnetics, and ground penetrating radar surveys can be found in one or more of the TtNUS SOPs included in the References (Section 8.0).

Electromagnetic Induction

Electromagnetic Induction (EMI) line locators operate either by locating a background signal or by locating a signal introduced into the utility line using a transmitter. A utility line acts like a radio antenna, producing electrons, which can be picked up with a radiofrequency receiver. Electrical current carrying conductors have a 60HZ signal associated with them. This signal occurs in all power lines regardless of voltage. Utilities in close proximity to power lines or used as grounds may also have a 60HZ signal, which can be picked up with an EM receiver. A typical example of this type of geophysical equipment is an EM-61.

EMI locators specifically designed for utility locating use a special signal that is either indirectly induced onto a utility line by placing the transmitter above the line or directly induced using an induction clamp. The clamp induces a signal on the specific utility and is the preferred method of tracing since there is little chance of the resulting signals being interfered with. A good example of this type of equipment is the Schonstedt® MAC-51B locator. The MAC-51B performs inductively traced surveys, simple magnetic locating, and traced nonmetallic surveys.

When access can be gained inside a conduit to be traced, a flexible insulated trace wire can be used. This is very useful for non-metallic conduits but is limited by the availability of gaining access inside the pipe.

Magnetics

Magnetic locators operate by detecting the relative amounts of buried ferrous metal. They are incapable of locating or identifying nonferrous utility lines but can be very useful for locating underground storage tanks (UST's), steel utility lines, and buried electrical lines. A typical example of this type of equipment is the Schonstedt® GA-52Cx locator. The GA-52Cx is capable of locating 4-inch steel pipe up to 8 feet deep.

Non-ferrous lines are often located by using a typical plumbing tool (snake) fed through the line. A signal is then introduced to the snake that is then traced.

Ground Penetrating Radar

Ground Penetrating Radar (GPR) involves specialized radar equipment whereby a signal is sent into the ground via a transmitter. Some portion of the signal will be reflected from the subsurface material, which is then recorded with a receiver and electronically converted into a graphic picture. In general, an object which is harder than the surrounding soil will reflect a stronger signal. Utilities, tunnels, UST's, and footings will reflect a stronger signal than the surrounding soil. Although this surface detection method may determine the location of a utility, this method does not specifically identify utilities (i.e., water vs. gas, electrical vs. telephone); hence, verification may be necessary using other methods. This method is somewhat limited when used in areas with clay soil types or with a high water table.

6.2 Passive Detection Surveys

Acoustic Surveys

Acoustic location methods are generally most applicable to waterlines or gas lines. A highly sensitive Acoustic Receiver listens for background sounds of water flowing (at joints, leaks, etc.) or to sounds introduced into the water main using a transducer. Acoustics may also be applicable to determine the location of plastic gas lines.

Thermal Imaging

Thermal (i.e., infrared) imaging is a passive method for detecting the heat emitted by an object. Electronics in the infrared camera convert subtle heat differentials into a visual image on the viewfinder or a monitor. The operator does not look for an exact temperature; rather they look for heat anomalies (either elevated or suppressed temperatures) characteristic of a potential utility line.

The thermal fingerprint of underground utilities results from differences in temperature between the atmosphere and the fluid present in a pipe or the heat generated by electrical resistance. In addition, infrared scanners may be capable of detecting differences in the compaction, temperature and moisture content of underground utility trenches. High-performance thermal imagery can detect temperature differences to hundredths of a degree.

6.3 Intrusive Detection Surveys

Vacuum Excavation

Vacuum excavation is used to physically expose utility services. The process involves removing the surface material over approximately a 1' x 1' area at the site location. The air-vacuum process proceeds with the simultaneous action of compressed air-jets to loosen soil and vacuum extraction of the resulting

debris. This process ensures the integrity of the utility line during the excavation process, as no hammers, blades, or heavy mechanical equipment comes into contact with the utility line, eliminating the risk of damage to utilities. The process continues until the utility is uncovered. Vacuum excavation can be used at the proposed site location to excavate below the "utility window" which is usually 8 feet.

Hand Excavation

When the identification and location of underground utilities cannot be positively confirmed through document reviews and/or other methods, borings and excavations may be cleared via the use of non-conductive hand tools. This should always be done in conjunction with the use of detection equipment. This would be required for all locations where there is a potential to impact buried utilities. The minimum hand-excavation depth that must be reached is to be determined considering the geographical location of the work site. This approach recognizes that the placement of buried utilities is influenced by frost line depths that vary by geographical region. Attachment 2 presents frost line depths for the regions of the contiguous United States. At a minimum, hand excavation depths must be at least to the frost line depth (see Attachment 2) plus two (2) feet, but never less than 4 feet below ground surface (bgs). For hand excavation, the hole created must be reamed large enough to be at least the diameter of the drill rig auger or bit prior to drilling. For soil gas surveys, the survey probe shall be placed as close as possible to the cleared hand excavation. It is important to note that a post-hole digger must not be used in this type of hand excavation activity.

Tile Probe Surveys

For some soil types, site conditions, and excavation requirements, non-conductive tile probes may be used. A tile probe is a "T"-handled rod of varying lengths that can be pushed into the soil to determine if any obstructions exist at that location. Tile probes constructed of fiberglass or other nonconductive material are readily-available from numerous vendors. Tile probes must be performed to the same depth requirements as previously specified. As with other types of hand excavating activities, the use of a non-conductive tile probe, should always be in conjunction with suitable utility locating detection equipment.

7.0 INTRUSIVE ACTIVITIES SUMMARY

The following list summarizes the activities that must be performed prior to beginning subsurface activities:

1. Map and mark all subsurface locations and excavation boundaries using white paint or markers specified by the client or property owner.

2. Notify the property owner and/or client that the locations are marked. At this point, drawings of locations or excavation boundaries shall be provided to the property owner and/or client so they may initiate (if applicable) utility clearance.

   Note: Drawings with confirmed locations should be provided to the property owner and/or client as soon as possible to reduce potential time delays.

3. Notify "One Call" service. If possible, arrange for an appointment to show the One Call representative the surface locations or excavation boundaries in person. This will provide a better location designation to the utilities they represent. You should have additional drawings should you need to provide plot plans to the One Call service.

4. Implement supplemental utility detection techniques as necessary and appropriate to conform utility locations or the absence thereof.

5. Complete Attachment 3, Utility Clearance Form. This form should be completed for each excavation location. In situations where multiple subsurface locations exist within the close proximity of one another, one form may be used for multiple locations provided those locations are noted on the Utility Clearance Form. Upon completion, the Utility Clearance Form and revised/annotated utility location map becomes part of the project file.

8.0 REFERENCES

OSHA Letter of Interpretation, Mr. Joseph Caldwell, Attachment 4
OSHA 29 CFR 1926(b)(2)
OSHA 29 CFR 1926(b)(3)
TtNUS Utility Locating and Clearance Policy
TtNUS SOP GH-3.1; Resistivity and Electromagnetic Induction
TtNUS SOP GH-3.2; Magnetic and Metal Detection Surveys
TtNUS SOP GH-3.4; Ground-penetrating Radar Surveys

ATTACHMENT 1
LISTING OF UNDERGROUND UTILITY CLEARANCE RESOURCES

American Public Works Association
2345 Grand Boulevard, Suite 500, Kansas City, MO 64108-2625
Phone (816) 472-6100 • Fax (816) 472-1610
Web www.apwa.net • E-mail apwa@apwa.net

ONE-CALL SYSTEMS INTERNATIONAL CONDENSED DIRECTORY

Alabama
Alabama One-Call
1-800-292-8525

Alaska
Locate Call Center of Alaska, Inc.
1-800-478-3121

Arizona
Arizona Blue Stake
1-800-782-5348

Arkansas
Arkansas One Call System, Inc.
1-800-482-8998

California
Underground Service Alert North
1-800-227-2600
Underground Service Alert of Southern California
1-800-227-2600

Colorado
Utility Notification Center of Colorado
1-800-922-1987

Connecticut
Call Before You Dig
1-800-922-4455

Delaware
Miss Utility of Delmarva
1-800-282-8555

Florida
Sunshine State One-Call of Florida, Inc.
1-800-432-4770

Georgia
Underground Protection Center, Inc.
1-800-282-7411

Hawaii
Underground Service Alert North
1-800-227-2600

Idaho
Dig Line Inc.
1-800-342-1585
Kootenai County One-Call
1-800-428-4950
Shoshone - Benewah One-Call
1-800-398-3285

Illinois
JULIE, Inc.
1-800-892-0123
Digger (Chicago Utility Alert Network)
312-744-7000

Indiana
Indiana Underground Plant Protection Service
1-800-382-5544

Iowa
Iowa One-Call
1-800-292-8989

Kansas
Kansas One-Call System, Inc.
1-800-344-7233

Kentucky
Kentucky Underground Protection Inc.
1-800-752-6007

Louisiana
Louisiana One Call System, Inc.
1-800-272-3020

Maine
Dig Safe System, Inc.
1-888-344-7233

Maryland
Miss Utility
1-800-257-7777
Miss Utility of Delmarva
1-800-282-8555

Massachusetts
Dig Safe System, Inc.
1-888-344-7233

Michigan
Miss Dig System, Inc.
1-800-482-7171

Minnesota
Gopher State One Call
1-800-252-1166

Mississippi
Mississippi One-Call System, Inc
1-800-227-6477

Missouri
Missouri One-Call System, Inc.
1-800-344-7483

Montana
Utilities Underground Protection Center
1-800-424-5555
Montana One Call Center
1-800-551-8344

Nebraska
Diggers Hotline of Nebraska
1-800-331-5666

Nevada
Underground Service Alert North
1-800-227-2600

New Hampshire
Dig Safe System, Inc.
1-888-344-7233

New Jersey
New Jersey One Call
1-800-272-1000

New Mexico
New Mexico One Call System, Inc.
1-800-321-2537
Las Cruces- Dona Ana Blue Stakes
1-888-526-0400

New York
Dig Safely New York
1-800-962-7962
New York City- Long Island One Call Center
1-800-272-4480

North Carolina
The North Carolina One-Call Center, Inc.
1-800-632-4949

North Dakota
North Dakota One-Call
1-800-795-0555

Ohio
Ohio Utilities Protection Service
1-800-362-2764
Oil & Gas Producers Underground Protect'n Svc
1-800-925-0988

Oklahoma
Call Okie
1-800-522-6543

Oregon
Oregon Utility Notification Center/One Call Concepts
1-800-332-2344

Pennsylvania
Pennsylvania One Call System, Inc.
1-800-242-1776

Rhode Island
Dig Safe System, Inc.
1-888-344-7233

South Carolina
Palmetto Utility Protection Service Inc.
1-888-721-7877

South Dakota
South Dakota One Call
1-800-781-7474

Tennessee
Tennessee One-Call System, Inc.
1-800-351-1111

ATTACHMENT 1 (Continued)

Texas
Texas One Call System
1-800-245-4545
Texas Excavation Safety System, Inc.
1-800-344-8377
Lone Star Notification Center
1-800-669-8344

Utah
Blue Stakes of Utah
1-800-662-4111

Vermont
Dig Safe System, Inc.
1-888-344-7233

Virginia
Miss Utility of Virginia
1-800-552-7001
Miss Utility (Northern Virginia)
1-800-257-7777

Washington
Utilities Underground Location Center
1-800-424-5555
Northwest Utility Notification Center
1-800-553-4344
Inland Empire Utility Coordinating Council
509-456-8000

West Virginia
Miss Utility of West Virginia, Inc.
1-800-245-4848

Wisconsin
Diggers Hotline, Inc.
1-800-242-8511

Wyoming
Wyoming One-Call System, Inc.
1-800-348-1030
Call Before You Dig of Wyoming
1-800-849-2476

District of Columbia
Miss Utility
1-800-257-7777

Alberta
Alberta One-Call Corporation
1-800-242-3447

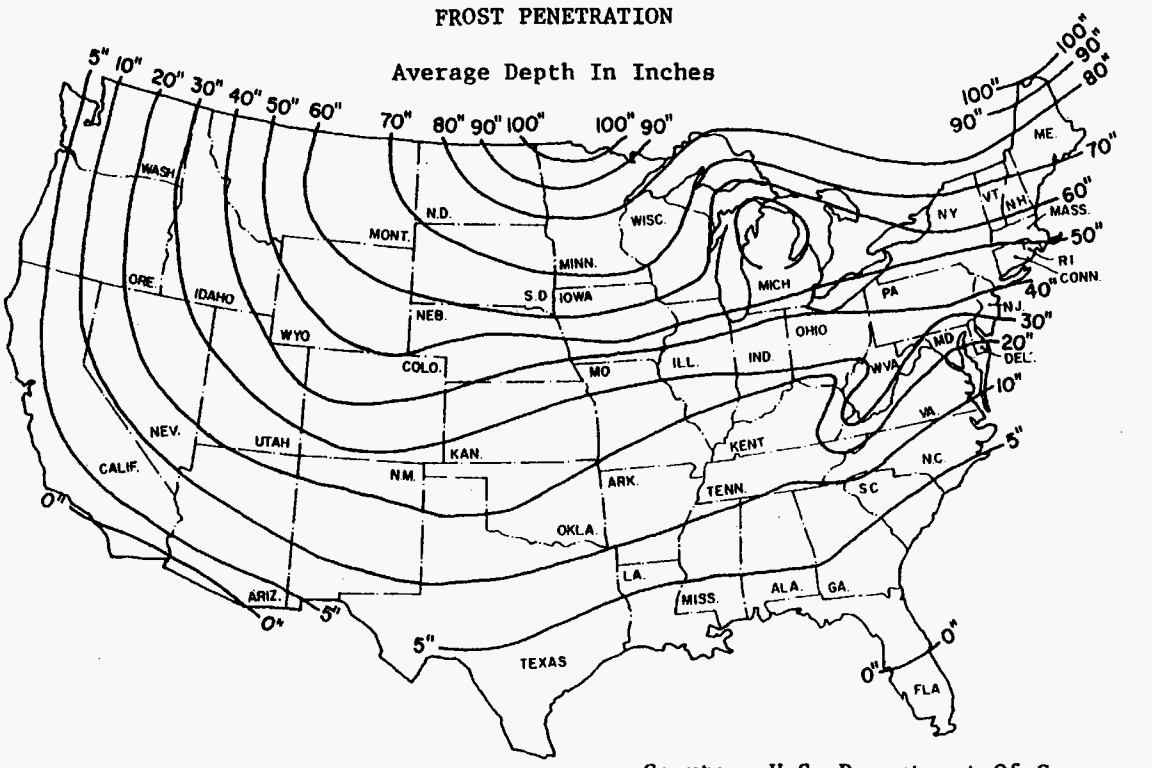
British Columbia
BC One Call
1-800-474-6886

Ontario
Ontario One-Call System
1-800-400-2255

Quebec
Info-Excavation
1-800-663-9228

ATTACHMENT 2

FROST LINE PENETRATION DEPTHS BY GEOGRAPHIC LOCATION



Courtesy U.S. Department Of Commerce

ATTACHMENT 3
UTILITY CLEARANCE FORM

Client: _____ Project Name: _____

Project No.: _____ Completed By: _____

Location Name: _____ Work Date: _____

Excavation Method/Overhead Equipment: _____

			Circle One
1.	Underground Utilities		
	a)	Review of existing maps?	yes no N/A
	b)	Interview local personnel?	yes no N/A
	c)	Site visit and inspection?	yes no N/A
	d)	Excavation areas marked in the field?	yes no N/A
	e)	Utilities located in the field?	yes no N/A
	f)	Located utilities marked/added to site maps?	yes no N/A
	g)	Client contact notified Name _____ Telephone: _____ Date: _____	yes no N/A
	g)	State One-Call agency called? Caller: _____ Ticket Number: _____ Date: _____	yes no N/A
	h)	Geophysical survey performed? Survey performed by: _____ Method: _____ Date: _____	yes no N/A
	i)	Hand excavation performed (with concurrent use of utility detection device)? Completed by: _____ Total depth: _____ feet Date: _____	yes no N/A
	j)	Trench/excavation probed? Probing completed by: _____ Depth/frequency: _____ Date: _____	yes no N/A

			Present Absent
2.	Overhead Utilities		
	a)	Determination of nominal voltage	yes no N/A
	b)	Marked on site maps	yes no N/A
	c)	Necessary to lockout/insulate/re-route	yes no N/A
	d)	Document procedures used to lockout/insulate/re-route	yes no N/A
	e)	Minimum acceptable clearance (SOP Section 5.2): _____	

3. Notes:

_____

_____

_____

Approval:

_____ _____

Site Manager/Field Operations Leader Date

c: PM/Project File
Program File

ATTACHMENT 4
OSHA LETTER OF INTERPRETATION

Mr. Joseph Caldwell
Consultant
Governmental Liaison
Pipeline Safety Regulations
211 Wilson Boulevard
Suite 700
Arlington, Virginia 22201

Re: Use of hydro-vacuum or non-conductive hand tools to locate underground utilities.

Dear Mr. Caldwell:

In a letter dated July 7, 2003, we responded to your inquiry of September 18, 2002, regarding the use of hydro-vacuum equipment to locate underground utilities by excavation. After our letter to you was posted on the OSHA website, we received numerous inquiries that make it apparent that aspects of our July 7 letter are being misunderstood. In addition, a number of industry stakeholders, including the National Utility Contractors Association (NUCA), have provided new information regarding equipment that is available for this work.

To clarify these issues, we are withdrawing our July 7 letter and issuing this replacement response to your inquiry.

Question: *Section 1926.651 contains several requirements that relate to the safety of employees engaged in excavation work. Specifically, paragraphs (b)(2) and (b)(3) relate in part to the safety of the means used to locate underground utility installations that, if damaged during an uncovering operation, could pose serious hazards to employees.*

Under these provisions, what constitutes an acceptable method of uncovering underground utility lines, and further, would the use of hydro-vacuum excavation be acceptable under the standard?

Answer

Background

Two sections of 29 CFR 1926 Subpart P (Excavations), 1926.651(Specific excavation requirements), govern methods for uncovering underground utility installations. Specifically, paragraph (b)(2) states:

> When utility companies or owners cannot respond to a request to locate underground utility installations within 24 hours * * * or cannot establish the exact location of these installations, the employer may proceed, provided the employer does so with caution, and provided detection equipment or other acceptable means to locate utility installations are used. (emphasis added).

Paragraph (b)(3) provides:

ATTACHMENT 4 (Continued)

> When excavation operations approach the estimated location of underground installations, the exact location of the installations shall be determined by safe and acceptable means. (emphasis added).

Therefore, "acceptable means" must be used where the location of the underground utilities have not been identified by the utility companies and detection equipment is not used.

Subpart P does not contain a definition of either "other acceptable means" or "safe and acceptable means." The preambles to both the proposed rule and the final rule discussed the rationale behind the wording at issue. For example, the preamble to the proposed rule, 52 Fed. Reg. 12301 (April 15, 1987), noted that a 1972 version of this standard contained language that specified "careful probing or hand digging" as the means to uncover utilities. The preamble then noted that an amendment to the 1972 standard later deleted that language "to allow other, *equally effective means* of locating such installations." The preamble continued that in the 1987 proposed rule, OSHA again proposed using language in section (b)(3) that would provide another example of an acceptable method of uncovering utilities that could be used *where the utilities have not been marked and detection equipment is not being used* – "probing with hand-held tools." This method was rejected in the final version of 29 CFR 1926. As OSHA explained in the preamble to the final rule, 54 Fed. Reg. 45916 (October 31, 1989):

> OSHA received two comments * * * and input from ACCSH [OSHA's Advisory Committee on Construction Safety and Health] * * * on this provision. All commenters recommended dropping 'such as probing with hand-held tools' from the proposed provision, because this could create a hazard to employees by damaging the installation or its insulation.

In other words, the commenters objected to the use of hand tools being used unless detection equipment was used in conjunction with them. OSHA then concluded its discussion relative to this provision by agreeing with the commentators and ultimately not including any examples of "acceptable means" in the final provision.

Non-conductive hand tools are permitted

This raises the question of whether the standard permits the use of hand tools alone -- without also using detection equipment. NUCA and other industry stakeholders have recently informed us that non-conductive hand tools that are appropriate to be used to locate underground utilities are now commonly available.

Such tools, such as a "shooter" (which has a non-conductive handle and a snub nose) and non-conductive or insulated probes were not discussed in the rulemaking. Since they were not considered at that time, they were not part of the class of equipment that was thought to be unsafe for this purpose. Therefore, we conclude that the use of these types of hand tools, when used with appropriate caution, is an "acceptable means" for locating underground utilities.

ATTACHMENT 4 (Continued)

Hydro-vacuum excavation

It is our understanding that some hydro-vacuum excavation equipment can be adjusted to use a minimum amount of water and suction pressure. When appropriately adjusted so that the equipment will not damage underground utilities (especially utilities that are particularly vulnerable to damage, such as electrical lines), use of such equipment would be considered a "acceptable means" of locating underground utilities. However, if the equipment cannot be sufficiently adjusted, then this method would not be acceptable under the standard.

Other technologies

We are not suggesting that these are the only devices that would be "acceptable means" under the standard. Industry stakeholders have informed us that there are other types of special excavation equipment designed for safely locating utilities as well.

We apologize for any confusion our July 7 letter may have caused. If you have further concerns or questions, please feel free to contact us again by fax at: U.S. Department of Labor, OSHA, Directorate of Construction, Office of Construction Standards and Compliance Assistance, fax # 202-693-1689. You can also contact us by mail at the above office, Room N3468, 200 Constitution Avenue, N.W., Washington, D.C. 20210, although there will be a delay in our receiving correspondence by mail.

Sincerely,

Russell B. Swanson, Director
Directorate of Construction

NOTE: OSHA requirements are set by statute, standards and regulations. Our interpretation letters explain these requirements and how they apply to particular circumstances, but they cannot create additional employer obligations. This letter constitutes OSHA=s interpretation of the requirements discussed. Note that our enforcement guidance may be affected by changes to OSHA rules. Also, from time to time we update our guidance in response to new information. To keep apprised of such developments, you can consult OSHA's website at http://www.osha.gov.

ATTACHMENT IV

EQUIPMENT INSPECTION CHECKLIST

Equipment Inspection Checklist for Drill Rigs

Company: _____ **Unit/Serial No#:** _____

Inspection Date: ____/____/____ **Time:** ____:____ **Equipment Type:** _____
(e.g., Drill Rigs Hollow Stem, Mud Rotary, Direct Push, HDD)

Project Name: _____ **Project No#:** _____

Yes	No	NA	Requirement	Comments
			Emergency Stop Devices	
☐	☐	☐	• Emergency Stop Devices (At points of operation)	
☐	☐	☐	• Have all emergency shut offs identified been communicated to the field crew?	
☐	☐	☐	• Has a person been designated as the Emergency Stop Device Operator?	
			Highway Use	
☐	☐	☐	• Cab, mirrors, safety glass?	
☐	☐	☐	• Turn signals, lights, brake lights, etc. (front/rear) for equipment approved for highway use?	
☐	☐	☐	• Seat Belts?	
☐	☐	☐	• Is the equipment equipped with audible back-up alarms and back-up lights?	
☐	☐	☐	• Horn and gauges	
☐	☐	☐	• Brake condition (dynamic, park, etc.)	
☐	☐	☐	• Tires (Tread) or tracks	
☐	☐	☐	• Windshield wipers	
☐	☐	☐	• Exhaust system	
☐	☐	☐	• Steering (standard and emergency)	
☐	☐	☐	• Wheel Chocks?	
☐	☐	☐	• Are tools and material secured to prevent movement during transport? Especially those within the cab?	
☐	☐	☐	• Are there flammables or solvents or other prohibited substances stored within the cab?	
☐	☐	☐	• Are tools or debris in the cab that may adversely influence operation of the vehicle (in and around brakes, clutch, gas pedals)	

Unit/Serial No#:_____ **Inspection Date:** ____/____/____

Yes	No	NA	Requirement	Comments
			Fluid Levels:	
☐	☐	☐	• Engine oil	
☐	☐	☐	• Transmission fluid	
☐	☐	☐	• Brake fluid	
☐	☐	☐	• Cooling system fluid	
☐	☐	☐	• Hoses and belts	
☐	☐	☐	• Hydraulic oil	
			High Pressure Hydraulic Lines	
☐	☐	☐	• Obvious damage	
☐	☐	☐	• Operator protected from accidental release	
☐	☐	☐	• Coupling devices, connectors, retention cables/pins are in good condition and in place	
			Mast Condition	
☐	☐	☐	• Structural components/tubing	
☐	☐	☐	• Connection points	
☐	☐	☐	• Pins	
☐	☐	☐	• Welds	
☐	☐	☐	• Outriggers	
☐	☐	☐	• Operational	
☐	☐	☐	• Plumb (when raised)	
			Hooks	
☐	☐	☐	• Are the hooks equipped with Safety Latches?	
☐	☐	☐	• Does it appear that the hook is showing signs of wear in excess of 10% original dimension?	
☐	☐	☐	• Is there a bend or twist exceeding 10% from the plane of an unbent hook?	
☐	☐	☐	• Increase in throat opening exceeding 15% from new condition	
☐	☐	☐	• Excessive nicks and/or gouges	
☐	☐	☐	• Clips	
☐	☐	☐	• Number of U-Type (Crosby) Clips (cable size 5/16 – 5/8 = 3 clips minimum) (cable size 3/4 – 1 inch = 4 clips minimum) (cable size 1 1/8 – 1 3/8 inch = 5 clips minimum)	

Unit/Serial No#:_____ **Inspection Date:** ____/____/____

Yes	No	NA	Requirement	Comments
☐	☐	☐	Power cable and/or hoist cable • Reduction in Rope diameter π (5/16 wire rope>1/64 reduction nominal size -replace) (3/8 to 1/2 wire rope>1/32 reduction nominal size-replace) (9/16 to 3/4 wire rope>3/64 reduction nominal size-replace)	
☐	☐	☐	• Number of broken wires (6 randomly broken wires in one rope lay) (3 broken wires in one strand)	
☐	☐	☐	• Number of wire rope wraps left on the Running Drum at nominal use (≥3 required)	
☐	☐	☐	- Lead (primary) sheave is centered on the running drum	
☐	☐	☐	• Lubrication of wire rope (adequate?)	
☐	☐	☐	• Kinks, bends – Flattened to > 50% diameter	
☐	☐	☐	Hemp/Fiber rope (Cathead/Split Spoon Hammer) • Minimum ¾; maximum 1 inch rope diameter (Inspect for physical damage)	
☐	☐	☐	• Rope to hammer is securely fastened	
☐	☐	☐	Safety Guards – • Around rotating apparatus (belts, pulleys, sprockets, spindles, drums, flywheels, chains) all points of operations protected from accidental contact?	
☐	☐	☐	• Hot pipes and surfaces exposed to accidental contact?	
☐	☐	☐	• High pressure lines	
☐	☐	☐	• Nip/pinch points	
☐	☐	☐	Operator Qualifications • Does the operator have proper licensing where applicable, (e.g., CDL)?	
☐	☐	☐	• Does the operator, understand the equipment's operating instructions?	
☐	☐	☐	• Is the operator experienced with this equipment?	
☐	☐	☐	• Is the operator 21 years of age or more?	

Unit/Serial No#:_____ **Inspection Date:** ____/____/____

Yes	No	NA	Requirement	Comments
			PPE Required for Drill Rig Exclusion Zone	
☐	☐	☐	• Hardhat	
☐	☐	☐	• Safety glasses	
☐	☐	☐	• Work gloves	
☐	☐	☐	• Chemical resistant gloves_____	
☐	☐	☐	• Steel toed Work Boots	
☐	☐	☐	• Chemical resistant Boot Covers	
☐	☐	☐	• Apron	
☐	☐	☐	• Coveralls Tyvek, Saranex, cotton)_____	
			Other Hazards	
☐	☐	☐	• Excessive Noise Levels? _____dBA	
☐	☐	☐	• Chemical hazards (Drilling supplies - Sand, bentonite, grout, fuel, etc.)	
☐	☐	☐	- MSDSs available?	
☐	☐	☐	• Will On-site fueling occur	
☐	☐	☐	- Safety cans available?	
☐	☐	☐	- Fire extinguisher (Type/Rating -_____ _ _____)	

Approved for Use ☐ Yes ☐ No ☐ See Comments

_____ _____

Site Health and Safety Officer Operator

ATTACHMENT V

SAFE WORK PERMITS

SAFE WORK PERMIT
MOBILIZATION AND DEMOBILIZATION ACTIVITIES
SITE 43
NAVAL SUPPORT FACILITY – INDIAN HEAD, MARYLAND

Permit No. _____ Date: _____ Time: From _____ to _____

I. Work limited to the following (description, area, equipment used): Mobilization and demobilization activities

II. Primary Hazards: Lifting; slips, trips and falls; vehicular and foot traffic; insect/animal bites and stings; poisonous plants; inclement weather.

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____TtNUS
Equipment Inspection required ☐ Yes ☐ No Initials of Inspector _____TtNUS

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐

Respiratory equipment required
Yes ☐ Specify on the reverse
No ☒

Modifications/Exceptions: Minimum requirement include sleeved shirt and long pants, or coveralls, safety, glasses and safety footwear. Hard hats and hearing protection will be worn when working near operating equipment.

VI. Chemicals of Concern	**Hazard Monitoring**	**Action Level(s)**	**Response Measures**
None anticipated given the nature of activities and limited contact with media	None	None	None

Primary Route(s) of Exposure/Hazard: NA

(Note to FOL and/or SHSO: Each item in Sections VII, VIII, and IX must be checked Yes, No, or NA)

VII. Additional Safety Equipment/Procedures

Hard-hat	☐ Yes ☐ No	Hearing Protection (Plugs/Muffs)	☐ Yes ☐ No
Safety Glasses	☐ Yes ☐ No	Safety belt/harness	☐ Yes ☒ No
Chemical/splash goggles	☐ Yes ☒ No	Radio/Cellular Phone	☐ Yes ☒ No
Splash Shield	☐ Yes ☒ No	Barricades	☐ Yes ☒ No
Splash suits/coveralls	☐ Yes ☒ No	Gloves (Type – Work)	☒ Yes ☐ No
Impermeable apron	☐ Yes ☒ No	Work/rest regimen	☐ Yes ☒ No
Steel toe work shoes/boots	☒Yes ☐ No	Chemical Resistant Boot Covers	☐ Yes ☐ No
High visibility vest	☐Yes ☐ No	Tape up/use insect repellent	☐ Yes ☐ No
First Aid Kit	☐Yes ☐ No	Fire Extinguisher	☐ Yes ☐ No
Safety Shower/Eyewash	☐Yes ☐ No	Other	☐ Yes ☐ No

Modifications/Exceptions: Tyvek coverall to protect against natural hazards (e.g., ticks) if working/walking through areas of high grass. Use insect repellants containing at least 10% DEET and tape up in such areas. Follow manufacturer's recommendations for proper application and reapplication. Hard hat when overhead hazards exist. Safety glasses when near eye hazards. Hearing protection when in high noise areas.

VIII. Site Preparation

	Yes	No	NA
Utility Locating and Excavation Clearance completed	☐	☐	☐
Vehicle and Foot Traffic Routes Established/Traffic Control Barricades/Signs in Place	☐	☐	☐
Physical Hazards Identified and Isolated (Splash and containment barriers)	☐	☐	☐
Emergency Equipment Staged (Spill control, fire extinguishers, first aid kits, etc).	☐	☐	☐

IX. Additional Permits required (Hot work, confined space entry, excavation etc.) ☐ Yes ☒ No
If yes, SHSO to complete or contact Health Sciences, Pittsburgh Office (412)921-7090

X. Special instructions, precautions: Preview work locations to identify potential hazards (slips, trips, and falls, natural hazards, etc.) Review PPE needs based on activities being performed and the associated hazards. Use safe lifting procedures and obtain assistance when handling heavy or awkward objects. Suspend site activities in the event of inclement weather. Observe site workers for signs and symptoms of heat/cold stress. Use sun block (SPF > 15) to prevent sunburn if necessary.

Permit Issued by: _____ Permit Accepted by: _____

SAFE WORK PERMIT
SAMPLING ACTIVITIES
SITE 43
NAVAL SUPPORT FACILITY – INDIAN HEAD, MARYLAND

Permit No. _____ Date: _____ Time: From _____ to _____

I. Work limited to the following (description, area, equipment used): Sampling activities, including monitoring well groundwater sampling (away from drill/DPT rig), soil gas with SUMMA canister.

II. Primary Hazards: Lifting; slips, trips and falls; vehicular and foot traffic; insect/animal bites and stings; poisonous plants; inclement weather. Chemical contamination.

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____TtNUS
Equipment Inspection required ☐ Yes ☐ No Initials of Inspector _____TtNUS

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐

Respiratory equipment required
Yes ☐ Specify on the reverse
No ☒

Modifications/Exceptions: Minimum requirement include sleeved shirt and long pants, or coveralls, safety, glasses and safety footwear. Hard hats and hearing protection will be worn when working near operating equipment.

VI. Chemicals of Concern	**Hazard Monitoring /Action Level(s)**	**Response Measures**
VOCs such as bromoform and TCE	PID w/10.06eV lamp reading of 30ppm for no more than 4 excursions of 5 minutes in any one work day.	Suspend site activities and report to an unaffected area.

Primary Route(s) of Exposure/Hazard: inhalation, dermal, ingestion

(Note to FOL and/or SHSO: Each item in Sections VII, VIII, and IX must be checked Yes, No, or NA)

VII. Additional Safety Equipment/Procedures

Hard-hat	☐ Yes ☐ No	Hearing Protection (Plugs/Muffs)	☐ Yes ☐ No
Safety Glasses	☐ Yes ☐ No	Safety belt/harness	☐ Yes ☒ No
Chemical/splash goggles	☐ Yes ☒ No	Radio/Cellular Phone	☐ Yes ☒ No
Splash Shield	☐ Yes ☒ No	Barricades	☐ Yes ☒ No
Splash suits/coveralls	☐ Yes ☒ No	Gloves (Type – Work)	☒ Yes ☐ No
Impermeable apron	☐ Yes ☒ No	Work/rest regimen	☐ Yes ☒ No
Steel toe work shoes/boots	☒ Yes ☐ No	Chemical Resistant Boot Covers	☐ Yes ☐ No
High visibility vest	☐ Yes ☐ No	Tape up/use insect repellent	☐ Yes ☐ No
First Aid Kit	☐ Yes ☐ No	Fire Extinguisher	☐ Yes ☐ No
Safety Shower/Eyewash	☐ Yes ☐ No	Other	☐ Yes ☐ No

Modifications/Exceptions: Minimum requirement include sleeved shirt and long pants, safety footwear, and nitrile gloves Tyvek coverall to protect against natural hazards (e.g., ticks) if working/walking through areas of high grass. Use insect repellants containing at least 10% DEET and tape up in such areas. Follow manufacturer's recommendations for proper application and reapplication. Hard hat when overhead hazards exist. Safety glasses when near eye hazards. Hearing protection when in high noise areas.

VIII. Site Preparation

	Yes	No	NA
Utility Locating and Excavation Clearance completed	☐	☐	☐
Vehicle and Foot Traffic Routes Established/Traffic Control Barricades/Signs in Place	☐	☐	☐
Physical Hazards Identified and Isolated (Splash and containment barriers)	☐	☐	☐
Emergency Equipment Staged (Spill control, fire extinguishers, first aid kits, etc).	☐	☐	☐

IX. Additional Permits required (Hot work, confined space entry, excavation etc.) ☐ Yes ☒ No
If yes, SHSO to complete or contact Health Sciences, Pittsburgh Office (412)921-7090

X. Special instructions, precautions: Preview work locations to identify potential hazards (slips, trips, and falls, natural hazards, etc.) Review PPE needs based on activities being performed and the associated hazards. Use safe lifting procedures and obtain assistance when handling heavy or awkward objects. Suspend site activities in the event of inclement weather. Observe site workers for signs and symptoms of heat/cold stress. Use sun block (SPF > 15) to prevent sunburn if necessary. If UXO is encountered, stop work, mark site with pin flag and notify NSF-IH EOD.

Permit Issued by: _____ Permit Accepted by: _____

SAFE WORK PERMIT
SOIL BORING ACTIVITIES
SITE 43
NAVAL SUPPORT FACILITY – INDIAN HEAD, MARYLAND

Permit No. _____ Date: _____ Time: From _____ to _____

I. Work limited to the following (description, area, equipment used): Drilling/soil boring, monitoring well installation and development and groundwater sampling (near drill/DPT rig), MIP via DPT rig, O Red Dye field testing of NAPL.

II. Primary Hazards: Lifting; slips, trips and falls; pinches; vehicular and foot traffic; insect/animal bites and stings; poisonous plants; inclement weather, chemical contamination; heavy equipment and underground utilities.

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____TtNUS
Equipment Inspection required ☐ Yes ☐ No Initials of Inspector _____TtNUS

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐

Respiratory equipment required
Yes ☐ Specify on the reverse
No ☒

Modifications/Exceptions: Minimum requirement include sleeved shirt and long pants, or coveralls, safety, glasses and safety footwear. Hard hats and hearing protection will be worn when working near operating equipment.

VI. Chemicals of Concern	Hazard Monitoring /Action Level(s)	Response Measures
VOCs such as bromoform and TCE	PID w/10.06eV lamp reading of 30ppm for no more than 4 excursions of 5 minutes in any one work day.	Suspend site activities and report to an unaffected area.

Primary Route(s) of Exposure/Hazard: inhalation, dermal, ingestion

(Note to FOL and/or SHSO: Each item in Sections VII, VIII, and IX must be checked Yes, No, or NA)

VII. Additional Safety Equipment/Procedures

Hard-hat	☒ Yes ☐ No	Hearing Protection (Plugs/Muffs)	☒ Yes ☐ No
Safety Glasses	☒ Yes ☐ No	Safety belt/harness	☐ Yes ☒ No
Chemical/splash goggles	☐ Yes ☐ No	Radio/Cellular Phone	☐ Yes ☒ No
Splash Shield	☐ Yes ☒ No	Barricades	☐ Yes ☐ No
Splash suits/coveralls	☐ Yes ☐ No	Gloves (Type – Work)	☒ Yes ☐ No
Impermeable apron	☐ Yes ☒ No	Work/rest regimen	☐ Yes ☒ No
Steel toe work shoes/boots	☒Yes ☐ No	Chemical Resistant Boot Covers	☐ Yes ☐ No
High visibility vest	☐Yes ☐ No	Tape up/use insect repellent	☐ Yes ☐ No
First Aid Kit	☐Yes ☐ No	Fire Extinguisher	☐ Yes ☐ No
Safety Shower/Eyewash	☐Yes ☐ No	Other	☐ Yes ☐ No

Modifications/Exceptions: Minimum requirement include sleeved shirt and long pants, safety footwear, and nitrile gloves Tyvek coverall to protect against splash and natural hazards (e.g., ticks) if working/walking through areas of high grass. Use insect repellants containing at least 10% DEET and tape up in such areas. Follow manufacturer's recommendations for proper application and reapplication. Hard hat when overhead hazards exist. Splash shield or goggles if splash hazard exists during O Red Dye field testing. Complete heavy equipment checklist

VIII. Site Preparation

	Yes	No	NA
Utility Locating and Excavation Clearance completed	☒	☐	☐
Vehicle and Foot Traffic Routes Established/Traffic Control Barricades/Signs in Place	☐	☐	☐
Physical Hazards Identified and Isolated (Splash and containment barriers)	☐	☐	☐
Emergency Equipment Staged (Spill control, fire extinguishers, first aid kits, etc).	☐	☐	☐

IX. Additional Permits required (Hot work, confined space entry, excavation etc.) ☐ Yes ☒ No
If yes, SHSO to complete or contact Health Sciences, Pittsburgh Office (412)921-7090

X. Special instructions, precautions: Preview work locations to identify potential hazards (slips, trips, and falls, natural hazards, etc.) Review PPE needs based on activities being performed and the associated hazards. Use safe lifting procedures and obtain assistance when handling heavy or awkward objects. Suspend site activities in the event of inclement weather. Observe site workers for signs and symptoms of heat/cold stress. Use sun block (SPF > 15) to prevent sunburn if necessary. If UXO is encountered, stop work, mark site with pin flag and notify NSF-IH EOD.

Permit Issued by: _____ Permit Accepted by: _____

SAFE WORK PERMIT
DECONTAMINATION ACTIVITIES
NAVAL SUPPORT FACILITY – INDIAN HEAD, MARYLAND

Permit No. _____ Date: _____ Time: From _____ to _____

I. Work limited to the following (description, area, equipment used): Decontamination of sampling equipment (i.e., reusable stainless steel trowels, etc.). Brushes and spray bottles will be used to decontaminate small sampling equipment.

II. Primary Hazards: Potential hazards associated with this task include chemical exposure, transfer of contamination, inclement weather, noise.

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____TtNUS
Equipment Inspection required ☐ Yes ☐ No Initials of Inspector _____TtNUS

V. Protective equipment required
Level D ☒ Level B ☐
Level C ☐ Level A ☐

Respiratory equipment required
Yes ☐ Specify on the reverse
No ☒

Modifications/Exceptions: Minimum requirement include sleeved shirt and long pants, safety glasses, safety footwear, and nitrile gloves. Impermeable aprons are preferred protection against soiling work clothes when lifting auger flights because of the need to carry close to the body. If it (impermeable apron) does not offer adequate protection, PVC rain suits or PE or PVC coated Tyvek should be employed. Chemical resistant boot covers if excessive liquids are generated or to protected footwear. PID with 10.6 or 11.04 eV lamp [Note: This instrument will be used to determine if any volatile contaminants have been removed. It will not be used for purposes of monitoring exposure.

VI. Chemicals of Concern	Hazard Monitoring	Action Level(s)	Response Measures
Decontamination Fluids	PID w/10.06 eV lamp	above BG levels	Re-wash and re-scan

Primary Route(s) of Exposure/Hazard: Inhalation and direct contact

(Note to FOL and/or SHSO: Each item in Sections VII, VIII, and IX must be checked Yes, No, or NA)

VII. Additional Safety Equipment/Procedures

Hard-hat	☐ Yes ☒ No	Hearing Protection (Plugs/Muffs)	☐ Yes ☒ No
Safety Glasses	☒ Yes ☐ No	Safety belt/harness	☐ Yes ☒ No
Chemical/splash goggles	☐ Yes ☒ No	Radio/Cellular Phone	☐ Yes ☒ No
Splash Shield	☒ Yes ☐ No	Barricades	☐ Yes ☒ No
Splash suits/coveralls	☐ Yes ☒ No	Gloves (Type – Nitrile)	☒ Yes ☐ No
Impermeable apron	☐ Yes ☐ No	Work/rest regimen	☐ Yes ☒ No
Steel toe Work shoes or boots	☒Yes ☐ No	Chemical Resistant Boot Covers	☐ Yes ☐ No
High Visibility vest	☐Yes ☐ No	Tape up/use insect repellent	☐ Yes ☐ No
First Aid Kit	☐Yes ☐ No	Fire Extinguisher	☐ Yes ☐ No
Safety Shower/Eyewash	☐Yes ☐ No	Other	☐ Yes ☐ No

Modifications/Exceptions: Chemical resistant boot covers if excessive liquids are generated or to protect footwear.

VIII. Site Preparation

	Yes	No	NA
Utility Locating and Excavation Clearance completed	☐	☐	☒
Vehicle and Foot Traffic Routes Established/Traffic Control Barricades/Signs in Place	☐	☐	☐
Physical Hazards Identified and Isolated (Splash and containment barriers)	☐	☐	☐
Emergency Equipment Staged (Spill control, fire extinguishers, first aid kits, etc).	☐	☐	☐

IX. Additional Permits required (Hot work, confined space entry, excavation etc.) ☐ Yes ☒ No
If yes, SHSO to complete or contact Health Sciences, Pittsburgh Office (412)921-7090

X. Special instructions, precautions: Suspend site activities in the event of inclement weather. Employ proper lifting techniques. When/where possible use heavy equipment to move and place containers.

Permit Issued by: _____ Permit Accepted by: _____

SAFE WORK PERMIT
IDW MANAGEMENT
NAVAL SUPPORT FACILITY – INDIAN HEAD, MARYLAND

Permit No. _____ Date: _____ Time: From _____ to _____

SECTION I: General Job Scope

I. **Work limited to the following (description, area, equipment used):** IDW management activities includes containerization, staging, monitoring for leaks of IDW accumulated wastes. Wastes types include soil cutting, purge and decontamination wash waters.

II. **Primary Hazards:** Lifting, pinches and compressions; flying projectiles; slips, trips, and falls and chemical contamination.

III. **Field Crew:** _____

IV. **On-site Inspection conducted** ☐ Yes ☐ No Initials of Inspector _____TtNUS
Equipment Inspection required ☐ Yes ☒ No Initials of Inspector _____TtNUS

SECTION II: General Safety Requirements (To be filled in by permit issuer)

V. **Protective equipment required**
Level D ☒ Level B ☐
Level C ☐ Level A ☐

Respiratory equipment required
Yes ☐ See Reverse
No ☒

Modifications/Exceptions: None anticipated

VI.

Chemicals of Concern	**Hazard Monitoring /Action Level(s)**	**Response Measures**
VOCs such as bromoform and TCE	PID w/10.06eV lamp reading of 30ppm for no more than 4 excursions of 5 minutes in any one work day.	Suspend site activities and report to an unaffected area.

Primary Route of Exposure/Hazard: NA

(Note to FOL and/or SHSO: Each item in Sections VII, VIII, and IX must be checked Yes or No)

VII. **Additional Safety Equipment/Procedures**

Hard-hat	☐ Yes ☐ No	Hearing Protection (Plugs/Muffs)	☐ Yes ☒ No
Safety Glasses	☐ Yes ☐ No	Safety belt/harness	☐ Yes ☒ No
Chemical/splash goggles	☐ Yes ☒ No	Radio/Cellular Phone	☐ Yes ☐ No
Splash Shield	☐ Yes ☒ No	Barricades	☐ Yes ☐ No
Splash suits/coveralls	☐ Yes ☒ No	Gloves (Type – Leather/Cotton)	☒ Yes ☐ No
Impermeable apron	☐ Yes ☒ No	Work/rest regimen	☐ Yes ☒ No
Steel toe work shoes/boots	☒ Yes ☐ No	Chemical Resistant Boot Covers	☐ Yes ☒ No
High visibility vest	☐ Yes ☐ No	Tape up/use insect repellent	☐ Yes ☐ No
First Aid Kit	☒ Yes ☐ No	Fire Extinguisher	☐ Yes ☐ No
Safety Shower/Eyewash	☐ Yes ☒ No	Other	☐ Yes ☐ No

Modifications/Exceptions: If you are using pneumatic/electric power to open drums – Safety glasses are required; If power equipment is employed to move drums or you are working near operating equipment hard hats will be employed. Tyvek coverall to protect against natural hazards (e.g., ticks) if working/walking through areas of high grass. Use insect repellants containing at least 10% DEET if necessary. Follow manufacturer's recommendations for proper application and reapplication. If working in areas where snakes are a threat, wear snake chaps to protect against bites. High visibility vest if near active traffic areas.

VIII. **Site Preparation**

	Yes	No	NA
Utility Locating and Excavation Clearance completed	☐	☐	☒
Vehicle and Foot Traffic Routes Established/Traffic Control Barricades/Signs in Place	☐	☐	☐
Physical Hazards Identified and Isolated	☐	☐	☐
Emergency Equipment Staged (Spill control, fire extinguishers, first aid kits, etc).	☐	☐	☐

IX. **Additional Permits required** (Hot work, confined space entry, excavation etc.) ☐ Yes ☒ No
If yes, SHSO to complete or contact Health Sciences, Pittsburgh Office (412)921-7090

X. **Special instructions, precautions:** Suspend site activities in the event of inclement weather. Employ proper lifting techniques. When/where possible use heavy equipment to move and place containers. When placing drums – Place the label and retention ring nut on the outside where it is readily visible. Place 4-drums to a pallet. Maintain a minimum distance of 4-feet between pallet rows. An IDW inventory shall be generated to provide the number of drums, contents, and volumes. This inventory should be provided to the facility contact. Inspect equipment prior to use.

Permit Issued by:_____ Permit Accepted by:_____

SAFE WORK PERMIT

SURVEYING ACTIVITIES

NAVAL SUPPORT FACILITY – INDIAN HEAD, MARYLAND

Permit No. _____ Date: _____ Time: From _____ to _____

I. Work limited to the following (description, area, equipment used): surveying activities

II. Primary Hazards: Lifting; slips, trips and falls; vehicular and foot traffic; insect/animal bites and stings; poisonous plants; inclement weather.

III. Field Crew: _____

IV. On-site Inspection conducted ☐ Yes ☐ No Initials of Inspector _____ TtNUS

Equipment Inspection required ☐ Yes ☐ No Initials of Inspector _____ TtNUS

V. Protective equipment required

Level D ☒ Level B ☐

Level C ☐ Level A ☐

Respiratory equipment required

Yes ☐ Specify on the reverse

No ☒

Modifications/Exceptions: Minimum requirement include sleeved shirt and long pants, or coveralls, safety, glasses and safety footwear. Hard hats and hearing protection will be worn when working near operating equipment.

VI. Chemicals of Concern	**Hazard Monitoring**	**Action Level(s)**	**Response Measures**
None anticipated given the nature of activities and limited contact with media	None	None	None

Primary Route(s) of Exposure/Hazard: NA

(Note to FOL and/or SHSO: Each item in Sections VII, VIII, and IX must be checked Yes, No, or NA)

VII. Additional Safety Equipment/Procedures

Hard-hat	☐ Yes ☐ No	Hearing Protection (Plugs/Muffs)	☐ Yes ☐ No
Safety Glasses	☐ Yes ☐ No	Safety belt/harness	☐ Yes ☒ No
Chemical/splash goggles	☐ Yes ☒ No	Radio/Cellular Phone	☐ Yes ☒ No
Splash Shield	☐ Yes ☒ No	Barricades	☐ Yes ☒ No
Splash suits/coveralls	☐ Yes ☒ No	Gloves (Type – Work)	☒ Yes ☐ No
Impermeable apron	☐ Yes ☒ No	Work/rest regimen	☐ Yes ☒ No
Steel toe work shoes/boots	☒ Yes ☐ No	Chemical Resistant Boot Covers	☐ Yes ☐ No
High visibility vest	☐ Yes ☐ No	Tape up/use insect repellent	☐ Yes ☐ No
First Aid Kit	☐ Yes ☐ No	Fire Extinguisher	☐ Yes ☐ No
Safety Shower/Eyewash	☐ Yes ☐ No	Other	☐ Yes ☐ No

Modifications/Exceptions: Tyvek coverall to protect against natural hazards (e.g., ticks) if working/walking through areas of high grass. Use insect repellants containing at least 10% DEET and tape up in such areas. Follow manufacturer's recommendations for proper application and reapplication. Hard hat when overhead hazards exist. Safety glasses when near eye hazards.

VIII. Site Preparation

	Yes	No	NA
Utility Locating and Excavation Clearance completed	☐	☐	☐
Vehicle and Foot Traffic Routes Established/Traffic Control Barricades/Signs in Place	☐	☐	☐
Physical Hazards Identified and Isolated (Splash and containment barriers)	☐	☐	☐
Emergency Equipment Staged (Spill control, fire extinguishers, first aid kits, etc)	☐	☐	☐

IX. Additional Permits required (Hot work, confined space entry, excavation etc.) ☐ Yes ☒ No

If yes, SHSO to complete or contact Health Sciences, Pittsburgh Office (412)921-7090

X. Special instructions, precautions: Preview work locations to identify potential hazards (slips, trips, and falls, natural hazards, etc.) Review PPE needs based on activities being performed and the associated hazards. Use safe lifting procedures and obtain assistance when handling heavy or awkward objects. Suspend site activities in the event of inclement weather. Observe site workers for signs and symptoms of heat/cold stress. Use sun block (SPF > 15) to prevent sunburn if necessary.

Permit Issued by: _____ Permit Accepted by: _____

ATTACHMENT VI

OSHA POSTER

Job Safety and Health

It's the law!

OSHA

Occupational Safety and Health Administration
U.S. Department of Labor

EMPLOYEES:

- You have the right to notify your employer or OSHA about workplace hazards. You may ask OSHA to keep your name confidential.
- You have the right to request an OSHA inspection if you believe that there are unsafe and unhealthful conditions in your workplace. You or your representative may participate in that inspection.
- You can file a complaint with OSHA within 30 days of retaliation or discrimination by your employer for making safety and health complaints or for exercising your rights under the *OSH Act*.
- You have the right to see OSHA citations issued to your employer. Your employer must post the citations at or near the place of the alleged violations.
- Your employer must correct workplace hazards by the date indicated on the citation and must certify that these hazards have been reduced or eliminated.
- You have the right to copies of your medical records and records of your exposures to toxic and harmful substances or conditions.
- Your employer must post this notice in your workplace.
- You must comply with all occupational safety and health standards issued under the *OSH Act* that apply to your own actions and conduct on the job.

EMPLOYERS:

- You must furnish your employees a place of employment free from recognized hazards.
- You must comply with the occupational safety and health standards issued under the *OSH Act*.

This free poster available from OSHA –
The Best Resource for Safety and Health

Free assistance in identifying and correcting hazards or complying with standards is available to employers, without citation or penalty, through OSHA-supported consultation programs in each state.

1-800-321-OSHA
www.osha.gov

OSHA 3165-12-06R

Appendix B

Laboratory SOPs

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP100 REVISION #: 21 EFFECTIVE DATE: 20100901

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: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/9/10

Changes Summary

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 litter bottles. Samples should be preserved with concentrated HNO_3 to a pH <2 immediately upon sampling. If dissolved metals are to be analyzed the sample should be filtered before the HNO_3 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.

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 (HNO_3). 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 (H_2SO_4). 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 HNO_3, 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 HNO_3, 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% HNO_3 and 5% HCl. Use 0.125 mLs HNO_3 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 transfered 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 then 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% HNO_3 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 HNO_3 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 HNO_3 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 HNO_3 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 HNO_3. **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 HNO_3 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 HNO_3 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 HNO_3 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 HNO_3 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 HNO_3 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 homogenity. 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 equivelent 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 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°C ± 5°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°C ± 5°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 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 aliquot must be added to the extract after filtration but before preservation.)

d. The CLH project requires that a high and a low spike be prepared and analyzed. Spikes shoud 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 LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 103 **REVISION #: 18** **EFFECTIVE DATE: 041110**

MERCURY ANALYSIS IN WATER
BY MANUAL COLD VAPOR TECHNIQUE
METHODS USEPA SW846 7470A and 245.1 CLP-M 4.1
(NJDEP DOES NOT ACCEPT CLPILM 04.1 AFTER JUNE, 2003),
ADDENDUM FOR USEPA CLP ILM 05.2

APPROVALS:

Lab Director: _____ Date: 4/12/10

Data Quality Manager: _____ Date: 4/11/10

Section Supervisor: Betty DeVille Date: 4/13/10

Changes Summary

Revision 18, 04/11/10

- The SOP is an update from Revision 17 dated 03/25/10
- 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.
- Tables have been updated to reflect the current limits/processes.

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1.0 Identification of the Test Method

This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury, and is compliant with SW846 Method 7470A, USEPA Method 245.1, and USEPA SOW ILM04.1.

2.0 Applicable Matrix or Matrices

This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

3.0 Detection Limit

Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

Limits Table

Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 7470A, SOW 4.1 & 5.2	**AQUEOUS MDL/DL (ug/L)**	**AQUEOUS LOD (ug/L)**	**AQUEOUS ERL/LOQ (ug/L)**	**AQUEOUS CRQL ILMO 4.1 (ug/L)**	**AQUEOUS CRQL ILMO 5.2 (ug/L)**
Mercury	0.080	0.16	0.20	0.20	0.20

Wavelength Table

ANALYTE	WAVELENGTH
Mercury	**253.7**

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. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

4.2 This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in mobility-procedure extracts, aqueous wastes, and ground waters. This method can also be used for sludge-type wastes. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

4.3 In addition to inorganic forms of mercury, organic materials may also be present. These organo-mercury compounds will not respond to the cold vapor atomic absorption technique unless they are first broken down and converted to mercuric ions. Potassium permanganate oxidizes many of these compounds, but recent studies have shown that a number of organic mercurials, including phenol mercuric acetate and methyl mercuric chloride, are only partially oxidized by this reagent. Potassium persulfate has been found to give approximately 100% recovery when used as the oxidant step following the addition of the permanganate has been included to insure that organo-mercury compounds, if present, will be oxidized to the mercuric ion before measurement. A heat step is required for methyl mercuric chloride when present in or spiked to a natural system. For distilled water the heat step is not necessary.

4.4 The range of the method may be varied through instrument and/or recorder expansion. Using a 30 mL sample, a detection limit of 0.2 µg Hg/L can be achieved.

4.3 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

The flameless AA procedure is a physical method based on the absorption of radiation at 253.7 nm by mercury vapor. Organic mercury compounds are oxidized and the mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of a flow injection Mercury system. Absorbance (peak height) is measured as a function of mercury concentration and recorded in the usual manner.

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.

6.2 Refer to SOP-431 for common definitions.

7.0 Interferences

7.1 Possible interference from sulfide is eliminated by the addition of potassium permanganate. Concentrations as high as 20 mg/L of sulfide as sodium sulfide do not interfere with the recovery of added inorganic mercury from distilled water.

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

7.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganate (as much as 6.25 mL in 30 mL of sample). During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253 nm. Care must be taken to assure that free chlorine is absent before the mercury is reduced and swept into the cell. This is accomplished by using an excess of hydroxylamine sulfate reagent (6.25 mL to 30 mL of sample).

7.4 Samples containing high concentrations of oxidizable organic materials, as evidenced by high chemical oxygen demand values, may not be completely oxidized of organic mercury will be low. The problem can be eliminated by reducing the sample volume or by increasing the amount of potassium persulfate (and consequently stannous chloride) used in the digestion.

8.0 Safety

8.1. Normal accepted laboratory practices should be followed while performing this procedure.

8.2. The toxicity and carcinogenicity of each reagent in this method have not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

8.3 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

8.4 The analyst should make sure that the system is vented to fresh permanganate in a bottle located at the back. Otherwise Hg vapors could be vented to the room.

9.0 Equipment & Supplies

9.1 Perken Elmer Flow injection Mercury system.

9.2 Mod Block Digester set to maintain 95$\pm$2°C for 2 hours.

9.3 Polypropylene sample digestion vessels with snap or screw caps or equivalent. Five vessels of each lot of digestion vessels must be taken through analysis to check for mercury.

10.0 Reagents and Standards

Reagent 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 Spex, Ultra Scientific 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 room temperature.

10.1 REAGENTS

10.1.1 Concentrated sulfuric acid suitable for Hg determination.
10.1.2 Concentrated nitric acid suitable for Hg determination.
10.1.3 Stannous chloride: In a 1000 mL volumetric flask add approximately 500 mLs D.I. water, 30 mLs concentrated HCl, add 11 grams stannous chloride crystals swirl to mix and dilute to 1000 mLs. Prepare fresh daily.
10.1.4 3% HCl Carrier Solution: Dilute 30 mLs of concentrated metals grade HCl to one liter. Prepare fresh daily.
10.1.5 Sodium chloride-hydroxylamine chloride solution: Dissolve 120 grams of sodium chloride and 120 grams of hydroxylamine hydrochloride (very high grade --Do not get from Tennessee Reagents) in D.I. water and dilute to 1 liter. Note: this is normally made up 2 Liters at a time.
10.1.6 Potassium permanganate: 5% solution, w/v: dissolve 200 grams of potassium permanganate in 4000 mLs of D.I. water. Should have "suitable for mercury determination" written on the side of the potassium permanganate bottle. This reagent takes overnight stirring (minimum of 3 hours if absolutely necessary). Use stirring bar already in the reagent bottle for this purpose. It is very easy to contaminate with mercury.
10.1.7 Potassium persulfate: 5% solution, w/v: dissolve 100 grams of potassium persulfate in 2000 mLs D.I. water. Slight heating with stirring may be necessary to completely dissolve. The formation of crystals in this solution is not a problem.

10.2 STANDARDS

10.2.1 Traceability

10.2.1.1 All reference materials are given a unique identifier within Element and labeled with the Element #. This 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 within Element as well as on the container's label.
10.2.1.2 All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded within Element. 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.
10.2.1.3. **NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All standards, blanks, and samples are taken through the digestion process.
10.2.1.4 Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique Element identifier.

10.2.1.5 Primary source and secondary source mercury standard solutions at 200 ug/L: dilute 2 mLs of stock solution to 1000 mLs in a 1000 mL volumetric flask, with 1.5 mLs concentrated HNO_3. This solution is recorded in Element and given a unique Element identifier.

10.2.2 Calibration Standards

Prepared from the primary source working standard. The preparation of the calibration standards, etc. is described below.

10.2.2.1 Dilute the volumes below to 30 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 10 mLs for the initial step of the digestion. From that point when 25 mLs of DI water are added to samples, 15 mLs of DI water is added to the standards.

ug/L Hg	mLs of 200 ug/L standard in 30 mLs
0.20	0.03
0.50	0.075
1.0	0.15
2.0	0.30
4.0	0.60
6.0	0.90
10.0	1.5

10.2.2.2 Appropriate reagents are added as below in the sample preparation section.

10.2.2.3 Prepare one vessel for each.

10.2.2.4 It is necessary to digest the calibration standards.

10.2.3 Calibration Verification Standards

10.2.3.1. Initial calibration verification (ICV) solution – 4.0 ug/L

10.2.3.1.1 Prepared by diluting 0.6 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel. (TV = 4.0 ug/L)

10.2.3.1.2 Appropriate reagents are added as below in the sample preparation section.

10.2.3.1.3 It is necessary to digest the ICV standards for Method 7470A, Method 245.1 does not require digestion of standards.

10.2.3.2 Continuing calibration verification (CCV) solution

10.2.3.2.1 Prepared from the primary source standard.

10.2.3.2.2 Prepared by diluting 0.3 mL of the primary standard at 200 ug/L to 30 mLs with reagent water in a 70 mL polypropylene vessel for 2.0 ug/L or 0.6 ml to 30 mls for 4.0 ug/L.

10.2.3.2.3 Appropriate reagents are added as below in the sample preparation section.

10.2.3.2.4 It is necessary to digest the CCV standards for Method 7470A, Method 245.1 does not require digestion of standards.

10.2.4 Digestion standards

10.2.4.1 Blank Spike

10.2.4.1.1 Prepared from the secondary source standard.

10.2.4.1.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with reagent water in a 70 mL polypropylene vessel.

10.2.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.1.4 This solution should be given a unique identifier within Element.

10.2.1.2 Matrix Spikes

10.2.1.2.1 Prepared from the secondary source working standard.

10.2.1.2.2 Prepared by diluting 0.3 mL of the second source standard to 30 mL with sample in a 70 mL polypropylene vessel. Project specific or method specific requirements may over-ride the spiking level.

10.2.1.2.3 Appropriate reagents are added as below in the sample preparation section.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 Samples are preserved by acidification with nitric acid to a pH of 2 or lower immediately at the time of collection, and refrigeration to 4°C.

11.2 The holding time for the mercury digestion is 28 days from time 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.

12.2 An initial demonstration must be performed by each analyst performing this method. Four BS’s are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 (Reference SW-846, 7470A Update III, USEPA CLP ILMO 4.1 or 245.1, Rev 3.0, 5/94 for further clarification)

12.4 Daily

12.4.1. **The instrument must be calibrated daily for all projects.**

12.4.2 Begin each analysis with an ICV(QCS) second source. The control limits are ±10% and IPC (CCV) for 245.1, limits are ± 5% and subsequent analyses are ± 10%.

12.4.3 Analyze ICB. Control limits (<±MDL for USACE or ±RL/CRDL for others and CLP)., depending on method. **No analyte detected >2xMDL for DOD.**

12.4.4 If the ICV (QCS) is not in control a new curve must be analyzed prior to sample analysis.

12.4.5 If the IPC (initial CCV) for 245.1 is not within the limits of ±5%, try preparing another undigested CCV and reanalyzing before recalibrating. If this fails then a recalibration is necessary.

12.4.6 Follow each set of 10 samples with a CCV and also must end up with a CCV after the last sample. The control limits are ±20% for SW846-7470 and ± 10% for 245.1.

12.6.7 A CCB must always follow a CCV, the control limit is (<±MDL for USACE or ±RL/CRDL for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte detected >2xMDL for DOD.**

12.5 Quarterly or as needed when doing straight CLP work.

12.5.1 IDL's for CLP 4.1.

12.6 Digestion

12.6.1 BS data should be maintained and available for easy reference or inspection.

12.6.2 BLK (<1/2 ±RL or ±RL/CRDL for common contaminates (DOD) and ±RL/CRDL for others and CLP).

12.6.2.1 Employ a minimum of one preparation blank (BLK) per sample batch to determine if contamination or any memory effects are occurring. The BLK is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be below the method detection limit. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit nor if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit but less than 10X the level found in the preparation blank, the sample must be re-digested and re-analyzed or the data must be qualified on the final report. The project manager or QA manager will make this determination.

12.6.3 Laboratory control sample (BS)

12.6.3.1. Employ a minimum of one laboratory control sample (BS) per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are ±20% for SW846-7470 and ±15% for 245.1. If the BS is not in control, the impact upon the client data should be evaluated and the associated sample(s) should be either re-digested or the data should be qualified. The project manager or QA Officer will make this determination.

12.7 Sample matrix:

12.7.1 Analyze one replicate sample for every twenty samples. 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. CLP does not allow this. Project specific requirements will take precedence in these situations.

12.7.2 Analyze one spiked sample and spiked sample duplicate for every twenty samples. A replicate sample is a sample brought through the whole sample preparation and analytical process in duplicate. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within ±25% of the true value (**± 20% for DOD projects**). If not, check with supervisor to determine appropriate action. The final analytical report must document this situation.

NOTE: For TCLP extracts, a matrix spike must be performed for each different matrix. The method of standard additions must be used if the sample spike recovery is not at least 50% and the concentration of Hg does not exceed the regulatory level and if the concentration of Hg measured in the extract is within 20% of the regulatory level.

12.7.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of ± 20% RPD shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation.

12.7.4 For 245.1 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be ±10%. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.

12.7.5 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

13.0 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.

13.1.1 Perkin Elmer Flow Injection Mercury System (FIMS).

13.1.1.1. Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the separator cover must not have any moisture in it. If it does replace it. **(Perkin-Elmer tygon tubing, waste**

and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)

13.1.1.2 Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)

13.1.1.3 Turn on PE 100 spectrophotmeter; (Note: this must be on in order to start up the software on the computer.)

13.1.1.4 Turn on computer and go to icon "AA Win LAB Analyst".

13.1.1.5 Go to method; select "Hg CAL 2" then OK.

13.1.1.6 Wavelength = 253.7; smoothing points =9; measurement = peak height; read time =18sec.; BCC time = 2 sec.

13.1.1.7 Go to "Sample Info" and enter the order of the samples and other information that may be needed.

13.1.1.8 Save entered sample list under "Save ….sample info file" Note: description and batch ID are normally the date of analysis.

13.1.1.9 Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information" File. The other is to select a results library. Double click on heading and choose.

13.1.1.10 Turn the printer on.

13.1.1.11 Connect all tubing to the pump and blocks.

13.1.1.12 Start the pump by going to "FIAS" and click the pump 1 Icon (120).

13.1.1.13 The pump will start, then lock down and tighten the tubes onto the pump.

13.1.1.14 Turn on the nitrogen tank, it should be above 500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.

13.1.1.15 The pressure gauge on the PE100 should be just below 100.

13.1.1.16 Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.

13.1.1.17 Adjust the spring tension tubing until there is a constant "bubble of low rate" coming out to the waste tube.

13.1.1.18 Place carrier tubes into carrier and stannous chloride tube into SnCl2. (Click the valve fill inject and make sure flow is correct and the line is rinsed).

13.1.1.19 Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.

13.1.1.20 Allow a few minutes for reagents to flow through the system before starting analysis.

13.1.1.21 Calibrate: Go to "Auto" click on "Analyze", click on "calibrate".

13.1.1.22 "Select Location" enter #'s to be ran, and then press "OK". Samples are done in increments of 10 samples

13.2 Analyze the calibration standards as below.

13.2.1 New calibration points must be analyzed when the ICV analysis is not within ± 5%. **A curve must be analyzed daily for all projects especially USACE and CLP projects.**

13.2.2 The curve should be linear with a calculated intercept with a minimum correlation coefficient (r) of $\geq$0.995 (USACE) or 0.998 (other). If not, a new curve must be analyzed.

14.0 Procedure

14.1 Glassware preparation

14.1.1 After use, samples are neutralized and disposed down an acid sink with running water and rinsed with tap water. Or the sample may be discarded into the Mercury waste drum.

14.1.2 Acid clean the glassware used for mercury prep as follows:

14.1.2.1 Rinse with low Hg content 1:1 HCl.

14.1.2.2 Rinse with D.I. water.

14.2 Label the vessels indicating which sample will be in each.

14.3 Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below.

14.4 Sample preparation

14.4.1. Transfer 30 mLs, or an aliquot diluted to 30 mLs of sample to the 30 mL mark on a 50 mL digestion vessel previously marked for this sample.

NOTE: Normally, **a**n automatic dilution of 10X to 100X is performed for all TCLP extracts. All TCLP samples get one matrix spike unless several come in at one time from the same client with the same matrix. Then one in ten of the same matrix gets spiked. Check with your manager.

14.4.2 Add 1.5 mLs of concentrated sulfuric acid to each vessel and mix.

14.4.3 Add 0.75 mL of concentrated nitric acid to each bottle and mix.

14.4.4 Add 4.5 mLs potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 7.5 mLs). If the purple color does not persist after the addition of 7.5 mLs $KMnO_4$ the sample must be diluted prior to digestion. Inform your manager that the minimum detection limit cannot be reached for that particular matrix.

NOTE: The same amount of $KMnO_4$ added to the samples should be present in the standards and blanks.

14.4.5 Add 2.4 mLs of potassium persulfate to each vessel and mix. Cover.

14.4.6 Heat for 2 hours in the block digester at 95$\pm$2°C (the block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool.

14.4.7 Samples may be saved at this point if there is not time to run the whole set that day.

NOTE: Stannous Chloride (VII. A 5.) and 3% HCl (VII. A 8.) are added by the instrument during analysis.

14.5 Sample analysis

14.5.1 Set up the instrument as described in the calibration section above.

14.5.2 When ready to run samples, add 1.8 mLs of sodium chloride-hydroxylamine chloride to reduce the excess permanganate. Sample analysis must be preceded by the analysis of an ICV with control limits of $\pm$10% for SW846-7470 and ±5 % for 245.1. Followed by the ICB (< ±MDL for USACE or ±RL/CRDL for others and CLP).

14.5.3 Each set of ten samples and at the end of the analytical run must be followed by a CCV with control limits of $\pm$20% for SW846-7470 and ±10% for 245.1.

14.5.4 CCB must always follow the CCV. Control limits are (<±MDL for USACE or ±RL/CRDL for others and CLP). CCB must be run at the beginning and end of a sequence and after every 10 samples. **No analyte must be detected >2xMDL for DOD.**

14.5.5 The auto-sampler log is set up to analyze 106 samples at a time.
Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ-ICV
10	SEQ-ICB
11	BS

AS LOC	Sample ID
12	BLK
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	SEQ-CCV
22	SEQ-CCB

23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	SEQ-CCV
34	SEQ-CCB

14.6 Data Reporting

14.6.1 Reduce data to result which will be reported.

14.6.2 Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

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 Apply a least squares fit to the calibration standards plotting µg Hg/L versus the absorbance. For the concentration of the standards, assume 30 mL of solution volume (the 0.1 µg Hg standard will be input as 1.0 µg Hg/L)
(0.1µg Hg / 0.030 L solution).

15.3 Input the sample absorbance into the mercury spreadsheet making sure that you are using the correct spreadsheet for the matrix of the sample.

15.4 Also make sure that the appropriate dilution factor is inputted in the correct space on the spreadsheet.

15.5 Report the data as µg Hg/L of sample.

16.0 Method Performance

16.1 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-BS samples. The data is calculated for accuracy and precision requirements. The DOC form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

DOC BS Preparation: Dilute 0.3 mL of the second source standard to 30 mLs with reagent water in a 70 mL polypropylene vessel. Follow SOP procedure for preparation and analysis steps.

DOC Accuracy and Precision Criteria: The four BS's for the DOC need to be within the methods recovery ranges. Duplicates should be below 20% relative percent difference.

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

19.1 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.

19.2 CORRECTIVE ACTIONS: INSTRUMENT RELATED

19.2.1 ICV (**QCS for 245.1**)- second source not within ± 10%.

A. If the problem is with the solution, re-prepare, obtain new stock if necessary.

B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and recheck ICV.

19.2.2 CCV not within ± 20% for SW846 and ±10% for (**245.1, ± 5% for initial IPC and + 10% for subsequent IPCs**)

A. If the problem is with the solution, re-prepare, obtain new stock if necessary.

B. If the problem is with the calibration, recalibrate through analysis of appropriate standards and re-prepare/reanalyze the previous ten sample according the following guidelines.

1. If the CCV was biased high, any of the previous ten samples which were below the detection limit do not require reanalysis.
2. If the CCV was biased low, the previous ten samples must be reanalyzed.

19.3 CORRECTIVE ACTION: DIGESTION RELATED

19.3.1 The preparation blank less than <1/2 RL or ±RL/CRDL for common contaminates (DOD) and ±RL/CRDL for others and CLP.

A. If the problem is with the instrument or stannous chloride.
Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, re-prepare the stannous chloride or determine if there are any problems with the instrument. Contact supervisor immediately.

B. If the problem is with the digestion.
All associated samples which are below the RL, CRDL or have a level of mercury greater than 5X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not BMDL nor greater than 5X the level found in the preparation blank, the sample must be re-digested/re-analyzed or reported as qualified. The project manager or QA manager will make this determination.

C. LCS not within control limits (or ±20%, ± 15% for **245.1**).

1. If the problem is with the instrument, reanalyze when instrument is in control if further sample bottles are available.
2. Is the problem is with the digestion.
   a. If biased low, associated samples must be re-digested.
   b. 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.

19.4 CORRECTIVE ACTION: SAMPLE MATRIX RELATED

19.4.1 Replicate analysis RPD not within ±20%
The associated sample data must be qualified on the final report.

19.4.2 Spike analysis recovery not within ±25%(**± 20% for DOD projects**)

A. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

B. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. TCLP extracts must be evaluated as in section XI.D.2 above. The associated sample data must be qualified on the final report.

19.4.3 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6 of SW846-7000A provides tests to evaluate the need for using the MSA.

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.

20.2 Please see Waste Disposal SOP-405, for instruction of proper 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.

21.0 References

21.1 *Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III)*; Method 7470A.

21.2 *USEPA Code of Federal Regulations, 40, CH 1,PT 136; Method 245.1; APX-B.*

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

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, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

APPENDIX:

ADDENDUM FOR USEPA SOW ILM05.2

1. The CCV concentration must be different from the ICV.

2. The same CCV shall be used throughout analysis for an SDG.

3. Calibration standards must be within 5% of the standard concentration.

4. A CRA must be analyzed after the ICV/ICB and after each batch of 20 samples, but before the final CCV/CCB. The control limit is ±30%.

5. Spike samples at 1 ug/L for water.

Table 1

Aqueous Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 7470A, SOW 4.1 & 5.2	**AQUEOUS MDL/DL (ug/L)**	**AQUEOUS LOD (ug/L)**	**AQUEOUS ERL/LOQ (ug/L)**	**AQUEOUS CRQL ILMO 4.1 (ug/L)**	**AQUEOUS CRQL ILMO 5.2 (ug/L)**
Mercury	0.080	0.16	0.20	0.20	0.20

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Initial calibration (ICAL)	• Daily ICAL prior to sample analysis • Low standard at the RL/LOD level	• If more than one calibration standard is used, r $\geq$ 0.995 • Must follow curve processing requirements from SOP QS08	• Re-run curve • Check instrument for maintenance needs Samples cannot be analyzed until there is a passing calibration
Second source calibration verification (ICV)	Once after each ICAL, prior to beginning a sample run.	Must be within $\pm$ 10% of true value	• Re-run ICV • Repeat ICAL
Continuing calibration verification (CCV)	• After every 10 field samples and at the end of analysis sequence.	• $\pm$ 20% of true value	• Correct problem, rerun CCV. If that fails, then repeat ICAL. Reanalyze all samples since the last successful CCV.
Method Blank (BLK)	One per prep batch	No analytes detected > ½ RL and greater that 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	• Re-analysis to confirm the positive value • Notify the PM for further action • Re-prep of samples associated with the BLK • NCR will be required for data reported
Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	Correct problem. Re-analyze calibration blank. All samples following the last acceptable calibration blank must be reanlyzed.
BS	One per prep batch	Most stringent criteria listed within the LIMS.	• Re-analyze to confirm failed. • Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. • NCR will be required for data reported
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year	• Calculated value must be less than the Spike level .	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • the response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	• Bias Requirement: Inorganics 50-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

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. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. 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)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):	
Batch Number(s):	**Sequence ID:**
Method:	**7470A/245.1 (Mercury)**

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 meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation Blank (BLK) below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath 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: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 104 **REVISION #: 19** **EFFECTIVE DATE: 041110**

MERCURY ANALYSIS IN SOIL/SEDIMENT BY MANUAL COLD VAPOR TECHNIQUE METHODS SW846 7471A 7471B, EPA 245.5 AND CLPILM 04.1 (NJDEP DOES NOT ACCEPT CLPILM 04.1 AFTER JUNE, 2003), ADDENDUM FOR USEPA CLP ILM 05.2

APPROVALS:

Lab Director: _____ Date: 4/12/10

Data Quality Manager: _____ Date: 4/11/10

Section Supervisor: Betty DeVille Date: 4/13/10

Changes Summary
Revision 19, 04/11/10

- The SOP is an update from Revision 18 dated 03/25/10.

Revision 18, 03/08/10

- The SOP is an update from Revision 17 dated 01/29/09.
- 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.
- Numerous improvements/modifications were made to this SOP. Details/specifications were added that require evaluation from start to finish.

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1.0 Identification of the Test Method

1.1 This SOP is compliant with USEPA method 245.5, SW-846 method 7471A&B and CLP SOW ILM04.1.

2.0 Applicable Matrix or Matrices

2.1 This procedure measures total mercury (organic and inorganic) in soils, sediments, bottom deposits and sludge type materials.

3.0 Detection Limit

3.1 The range of the method is 0.2 to 2 µg/g. The range may be extended above or below the normal range by increasing or decreasing sample size or through instrument and recorder control.

3.2 Method Detection Limit (MDL), Empirical Laboratories' Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength:

Limits Table

Soil/Solid Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)					
Mercury by EPA 245.1, 245.5 7471A, SOW 4.1 & 5.2	**SOLID/SOIL MDL/DL (mg/Kg)**	**SOLID/SOIL LOD (mg/Kg)**	**SOLID/SOIL ERL/LOQ (mg/Kg)**	**SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)**	**SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)**
Mercury	0.013	0.026	0.033	0.10	0.10

Wavelength Table

ANALYTE	WAVELENGTH
Mercury	**253.7**

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. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

4.2 This method is a cold-vapor atomic absorption procedure for determining the concentration of mercury in soils, sediments, bottom deposits, and sludge-type materials. All samples must be subjected to an appropriate dissolution procedure prior to analysis.

4.3 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 A weighed portion of the sample is acid digested for 2 minutes at 95±2°C, followed by oxidation with potassium permanganate and with a secondary digestion at 95°C for 30 minutes. Mercury in the digested sample is then measured by the conventional cold vapor technique.

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.

6.2 Refer to SOP-431 for common definitions.

7.0 Interferences

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

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

7.3 Samples high in chlorides require additional permanganate (as much as 12.5 mLs) because, during the oxidation step, chlorides are converted to free chlorine, which also absorbs radiation of 253 nm. Care must therefore be taken to ensure that free chlorine is absent before the mercury is reduced and swept into the cell.

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

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 Normal accepted laboratory practices should be followed while performing this procedure.

8.3 The toxicity and carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure to these compounds should be minimized by good laboratory practices. Normal accepted laboratory safety practices should be followed during reagent preparation and instrument operation. Always wear safety glasses or full-face shield for eye protection when working with these reagents. Each laboratory is responsible for maintaining a current safety plan, a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method.

8.4 Mercury compounds are highly toxic if swallowed, inhaled, or absorbed through the skin. Analyses should be conducted in a laboratory exhaust hood. The analyst should use chemical resistant gloves when handling concentrated mercury standards.

9.0 Equipment & Supplies

9.1 Perkin Elmer Flow Injection Mercury System (FIMS).
9.2 Perkin Elmer AS 90.
9.3 Mercury lamp.
9.4 Environmental Express Mod-Block digestion block capable of holding 95+2°C for 2 hours.
9.5 A scale or balance capable of weighing to 0.01 + 0.02 gram.
9.6 Snap cap digestion polypropylene vessels for use with the mod block digester. Five vessels of each lot must be taken through analysis to check for mercury.
9.7 Polypropylene watch glasses suitable for use with the above vessels in F above.
9.8 Manual Sample Mill
9.9 Wiley Sample Mill
9.10 Clippers for cutting vegetation

10.0 Reagents and Standards

10.0.1 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:

10.0.2 Reagent 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 Spex, 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.

10.1 REAGENTS

10.1.1 Reagent Water: Reagent water will be interference free. All references to water in this method refer to reagent water unless otherwise specified.

10.1.2 Aqua Regia: Prepare immediately before use by carefully adding three volumes of concentrated HCl to one volume of concentrated HNO_3. Both HNO_3 and HCl must be of the reagent grade suitable for mercury determinations.
NOTE: This reagent is required for use when USACE project samples are being digested.

10.1.3 Concentrated HCl.

10.1.4 Concentrated HNO_3.

10.1.5 Stannous chloride in a one liter volumetric flask add ~500 mL D.I. H_2O, 30 mL concentrated HCl, and 11g stannous chloride crystals. Swirl to mix and dilute to 1 L.

10.1.6 Sodium chloride-hydroxylamine chloride solution: Dissolve 120 g of sodium chloride and 120 g of hydroxylamine sulfate in reagent water and dilute to 1 L. Note: this is normally made up 2 liters at a time.

10.1.7 Potassium permanganate, mercury-free, 5% solution (w/v): Dissolve 200 g of potassium permanganate in 4 L of reagent water.

10.1.8 3 % HCl carrier solution: 30 mLs HCl – 1 L DI H20; Prepare fresh daily.

10.1.9 Potassium persulfate 5% solution: Dissolve 100g in 2 liters of D.I. water. Used with digestion of CLP soils.

10.2 STANDARDS

10.2.1 Traceability

10.2.1.1 All reference materials are given a unique identifier within Element and labeled with the Element #. This 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 within Element as well as on the container's label.

10.2.1.2 All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded within Element. 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.

10.2.2 Preparation

10.2.2.1. **NOTE:** All standard solutions should be prepared using class A volumetric flasks, class A volumetric pipettes (or calibrated Eppendorfs). All Standards, blanks, and samples are taken through the digestion process.

10.2.2.2 Stock mercury solution: (100 µg/mL). Order from manufacturer already prepared. This solution is given a unique identifier.

10.2.2.3 Primary source and secondary source mercury standard solutions: dilute 2 mLs of stock solution to 1000 mLs in a 1000 mL volumetric flask, with 1.5 mLs concentrated HNO_3 (200 ug/L).

10.2.3 Calibration standards:

Prepared from the primary source standard. The preparation of the calibration standards, etc. is described below.

10.2.3.1 Dilute the volumes below to 5 mLs in a 70 mL polypropylene vessel. (Note: The standards are diluted to 5 mLs for the initial step of the digestion.)

ug/L Hg	mLs of 200 ug/L standard in 50 mL

0.20	0.050
0.50	0.125
1.0	0.25
ug/L Hg	mLs of 200 ug/L standard in 50 mL
2.0	0.50
4.0	1.0
6.0	1.5
10.0	2.5

10.2.3.2 Appropriate reagents are added as below in the sample preparation section.

10.2.3.3 Prepare one vessel of each.

10.2.3.4 It is necessary to digest the calibration standards when following all mercury methods.

10.2.4. Calibration verification standards:

10.2.4.1. Initial calibration verification (ICV) solution – 4.0 ug/L.

10.2.4.1.1 Prepared from the secondary source mercury standard (200 ug/L).

10.2.4.1.2 Prepared by diluting 1.0 mL of the second source mercury standard to 5 mLs in a polypropylene digestion vessel.

10.2.4.1.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.1.4 It is necessary to digest the ICV standards when using all mercury methods for soil.

10.2.4.2 Continuing calibration verification (CCV) solution:

10.2.4.2.1 Prepared from the primary or secondary source mercury standard. The concentration is alternated from 2.0 ug/L to 4.0 ug/L every 20 samples.

10.2.4.2.2 Prepared by diluting 0.50 for a 2.0 ug/L and 1.0 mL for a 4.0 ug/L of the secondary 200 ug/L standard to 5.0 mLs with reagent water in a polypropylene digestion vessel.

10.2.4.2.3 Appropriate reagents are added as below in the sample preparation section.

10.2.4.2.4 It is necessary to digest the CCV standards when following all mercury methods for soil.

10.2.5 Digestion standards:

10.2.5.1 Laboratory control sample:

10.2.5.1.2 The Laboratory Control Sample (BS) is prepared from the secondary source mercury standard (200 ug/L) and added to ~ 0.3 grams of teflon chips.

10.2.5.1.3 Prepared by diluting 0.50 mL of the secondary mercury standard (200 ug/L) to 5 mLs in a polypropylene digestion vessel with 0.30 grams of teflon chips.

10.2.5.1.4 Appropriate reagents are added as below in the sample preparation section.

10.2.5.1.5 This solution is given a unique identifier in Element.

10.2.5.2 Matrix Spikes

10.2.5.2.1 Prepared from the primary or secondary source mercury standard (200 ug/L).

10.2.5.2.2 Prepared by adding 0.50 mL of the mercury standard (200 ug/L) to the sample in a polypropylene digestion vessel. Project specific requirements may over-ride the spiking level.

C10.2.5.2.3 Appropriate reagents are added as below in the sample preparation section.

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 Because of the extreme sensitivity of the analytical procedure and the omnipresence of mercury, care must be taken to avoid extraneous contamination. Sampling devices and sample containers should be ascertained to be free of mercury; the sample should not be exposed to any condition in the lab that may result in contact with solid, liquid or airborne mercury.

11.3 Refrigerate solid samples at 4°C (±2°C) upon receipt until digestion and analysis.

11.4 The sample should be analyzed without drying. A separate percent solids determination is required

11.5 The holding time for digestion of mercury samples is 28 days.

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. Four BSs are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 QUALITY CONTROL (Reference SW-846, 7471A Update III, 7471B Revision 2 February 2007, USEPA CLP ILMO 4.1 or EPA 245.5 for further clarification)

12.3.1 Daily

12.3.1.1 The instrument must be calibrated daily for all projects.

12.3.1.2 Begin each analysis with an ICV(concentration at or near mid range). The control limits are +10% for 7471A and 245.5, ±20% for 7471B and ±5% for 245.5..

12.3.1.3 Analyze ICB. Control limit is <± MDL or ±RL/CRDL for other or CLP. For DOD, no analyte detected > 2x MDL.

12.3.1.4 If the ICV is not in control a new curve must be analyzed prior to sample analysis.

12.3.1.5 Follow each set of 10 samples with a CCV and also must end up with CCV after last sample. The control limits are +20% for SW846-7471A, SW846 7471B and ±10% for 245.5. If an exceedance occurs, analyze another CCV, if the second CCV fails, then a new calibration curve should be generated and all affected samples should be reanalyzed.

12.3.1.6 Follow each CCV with a CCB. Control limit is < ±MDL or ±RL/CRDL for others or CLP. For DOD, no analyte detected > 2x MDL.

12.3.2 Quarterly

12.3.2.1 IDLs for CLP (Follow SOP - 414).

12.3.3 Annually

A. MDLs must be analyzed for all matrixes (Follow SOP - 414).

12.3.4 Digestion

12.3.4.1 BS data should be maintained and available for easy reference or inspection.

12.3.4.2 BLK (< ±½ RL or ±RL for common contaminates or ±RL/CRDL for others or CLP)

12.3.4.2.1 Employ a minimum of one BLK per sample batch to determine if contamination or any memory effects are occurring. The preparation blank is taken through the same digestion/preparation steps as the samples being tested. The result for the preparation blank must be < ± ½ RL for USACE or ±RL/CRDL for others or CLP. If not, the analyst must use good judgment to evaluate the impact upon the associated samples. There is no impact if an associated sample is below the method detection limit or if the level in the sample is greater than 10X the level found in the preparation blank. If the level of mercury in a sample is above the method detection limit, but less than 10X the level found in the preparation blank, the sample must be redigested and reanalyzed or the data must be qualified on the final report. The project manager or QA officer will make this determination.

12.3.4.3 Laboratory control sample (BS).

12.3.4.3.1 Employ a minimum of one BS per sample batch to verify the digestion procedure. The BS is taken through the same digestion/preparation steps as the samples being tested. The minimum control limits are +20% for SW846-7471A, 7471B and 245.5 solid samples. A BS will accompany each batch of soil samples. If the BS is not in control, the Inorganic Manager and QA Officer must be notified immediately. Several possibilities exist at this point and a thorough investigation and data evaluation is essential. The first question is to evaluate the impact upon the

data. All samples may need to be retested or flagged with the appropriate qualifier. The next question is to find out why it occurred and to proceed with a corrective action plan to prevent reoccurrence. This corrective action is documented in a CAR.

12.3.5 Sample matrix

12.3.5.1 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.

12.3.5.2 Analyze one spiked sample and spiked sample duplicate 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. Project specific requirements will take precedence in these situations. CLP requires 1 duplicate and 1 spike per batch. If the analyte level in the sample is not greater than four times the spiking level, the spike recoveries should be within +25% for 7471A and ±20% for 7471B of the true value (+20% for DOD projects). If results do not fall within the control limit redigestion/reanalysis may be required. If reanalysis is not required, the associated batch of samples will be flagged accordingly. Discuss the situation with your supervisor. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.

12.3.5.3 The relative percent difference (RPD) between replicate determinations is to be calculated as referenced in the laboratory QA manual. (A control limit of + 20% RPD (non-aqueous samples may routinely exceed this amount) shall be used for sample values greater than ten times the instrument detection limit.) Supervisor must be notified if the control limit is not met. Supervisor will determine corrective action if required. The final analytical report must document this situation. A Corrective Action Report (CAR) must be filled out and attached to the data as well as emailed or sent to the supervisor when the control limits are exceeded.

12.3.5.4 For 245.5 analyze one serial dilution (1 to 5 dilution) for every 20 samples or per analytical batch, whichever is more frequent. Percent recovery should be 10%. The concentration of the original sample should be a minimum of 50X the IDL in order to apply the recovery criterion; if not, the serial dilution approach is not used.

12.3.5.5 When the sample matrix is so complex that viscosity, surface tension, and components can not be accurately matched with standards, the method of standard addition (MSA) is recommended. Section 8.6

13.0 Calibration and Standardization

13.0.1 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.

13.0.2 Set up the instrument with proper operating parameters.

13.0.3 Perkin Elmer Flow Injection Mercury System (FIMS).

13.0.3.1 Prepare the instrument for calibration by the following steps:

13.0.3.1.1 Replace any old tubing that is around the pump cylinder. The sample transfer tubing connected to the seperator cover must not have any moisture in it, if it does replace it. (Perkin-Elmer tygon tubing, waste and carrier 1.52mm I.D., waste only 3.17mm I.D., stannous chloride 1.14mm I.D.)

13.0.3.1.2 Also replace the filter membrane with the rough side up. (for instructions refer to page 1-22 in maintenance manual.)

13.0.3.1.3 Turn on PE 100 spectrophotometer; (Note: this must be on in order to start up the software on the computer.)

13.0.3.1.4 Turn on computer and go to icon "AA Win LAB Analyst"

13.0.3.1.5 Go to method; select "Hg CAL 2" then OK.

13.0.3.1.6 Wavelength = 253.7; smoothing points =9; measurement = peak height; read time = 18 sec.; BCC time = 2 sec.

13.0.3.1.7 Go to "Sample Info" and enter the order of the samples and other information that may be needed.

13.0.3.1.8 Save entered sample list under "Save …sample info file" Note: description and batch ID are normally the date of analysis.

13.0.3.1.9 Go to "auto"; then to set-up. Select Browse in both spaces. One is to bring up your saved "Sample Information." File. The other is to select a results library. Double click on heading and choose.

13.0.3.1.10 Turn the printer on.

13.0.3.1.11 Connect all tubing to the pump and blocks.

13.0.3.1.12 Start the pump by going to "FIAS" and click the pump 1 Icon (120).

13.0.3.1.13 The pump will start, then lock down and tighten the tubes onto the pump.

13.0.3.1.14 Turn on the nitrogen tank, it should be >500 psi on the gauge. Replace the nitrogen tank when it is at 500 psi.

13.0.3.1.15 The pressure gauge on the PE100 should be just below 100.

13.0.3.1.16 Use the tension adjuster to press down the tubing magazine to the pump head on the top and bottom. Start the pump and then lock them down. This technique needs to be demonstrated so that a new user will be able to understand what is needed here and how to do it.

13.0.3.1.17 Adjust the spring tension tubing until there is a constant "bubble of low rate" coming out to the waste tube.

13.0.3.1.18 Place carrier tubes into carrier and stannous chloride tube into SnCl2. (click valve fill inject and make sure flow is correct and the line is rinsed)

13.0.3.1.19 Make sure the permanganate waste bottle is bubbling in order to absorb any Hg vapors which could be vented into the room.

13.0.3.1.20 Allow a few minutes for reagents to flow through the system before starting analysis.
13.0.3.1.21 Calibrate: Go to "Auto" click on "Analyze", click on "calibrate".
13.0.3.1.22 "Select location" enter the #'s of the samples to be analyzed, then "OK".

13.0.3.2 Analyze the calibration standards as below.
13.0.3.2.1 A curve must be analyzed daily for all projects. A new curve must be analyzed when the ICV analysis is not within ±10% for SW846 7471A and ±5 % for 245.5 methods, or ±20% for 7471B.

13.0.3.2.1 The curve should be linear with a calculated intercept with a minimum correlation coefficient of >0.995 (USACE) or 0.998 (other). If not, a new curve must be analyzed.
13.0.3.2.2 CLP requires a blank + 5 calibration standards (0, .02, .05, .1, .5 and 1.0 µg). (One standard must be at CRDL or IDL whichever is greater.)

14.0 Procedure

14.1 Prepare calibration standards as detailed above. Add all reagents to the standards which are added to the samples as outlined below. Record the standard preparation in the standard log.
14.2 Sample preparation:
14.2.1 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:
14.2.1.1 The material in the sample pan (inorganic-plastic/organic-aluminum) should be divided into quarters and each quarter should be mixed individually.
14.2.1.2 Two quarters should then be mixed to form halves.
14.2.1.3 The two halves should be mixed to form a homogenous matrix.
14.2.1.4 This procedure should be repeated several times until the sample is adequately mixed.
14.2.1.5 NOTE: Samples that are clay type materials must 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.
14.2.2 Grinding of Vegetation Samples
14.2.2.1 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.

14.2.2.2 Transfer 0.30 g (for USACE work use anywhere from 0.20 to 1.0 g and record the weight in the digestion log) of sample to a polypropylene digestion vessel previously marked for this sample. Record the exact sample mass on the bottle and on the Element Batch Sheet. (Note: the balance must be calibrated for the specific task. Calibrate by weighing a 0.5 and a 0.1g weight on the balance along with a digestion vessel. (Record in specific balance calibration log.)

14.2.2.3 Add 2.5 mLs of reagent water, and 2.5 mLs of aqua regia and mix for samples. Add 2.5 mLs of aqua regia to standards and mix.

14.2.2.4 Cover samples and standards with watch glasses and heat for 2 minutes in the hot block at 95+2°C (The hot block temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature).

14.2.2.5 Cool, bring to 30 ml with D.I. water.

14.2.2.6 Add 7.5 mLs potassium permanganate solution to each vessel and mix. For sewage samples additional permanganate may be required. Shake and add additional portions of potassium permanganate to the solution if necessary, until the purple color persists for at least 15 minutes (not more than 12.5 mLs).

NOTE: The same amount of $KMnO_4$ added to the samples should be present in the standards and blanks.

14.2.2.7 Heat for 30 minutes on the hot block at 95+2°C (The temperature must be monitored and documented. Record observed temperature, correction factor, and the corrected temperature), cool. Samples may be saved at this point if there is not time to run the whole set that day.

14.2.2.8 Add 3 mLs of sodium chloride-hydroxylamine chloride solution to each vessel.

14.2.2.9 Bring to 50 mLs with D.I. water both standards and samples. Cap mix and vent to decolor and release Cl gas. The samples are now ready for analysis.

14.2.2.10 NOTE: Stannous Chloride (10.1.5) and 3% HCl (10.1.8) are added by the instrument during analysis.

14.2.3 Sample analysis

14.2.3.1 Set up the instrument as described in the calibration section above.

14.2.3.2 When ready to run samples, transfer samples and standards to autosampler tubes and load the auto sampler according to the sample information sheet set up previously. If chlorides are suspected, purge the head space in the polyethylene tube for at least 1 minute to get rid of any chlorine gas collected there. After a delay of at least 30 seconds the sample is ready for step “3”. NOTE: When aqua-regia is added assume that all samples and standards have chlorine and treat accordingly. Purging the samples of chlorine is accomplished by putting a pasteur pipette on the end of some air tubing hooked to a fish pump. The

pasteur pipette is then placed at an angle into the top of the polyethylene vessel without breaking the surface of the sample. It takes about one minute to purge the air above the sample of chlorine.

14.2.3.3 Analysis must be preceded by the analysis of an ICV (concentration at or near mid range) with control limits of +10% for SW846-7471A or ±20% for 7471B and ±5% for 245.5 methods.

14.2.3.4 The ICB must follow the calibration standards (<±MDL (USACE) or ±RL/CRDL for other or CLP), but not before the ICV. No analyte must be detected > 2xMDL for DOD.

14.2.3.5 Each set of ten samples must be followed by a CCV with control limits of +20% for SW846-7471A and B and ±10% for 245.5 method. The run must also end with a CCV, then CCB.

14.2.3.6 Analyze CCB after calibration and each CCV. The CCB frequency is 10% or every 2 hours whichever is more frequent. (control limit is < ±MDL or ±RL/CRDL for other or CLP). For DOD, CCB at beginning and end of sequence and after every 10 samples. No analyte detected > 2xMDL.

14.2.3.7 Instrument Run Log example:

AS LOC	Sample ID
0	Wash
1	0.0
2	0.02
3	0.05
4	0.1
5	0.2
6	0.4
7	0.6
8	1.0
9	SEQ- ICV
10	SEQ-ICB
11	BS
12	BLK
13	Sample
14	Sample
15	Sample
16	Sample
17	Sample
18	Sample
19	Sample
20	Sample
21	SEQ-CCV
22	SEQ-CCB
23	Sample
24	Sample
25	Sample
26	Sample
27	Sample
28	Sample
29	Sample
30	Sample
31	MS
32	MSD
33	SEQ-CCV
34	SEQ-CCB

14.2.3.8 Sample analysis:

14.2.3.8.1 Go to "Analyze", "select location" and type in the range of numbers needed to complete analysis. (ie. 9-54). Press enter and the autosampler will proceed to enter the selected range. NOTE: Check standards are loaded as part of the tray.

14.2.3.8.2 Make sure that the sample wash beaker is filled with 3% HCl.

14.2.3.8.3 Dilute and reanalyze samples that are more concentrated than within 10% of the high standard. Soil sample dilutions are

made from the digested aliquot. Sample concentration results that are below the calibration curve but above the MDL are reported flagged as estimated, ("B" flag).

14.2.4 Data reporting

14.2.4.1 Reduce data to result which will be reported using the soil spreadsheet found on the network..

14.2.4.2 Complete the data review checklist (attached). Must be completed and attached to each set of USACE data.

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 Pull up the blank spreadsheet at V: lab\metals\tests\mercury and transfer all the information pertinent to the current analysis. Save as the date of analysis. This information can be obtained from your mercury batch sheet.

15.3 Transfer the sample absorbance into the excel spreadsheet in the appropriate cell. The spreadsheet uses the current calibration to calculate the Hg results.

15.4 Make sure that the appropriate dilution factors are entered into the spreadsheet in the correct cells.

15.5 The spreadsheet should divide the result which is the µg Hg obtained from the sample mass by the sample mass in grams. This will yield a result of µg Hg/g sample on a wet weight basis. Calculations in the spreadsheet should be checked occasionally to make sure that they are working correctly.

15.6 If available, divide the result by the %solids to obtain the result on a dry weight basis.

15.7 Report the data as µg Hg/g of sample (mg/kg wet or mg/kg dry when % solids are available).

16.0 Method Performance

16.1 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 is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

DOC BS Preparation: Dilute 0.5 mL of the second source standard (200 ug/L) add to ~0.3g to 5 mLs with reagent water/aqua-regia in a 70 mL polypropylene vessel. Follow SOP procedure for preparation and analysis steps.

DOC Accuracy and Precision Criteria: The four BS's for the DOC need to be within the methods recovery ranges. Duplicates should be below 20% relative percent difference.

17.0 Pollution Prevention

14.1 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

14.2Quality 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

19.1 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.

19.2 **CORRECTIVE ACTIONS: INSTRUMENT RELATED**

19.2.1 ICV not within + 10% (SW846) and (245.5)

19.2.1.1 If the problem is with the solution, re-prepare, obtain new stock if necessary.

19.2.1.2 If the problem is with the calibration, recalibrate thru analysis of appropriate standards and recheck ICV.

19.2.2 CCV not within + 20% (SW846) and (245.5)

19.2.2.1 If the problem is with the solution, reprepare, obtain new stock if necessary.

19.2.2.2 If the problem is with the calibration, recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten sample according the following guidelines.

19.2.2.2.1 If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.

19.2.2.2.2 If the CCV was biased low, the previous ten samples must be reanalyzed.

19.3 **CORRECTIVE ACTION: DIGESTION RELATED**

19.3.1 The preparation blank less than ± ½ RL for DOD or ±RL/CRDL for others or CLP.

19.3.1.1. If the problem is with the instrument or stannous chloride.

19.3.1.1.1 Analyze a reagent blank to determine the stannous chloride and the instrument are behaving properly. If this check has detectable mercury, reprepare the stannous chloride or determine if there are any problems with the instrument.

19.3.1.1.2 If the problem was with the instrument or the stannous chloride and the situation is corrected continue analysis with a second aliquot of the preparation blank.

19.3.1.2 If the problem is with the digestion, all associated samples which are below the method detection limit (MDL) or have a level of mercury

greater than 10X the level found in the preparation blank can be reported. If the level of mercury in an associated sample is not <MDL nor greater than 10X the level found in the preparation blank, the sample must be redigested/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

19.3.2 BS not within control limits.

19.3.2.1 If the problem is with the instrument, reanalyze when instrument is in control with another aliquot of the sample.

19.3.2.2 If the problem is with the digestion.

19.3.2.2.1 If biased low, associated samples must be redigested.

19.3.2.2.2 If biased high, the impact upon the data user must be evaluated. The samples will be redigested or the data will be qualified on the final report.

19.4 **CORRECTIVE ACTION: SAMPLE MATRIX RELATED**

19.4.1 Replicate analysis RPD not within +20%

19.4.1.1 The associated sample data must be qualified on the final report.

19.4.2 Spike analysis recovery not within +25% 7471A and ±20% 7471B (+20% for DOD projects)

19.4.2.1 If the analyte level in the sample is greater than 4X the spiking level, the % recovery can not be evaluated and no action is taken.

19.4.2.2 If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

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.

20.2 Please see Waste Disposal SOP-405, for instruction of proper disposal of waste generated from this area.

21.0 References

21.1 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III/IV); Method 7471A, 7471B

21.2 USEPA Code of Federal Requlations, 40, CH 1,PT 136; Method 245.1; APX-B

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

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, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

APPENDIX:

Addendum for USEPA CLP ILM 05.2

1. CCV concentration must be different from ICV.

2. The same CCV shall be used throughout analysis for a sample delivery group.

3. Calibration standards must be within 5% of the standard concentration.

4. 0.2 grams of sample must be used for the sample aliquot, add enough reagent water to each sample to make a total volume of 10 mL. Proceed with method as in the water method SOP 103.0 Revision 9.

5. The ICV and CCV must be at ±20% recovery.

6. A CRA must be analyzed at the beginning and end of each batch of 20 samples. Right after the ICV/ICB and right before the final CCV/CCB. The control limit is ±30%.

7. The matrix spike must be analyzed at the concentration of 0.5 mg/Kg.

Table 1

Soil/Solid Method Detection Limits(MDL)/Detection Limit(DL), Limit of Detection(LOD) Empirical Laboratories' Reporting Limits(ERL)/Limit of Quantitation(LOQ), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)

Mercury by EPA 245.1, 245.5 7471A, SOW 4.1 & 5.2	SOLID/SOIL MDL/DL (mg/Kg)	SOLID/SOIL LOD (mg/Kg)	SOLID/SOIL ERL/LOQ (mg/Kg)	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	SOLID/SOIL CRQL ILMO 5.2 (mg/Kg)
Mercury	0.013	0.026	0.033	0.10	0.10

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Initial calibration (ICAL)	• Daily ICAL prior to sample analysis • Low standard at the RL/LOD level	• If more than one calibration standard is used, r $\geq$ 0.995 • Must follow curve processing requirements from SOP QS08	• Re-run curve • Check instrument for maintenance needs Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	Must be within $\pm$ 10% for SW846 7471A,±20% for 7471B, or ±5% for 245.5 of true value	• Re-run ICV • Repeat ICAL
CCV	• After every 10 field samples and at the end of analysis sequence.	• $\pm$ 20% for SW846-7471A&B, ±10% for 245.5of true value	• Follow guidelines for SOP QS05
Closing CCV	• At the end of every sequence	• $\pm$ 20% for SW846-7471A&B, ±10% for 245.5of true value	• Follow guidelines for SOP QS05
BLK	One per prep batch	No analytes detected > ½ RL and greater that 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	• Re-analysis to confirm the positive value • Notify the PM for further action • Re-prep of samples associated with the BLK • NCR will be required for data reported
BS	One per prep batch	Most stringent criteria listed within the LIMS.	• Re-analyze to confirm failed. • Re-prep and reanalyze BS and all samples in the associated prep batch for failed analytes, if sufficient sample material is available. • NCR will be required for data reported • Follow guidelines from SOP QS05
Calibration Blank	Before beginning a sample run, after every 10 samples, and at end of the analysis sequence.	No analytes detected > LOD.	• Correct problem. Re-analyze calibration blank. All samples following the last acceptable calibration blank must be reanalyzed.
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year	• Calculated value must be less than the Spike level •	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • the response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	• Bias Requirement: Inorganics 50-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

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. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. 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)

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: SW846 7471A/B, EPA245.5 (Mercury)

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 blank spike(BS) meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation blank (BLK) below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was water bath 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: _____

Second-Level Review: _____ Date: _____

1.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

METALS: SOP 105 REVISION #: 16 EFFECTIVE DATE: 041110

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

References: SW-846, Method 6010B, December 1996; SW-846, Method 6010C, Revision 3 February 2007; USEPA, Method 200.7, June 1991; Standard Methods 19th Edition 2340B; 1995 USEPA CLP, ILM 04.1. See Addendum for USEPA CLPILM 05.2

APPROVALS:

Lab Director: _____ Date: 4/12/10

Data Quality Manager: _____ Date: 4/11/10

Section Supervisor: Betty DeVille Date: 4/13/10

Changes Summary

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.

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1. Identification of the Test Method

This SOP is compliant with methods – SW846 6010B, SW846 6010C, EPA 200.7, (SM 19th Edition 2340B) 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, 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 ± 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 1000µL; 5000µL

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), HNO_3. 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% HNO_3 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 10.5.1. 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 <u>CLP-CAL-1 solution A</u>: 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; <u>solution B</u>: 250 ug/mL Ag; <u>CLP-CAL-2</u>: 1000 ug/L Sb; <u>CLP-CAL-3</u>: 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 1gram 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 HNO_3 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 19th Edition 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 ±5% for 200.7 and within ±10% for all other methods.

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

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

12.1.6. IFA/IFB analyzed daily. IFA must be less than two times ±MDL or less than ±LOD unless verified standard contamination for DOD. The IFB must recover within ±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 ±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 ±MDL or <±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 ±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 +/-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 ± ½ RL for DOD or ±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 ± 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 ±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% HNO_3 / 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 (± 10%) for 200.7 (± 5%) and ICB (< ±2xMDL, <±LOD-DOD or ±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 ±20% for DOD projects and ±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: < ±MDL or ±RL/CRDL for others or CLP, or twice during every 8-hour work shift, whichever is more frequent. Results should be within ±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 < ±2xMDL or <±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 <LOD (unless they are verified trace impurity from one of the spiked analytes) for DOD. Results must be within ±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) Sample10
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. **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% HNO_3 / 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 ±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 ±MDL, for CLP <RL, for **DOD no analytes detected >±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-413 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 HNO_3 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 $CaCO_3$.

15.2. The instrument will generate data results in mg/L or µg/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 (ug/g or mg/kg) = IR*DF*FED/SM

SR = Sample result
IR = Instrument result (µg/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 Manuel 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 through10.7.3 above.
DOC Accuracy and Precision Criteria: The LOD is analyzed at 2 times the MDL and must result in an concentration 3 times the noise. The LOQ is analyzed at the RL or 2 times the RL and must be recovered within ±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 ± 10% or ± 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 ±MDL or within ± 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 ± 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 ± 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 <±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 ± 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 ± 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 ±2xMDL 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. Re-calibrate and reanalyze.

19.2. DIGESTION RELATED

19.2.1. Preparation blank (BLK) not within ± ½ RL and ± 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 ±20% (if both are >5X CRDL) or ± 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 ±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 ±25% for SW6010B, DOD or ± 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 ±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.

iii. 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 Requlations, 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 4.1. (Based on NELAC Voted Revision June 5, 2003. 4/22/09

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	• once per calibration	• IFA less than LOD if not verified contamination of standard. IFB must be within ±20%.	• Check IEC corrections for metals in the IFA.
Calibration Curve	• 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).	• Linear calibration Corr. of 0.998 • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICB	At the beginning of every sequence	Must meet the <±LOD for DOD or < 2xMDL	Re-run
ICV	Alternate source standard to be analyzed after every calibration curve	• Must be in the range 90 to 110% for 6010B&C, or 95 to 115% for 200.7.	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • For every 10-client samples	• Must be in the range 90 to 110%	• Samples must be reanalyzed if possible, if not samples are flagged with a “Q”.
Closing CCV	• At the end of every sequence	• Must be in the range 90 to 110%	• Samples must be reanalyzed if possible, if not samples are flagged with a “Q”.
BLK	One per prep batch	• Must be less than ½ ±RL.	• 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-prep 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.	• 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	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the BS for average accuracy	• 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 145 REVISION #: 07 EFFECTIVE DATE: 20100325

DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING THE DIONEX dx-500 ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX AS18 COLUMN

References:
USEPA METHOD 300.0/ SW846 Method 9056

APPROVALS:

Lab Director: _____ Date: 3/30/10

Data Quality Manager: _____ Date: 3/30/10

Section Supervisor: _____ Date: 3/30/10

Changes Summary:

Revision Date: 03/25/2010

- The SOP has been reviewed for accuracy and completeness.
- All references to analysis of ortho-phosphorus by this method have been removed.

DETERMINATION OF INORGANIC ANIONS IN WATER BY ION CHROMATOGRAPH USING THE DIONEX dx-500 ION CHROMATOGRAPH WITH HYDROXIDE ELUENT AND DIONEX AS18 COLUMN

References:
USEPA METHOD 300.0/ SW846 Method 9056

I. SCOPE AND APPLICATION:

1. This method covers the determination of the following inorganic common anions in reagent water, surface water, ground water, and other aqueous matrixes.

PART A.--Common Anions

Chloride	Nitrate	Fluoride	Sulfate
Nitrite	Bromide		

2. Single laboratory Method Detection Limit for the above analytes is listed in Tables 1A, 1B and 1C from method 300.0. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample and the specific instrumentation employed.

   A. In order to achieve comparable detection limits, an ion chromatographic system must utilize suppressed conductivity detection, be properly maintained and must be capable of yielding a baseline with no more then a 5 nS noise/drift per minute of monitored response over the background conductivity.

3. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatograms.

4. When the method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of a laboratory fortified matrix sample covering the anions of interest. The fortification procedure is described in the Quality Control section.

5. Users of the method data should state the data-quality objectives prior to analysis. Analyst using this method must demonstrate the ability to generate acceptable results with the method, using the procedures described in the Quality Control section.

II. SUMMARY OF METHOD

1. A small volume of sample, 50 uL for Part A is introduced into an ion chromatograph (IC). The anions of interest are separated and measured, using a system comprised of a guard column, analytical column, suppressor device, and conductivity detector.

III. DEFINITIONS

1. **ANALYSIS BATCH** -- A group of no more than 20 field samples (Field sample analyses include only those samples derived from a field sample matrix. These include the initial and duplicate field samples as well as all Laboratory Fortified Sample Matrices (MS/MSD)). The analysis batch must include an Initial Calibration Check Standard (CCV), and End Calibration Check Standard (ending CCV), Laboratory Reagent Blank (BLK), and a

Laboratory Fortified Blank (BS). Within an ANALYSIS BATCH, for every group of **ten** field samples at least one Laboratory Fortified Matrix (MS) and either a Field Duplicate or a Laboratory Duplicate must be analyzed after the tenth field sample analysis. MSD does not count as a laboratory duplicate for anions.

2. **CALIBRATION STANDARD (CAL)** -- A solution prepared from the primary dilution standard solution or stock standard solutions and the surrogate analyte. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

   A. **INITIAL CALIBRATION STANDARDS --** A series of CAL solutions used to initially establish instrument calibration and develop calibration curves for individual target anions.

   B. **INITIAL CALIBRATION CHECK STANDARD** -- An individual CAL solution, analyzed initially, prior to any sample analysis, which verifies previously established calibration curves.

   C. **CONTINUING CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after every tenth field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for the previous ten field samples analyzed.

   D. **END CALIBRATION CHECK STANDARD** -- An individual CAL solution which is analyzed after the last field sample analyses which verifies the previously established calibration curves and confirms accurate analyte quantitation for all field samples analyzed since the last continuing calibration check.

3. **FIELD DUPLICATES (FD) --**Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout the field and laboratory procedures. Analyses of field duplicates indicate the precision associated with sample collection, preservation and storage, as well as with laboratory procedures.

4. **INSTRUMENT PERFORMANCE CHECK SOLUTION (ICV) --** A solution of one or more method analytes, surrogates or other test substances used to evaluate the performance of the instrument system with respect to a defined set of criteria.

5. **LABORATORY DUPLICATE (DUP) --** Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analyses of sample and DUP1 indicate precision associated specifically with the laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.

6. **LABORATORY FORTIFIED BLANK (BS) --**An aliquot of reagent water or other blank matrices to which known quantities of the method analytes are added in the laboratory. The BS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements.

7. **LABORATORY FORTIFIED SAMPLE MATRIX (MS) --** An aliquot of an environmental sample to which known quantities of the method analytes are added in the

laboratory. The MS and or MSD are analyzed exactly like a sample, and their purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the MS corrected for background concentrations.

8. **LABORATORY REAGENT BLANK (BLK) --** An aliquot of reagent water or other blank matrices that are treated exactly as a sample including exposure to all glassware, equipment, solvents, reagents, internal standards, and surrogates that are used with other samples. The BLK is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents, or the apparatus.

9. **LINEAR CALIBRATION RANGE (LCR) --** The concentration range over which the instrument response is linear.

10. **MATERIAL SAFETY DATA SHEET (MSDS) --** Written information provided by vendors concerning a chemical's toxicity, health hazards, physical properties, fire, and reactivity data including storage, spill, and handling precautions.

11. **METHOD DETECTION LIMIT (MDL) --** The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

12. **MINIMUM REPORTING LEVEL (MRL) --** The minimum concentration that can be reported for an anion in a sample following analysis. This defined concentration can be no lower than the concentration of the lowest calibration standard and can only be used if acceptable quality control criteria for this standard are met.

13. **PERFORMANCE EVALUATION SAMPLE (PE) --** A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst.

14. **QUALITY CONTROL SAMPLE (QCS) --** A solution of method analytes of known concentrations that is used to fortify an aliquot of LRB or sample matrix. The QCS is obtained from a source external to the laboratory and different from the source of calibration standards. It is used to check laboratory performance with externally prepared test materials.

15. **STOCK STANDARD SOLUTION (SSS) --** A concentrated solution containing one or more method analytes prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.

IV. INTERFERENCES

1. Interferences can be divided into three different categories: **direct chromatographic coelution**, where an analyte response is observed at very nearly the same retention time as the target anion; **concentration dependant coelution**, which is observed when the response of higher than typical concentrations of the neighboring peak overlap into the retention window

of the target anion; and, **ionic character displacement**, where retention times may significantly shift due to the influence of high ionic strength matrices (high mineral content or hardness) overloading the exchange sites in the column and significantly shortening target analytes' retention times.

A. A direct chromatographic coelution may be solved by changing columns, eluant strength, modifying the eluant with organic solvents (if compatible with IC columns), changing the detection systems, or selective removal of the interference with pretreatment. Sample dilution will have little to no effect. The analyst must verify that these changes do not negatively affect performance by repeating and passing all the criteria in the Quality Control Section.

B. Sample dilution may resolve some of the difficulties if the interference is the result of either concentration dependant coelution or ionic character displacement, but it must be clarified that sample dilution will alter your Minimum Reporting Limit (MRL) by a proportion equivalent to that of the dilution. Therefore, careful consideration of project objectives should be given prior to performing such a dilution. An alternative to sample dilution, may be dilution of the eluant.

C. Pretreatment cartridges can be effective as a means to eliminate certain matrix interferences. Prior to using any pretreatment, the analyst should be aware that all **instrument calibration standards must be pretreated in exactly the same manner** as the pretreated unknown field samples. The need for these cartridges has been greatly reduced with recent advances in high capacity anion exchange columns.

    1. Extreme caution should be exercised in using these pretreatment cartridges. Artifacts are known to leach from certain cartridges, which can foul the guard, and analytical columns causing loss of column capacity indicated by shortened retention times and irreproducible results. Frequently compare your calibration standard chromatograms to those of the column test chromatogram (received when the column was purchased) to insure proper separation and similar response rations between the target analytes is observed.

D. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baselines in an ion chromatogram. These interferences can lead to false positive results for target analytes as well as reduced detection limits as a consequence of elevated baseline noise.

E. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems.

F. Any anion that is only weakly retained by the column may elute in the retention time window of fluoride and potentially interfere. At concentrations of fluoride above 1.5 mg/L, this interference may not be significant; however, it is the responsibility of the analyst to generate precision and accuracy information in each sample matrix.

G. Close attention should be given to the potential for carry over peaks from one analysis which will effect the proper detection of analytes of interest in a second, subsequent analysis. The elution of nitrate (retention time of ~9.0 min.) indicates the end of a

chromatographic run. A run time of 12 minutes is recommended to allow for the proper elution of any potentially interferrant late peaks. It is the responsibility of the analyst to confirm that no late eluting peaks have carried over into a subsequent analysis thereby compromising the integrity of the analytical results.

2. **SAFETY**

A. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

B. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be warn when handling samples and chemicals.

V. EQUIPMENT AND SUPPLIES

1. Ion Chromatograph (IC) – Analytical system complete with eluant generator, an ion chromatographic pump, injection valves, both guard and analytical separator columns, suppressor, conductivity detector, and computer based data acquisition system. Dionex DX-500 or equivalent. (See letter from EPA to Dionex on discussion of alternate hydroxide eluant for anions. Also since hydroxide eluant cannot be run on the traditional column Dionex AS18, 4mm (P/N 060549) or equivalent should be used.

a. Anion guard column--Dionex Ion Pac AG18 4mm (P/N 060551), or equivalent. This column functions as a protector of the separator column. If omitted from the system, the retention times will be shorter.

b. Anion separator column--Dionex Ion Pac AS18, 4mm (P/N 060549), or equivalent. An optional column (2mm or 4 mm) may be used if comparable resolution of peaks is obtained, and the quality control requirements can be met.

i. When a 4 mm column is employed, the injection volume should be 50 uL.

ii. Comparable results can be attained using the Dionex, AS17, 4 mm column.

2. Anion suppressor device--The data presented in this method were generated using an Ultra 4 mm Dionex Anion Self Regenerating Suppressor (ASRS, P/N 53946). An equivalent suppressor device may be utilized provided comparable conductivity detection limits are achieved and adequate baseline stability is attained as measured by a combined baseline drift/noise of no more than 5 nS per minute over the background conductivity. Proper suppressor performance is essential to analytical data reproducibility and sensitivity of the conductivity detector.

a. The ASRS was set to perform electrolytic suppression at a current setting of 300 ma using the external water mode. External water was delivered to the suppressor directly from a pressurized source at a flow rate of 5 mL/min. It should be noted that while Empirical

Laboratories has the suppressor currently set at 300 mA, no external water is being used at this time.

3. Detector--Conductivity cell (Dionex CD20, or equivalent) capable of providing data as required in the Quality Control section of this SOP.

4. Data Acquisition System--The Dionex Peaknet Data Chromatography Software version 5.2 or equivalent is used by Empirical Laboratories.

5. Analytical balance--Mettler Used to accurately weigh target analyte salt for stock standard preparation (±0.1 mg sensitivity).

6. Micro beakers -- Plastic, disposable - used during sample preparation.

7. Syringes--Plastic, disposable, 10 mL - used during sample preparation.

8. Eppendorfs with variable settings- 1mL and 5 mL. Must be calibrated quarterly.

9. Bottles -- High density polyethylene (HDPE) or glass, amber or clear, 30 mL, 125 mL, 250 mL. For sampling and storage of calibration solutions.

10. Particulate filters-- 0.45 micron syringe filters, specifically designed for IC applications (Gelman IC Acrodisc, PN 4485, or equivalent). These cartridges are used to remove particulates from the sample matrix while loading the sample manually or if the autosampler employed does not filter the sample during loading.

NOTE: See method for several types of pretreatment cartridges that are available and may be useful depending on the matrices of the samples normally processed.

11. Autosampler PolyVials 5-mL size, with filtercaps, 250 each --Dionex cat log # 38141.

12. Shaker for use when extracting soil samples.

13. Centrifuge to aid in separation after extraction.

14. Centrifuge tubes--50 mL capacity

VI. REAGENTS AND STANDARDS

1. Reagent water-- Distilled or deionized water 17.8 Mohm or better, free of anions of interest. Water should contain particles no larger than 0.20 microns.

2. A system or apparatus which automatically generates the hydroxide eluant (Dionex EG40, or equivalent) is an acceptable alternative to physically preparing the hydroxide eluant.

3. Stock standard solutions, 1000 mg/L (1mg/mL): Stock standard solutions are purchased as certified solutions from selected vendors.

NOTE: Stability of standards: Stock standards for most anions are stable for at least 6 months when stored at 4 °C. Dilute working standards should be prepared monthly.

VII. SAMPLE COLLECTION, PRESERVATION AND STORAGE

1. Samples should be collected in plastic or glass bottles. All bottles must be thoroughly cleaned and rinsed with reagent water. The volume collected should be sufficient to insure a representative sample, allow for replicate analysis, if required, and minimize waste disposal.

2. Sample preservation and holding times for the anions that can be determined by this method are as follows:

PART A: Common Anions

Analyte	Preservation	Holding Time
Bromide	None required	28 days
Chloride	None required	28 days
Fluoride	None required	28 days
Nitrate-N	**Cool to 4 °C**	**48 hours**
Nitrite-N	**Cool to 4 °C**	**48 hours**
Sulfate	Cool to 4 °C	28 days

3. When collecting a sample from a treatment plant employing chlorine dioxide, the sample must be sparged with an inert gas (helium, argon, nitrogen) prior to addition of the addition of the EDA preservative at time of sample collection.

VIII. QUALITY CONTROL

1. The laboratory is required to operate a formal quality control (QC) program. The requirements of this program consist of an initial demonstration of laboratory performance, and subsequent analysis in each analysis batch of a Laboratory Reagent Blank, Laboratory Fortified Blank, Instrument Performance Check Standard, calibration check standards, Laboratory Fortified Sample Matrices (LFM) and either Field, Laboratory or LFM duplicate sample analyses. This section details the specific requirements for each of these QC parameters. The laboratory is required to maintain performance records that define the quality of the data that are generated.

2. INITIAL DEMONSTRATION OF PERFORMANCE

   A. The initial demonstration of performance is used to characterize instrument performance (determination of accuracy through the analysis of the QCS) and laboratory performance (determination of MDLs) prior to performing analysis by this method.

   B. Quality Control Sample (QCS) – When beginning the use of this method, on a quarterly basis or as required to meet data-quality needs, verify the calibration standards and acceptable instrument performance with the preparation and analyses of a QCS. If the determined concentrations are not within ±10% of the stated values, performance of the determinative step of the method is unacceptable. The source of the problem must be identified and corrected before either proceeding with the initial determination of MDLs or continuing with on-going analyses.

C. Method Detection Limit (MDL)—MDLs are established for all analytes, using reagent water (blank) fortified at a concentration of three to five times the estimated instrument detection limit. To determine MDL values, take seven replicate aliquots of the fortified reagent water and process through the entire analytical method over at least three separate days. Perform all calculations defined in the method and report the concentration values in the appropriate units. Calculate the MDL as follows:

$$MDL = (t) \times (S)$$

Where,

t = Student's t value for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom [t = 3.14 for seven replicates].

S = standard deviation of the replicate analyses.

D. MDLs should be determined every 6 months or at least annually, when a new operator begins work or whenever there is a significant change in the background, or instrument response. MDL check samples are used in connection with confirming that the MDL determined is legitimate and to monitor the instrument sensitivity periodically. MDL checks are analyzed whenever a new MDL is generated and at a minimum quarterly to monitor for any shifts in sensitivity.

3. ASSESSING LABORATORY PERFORMANCE

A. Laboratory Reagent Blank (BLK) – The laboratory must analyze at least one LRB with each analysis batch. Data produced are used to assess contamination from the laboratory environment. Values that exceed the MDL (**For DOD QSM Ver. 3 no analytes detected ≥ ½ RL or for common lab contaminants no analyte detected ≥ RL**) indicate laboratory or reagent contamination should be suspected and corrective actions must be taken before continuing the analysis.

B. Laboratory Fortified Blank (BS)—The BS should be prepared at concentrations similar to those expected in the field samples and ideally at the same concentration used to prepare the MS/MSD. Calculate accuracy as percent recovery. If the recovery of any analyte falls outside the required concentration dependant control limits that analyte is judged out of control, and the source of the problem should be identified and resolved before continuing analyses.

i. Control Limits for the BS are 90 to 110%.

ii. The laboratory uses the BS to assess laboratory performance against the required control limits listed in the QC section. When sufficient internal performance data becomes available (usually a minimum of 20-30 analyses), optional control limits can be developed from the percent mean recovery (x) and the standard deviation (S) of the mean recovery. These data can be used to establish the upper and lower control limits as follows:

$$\text{UPPER CONTROL LIMIT} = x + 3S$$
$$\text{LOWER CONTROL LIMIT} = x - 3S$$

The optional control limits must be equal to or better than those listed in the QC section (±10%). After each five to ten new recovery measurements, new control limits can be calculated using only the most recent 20-30 data points. Also, the standard deviation (S) data should be used to establish an on-going precision statement for the level of concentrations monitored. These data must be on file and be available for review.

i. Instrument Performance Check Solution (ICV) – The Initial Calibration Check Standard is to be evaluated as the instrument performance check solution in order to confirm proper instrument performance. The acceptable limits for this standard is 90 to 110%. Small variations in retention time can be anticipated when a new solution of eluant (or when the KOH cartridge is changed) is prepared but if shifts of more that 2% are observed in the IPC retention time, some type of instrument problem is present. Potential problems improperly prepared eluant, erroneous method parameters programmed such as flow rate or some other system problem. The chromatographic profile (elution order) of the target anions following an ion chromatographic analysis should closely replicate the profile displayed in the test chromatogram that was shipped when the column was purchased. As a column ages, it is normal to see a gradual shift and shortening of retention times, but if after several years of use, extensive use over less than a year, or use with harsh samples, this retention time has noticeably shifted to any less than 80% of the original recorded value, the column may require cleaning or replacement. Particularly if resolution problems are beginning to become common between previously resolved peaks. A laboratory must retain a historic record of retention times for all the target anions in the ICV to provide evidence of an analytical column's vitality.

4. ASSESSING ANALYTE RECOVERY AND DATA QUALITY

A. Laboratory Fortified Sample Matrix (MS) – The laboratory adds a known amount of analyte to a minimum of 10% of the field samples within an analysis batch. The MS sample is prepared from a sample matrix which has been analyzed prior to fortification. The analyte concentration must be high enough to be detected above the original sample and should adhere to the QC requirements. It is recommended that the solutions used to fortify the MS be prepared from the same stocks used to prepare the calibration standards and not from external source stocks. This will remove the bias contributed by an externally prepared stock and focus on any potential bias introduced by the field sample matrix.

i. If the fortified concentration is less than the observed background concentration of the unfortified matrix, the recovery should not be calculated. This is due to the difficulty in calculating accurate recoveries of the fortified concentration when the native sample concentration is so high.

ii. The MS should be prepared at concentrations no greater than five times the highest concentration observed in any field sample. If no analyte is observed in any field sample, the MS must be fortified no greater than five times the lowest calibration level which as outlined in this method is the minimum reported level (MRL). For example, if chloride is not detected in

any field samples above the lowest calibrations standard concentration of 5.00 ug/L, the highest MS fortified concentration allowed is 25.0 ug/L.

iii. Calculate the percent recovery for each analyte, corrected for concentrations measured in the unfortified sample. Percent recovery should be calculated using the following equation:

$$R = \frac{C_s - C}{s} \times 100$$

where, R = percent recovery.
C_s = fortified sample concentration
C = sample background concentration
S = concentration equivalent of analyte added to sample.

iv. Until sufficient data becomes available (usually a minimum of 20 to 30 analysis), assess laboratory performance against recovery limits of 80 to 120%. When sufficient internal performance data becomes available develop control limits from percent mean recovery and the standard deviation of the mean recovery. The optional control limits must be equal to or better than the required control limits of 80 –120%.

v. If the recovery of any analyte falls outside the designated LFM recovery range and the performance for that analyte is shown to be in control, the recovery problem encountered with the LFM is judged to be matrix induced and the results for that sample and the LFM are reported with a "matrix induced bias" qualifier.

B. FIELD OR LABORATORY DUPLICATES –Analyze either a field, matrix spike duplicate or a laboratory duplicate for a minimum of 10% of the collected field samples or at least one with every analysis batch, whichever is greater. The sample matrix selected for this duplicate analysis must contain measurable concentrations of the target anions in order to establish the precision of the analysis set and insure the quality of the data. If none of the samples within an analysis batch have measurable concentrations, the LFM should be employed as a laboratory duplicate.

i. Calculate the relative percent difference (RPD) of the initial quantitated concentration (I_c) and duplicate quantitated concentration (D_c) using the following formula,

$$RPD = \frac{(I_c - D_c)}{([I_c + D_c]/2)} \times 100$$

ii. Duplicate analysis acceptance criteria

Concentration range	RPD Limits
MRL to 10xMRL	± 20%
10xMRL to highest calibration level	± 10%

iii. If the RPD fails to meet these criteria, the samples must be reported with a qualifier identifying the sample analysis result as yielding a poor duplicate analysis RPD. This should not be a chronic problem and if it frequently recurs (>20% of duplicate analyses) it indicates a problem with the instrument or individual technique.

C. Where reference materials are available, they should be analyzed to provide additional performance data. The analysis of reference samples is a valuable tool for demonstrating the ability to perform the method acceptably.

D. In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options, such as the use of different columns, injection volumes, and/or eluants, to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in the QC section and adhere to the condition of baseline stability.

E. The laboratory adopts additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the client and the nature of the samples. Whenever possible, the laboratory performs analysis of quality control check samples and participate in relevant performance evaluation sample studies.

5. CALIBRATION AND STANDARDIZATION

A. Establish ion chromatographic operating parameters equivalent to those indicated in Tables 1C for a 4-mm column.

i. Estimate the Linear Calibration Range (LCR) – The LCR should cover the expected concentration range of the field samples.

ii. For an individual calibration curve, a minimum of eight calibration standards is required for a curve.

B. Prepare the calibration standards by carefully adding measured volumes of one or more stock standards to a volumetric flask and diluting to volume with reagent water. Chloride and sulfate are calibrated from 0.5-200 mg/L; fluoride, nitrate, and nitrite from 0.05-20 mg/L.

C. Using a 4mm column, inject 50 uL (Part A) of each calibration standard. Tabulate peak area responses against the concentration. The results are used to prepare calibration curves using a linear least squares fit for each analyte. Acceptable calibration curves are confirmed after reviewing the curves for linearity and passing the criteria for the initial calibration check standard. Alternately, if the ratio of response to concentration (response factor) is constant over the LCR (indicated by < 15% relative standard deviation (RSD)), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

i. Peak areas strongly recommended since they have been found to be more consistent, in terms of quantitation, than peak heights. Peak height can tend to be suppressed as a result of high levels of common anions in a given matrix which can compete for exchange sites. Using peak areas, it is the analyst responsibility to review all chromatograms to insure accurate baseline integration of target analyte peaks since poorly drawn baselines will more significantly influence peak areas than peak heights.

D. Once the calibration curves have been established they must be verified prior to conducting any sample analysis using an initial calibration check standard. This verification must be performed on each analysis day or whenever fresh eluant has been prepared. A continuing calibration check standard must be analyzed after every tenth sample and at the end of the analysis set as an end calibration check standard. The response for the initial, continuing and end calibration check must satisfy the QC criteria of 90 to 110%. If during the analysis set, the response differs by more than the calibration verification criteria, or the retention times shift more than ±5% from the expected values for any analyte, the test must be repeated, using fresh calibration standards. If the results are still outside these criteria, sample analysis must be discontinued, the cause determined and/or in the case of drift, the instrument recalibrated. All samples following the last acceptable calibration check standard must be reanalyzed.

i. Control limits for calibration verification are 90 to 110%.

F. After satisfying the requirements, the levels selected for the other calibration check standards should be varied between a middle calibration level and the highest calibration level.

G. Software Entry of new curve

i. Integrate curve data and print results.
ii. Open Current method (Go to Peaknet Main Menu, Method) and do a save as the new date of curve.
iii. Open the schedule with the curve (Go to PeakNet Main Menu, Schedule). Change the method to the new method that was just created and change the sample type to Calibration Standard 1, 2, 3, etc… and save.
iv. Go to main menu, batch, processing, input tab and select the schedule with the curve. Then go to output tab and check update methods from calibration standards and update raw data files. Then press ok and F5. **EXIT, DO <u>NOT</u> SAVE CHANGES**.
v. Open new method and click the 5th data processing icon (blue), then click details and go to each component (analyte) (there's a drop down menu) and check areas with the printouts to make sure all data was pulled in correctly.
vi. Go through each analyte making sure the correlation is >0.995. Also the MDL point cannot be used as part of curve so it must be dropped. To drop a point, deselect it and put in comment NOT USED. Also curvefit = Linear; Origin = Include; Calibrate by = Area. Then take the retention times from the middle point of the curve. Go out of details and go to the identification tab, change the retention times and click apply then ok and save.

vii. Once changes have been made go to main menu, batch, processing, input tab and choose schedule, then output tab and check update raw data files. (**DO NOT CHECK UPDATE CALIBRATION STANDARDS or it will erase all the changes that were just made).** Then click OK then F5.

viii. Go to main menu – optimize, open the QC files that were ran after curve and check. ICV must be ±10%.

ix. When curve is complete, gather raw data before and after and print out the curve. To do this open a blank EXCEL sheet, then go to main menu – method – open current method, go to each analyte and press ALT PRINT SCREEN then paste into the EXCEL file. A copy of all this will go to the reporting department.

6. PROCEDURE

A. Other columns, chromatographic conditions, or detectors may be used if the requirements of the QC section are met.

B. Check system calibration daily and, if required, recalibrate as necessarily.

C. Sample Preparation

i. For refrigerated or samples arriving to the laboratory cold, ensure the samples have come to room temperature prior to conducting sample analysis by allowing the samples to warm on the bench for at least 1 hour.

D. Using a Luer lock, plastic 5 to 10 mL syringe, withdraw the sample from the micro beaker and attach a 0.45 um particulate filter (demonstrated to be free of ionic contaminants) directly to the syringe. Filter the sample into an autosampler vial.

E. Using a 4 mm column, inject 50 uL of each sample. Tabulate peak area responses against the concentration. During this procedure, retention times must be recorded. Use the same size loop for standards and samples. Record the resulting peak size in area units. An automated constant volume injection system may also be used.

F. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time can be used to calculate a suggested window size for each analyte. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

G. If the response of a sample analyte exceeds the calibration range, the sample may be diluted with an appropriate amount of reagent water and reanalyzed. If this is not possible then three new calibration concentrations must be employed to create a separate high concentration curve, one standard near the estimated concentration and the other two bracketing around an interval equivalent to ±25% the estimated concentration. The latter procedure involves significantly more time than a simple sample dilution therefore; it is advisable to collect sufficient sample to allow for sample dilution or sample reanalysis, if required.

H. Shifts in retention time are inversely proportional to concentration. Nitrate, phosphate and sulfate will exhibit the greatest degree of change, although all anions can be

affected. In some cases this peak migration may produce poor resolution or make peak identification difficult.

I. Should more complete resolution be needed between any two coeluting peaks, the eluant can be diluted. This will spread out the run, however, and will cause late eluting anions to be retained even longer. The analysts must verify that this dilution does not negatively affect performance by repeating and passing all the QC criteria.

i. Eluant dilution will reduce the overall response of an anion due to chromatographic band broadening which will be evident by shortened and broadened peaks. This will adversely affect the MDLs for each analyte.

7. **DATA ANALYSIS AND CALCULATIONS**

A. Prepare a calibration curve for each analyte by plotting instrument response, as peak area, against standard concentration. Compute sample concentration by comparing sample response with the standard curve. If a sample has been diluted, multiply the response by the appropriate dilution factor.

B. Report ONLY those values that fall between the lowest and the highest calibration standards. Samples with target analyte responses exceeding the highest standard should be diluted and reanalyzed. Samples with target analytes identified but quantitated below the concentration established by the lowest calibration standard should be reported as below the minimum reporting limit (MRL).

C. Report results for Part A anions in mg/L.

D. Report NO_3^- as N

Traceability

A record shall be maintained on all reference materials within Element. 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 Element as well as on the container's label.

All working standards made from reference materials shall be labeled with a unique Element ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date, date opened, and the logbook where information is recorded. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded within Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

IX. INSTRUMENT INFORMATION

Analyst should confirm the following:

1. **Start Up routine for instrument is as follows:**

a. **Turn Power on to autosampler, conductivity detector, eluant generator and pump in any order.**

b. **Turn on the Helium gas supply~ 80 to 100. Ensure gas lines to bottles not in use are off. Ensure air supply to the injection valve is on.**

c. **Close the vent valves on sparging bottles and allow head pressure to build for a few moments. DI H2O bottles should be 7 to 10 psi.**

d. **Open the eluant supply valve(s) for the bottles in use. Check for sputter after eluant flow has started.**

e. **Inject 5 mL of DI water into the pump head and clean the pump head. Also check the frits on the guard column and change if necessary. If necessary to change guard column frits check primary column frits as well.**

f. **Load the auto sampler cartridges and put autosampler into RUN state.**

g. **To vent airlocks, Run eluant with pump on and SRS off, open waste valve-> bottom door top black knob, just for a few seconds and close it.**

h. **Turn on the SRS power. NOTE: NEVER TURN ON THE SRS POWER SUPPLY WITHOUT THE PUMP GOING FIRST!!! Use either LOCAL/DIRECT CONTROL to enter commands at keypads, or REMOTE/DIRECT CONTROL to use the direct control option from the RUN menu within PEAKNET.**

i. **After System has come to equilibrium, load sequence and run.**

2. **Shut Down routine for instrument is as follows:**

   a. **If the instrument is not going to be operated for a period of time, run deionized water through the eluant lines for ~ 30 minutes to an hour to rinse the lines.**

   b. **Stop the OFF/ON pump and then select SRS-OFF.**

   c. **Close gas supply valves and eluant valves on the eluant bottles. Turn off the supply. Is not necessary to vent the eluant bottles.**

   d. **Power down the modules in any order.**

3. **General Sample Loading and Run Set-up.**

   a. **Enter Peak-net Software from Desktop.**

   b. **Loading a Run: Click on <u>Schedule</u>. The headings within the Schedule Editor are SAMPLE, SAMPLE TYPE, LEVEL, METHOD and DATA FILE.**

      i. **Name each sample under the SAMPLE heading column. (, CCV, CCB, CRL, BS, BLK, sample #'s, etc.)**

      ii. **SAMPLE TYPE is sample unless loading a calibration curve.**

iii. LEVEL designations are used only when assigned to a calibration curve.

iv. Enter method name under METHOD heading. In most cases, date of most recent calibration in Anions Method file will be used.

v. Enter the date under the DATA FILE heading. The program will then sequentially assign the data file names based on the date.

vi. All other column headings are defaulted to enter "1". Samples requiring dilution should be left at "1" and manual calculation is required.

vii. To include a command to Shut Down the pump at the end of the run: Name the row following the last sample, Pump Off under the sample heading. It is not necessary to include a vial in the corresponding position in the autosampler. Sample type is Sample, and Method is entered as <pumpoff.met>.

Typical run-log:

1	**Blank**
2	**CCV**
3	**CCB**
4	**CRL1**
5	**CRL2**
6	**BS1**
7	**BLK1**
8	**Sample**
9	**Sample @ 10X**
10	**Sample @ 50X**
11	**Sample**
12	**Sample**
13	**Sample**
14	**CCV**
15	**CCB**
16	**Sample**
17	**Sample**
18	**Sample**
19	**Sample**
20	**Sample**
21	**Sample**
22	**Batch #-MS1**
23	**Batch #-MSD1**
24	**Batch #- DUP1**
25	**Sample @ 10X**
26	**Sample**
27	**Sample**
28	**Sample**
29	**CCV**
30	**CCB**
31	**Sample**
32	**Sample**

33 Sample
34 Sample
35 Sample
36 Batch #-MS2
37 Batch #-MSD2
38 Batch #- DUP2
39 Sample
40 Sample
41 Sample
42 Sample
43 CCV
44 CCB
45 Sample
46 Sample
47 Sample
48 CCV
49 CCB
50 pumpoff

c. Save a schedule under File/Save as, using the date as the title of the Schedule.

d. When saved, exit out of Schedule Editor.

e. Load autosample cartridges in the same order as the scheduled run. After putting the cartridges in the autosampler, switch the autosampler to RUN using the Hold/Run button.

f. Assuming that the Pump is equilibrated with steady eluant baseline/uniform conductivity, enter into the <u>RUN</u> page.

g. Go to File to Open Method. Open correct method <date> of most recent calibration. This will begin pumping eluant at 1.0 mL/minute and turn on the SRS pump at 300 *u*amps voltage.

h. Next, go to file to Open Schedule. Open newly created schedule for the day.

i. When Method and Schedule are opened, go to Run and click on Start. The autosampler will inject into the first sample and the run should continue until completion.

X. <u>POLLUTION PREVENTION</u>

A. Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option.

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.

C. For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

XI. WASTE MANAGEMENT

A. The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3 from method 300.1.

XII. CORRECTIVE ACTIONS

A. INSTRUMENT RELATED

1. ICV not within $\pm$ 10%

    a. If the problem is with the solution.

        i. Reprepare, obtain new stock if necessary.

    b. If the problem is with the calibration. Recalibrate thru analysis of appropriate standards and recheck ICV.

2. CCV not within $\pm$ 10%

    a. If the problem is with the solution.

        i. Reprepare, obtain new stock if necessary.

    b. If the problem is with the calibration.

        i. Recalibrate thru analysis of appropriate standards and reprepare/reanalyze the previous ten samples according the following guidelines.

            a. If the CCV was biased high, any of the previous ten samples which were BMDL do not require reanalysis.

            b. If the CCV was biased low, the previous ten samples must be reanalysed.

3. CCB not >+ MDL (USACE) (**For DOD QSM Ver. 3 no analyte detected >2xMDL, frequency- beginning and ending a run and every 10 samples**) or + RL or CRDL for others and CLP

   a. If the CCB is biased high.

      i. Any samples BDL or greater than 10X the CCB bias need not be reanalyzed.
      ii. Any samples above the detection limit but less than 10X the CCB level must be reanalyzed after the problem is corrected.

   b. If the CCB is biased low.

      i. Any samples greater than 10X the absolute CCB bias need not be reanalyzed.
      ii. All other samples must be reanalyzed after the problem is corrected.

4. BS not within our in-house generated control limits (or +10%).

   a. If the problem is with the instrument.

      i. Reanalyze when instrument is in control.

      ii. If biased high, the impact upon the data user must be evaluated. The samples will be re-extracted or the data will be qualified on the final report.

C. SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within +20%

   i. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within +20%

   i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

   ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report.

XIII. SOURCES/REFERENCES:

1. Standard Methods for the Examination of Water and Wastewater, Method 4110B, "Anions by Ion Chromatography", 18th Edition of Standard Methods (1992).

2. Dionex, System DX500 Operation and Maintenance Manual, Dionex Corporation, Sunnyvale, California 94086, 1996.

3. Method Detection Limit (MDL) as described in "Trace Analyses for Wastewater," J. Glaser, D. Foerst, G. McKee, S. Quave, W. Budde, Environmental Science and Technology, Vol. 15, Number 12, page 1426, Decemvber, 1981.

4. American Society for Testing and Materials. Test Method for Anions in Water by Chemically – Suppressed Ion Chromatography D4327-91. Annual Book of Standards, Vo. 11.01 (1993).

5. Code of Federal Regulations 40, Ch. 1, Pt. 136, Appendix B; MDL determination.

6. Hautman, D.P. & Bolyard, M. Analysis of Oxyhalide Disinfection By-products and other Anions of Interest in Drinking Water by Ion Chromatography. Jour. Of Chromatog., 602, (1992), 65-74.

7. USEPA Methods 300.0; *Method for Determination of Inorganic Substances*(EPA/600/R-93/100) / *Method for the Determination of Organic and Inorganic Compounds in Drinking Water* (Vol. 1, EPA 815-R-00-014).

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

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: EPA 300.0 Anions by Ion Chromatography
Instrument is a Dionex DX-500 system. Equipped with Guard Column, Analytical Column, Conductivity Suppressor, Conductivity Detector, and Eluant Generator.

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 CRL meet control limits?				
6. Did BS, Laboratory Fortified Blank or blank spike meet control limits?				
7. Did MS/MSD meet control limits? Did Duplicate meet control limits?				
8. Was the Blank below the project required detection limits?				
9. Did you return samples back to cold storage immediately after use?				
10. Were samples analyzed for Nitrate (as N) and Nitrite (as N) done within the 48-hr holding time?				
11. Sample preparation information is correct and complete.				
12. Were all samples filtered through a 0.45*u*m filter?				
13. Analytical results are correct and complete.				
14. The appropriate SOP's have been used and followed.				
15. Raw data" including all manual integration's have been correctly interpreted.				
16. "Special" sample preparation and analytical requirements have been met.				
17. 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: _____

Second-Level Review: _____ Date: _____

ADDITIONAL INFORMATION:

All manual integrations of standards have been checked and confirmed by supervisor or qualified personnel.

____yes ____no Data confirmed by_____Date:_____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP153 **REVISION #: 04** **EFFECTIVE DATE: 20100907**

SULFIDE by METHOD 376.1 and STANDARD METHODS SM4500S F (21st ED, TITRIMETRIC, IODINE) WITH SAMPLE PRETREATMENT TO REMOVE INTERFERING SUBSTANCES OR TO CONCENTRATE THE SULFIDE

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/9/10

Changes Summary

Revision 04, 09/07/10

- The SOP is an update from Revision 03 dated 05/27/09
- 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.

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1.0 Identification of the Test Method

1.1 This SOP is compliant with EPA method 376.1 and SM4500S F (21st ED).

2.0 Applicable Matrix or Matrices

2.1 This method is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.

3.0 Detection Limit

3.1 All limits reported for various programs are listed in Table 1 of this SOP.

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. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

4.2 This method is applicable to the measurement of total and dissolved sulfides in drinking, surface and saline waters, domestic and industrial wastes.

4.3 Acid insoluble sulfides are not measured by the use of this test. (Copper sulfide is the only common sulfide in this class.).

4.4 This method is suitable for the measurement of sulfide in concentrations above 1 mg/L.

5.0 Summary of the Test Method

5.1 Excess iodine is added to a sample which has been treated with zinc acetate to produce zinc sulfide. The iodine oxidizes the sulfide to sulfur under acidic conditions. The excess iodine is back-titrated with sodium thiosulfate.

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. Some definitions are included within this section.

6.2 **Preparation Blank (PB)-** Laboratory reagent water that is treated exactly as a sample including exposure to all glassware, equipment, and reagents that are used with other samples. The PB is used to determine if method analytes or other interferences are present in the laboratory environment, the reagents or the apparatus.

6.3 **Blank Spike (BS)-** An aliquot of reagent water or other blank matrices to which known quantities of the method analyte is added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The LCS is given a unique identifier so that it is traceable to its source and concentration and expiration date.

6.4 **Analysis Batch-** An analysis batch is a group of twenty field samples, a preparation blank, a laboratory control sample, a matrix spike, and either a matrix spike duplicate or a sample duplicate.

6.5 **Sample Duplicate-** Two sample aliquots, taken in the laboratory from a single sample bottle, and analyzed separately with identical procedures. Analysis of sample one and sample two indicate precision associated specifically with the

laboratory procedures, removing any associated variables attributed by sample collection, preservation, or storage procedures.

6.6 **Matrix Spike-** An aliquot of an environmental sample to which known quantities of the method analyte is added in the laboratory. The matrix spike is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analyte in the sample matrix must be determined in a separate aliquot and the measured values in the matrix spike corrected for background concentrations.

6.7 **Method Detection Limit (MDL)--** The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.

6.8 **Performance Evaluation Sample (PE)--** A certified solution of method analytes whose concentration is unknown to the analyst. Often, an aliquot of this solution is added to a known volume of reagent water and analyzed with procedures used for samples. Results of analyses are used to determine statistically the accuracy and precision that can be expected when a method is performed by a comptetent analyst.

7.0 Interferences

7.1 The iodometric method suffers interferences from reducing substances that react with iodine, including thiosulfate, sulfite and various organic compounds, both solid and dissolved. Interferences due to sulfite, thiosulfate, iodide and many other soluble substances are eliminated by first precipitating ZnS in the samples, removing the supernatant, and replacing it with distilled water.

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 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.3 Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be warn when handling samples and chemicals.

8.4 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.

8.5 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.

8.6 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

9.1 25 mL burette

9.2 250 mL Bel-Arte plastic bottle

9.3 Vacuum pump
9.4 Magnetic stirrer with Teflon coated stirring bars
9.5 Buchner funnel
9.6 500 mL Erlenmeyer flask

10.0 Reagents and Standards

10.1 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.

10.2 All working standards made from reference materials shall be labeled with unique ID number with complete information on preparation date, concentration of each compound, solvent, preparer's name, expiration date and the LIMS where information is recorded. 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 weighting 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.

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

10.4 Use DI water to prepare reagents.

10.5 Zinc acetate, 2N: dissolve 220 grams Zn $(C_2H_3O_2)_2 \cdot 2\ H_2O$ in 870 mL D.I. water; this makes 1 liter solution.

10.6 Sodium Hydroxide, 6N: dissolve 240 grams of NaOH in about 600 mL of D.I. water. Dilute to 1 liter.

10.7 Hydrochloric acid, 6N: add 250 mL concentrated HCl to 250 mL D.I. water, mix well.

10.8 Standard iodine solution, 0.025N: dissolve 20 to 25 grams KI in a little water and add 3.2 grams iodine. After iodine has dissolved, dilute to 1000 mL in a volumetric flask. Standardize against 0.0250N Na_2S0_3, using thyodene as indicator. **(Purchased commercially)**

10.9 Standard sodium thiosulfate titrant solution, 0.025N: dissolve 6.205 grams $Na_2S_20_3 \bullet 5H_20$ in distilled water. Add 1.5 mL 6N NaOH or 0.4 grams solid NaOH and dilute to 1000 mL. Standardize with bi-iodate solution. **(Purchased commercially)**

10.10 Standard potassium bi-iodate solution, 0.025ON: dissolve 812.4 mg $KH(IO_3)_2$ in distilled water and dilute to 1000 mL. Standardization of thiosulfate--dissolve approximately 2 grams KI, free from iodate, in an Erlenmeyer flask with 100 to 150 mL distilled water. Add 1 mL 6N H_2SO_4 or a few drops of concentrated H_2SO_4 and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate liberated iodine with thiosulfate titrant, adding 1 scoop thyodene toward end of titration, when a pale straw color is reached. When the solutions are of equal strength, 20.00 mL 0.0250N $Na_2S_20_2$ should be required. If not, adjust the $Na_2S_20_3$ solution to 0.025ON using N1V1 = N2V2 equation.

10.11 Thyodene.

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 must be taken with a minimum of aeration. Sulfide may be volatilized by aeration and any oxygen inadvertently added to the sample may convert the sulfide to an unmeasurable form.

11.3 Samples are taken in 250 ml Bel-Arte bottles. Preserve with zinc acetate 2N and 6N NaOH. There should be no air-space in the container.

11.4 The holding time for sulfides preserved in this manner is 7 days.

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. Four Blank Spikes (BS) are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.2.1. The BS is made by diluting 33.5 mL of 5% sodium Sulfide solution to 200 mL with de-ionized water and preserving with NaOH. The true value is determined by titration using 3 mLs of this solution and diluting to 250mLs.

12.3 Analyze a duplicate and second source check with each analytical batch. The second source check standard can be purchased commercially.

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 Traceability: All reference materials must be assigned an Element # and labeled accordingly. 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. 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 and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded in the batch sheet in Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

14.0 Procedure

14.1 Pretreatment:

14.1.1 If sample has not been preserved with zinc acetate and NaOH, place 3 to 5 pasteur pipettes of 2N zinc acetate solution into a 500 mL glass bottle, fill with sample, and add 10 drops 6N NaOH solution. Stopper with no air bubbles under stopper and mix by rotating back and forth vigorously about a transverse axis. Vary volume of reagents added according to sample so that the resulting precipitate is not excessively bulky and settles readily. Add enough NaOH to produce a pH above 9.

14.1.2 Let precipitate settle for 30 minutes. The treated sample is relatively stable and can be held for several hours; however, if much iron is present, oxidation may be fairly rapid. Holding time for this sample is 7 days.

14.2 Preparation and Titration of Sample:

14.2.1 Mark meniscus of sample volume on side of bottle so you can measure sample volume. Filter precipitate through glass fiber filter paper 11.0 to 12.5 cm in a Buchner funnel. Save the filter and all precipitate and discard filtered sample.

14.2.2 Measure exactly, amount of standard iodine solution (estimated to be an excess over the amount of sulfide present in the sample--usually 2 to 10 mL) into a 500 mL Erlenmeyer flask or original sample bottle. Add distilled water, if necessary, to bring volume and iodine solution to 20 mL.

14.2.3 Add 2 mL 6N HCl.

14.2.4 Place filter with precipitate, making sure you wipe sides of Buchner funnel to get any precipitate clinging to the sides, into bottle with iodine solution and acid. Add 200 mL D.I. water.

14.2.5 Fill a 25 mL Burette with 0.0250 N sodium thiosulfate solution.

14.2.6 Put a small stirring bar in sample and place on magnetic stirrer. Stir bar shaking slowly.

14.2.7 Titrate slowly with sodium thiosulfate titrant adding 1 scoop thyodene reagent toward the end of the titration, when a pale straw color is reached. The sample will go from straw yellow to a dark blue when thyodene is added. It should take only 2 or 3 drops of titrant to bring sample to the endpoint of clear at this point. Record mL of titrant used. Sample will turn back blue but the first change from blue to clear is the endpoint.

14.2.8 Do a blank with each set of samples taken D.I. water through the entire procedure the same as the samples including pretreatment. D.I. water is usually ~1.0 mg/L sulfide.

14.2.9 Discard titrated sample. Rinse out bottle and measure volume of sample used by filling bottle to the calibration mark on the side of bottle with water and pouring water into a graduate cylinder.

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 One milliliter 0.0250N iodine solution reacts with 0.4 mg S2-:

$$\text{Mg S2-/L} = \frac{\text{(AXB) - (CXD)}}{\text{Sample}} \text{ x 16000 x (Ratio of final to mL initial volume)}$$

where:

A = mL standard iodine solution
B = normality of iodine solution
C = mL $Na_2S_2O_3$ solution
D = normality of $Na_2S_2O_3$ solution
Final volume = 200 mL
Initial volume = measured volume of original sample

16.0 Method Performance

16.1 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. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

16.2 Four LCS's are reported with a precision and accuracy sheet for the DOC. See section 12.2 for LCS preparation.

17.0 Pollution Prevention

17.1 Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The EPA has established a preferred hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the Agency recommends recycling as the next best option. 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.

17.2 For information about pollution prevention that may be applicable to laboratories and research institutions, consult "Less is Better: Laboratory Chemical Management for Waste Reduction," available from the American Chemical Society's Department of Government Regulations and Science Policy, 1155 16th Street N.W., Washington D.C. 20036, (202) 872-4477.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 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.

18.2 If the LCS fails (exceeds 80-120 %). Contact Lab supervisor.

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

19.1 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

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

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.

20.2 The Environmental Protection Agency requires that laboratory waste management practices be conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes should be characterized and disposed of in an acceptable manner. The Agency urges laboratories to protect the air, water, and land by minimizing and controlling all releases from hoods and bench operations, complying with the letter and spirit of any waste discharge permit and regulations, and by complying with all solid and hazardous waste regulations, particularly the hazardous waste identification rules and land disposal restrictions. For further information on waste management consult the "Waste Management Manual for Laboratory Personnel," available from the American Chemical Society at the address listed in Section 14.3 from method 300.1.

21.0 References

21.1 Methods 376.1 and Standard Methods 21st Edition SM4500S F.

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters with the applicable RL and lowest calibration standard.

22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

Table1

Analytes by EPA SM 21st Ed 4500S2-F, Method 376.1	AQUEOUS RL (mg/L)
Sulfide	2.0

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Useability**
BLK	One per prep batch	• Must be less than the LOD	• 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-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
BS	One per prep batch	80 to 120% difference	• Follow guidelines from SOP QS05
BSD	One per prep batch, when MS/MSD not included.	80 to 120% difference	• Follow guidelines from SOP QS05
MS	One per prep batch, if sample volume available.	75 to 125% difference	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	75 to 125% difference	• Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year to meet specific state requirements.	• Parameter must be detected • the response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• NA to titration and gravimetric tests	
LOQ Verification	Every quarter	• NA to titration and gravimetric tests	

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. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. 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

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP 154 **REVISION #: 06** **EFFECTIVE DATE: 20100907**

TOTAL ALKALINITY , CARBONATE, BICARBONATE

METHOD USEPA 310.1, SM2320B
20th and 21st Edition

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: Betty DeVille Date: 9/9/10

Changes Summary

Revision 06, 09/07/10

- The SOP is an update from Revision 05 dated 05/27/2009
- 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.

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1.0 Identification of the Test Method

1.1 The GC/FID system is used to analyze TOTAL ALKALINITY, CARBONATE, BICARBONATE utilizing Method EPA 310.1 & Standard Methods 2320B

2.0 Applicable Matrix or Matrices

2.1 This method is applicable to drinking, surface, and saline waters, and domestic and industrial wastes. Soils are leached 10 grams to 100 mLs and the analysis performed on the leachate.

2.2 The method is suitable for all concentration ranges of alkalinity; however, appropriate aliquots should be used to avoid a titration volume greater than 10 mL.

3.0 Detection Limit

3.1 The detection limit is 1.0 mg/L $CaCO_3$.

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. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5.0 Summary of the Test Method

5.1 An unaltered sample is titrated to an electrometrically-determined endpoint of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way. The calculation for total alkalinity (in calculation section of this SOP) is then used to calculate.

5.2 When the sample is being analyzed for phenolthalein alkalinity, carbonate, bicarbonate and total alkalinity, method 2320B is used. With this method, an unaltered sample is titrated to an electrometrically-determined endpoint of pH 4.5. The sample must not be filtered, diluted, concentrated, or altered in any way. The sample is then titrated to a pH exactly 0.3 pH units lower and the calculation for 2320B (in calculation section of this SOP) is used to calculate the samples for phenolthalein alkalinity, total alkalinity, carbonate and bicarbonate results. See note at the end of Section 14 (Procedure) after step 14.14 for samples with pH greater than 8.3.

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 Substances, such as salts or weak organic and inorganic acids present in large amounts, may cause interference in the electrometric pH measurements.

7.2 For samples having high concentrations of mineral acids, such as mine wastes and associated receiving waters, titrate to an electrometric endpoint of pH 3.9, using the procedure in *Annual Book of ASTM Standards*, Part 31, "Water," p. 115, D-1067, Method D (1976).

7.3 Oil and grease, by coating the pH electrode, may also interfere, causing sluggish response.

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 Care should be used in handling all samples.

8.3 Safety glasses must be worn in the lab at all times. The use of latex 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 that need special consideration have applicable notes on the sample logs.

8.5 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.

8.6 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

8.7 Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be warn when handling samples and chemicals.

9.0 Equipment & Supplies

9.1 pH meter that uses a glass electrode and can be read to 0.01 pH units. The analyst will note on the data which pH meter (either the Corning 240 or Orion 420A) is used.

9.2 50 mL disposable beakers with wide enough mouths to allow room for burette tip and pH probe.

9.3 10 mL Class A microburette.

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:

Reagent 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 Ultra Scientific, NSI, Ricca 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.

10.2 Sodium carbonate solution, approximately 0.05N: Place 2.5 $\pm$0.2 g (to nearest mg) Na_2CO_3 (dried at 250°C for 4 hours and cooled in desiccator) into a 1-liter, Class A, volumetric flask and dilute to the mark. The Na_2CO_3 solution must be disposed of after one week.

10.3 Standard Acid (sulfuric or hydrochloric), 0.1N (high titrant): **May be purchased from a vendor, make sure that ACS grade or better is purchased. Also a Certificate of Analysis must be obtained and kept on file when a purchased solution is used.** Dilute 3.0 mL concentrated H_2SO_4 or 8.3 mL concentrated HCl to 1 liter with distilled water. Dilute 40 mL of 0.05N Na_2CO_3 solution to 100 mL with deionized water and titrate potentiometrically with the Standard Acid to a pH of about 5. Lift electrode and rinse into beaker. Boil solution gently for 3 to 5 minutes under a watch glass cover. Cool to room temperature. Rinse cover glass into beaker. Continue titration to the pH Inflection point (3 units lower). This standardization must be done at least every three months. Calculate normality using:

$$N = \frac{A \times B}{53.00 \times C}$$

where: A = g Na_2CO_3 weighed into 1 liter
B = mL Na_2CO_3 solution
C = mL acid used to inflection point

10.4 Standard Acid (sulfuric or hydrochloric), 0.02N (low titrant): **May be purchased from a vendor, make sure that ACS grade or better is purchased. Also a Certificate of Analysis must be obtained and kept on file when a purchased solution is used.** Dilute 200.0 mL of 0.1000 N Standard Acid to 1 liter with distilled water. Standardize by potentiometric titration of 15.0 mL 0.05N Na_2CO_3 solution every three months as above.

11.0 Sample Collection, Preservation, Shipment, and Storage

11.1 No preservation necessary except to keep chilled to 4°C until sample is analyzed. Do not open sample bottle until analysis.

11.2 The holding time for these samples is 14 days. Example: If sampled on November 1 at 10 a.m., analysis must be performed by November 14 at 10 a.m.

12.0 Quality Control

12.1 Run a laboratory control sample (LCS) for each batch of samples (maximum of 20 samples per day). If the LCS does not fall in the range of 80 to 120%, corrective action must be taken to find the problem and correct it.

12.2 Run a preparation blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than the reporting limit.

12.3 Analyze a sample duplicate every 10 to 20 samples (depending on specific client requirements). Relative percent difference (RPD) on duplicates should be less than 20%.

12.4 The Excel file for calculations is located in "V:\Wetchem\TESTS\CarbonDioxide Alkalinity\".

12.5 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

<u>Spike – Sample</u>

% Recovery = True Value x100

Relative percent difference is calculated as follows, with everything in concentration:

$$RPD = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

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.

14.0 Procedure

14.1 Write down time test started.

14.2 Fill 10 mL microburette with Standard Acid.

14.3 Pick titrant according to estimated total alkalinity. For example, a drinking water or groundwater sample would probably use the 0.020 N titrant and a wastewater sample would probably use the 0.10 N titrant. Historical data is very useful for this.

A sample size of 25 mL is usually appropriate. If you use less than 1 mL of your high titrant, then you must titrate a new sample using low titrant. Using less than 1 mL of your low titrant is valid. When the samples are soils a 12 gram portion diluted to 120 mLs is used.

14.4 Sample size should be such that a sufficiently large volume of titrant is used (1 to 10 mL titrant).

14.5 Standardize and calibrate the pH meter according to laboratory procedures as explained. Using the Corning 240 pH meter, first calibrate the meter by putting the probe (which has been filled with the correct filling solution) in 7 buffer, setting the pH at 7; then in 4 buffer, setting the pH at 4, and then checking 7 again to make sure it still reads 7, if an adjustment is made to the 4 buffer then check that buffer again as well. The calibration buffers must be within ±0.05 pH units of the true value. Then check the 10 buffer. The reading should be within ±0.10 pH units. If not, recalibration is necessary. Record this information in the appropriate log book. If automatic temperature compensation is not provided, make titration at 25 $\pm$2°C. Check the buffer every 3 hours after calibration. The reading should be within ±0.20 pH units.

14.6 Carefully pour 25 mLs into disposable beaker by gently pouring down the side of the vessel so as to have the least aeration to sample as possible. Place a small magnetic stirring bar in vessel and start magnetic stirrer at medium to slow stirring.

Note 1: When soils are being analyzed 12 grams is weighed into a 120 mL bottle and diluted to 120 mLs. Place in the shaker for one hour. Mix the sample well and use 25 mLs to analyze. Make sure you get a representative sample for analysis.

Note2: Where sample volume is adequate when using low titrant, a sample volume of 100 to 200 mL should be used and titration should be performed using a 10 mL microburette.

14.7 Place pH probe (which has been rinsed with DI water and patted dry with a Kimwipe) in the sample such that the probe tip is not touching the sides or bottom of the flask or beaker. If the probe has a protective cover, this is not a consideration.

14.8 Make sure there are no air bubbles at the bottom of filled burette. Wipe tip of burette so that no extra drops are clinging to it. Place tip of burette into mouth of vessel so that it is above the surface of the sample but is not touching the sides of the flask and drops can go nowhere but into the sample (e.g., drops from burette are not going onto pH probe or walls of flask but directly into sample).

14.9 Titrate a blank and an LCS first. This will let you know that the normalities of titrant are correct. If the result is out of the acceptable range of the LCS, run a duplicate LCS. If still out of range, find another second source and if still incorrect, restandardize titrant. First double-check titrant normality.

14.10 Record sample pH after reading is stable for 5 to 10 seconds.

14.11 Titrate sample to pH 4.5. This must be done slowly so as not to miss the exact pH. Record titrant volume.

14.12 The minimum titrant volume to be employed using high titrant is 1 mL. If high titrant is being used, go to low titrant; if low titrant doesn't work, use more sample. Be aware of sample volume that may be needed for other analyses. Do not dilute.

14.13 When titrating the sample, be sure to allow time for the pH to equilibrate so that the inflection point will not be passed.

14.14 Place pH probe in 7 buffer between samples. If this does not read 7, recalibrate between 4 and 7.

14.15 Potentiometric titration of low alkalinity

14.15.1 For alkalinity of < 20 mg/L titrate 100 – 200 mL as above using a 10 mL microburet and 0.02 N acid solution.

14.15.2 Stop titration at pH in range of 4.3-4.7, record volume and exact pH. Very carefully add titrant to lower pH exactly 0.3 pH units and record volume. See note below.

Note : For method 2320B, if the pH of the sample is above 8.3, the sample needs to be titrated for phenolthalein alkalinity, first check original pH. If it is not above 8.3, the phenolthalein result will be below the minimum detection limit. If the original pH is above 8.3, titrate sample as in above procedure but down to 8.3 instead of 4.5, and record titrant volume in box A on the alkalinity bench sheet. Then proceed with the regular procedure titrating to pH 4.5, and recording this result in box B on the alkalinity bench sheet, then carefully titrate exactly 0.3 pH units lower to pH 4.2 and record titrant volume at this level, in box C on the alkalinity bench sheet.

14.16 Potentiometric titration of high alkalinity: Use a sufficiently large volume of titrant (>20 mL in a 50 mL buret) to obtain good precision while keeping volume low enough to permit sharp endpoint.

14.16.1 For >1000 mg $CaCO_3$/L use 0.1 N titrant

A. For alkalinity of > 1000 mg $CaCO_3$/L, titrate 25 – 50 mL as above using a 50 mL burette and 0.10 N acid solution.

B. Stop titration at pH in range of 4.5, record volume and exact pH. See note above.

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 detection limit is 1.0 mg/L $CaCO_3$.

15.3 Potentiometric titration to pH 4.5 (high alkalinity)

$$\text{Total Alkalinity mg/ L } CaCO_3 = \frac{A \times N \times 50{,}000}{\text{mL of Sample}}$$

Where: A = mL Standard Acid to pH 4.5
N = Normality Standard Acid

Potentiometric titration of low alkalinity = T below

15.4 Method 2320B calculations

Use the following notation in below calculations:

P = Phenolthalein alkalinity
T = Total alkalinity

$$P = \frac{A \times N \times 50{,}000}{\text{mL of Sample}} \qquad T = \frac{(2B-C) \times N \times 50{,}000}{\text{mL of Sample}}$$

If P	= 0	Carbonate	= 0	Bicarbonate	= T
If P	< 1/2T	Carbonate	= 2P	Bicarbonate	= T-2P
If P	= 1/2T	Carbonate	= 2P	Bicarbonate	= 0
If P	> 1/2T	Carbonate	= 2(T-P)	Bicarbonate	= 0
If P	= T	Carbonate	= 0	Bicarbonate	= 0

Where: A = mL titrant to pH 8.3
B = mL titrant to pH 4.5
C = mL titrant to pH 4.2
N = normality Standard Acid

16.0 Method Performance

16.1 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 is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

16.2 Forty analysts in seventeen laboratories analyzed synthetic water samples containing increments of bicarbonate, with the following results:

Increment as Alkalinity mg/L, CaCO3	Precision as Standard Deviation mg/L, CaCO3	Accuracy as Bias, %	Accuracy as Bias, mg/L, CaCO3
8	1.27	+10.61	+0.85
9	1.14	+22.29	+2.0
113	5.28	-8.19	-9.3
119	5.36	-7.42	-8.8

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. Please see Waste Disposal SOP QS14, for instruction of proper disposal of waste generated from this area.

18.0 Data Assessment and Acceptance Criteria for Quality Control Measures

18.1 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

19.1 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.

19.2 If the preparation blank is higher than the reporting limit, all samples less than ten times the concentration of the blank must be reanalyzed.

19.2.1 If the laboratory control sample (LCS) is out of the range of 80 to 120%, and the % recovery is high (higher than 120%), only sample concentrations less than the reporting limit are acceptable data. Otherwise, all data with concentrations above the method detection limit must be reanalyzed with an LCS in the range of 80 to 120%. If the LCS is low (less than 80%), all samples must be reanalyzed.

19.2.2 If the relative percent difference (RPD) between the sample and the sample duplicate are out of the range of 20%, the sample should be repeated one more time to make sure that the problem is not caused by analyst's error. If the RPD is still higher than 20% the sample is flagged on the final report with a "*".

20.0 Waste Management

20.1 Please see Waste Disposal SOP QS14, for instruction of proper 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.

21.0 References

21.1 Methods for the Chemical Analysis of Water and Wastes, EPA Series Method 310.1.

21.2 Standard Methods for the Examination of Water and Wastewater, 20th and 21st Edition, p. 2-28, method 2320B, (2005).

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, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.2 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

DEFINITIONS

°C - degrees centigrade
CRDL - contract required detection limit
%D - percent difference
DOD - Department of Defense
LCS - laboratory control sample
LCSD - laboratory control sample duplicate
MDL - method detection limit
mg/KG - milligrams per kilogram
mg/L - milligrams per liter
µL - microliter
µm - micrometer
ml - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
%RSD - percent relative standard deviation
SOP - standard operating procedure

Refer to SOP QS08 for further definitions.

TABLE 1 – Analytes, Reporting Limit (RL), & Low Calibration Standard (Units mg/L)

Parameter	RL	LowCal
TOTAL ALKALINITY , CARBONATE, BICARBONATE	1.0	NA

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
MB	One per prep batch	• Must be less than the LOD	• 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-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
LCS	One per prep batch	Most stringent criteria listed within the LIMS.	• Follow guidelines from SOP QS05
LCSD	One per prep batch, when MS/MSD not included.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MS	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	Criteria listed within LIMS or specified by client.	• Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
MDL Study	Once per year as required for state certifications.	• Calculated value must be greater than 10% of the Spike Level • Calculated value must be less than the Spike level • Percent recovery must be between 60-140% of the true value of each analyte.	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

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. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. 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

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 164 REVISION #: 15 EFFECTIVE DATE: 20100325

DISTILLATION OF AQUEOUS/SOLID SAMPLES FOR CYANIDE, TOTAL AND AMENABLE

REFERENCE:
SW846 METHOD 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM 4500-CN C,G, 18th, 19th ED. / CLP ILMO 4.1
See Addendum for USEPA CLPILM 05.2 (Aqueous, Soil/Sediment)

APPROVALS:

Lab Director: _____ Date: 3/30/10

Data Quality Manager: _____ Date: 3/30/10

Section Supervisor: _____ Date: 3/30/10

Changes Summary:

Revision Date: 03/25/2010

- The SOP has been reviewed for accuracy and completeness. No changes have been made.

DISTILLATION OF CYANIDE, TOTAL AND AMENABLE

REFERENCE:
SW846 METHOD 9012A, USEPA Methods 335.1, 335.4, Standard Methods SM 4500-CN C,G, 18th, 19th ED. / CLP ILMO 4.1
See Addendum for USEPA CLPILM 05.2 (Aqueous, Soil/Sediment)

I. SCOPE AND APPLICATION

A. This method is applicable to the distillation of cyanide from drinking, surface, and saline waters, domestic and industrial wastes, and soil/sediments.

B. The limit of detection for waters is 0.0050 mg/L and the limit of quantitation is 0.010 mg/L. The limit of detection for soils is 0.13 mg/kg and the limit of quantitation is 0.25 mg/kg.

II. SUMMARY OF METHOD

A. The cyanide as hydrocyanic acid is released from cyanide complexes by means of a reflux distillation operation, and absorbed in a scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined colorimetrically.

B. Cyanide is defined as cyanide ion (CN^-) and complex cyanides converted to hydrocyanic acid (HCN) by reaction in a reflux system of a mineral acid in the presence of magnesium ion.

III. SAMPLE HANDLING AND PRESERVATION

A. The sample should be collected in plastic or glass bottles of 100 mLs. All bottles must be thoroughly cleansed and thoroughly rinsed to remove soluble material from containers.

B. Oxidizing agents such as chlorine decompose most of the cyanides. Test a drop of the sample with potassium iodide-starch test paper (KI starch paper); a blue color indicates the need for treatment. Add ascorbic acid, a few crystals at a time, until a drop of sample produces no color on the indicator paper. Then add an additional 0.06 g of ascorbic acid for each liter of sample volume.

C. Samples (not including soils/sediments) must be preserved with 2 mL of ten normal sodium hydroxide per liter of sample (pH $\geq$12) at time of collection.

D. Samples should be analyzed as rapidly as possible after collection. If storage is required, the samples should be stored in a refrigerator to maintain temperature at 4°C.

E. The holding time for cyanide is 14 days counting the day of sampling as the first day. This applies for soils as well as waters. Samples must be distilled and analyzed within 14 days of sampling.

IV. INTERFERENCES

A. Interferences are eliminated or reduced by using the distillation procedure.

B. Sulfides adversely affect the colorimetric procedures. Samples that contain hydrogen sulfide, metal sulfides, or other compounds that may produce hydrogen sulfide during the distillation, must be treated by adding lead acetate, or if the sulfide concentration is too high, add powdered lead carbonate [$Pb(CO_3)_2$] to avoid significantly reducing pH. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper. Filter sample before raising pH for stabilization.

C. High results may be obtained for samples that contain nitrate and/or nitrite. During the distillation, nitrate and nitrite will form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose under test conditions to generate HCN. Nitrate and nitrite interference is eliminated by adding 0.5 ml of sulfamic acid solution (VII.A.7) before distillation.

D. The presence of surfactants may cause the sample to foam during refluxing. If this occurs, the addition of an agent such as Dow Corning 544 antifoam agent will prevent the foam from collecting in the condenser.

V. SAFETY

A. **CAUTION: KCN is highly toxic. Avoid contact with standard solutions.**

B. If an alkaline solution containing simple cyanide is acidified, the simple cyanide will be released.

C. The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential heath hazard and exposure should be as low as reasonably achievable. Cautions are included for known extremely hazardous materials or procedures.

D. Your laboratory manager and/or Safety Officer is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of Material Safety Data Sheets (MSDS) are made available to all personnel involved in the chemical analysis. A formal safety plan is also available. Use proper personal protection equipment, PPE, such as safety glasses, gloves and laboratory coats should be warn when handling samples and chemicals.

VI. EQUIPMENT/APPARATUS

A. Reflux distillation apparatus is a midi-distillation apparatus which uses Hammett Scientific Glassware Note: This glassware is made to fit our specific block digester for cyanide. Glassware orders must be made in advance since they are not made until ordered.

VII. REAGENTS AND STANDARDS PREPARATION

A. Reagents

1. **Sodium Hydroxide, 0.25N**: Dissolve 10 g grams of NaOH in DI water and dilute to 1 liter with DI water. Store in a labeled plastic container at room temperature.

2. **Sulfuric Acid 1:1:** Slowly add 500 mL of concentrated H_2SO_4 to 500 mL DI water. Use caution because solution will become extremely hot!! Allow to cool while continuing to stir. Store in a labeled container at room temperature.

4. **Magnesium Chloride Solution**: Weigh 510 g of $MgCl_2 \cdot 6H_2O$ into a 1 liter flask; dissolve and dilute to 1 liter with DI water. Store in a labeled plastic container at room temperature.

5. **Chlorine Bleach Made with Sodium Hypochlorite**, such as Chlorox.

6. **Rhodanine Indicator**:
   Dissolve 20 mg of p-dimethyl-amino-bensalrhodanine in 100 mL acetone.

7. **Sulfamic Acid:** Dissolve 40 g of sulfamic acid in DI water, dilute to 100 mL (saturated solution). **NOTE:** When making this reagent for use with the maxi-distillation system the 40 g of sulfamic are diluted to one liter with DI water

8. **Phenolphthalein solution:** Commercially prepared.

9. **Lead Acetate Test Paper**: Commercially prepared.

10. **Acetate Buffer Solution, pH 4.0:** Dissolve 146g anhydrous $NaC_2H_3O_2$, or 243 g $NaC_2H_3O_2.3H_2O$, in 400 mL distilled water, add 480 g conc. acetic acid, and dilute to 1 L with chlorine-demand-free water.

11. **Lead Acetate**

12. **Powdered Lead Carbonate [$Pb(CO_3)_2$]**

B. Standards

1. Traceability

a. All reference materials must be recorded in Element and given an element identification number. 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 Element as well as on the container's label.

b. All working standards made from reference materials shall be labeled with a unique ID number from Element with complete information on preparation date, concentration of

each compound, solvent, preparer's name, expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded on the bench sheet. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

2. **Preparation**

a. Stock Cyanide Solution at 1000 mg/L: Commercially prepared.

b. Working Standard Cyanide Solution: Prepare fresh daily by diluting 1 mL of stock cyanide solution to 100 mL in a volumetric flask with using 0.25N NaOH as diluent. 1 mL - 10 µg CN. Record the preparation in Element.

c. Performance Evaluation (PE) samples which we have analyzed and received true values for are used for the Laboratory Control Samples. These PE samples are given a unique identifier and all information such as expiration date (typically one year from the date made), source, concentration and preservation are recorded in Element and the Element # assigned is used to label this PE sample and is used on all data to trace it back to its source and true concentration.

VIII. CALIBRATION

A. Not applicable. See SOP-175 "**POST DISTILLATION ANALYSIS OF CYANIDE BY THE LACHAT"** for analysis.

IX. PROCEDURE

A. Pretreatment for cyanides amenable to chlorination

1. Two sample aliquots are required to determine cyanides amenable to chlorination. Place one 25 mL aliquot or a volume diluted to 25 mL, in a 100 mL beaker, under the hood, add a stirring bar, and place on a magnetic stirrer, and start stirring bar turning.

2. Check pH and adjust to between 11 and 12 units, with 1.25N NaOH. Use NaOH pellets if solution is highly buffered.

3. Add sodium hypochlorite chlorine bleach (Chlorox) solution dropwise until a drop of well-mixed sample turns KI starch paper a distinct blue color. Add about 10 drops excess bleach. (Caution: the initial reaction product of alkaline chlorination is the very toxic gas cyanogen chloride.) Maintain this excess for one hour, continuing agitation. Check periodically during the hour (15 minute intervals) to make sure an excess is maintained. If necessary, add additional hypochlorite bleach solution. Also check the pH and adjust back up to 11 to 12 units if necessary.

4. After one hour, add 0.025 g portions of ascorbic acid until KI starch paper shows no residual chlorine. Add an additional 0.025 g of ascorbic acid to ensure the presence of excess reducing agent.

5. Turn off stirring plate and remove stirring bar. Proceed with distillation as in the procedure.

6. Test for total cyanide in both the chlorinated and unchlorinated aliquots. (The difference of total cyanide in the chlorinated and unchlorinated aliquots is the cyanide amenable to chlorination.)

B. Spikes and high and low check standards

1. **Spikes**: Matrix spikes are prepared by diluting 2.5 mL of the High DCV solution in #3 below, to 25 mL with sample (adding 22.5 mL of sample and 2.5 mL of DCV high to the distillation tube). The concentration of this spike is 0.10 mg/L.

2. **Low Distilled Check standard (low DCV).** Dilute 2.0 mL of the 10 mg/L working standard to 100 mLs in a volumetric flask. Then dilute 6.0 mLs of this solution to 25 mLs with 0.25N NaOH, to make a 0.048 mg/L standard.

3. **High Distilled Check standard (high DCV).** Dilute 10 mLs of the 10 mg/L working standard to 100 mLs in a volumetric flask with DI water. Then dilute 5 mLs of this solution to 25 mLs with 0.25N NaOH, to make a 0.20 mg/L standard.

C. Distillation

1. For Waters: Place 25 mL of sample, or an aliquot diluted to 25 mL, in a sample tube. 0.25 N NaOH must be used for all sample dilutions following the distillation.

2. For Soils or Sludges:

   a.) 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.

b.) Accurately weigh a representative 1-g minimum (record actual weight) portion of wet-weight sample (unless there are project-specific requirements where a lower detection limit is required) and transfer it to a sample tube. Add 25 mL of 0.25 N NaOH. Shake or stir the sample so that it is dispersed.

3. Add one drop of Phenolphthalein solution to verify preservation. A positive test is indicated by a bright pink color. If not add a small amount of NaOH.

4. To test for sulfide, place a drop of sample on lead acetate paper, previously moistened with acetic acid buffer solution (pH 4). A positive test is indicated by a black color on the paper.

   a.) If positive the sample will need to be filtered through 934AH filter paper. (4.5cm size) The filtrate will be treated and the filter added back to the treated filtrate before distillation.

   b.) Treat filtrate with powdered lead carbonate [$Pb(CO_3)_2$]. Repeat test until a drop of treated sample no longer darkens the acidified lead acetate test paper.

   c.) Filter the resulting precipitate (should form a lead sulfide which will be black in appearance).

   d.) Once the filtrate is treated and filtered, the filter with the original solids from the pretreated sample will be added back to the treated filtrate and then distilled.

5. Add 25 mL of sodium hydroxide solution 0.25N to the absorber and assemble the apparatus as shown in Fig. 1.

6. Start a slow stream of air entering the boiling flask by adjusting the vacuum source. Adjust the vacuum such that air bubbles from the thistle tube in the flask at a rate of 3 to 5 per second.

   Note: The bubble rate will not remain constant after the reagents have been added and while heat is being applied to the flask. It may be necessary to readjust the air rate occasionally to prevent the solution in the sample tube from boiling and spewing out through the thistle tube.

7. Turn on condenser water.

8. Add 0.5 mLs of sulfamic acid through the thistle tube.

9. Slowly add 2 mL 1:1 H_2SO_4 through the thistle tube.

10. Add 2 mL 2.5 M $MgCl_2$ through the thistle and rinse it down with deionized water.

11. Reflux at 100°C for 90 minutes, turn off heat, allow to cool with pump on for 15 minutes.

12. Disconnect absorber and transfer the solution from the absorber into a storage container.

14. Record distillation information in the distillation log. Include date, analyst, sample number, client, matrix, position, sample mass / volume, distillation time, cool down time, when transferred to analyst, when analyzed.

X. CALCULATIONS

A. Sample volumes / masses must be recorded accurately in the distillation log to allow the analyst to correctly calculate the final concentration of cyanide correctly.

XI. DISTILLATION GLASSWARE CLEANING & LABELING

A. The distillation glassware is cleaned thoroughly with hot soapy water between distillations. 1:1 HCl is aerated through the frits on the block to ensure that the frits are clean. Deionized water is forced up through the glassware to rinse it. The cold fingers are dipped in mild acid, rinsed and wiped off.

B. All glassware must be labeled and each position's label must be recorded in the distillation log with the sample that was distilled in that particular vessel.

C. When sample concentrations are at or exceed 1000 mg/L a deionized water blank must be distilled in the exact glassware used for that sample before processing any other client's sample to ensure that the glassware is cleaned and has no residual cyanide to carryover.

XII. QUALITY CONTROL

A. With each distillation batch of samples (maximum of 20 samples) a preparation blank (BLK) must be distilled.

B. Each distillation batch of aqueous samples **requires a laboratory control sample (BS) each day or per 20 samples whichever is more frequent.** Under CLP the BS serves as the distilled ICV.

C. With each distillation batch of soil samples a soil BS (boiling chips are used) **each day or per 20 samples whichever is more frequent.**

D. A high and low check standard (DCV/ICV) must be distilled each day or per 20 samples whichever is more frequent. A non-distilled check standard must be prepared daily. This will be used to verify the preparation of the DCV if the DCV results are outside the specification limits. Under CLP the aqueous BS serves as the ICV.

E. A sample spike (MS) and sample spike duplicate (MSD) are performed with each set of twenty samples. CLP requires 1 duplicate and 1 spike per batch. All QC samples should be taken through the whole procedure including distillation. Spikes are added to the boiling flask in the same manner as standards adding 25 mL of samples or a dilution as used for the unspiked

sample. Spike concentration is listed in the method. The addition of the spiking solution must be witnessed and such must be noted in the digestion log.

XIII. CORRECTIVE ACTION

A. If any problems, such as low or high recovery for check standards or BSs occur, contact the Group Leader. Problems most frequently encountered (for the distillation procedure) are listed below.

B. Recoveries are affected in various ways.

1. If the spiking solution is not prepared properly (e.g., making the proper dilutions), the check standard will be out of control. Caution should be used in calculating the dilution and performing the pipetting.

2. Also, the air flow from the vacuum pumps can cause low recovery. Pay attention to the air flow (it should be in the area of 1 to 2 bubbles per second). If it is too great the HCN does not have an adequate amount of time to be absorbed by the NaOH solution.

3. If the heating block is not heating properly, this will also lead to low recovery. During the distillation, periodically check the sample tubes for the presence of heat (tubes should be very hot to the touch).

4. The addition of all reagents, including the spiking solution, is essential. Without the $MgCl_2$, 1:1 H_2SO_4, or spiking solution, the percent recovery will suffer greatly for complex cyanide. The recovery of simple cyanides will usually be unaffected.

5. If the volume of 0.25 N NaOH and reagent water is not accurate in the NaOH absorber tubes, low recoveries will usually occur.

B. Spike recoveries are affected in similar ways. Refer to the above suggestions. Also, if possible, do not spike a sample that is known to be high in cyanide. This may cause recovery and calculation problems.

XIV. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper 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. The ICV shall be distilled.

2. Boiling chips are to be added to each sample. Midi-Distillation is reflux 1.5 hours then the heat and vacuum is turned off and the samples cool an additional 15 minutes.

3. The QC criteria for both the Distillation Check QC and the distilled ICV is ±15%.

4. A CRI is required at the beginning and end of each run and for every 20 samples. The QC criteria for the CRI is ±30%.

5. Rounding rule for the appropriate level of precision is that the figure following those to be retained is $\geq$ 5, round up: otherwise round down. (examples: 1.5 and 2.5 would be 2 and 3 respectively rounded up; 1.4 and 2.4 would be 1 and 2 respectively rounded down) Please see Exhibit B, Section 3, (3.3.9.1) of SOW ILM0 5.2 for more guidance on rounding significant figures.

XV. DEFINITIONS

See SOP-431 for a list of definitions

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

INORGANICS: SOP175 **REVISION #: 11** **EFFECTIVE DATE: 20100907**

POST-DISTILLATION ANALYSIS FOR CYANIDE USING LACHAT FLOW INJECTION ANALYZER METHODS 335.4;(SW846) 9012A, Standard Methods 21st Edition USEPA-CLP 4.1, (NJDEP does not accept CLPILM 04.1 after June, 2003) Addendum for USEPA CLPILM 05.2 AQUEOUS &SOIL/SEDIMENT

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/9/10

Section Supervisor: Betty DeVille Date: 9/9/10

Changes Summary

Revision 11, 09/07/10

- The SOP is an update from Revision 10 dated 12/15/09
- 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.

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19. Contingencies for Handling out-of-control or unacceptable data
20. Waste Management
21. References
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1.0 Identification of the Test Method

1.1 This SOP is compliant with EPA method 335.4 and SW9012A/B.

2.0 Applicable Matrix or Matrices

2.1 This SOP is applicable to the determination of Cyanide in Aqueous and Soil / Sediment matrices.

3.0 Detection Limit

3.1 All limits reported for various programs are listed in Table 1 of this SOP.

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. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte.

5.0 Summary of the Test Method

5.1 Cyanide from alkaline distillates is converted to cyanogen chloride, CNCl, by reaction with chloramine-T at pH lower than 8. The CNCl then forms a red-blue dye by reacting with pyridine-barbituric acid reagent. The color is read at 570 nm.

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 For total cyanide, most interferences are eliminated or minimized by the distillation procedure. Sulfides are removed by treatment with lead acetate or powdered lead carbonate. See Empirical Laboratories Method SOP-164 for distillation procedure.

Note: method 9012A/B says that sulfides "should" be treated with bismuth nitrate. Empirical Laboratories attempted to use that procedure and found that bismuth nitrate also removes cyanide from standard solutions as well. This sulfide treatment is not used. See Empirical Laboratories Method SOP-164 for distillation procedure.

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 & Supplies

9.1 Lachat Quick Chem AE Automated Ion Analyzer
9.2 Cyanide manifold (Lachat)
9.3 Instrument Information

Analyst should confirm the following:

A. Timing:

Sample throughput: 80 samples/h; 40 s/sample
Pump speed: 35 RPM

Cycle Period: 45 s
Inject to start of peak period: 28 s
Inject to end of peak period: 61 s

B. QuikChem AE Settings:

1) Parameter, Data Window:

Top Scale Response: 0.50 abs
Bottom Scale Response: 0.00 abs

2) Segment/Boundaries

A - 0.50 mg CN/L
F - 0 mg CN/L

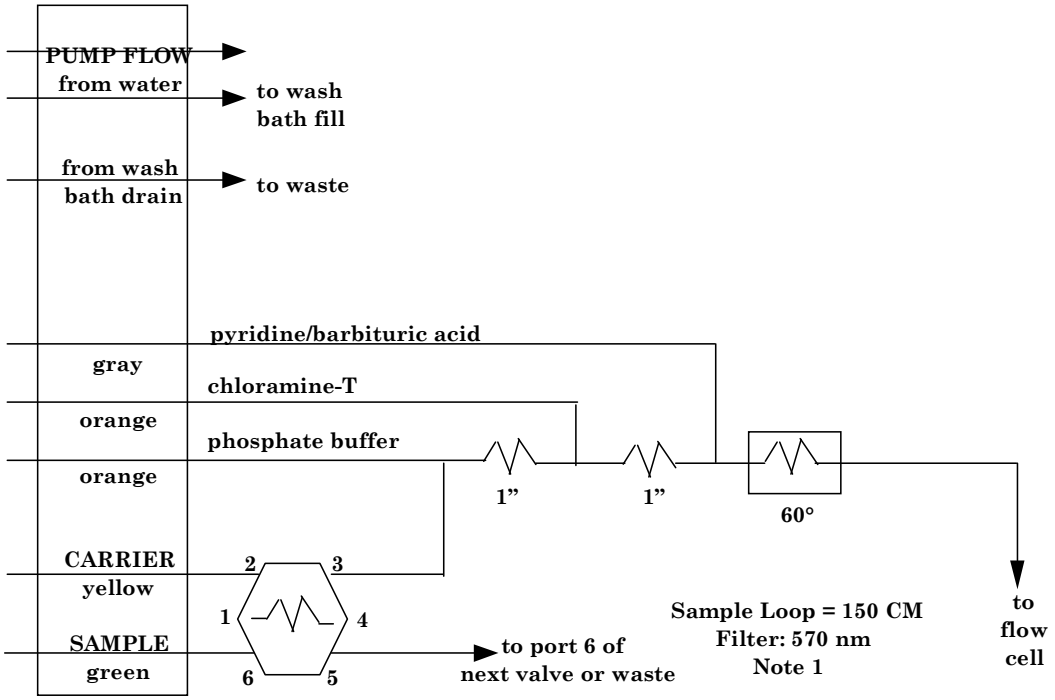
3) Results/Approval, Reports

In the default Report definition file (RDF), change:
-Set Default Chord 0 to
-Set Default Chord 3
(Peak should be centered in chord 3)
This change must be made to both the sample and the calibration RDF's.

C. System Notes:

1. Allow enough time for heating unit to warm¬ up to 60°C with D.I. water pumping through all reagent lines at full speed. (approximately 30 min.)

MANIFOLD DIAGRAM



CARRIER is 0.25 M Sodium Hydroxide

1" is 70.0 cm of tubing on a 1 in coil support

Note1: A 3.0 m x 0.022" id back pressure loop is placed at the exit of the flow cell.

Manifold Diagram Revision Date: 13 Ma

APPARATUS: Standard valve, flow cell, and detector head modules are used. The box shows 650 cm of heated tubing.

All manifold tubing is 0.8 mm (0.032 in) i.d., This is 5.2 µL/cm.

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:

Reagent 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.

10.2 Use deionized water (<10 megaohm) for all solutions.

10.3 **Reagent 1. Carrier, 0.25 M Sodium Hydroxide:** In a 1L vol. flask add 200 mL of 1.25N NaOH (which is used in the SOP 164 distillation method) to approximately 600 mL of D.I. water. Dilute to 1 L, invert to mix. This will ensure that the concentration of the NaOH used for the distillates will be the same concentration used for the carrier solution.

10.4 **Reagent 2. Phosphate Buffer, 0.71 M**: In a 1 L volumetric flask, dissolve 97 g anhydrous potassium dihydrogen phosphate (potassium phosphate, monobasic, anhydrous, KH2PO4) in approximately 800 mL water. Dilute to the mark and invert to mix. Prepare fresh monthly.

10.5 **Reagent 3. Chloramine-T Hydrate:** To a 500 mL volumetric flask add about 250 mL water, then add 2.0 g chloramine-T [CH3C6H4SO2N(Cl)Na x H2O]. Dilute to the mark and invert to mix. Prepare fresh daily. The shelf life of the chloramine-T can be a critical factor in the Lachat run. New Chloramine-T should be ordered every 6 months. (Analyst discretion should be used).

10.6 **Reagent 4. Pyridine-Barbituric Acid Reagent:** In the fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100.0 mL water, rinsing down the sides of the beaker to wet the barbituric acid. Add 75 mL pyridine (C5H5N) with stirring and mix until the barbituric acid dissolves. Add 15 mL concentrated hydrochloric acid (12 M HCl) and mix. Transfer to a 1 L volumetric flask, dilute to the mark, and invert to mix. This reagent is stable for approximately six months if stored in a cool, dark place.

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 The color reaction is pH sensitive. Therefore, distillates and standards should be carefully matched with respect to NaOH concentration.

11.3 The distillates are 0.25 N NaOH.

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. Four LCSs are analyzed at 0.10ug/L. See **Table 2** for acceptance criteria.

12.3 QA/QC, distilled:

12.3.1 PB (preparation blank):

25 mL of DI water is distilled. When brought up to 25 mL volume following the distillation process a 0.25 M NaOH solution is obtained. A blank is distilled every batch of samples. If the absolute value of the PBW is not below the CRDL or the RL or ½ the CRDL for Navy Projects the analyst's supervisor should be consulted before proceeding.

12.3.2 DCV (distilled calibration verification)/ICV (distilled/CLP)

a. The DCV high and low are check standards generally of a concentration of about 0.200 mg/L for the high and 0.050 mg/L for the low. A DCV high and low are distilled with every batch of distilled samples. There is a maximum of 20 samples in a batch. If the DCV is not within +15 % of the

undistilled value the analyst's supervisor should be consulted before proceeding. A NCR will be required if the limit is exceeded.

b. CLP requires the ICV to be distilled and from a second source. Must be analyzed immediately after calibration. Since the matrix for the soil LCS is Teflon chips the LCS serves as a distilled ICV. The control limit is + 15%.

12.3.3 Blank Spike (BS or laboratory control sample)

The laboratory control sample is a second source check on the calibration and must be run once every 20 aqueous samples. The control limits are 80 to 120%. If the BS is not within control limits the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in the distillation log.

12.3.4 BS (BS or laboratory control sample-**soil)**

The LCSS is a second source check with a soil matrix (Teflon chips are used for this purpose) and must be run once every 20 solid phase samples. If the BS is not within control limits the analyst's supervisor should be consulted before proceeding. This sample is given a unique identifier in Element.

12.3.5 MS/MSD (matrix spike/matrix spike duplicate)

A spike and spike duplicate (CLP requires 1 duplicate and 1 matrix spike) are done with each batch or 20 samples. The spike concentration is 0.10 mg/L. The analyst's supervisor should be consulted before proceeding if the percent recovery for the MS or MSD is not within ±20 percent of the spike concentration,.

12.3.6 Documentation of Capability (DOC) - Each analyst must perform a DOC to demonstrate proficiency with this method. Refer to SOP QS05 for guidance.

12.4 QA/QC, undistilled:

12.4.1 ICV (initial calibration verification)

The ICV is an undistilled second source check standard (except when analyzing by CLP methods and then the ICV is distilled) made from a second source working standard solution. The concentration is usually around the midpoint of the calibration curve (about 0.500 mg/L). The ICV is analyzed at the beginning of each Lachat run. The analyst's supervisor should be consulted before proceeding, if the ICV is not within ±10 percent of its true value. This calibration verification standard must be prepared fresh daily.

12.4.2 ICB (initial calibration blank)

The ICB is 0.25 M NaOH solution and follows the ICV at the beginning of each Lachat run. If the absolute value of the ICB is not <± 2x MDL or < + CRDL or ½ the RL for Navy Projects, the analyst's supervisor should be consulted before proceeding.

12.4.3 CCV (continuing calibration verification) from primary std. that curve is made from.

The CCV will be analyzed after every 10 samples and at the end of the tray. The sampling of the CCV's will be done automatically by the Lachat if it is included in the tray definition. The CCV scheduling can be done under the Data Quality Management option in the Tray Definition screen. If the CCV is not within +10 percent of its true value a CHK STD failure signal will be given by the Lachat and the analyst's supervisor should be consulted before proceeding.

12.4.4 CCB (continuing calibration blank)

This sample verifies the instrumental baseline and must be analyzed immediately after every ICV/CCV. If the absolute value of the ICB/CCB is not below the LOD the analyst's supervisor should be consulted before proceeding.

12.5 Calibration Approval:

12.5.1 For a cyanide calibration to be approved, segment (A-F) of the curve must have a correlation of at least 0.995 in chord 3.

12.5.2 Method Detection Limit (MDL), Empirical Laboratories Reporting Limit (ERL), Contract Required Quantitation Limit (CRQL) and Analyte Wavelength: (MDLS are performed annually to meet specific state requirements or whenever a change in the method is 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 Traceability: All reference materials must be assigned Element # and labeled accordingly. 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. 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 and expiration date. Reagents shall be labeled with date received and expiration date, if applicable. All of the information described above shall also be recorded in Element. Measurements made during standards preparation (e.g., from weighing operations, volume diluted to, etc.) shall also be recorded in the batch sheet in Element. There should be no container with sample, sample extract, standard solution, etc. that is not correctly labeled and properly stored.

13.3 Standard 1.

1000 mg CN/L [~1,000 mg CN-/L (Stock)]: The Stock Standard is purchased from a vendor. CAUTION: KCN is highly toxic. Avoid contact with standard solutions. Use gloves and protective equipment when handling standards and samples. This solution is given a unique identifier. Record preparation in Element and label standards with appropriate Element #'s.

13.4 Standard 2. Working Standard (10.0 mg CN/L)

Pipette 2.00 mL Standard 1 into a 200 mL volumetric flask. Dilute to the mark with Reagent 1, 0.25 M Sodium Hydroxide. Invert to mix.

13.5 Set of six calibration Standards:

10 mL of 10 mg/L diluted to 100 mL = 1.0 mg/L std
5.0 mL of 10 mg/L diluted to 100 mL = 0.500 mg/L std
2.0 mL of 10 mg/L std diluted to 100 mL = 0.200 mg/L std
1.0 mL of 10 mg/L std diluted to 100 mL = 0.100 mg/L std
4 mL of 0.500 mg/L std diluted to 100 mL = 0.020 mg/L std
2 mL of 0.500 mg/L std diluted to 100 mL = 0.010 mg/L std
Blank consists of 0.25 M NaOH
10 mL of 10.0 mg/L std diluted to 200 mL = 0.500 mg/L chk std

13.6 The diluent for all standards is 0.25 M NaOH.

14.0 Procedure

14.1 The samples for analysis come directly from the midi scale distillation procedure.
14.2 See "Equipment and Supplies" for how to set up the manifold for use for cyanide analysis.
14.3 Be sure to read through all components of this SOP for procedural instructions.

14.4

ANALYTE	WAVELENGTH
Cyanide	570.0

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. The ICV shall be distilled.

2. Boiling chips are to be added to each sample. Midi-Distillation is reflux 1.5 hours then the heat and vacuum is turned off and the samples cool an additional 15 minutes.

3. The QC criteria for both the Distillation Check QC and the distilled ICV is ±15%.

4. A CRI is required at the beginning and end of each run and for every 20 samples. The QC criteria for the CRI is ±30%.

5. Rounding rule for the appropriate level of precision is that the figure following those to be retained is ≥ 5, round up: otherwise round down. (examples: 1.5 and 2.5 would be 2 and 3 respectively rounded up; 1.4 and 2.4 would be 1 and 2 respectively rounded down) Please see Exhibit B, Section 3, (3.3.9.1) of SOW ILM0 5.2 for more guidance on rounding significant figures.

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 Aqueous Samples: The Lachat reports the concentration of the final distillate in mg/L. The method detection limit (MDL) is 0.005 mg/L and the reporting limit (RL) is 0.010 mg/L.

15.3 Solid Samples: The Lachat reports the concentration of the final distillate in mg/L. Results for solid samples should be in mg/kg and can be obtained by entering the appropriate dilution factor when defining the tray. Conversion from mg/L to mg/kg can be accounted for by the following formula:

From this formula a dilution factor can be obtained. For example if 2 grams of soil were used then this would be a x 250 dilution. When the x 0.50 correction factor is included a final manual dilution factor of x 125 is obtained.

N would be the value obtained from the Lachat without a correction factor. The minimum value for N is 0.01 mg/L soln.

15.4 Data reporting

1. Reduce data to the result which will be reported.
2. Complete the data review checklist (attached). This must be completed and attached to each set of USACE data.

16.0 Method Performance

16.1 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 is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

16.2 The IDOC is analyzed at 4 times the LOQ or 0.080 mg/L for waters and 3.0 mg/kg. All standard results should be in the range of 80 to 120%. The continuing DOC is four LCS's analyzed and a completed precision and accuracy sheet with all LCS within the control limits of 80 to 120%.

16.3 LOD/LOQ are analyzed quarterly and an MDL study is performed annually for specific state requirements. The LOD must be three times the noise and is analyzed at 0.010 mg/L for waters and 0.25 mg/kg for soils. The LOQ must be in the range of 50 to 150% and is analyzed at a concentration of 0.020 mg/L for waters and 0.75 mg/kg for soils.

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

18.1 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.

18.2 **Instrument Related**

1. ICV not within ± 10%
    a. If the problem is with the solution.
        i. Re-prepare, obtain new stock if necessary.
    b. If the problem is with the calibration.
        i. Recalibrate thru analysis of appropriate standards and recheck ICV.
2. CCV not within ± 10%
    a. If the problem is with the solution.
        i. Re-prepare, obtain new stock if necessary.
    b. If the problem is with the calibration.
        i. Recalibrate thru analysis of appropriate standards and re-prepare/reanalyze the previous ten sample according the following guidelines.
            a. If the CCV was biased high, any of the previous ten samples which were BMDL do not require reanalysis.
            b. If the CCV was biased low, the previous ten samples must be re-analysed.
3. Ending CCB not ± RL or CRDL **or ½ the CRDL for Navy Projects**
    a. If the CCB is biased high.
        i. Any samples BDL or greater than 10X the CCB bias need not be reanalyzed.

ii. Any samples above the detection limit but less than 10X the CCB level must be reanalyzed after the problem is corrected.

b. If the CCB is biased low.

i. Any samples greater than 10X the absolute CCB bias need not be reanalyzed.
ii. All other samples must be reanalyzed after the problem is corrected.

18.4 Distillation Related

1. The preparation blank is not less than the ± RL or CRDL **or ½ the CRDL for Navy Projects**.

a. If the problem with the instrument.

i. Analyze a CCB to determine this.
ii. If the problem was with the instrument correct the situation and reanalyze the preparation blank.

b. If the problem is with the distillation.

i. All associated samples which are <MDL or have a level of cyanide greater than 10X the level found in the preparation blank can be reported. If the level of cyanide in an associated sample is not <MDL nor greater than 10X the level found in the preparation blank, the sample must be redistilled/reanalyzed or reported as qualified. The project manager or QA manager will make this determination.

2. LCS not within control limits (±20%).

a. If the problem is with the instrument.

i. Reanalyze when instrument is in control.

b. If the problem is with the distillation.

i. If biased low, associated samples must be redistilled.
ii. If biased high, the impact upon the data user must be evaluated. The samples will be redistilled or the data will be qualified on the final report.

3. Distilled check standard not within control limits of ±15%.

a. If the problem is with the instrument.

i. Reanalyze when instrument is in control.

b. If the problem is with the distillation.

i. If biased low, associated samples must be redistilled.
ii. If biased high, the impact upon the data user must be evaluated. The samples will be redistilled or the data will be qualified on the final report.

18.5 SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20%

    i. The associated sample data must be qualified on the final report.

2. Spike analysis recovery not within ±20%

    i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.

    ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report.

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

19.1 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

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 Lachat manual; Quick Chem method 10-204-00-1-A.
21.2 U.S. Environmental Protection Agency, Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, Revised March 1983, Method 335.4.
21.3 Standard Methods for the Examination of Water and Wastewater, 21st Edition, APHA-AWWA-WCPC, Park 413D, pp. 370-372.
21.4 SW-846, 9012A, Revision 1, December 1996.
21.5 SW-846, 9012B, Revision 2, November 2004.
21.6 U.S. Environmental Protection Agency, C.L.P. S.O.W. ILMO 4.1.
21.7 QuickChem AE, Automated Ion Analyzer Training Manual, Lachat Instruments.
21.8 QuickChem AE, Automated Ion Analyzer Software Reference Manual, Lachat Instruments.

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, all applicable parameters, with the applicable RL and lowest calibration standard.
22.2 Table 2, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.
22.3 Table 3, Technical Completeness / Accuracy Checklist
22.4 Table 4, Data Reviewers Checklist
22.5 Validation data would be actual documentation (eg: a pdf email from a regulator explaining the approach to a method, etc.) or a side by side study performed to reach to our approach on how we handle the method.

Table 1
Aqueous and Soil Method Detection Limits(MDL), Empirical Laboratories Reporting Limits(ERL), CLP OLM04.1 & OLM05.2 Contract Required Quantitation Limits (CRQL)

Analytes by EPA 335.1, 335.3, 9012A/B, SOW 4.1 & 5.2	AQUEOUS MDL(ug/L)	AQUEOUS ERL(ug/L) And lowest calibration standard	AQUEOUS CRQL ILMO 4.1 (ug/L)	AQUEOUS CRQL ILMO 5.2 (ug/L)	SOLID/SOIL MDL (mg/Kg)	SOLID/SOIL ERL (mg/kg) And lowest calibration standard	SOLID/SOIL CRQL ILMO 4.1 (mg/Kg)	OLID/SOIL CRQL ILMO 5.2 (mg/Kg)
Cyanide	5.0	10	10	10	0.125	0.25	0.25	0.25

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
Calibration Curve	• Prior to analyzing any samples • A minimum of 5-points for linear fits • Low standard at the RL/LOD level	• For Linear fits a RF of 0.995 • Average RSD ≤ 20% • Must follow curve processing requirements from SOP QS08	• Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards Samples cannot be analyzed until there is a passing calibration
ICV	Alternate source standard to be analyzed after every calibration curve	Must meet 85 to 115%	• Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources
CCV	• At the beginning of every sequence • For every 10-client samples	• ± 10% difference	• Follow guidelines for SOP QS05
Closing CCV	• At the end of every sequence	• ± 10% difference	• Follow guidelines for SOP QS05
BLK/CCB	One per prep batch/ after every CCV	• Must be less than ½ RL/Must be less than the LOD	• 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-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
BS	One per prep batch	• ±20% difference	• Follow guidelines from SOP QS05
BSD	One per prep batch, when MS/MSD not included.	• ±20% difference	• Follow guidelines from SOP QS05

Table 2 - Method Quality Control Requirements Summary

QC Check	Minimum Frequency / Requirements	Acceptance Criteria	Corrective Action for Failures / Data Useability
MS	One per prep batch, if sample volume available.	• ±20% difference	• Follow guidelines from SOP QS05
MSD	One per prep batch, if sample volume available.	• ±20% difference	• Follow guidelines from SOP QS05
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Average percent recovery should be between 80-120%, with a 20% standard deviation.	• Re-prep and / or re-analysis
MDL Study	Once per year for specific state requirements	• Calculated value must be greater than 10% of the Spike Level • Calculated value must be less than the Spike level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOD Verification	Every quarter	• Parameter must be detected • 2nd column / detector confirmation is required • the response must be 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	Every quarter	• Bias Requirement: 50-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05

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. If the data was entered into LIMS manually, was a check of all entered values performed
7. Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
8. Were proper data qualifiers applied to the data in LIMS
9. 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:	**9012A/B (Cyanide)**

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 LCS or blank spike meet control limits?				
6. Did MS/MSD meet control limits?				
7. Was the preparation (Method) Blank below the project required detection limits?				
8. Did you return samples back to cold storage immediately after use?				
9. Was distillation 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.				

ANALYST DATA REVIEW CHECKLIST
9012A/B (Cyanide)

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 202 **REVISION #: 23** **EFFECTIVE DATE: 20100909**

GC/MS VOLATILES BY EPA METHOD E624 & SW846 METHOD 8260B INCLUDING APPENDIX IX COMPOUNDS

APPROVALS:

Lab Director: _____ Date: 9/9/10

Data Quality Manager: _____ Date: 9/9/10

Section Supervisor: _____ Date: 9/9/10

Changes Summary

Revision 23, 09/09/10

- This SOP is an update from Revision 22 dated 09/30/09.
- Tables 1 and 2 have been updated with appropriate reference updates.
- Tables 5-7 have been added.

Revision 22, 9/30/09

- The SOP is an update from Revision 21 dated 09/11/08
- 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.

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1. Identification of the Test Method

1.1 This SOP is compliant with methods – EPA Method 624 and SW-846 Method 8260B

2. Applicable Matrix or Matrices

2.1 This SOP is applicable to – The analysis of volatile organic compounds in a variety of matrices including but not limited to soils, sediments, ground and surface waters, aqueous sludge, oily wastes, etc.

3. Detection Limit: See **Table 1** of this SOP.

4. Scope of Application, Including components to be Analyzed

4.1 This SOP is based primarily on SW-846 Method 8260B. Methods SW-846 Method 8000B; *Federal Register* Method 624; and CLP Method for Volatiles have also been used in the development of this SOP. The analyses by these various methods are clearly defined in the respective regulatory manuals. A good understanding of these different methods is essential to the performance of each method. Each parameter that is analyzed and reported under the scope of this SOP is listed in **Table 1** of this SOP. When applicable, surrogate and Internal Standard Analytes are listed and indicated as such within this table.

5. Summary of the Test Method

5.1 After sample preparation, the sample is introduced into the GC/MS generally using purge and trap but sometimes using direct injection (see SW-846 Methods 5030B, 5035 and 3585 for preparation). In purge and trap, the analytes are stripped from the sample using helium and trapped on an adsorbent tube. The tube is heated while being backflushed with helium to carry the analytes to the GC/MS system. The analytes are separated in the gas chromatograph by a combination of the temperature 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 of the sample. Analytes are quantitated relative to known standards using the internal standard method.

6. 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. Interferences

7.1 Section 3.0 of SW-846 Method 8260B details interferences and potential problems which may be encountered when dealing with volatile analyses.

8. Safety

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

9. Equipment & Supplies

9.1 GC : HP 5890 or 6890, temperature programmable, suitable for split or splitless injection.

9.2 Column: DB-VRX 60 meter x 0.25 mm I.D. 1.4 µm film thickness or 20 meter x 0.18 mm ID 1.0 µm film thickness silicon coated fused silica capillary column or equivalent.

9.3 M.S.: HP 5971, 5972 or 5973 capable of scanning 35 to 500 amu every one second or less, using 70 volts electron energy in electron impact ionization mode. The MS is capable of producing a mass spectrum for p-Bromofluorobenzene, BFB, which meets all tuning criteria for EPA methods [when 1 µL (50 ng) of the GC/MS tuning standard is introduced to the GC.]

9.4 Purge and Trap Unit

9.4.1 Concentrators: Tekmar LSC 2000 or Tekmar/Dohrmann 3000/3100 Sample Concentrator equipped with Supelco trap number 2-1066-U or 2-4920-U VOCARB 3000 providing good delivery for all target compounds.

9.4.2 Autosamplers: Varian Archon 51 position programmable autosampler with 5ml to 25ml water and heated soil capability.

9.5 Acquisition Software: HP chemstation system interfaced to the GC/MS. The system acquires and stores data throughout the chromatographic programs.

9.6 Data Processing Software: TargetDB on Windows NT data system 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 of any EICP between specified time or scan-number limits. NBS75K mass spectral library is installed.

9.7 Microsyringes – 1.0, 5.0,10, 25, 100, 250, 500 and 1000 µL.

9.8 Syringes – 5, 25 and 50 mL, gas-tight with Luer end.

9.9 Balance - analytical, 0.0001 g; top-loading, 0.01 g.

9.10 Disposable pasteur pipets.

9.11 Volumetric flasks, Class A - 2 mL, 5 mL, 10 mL, 50 mL, 100 mL and 250 mL with ground-glass stoppers.

9.12 Spatula - stainless steel.

9.13 Glass scintillation vials - 20mL with screw caps.

9.14 Nitrile Gloves

9.15 pH paper (measures pH from 0-14).

10. 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 Organic-free reagent water - obtained from the charcoal filter system in the VOA laboratory.

10.3 Methanol - Purge and trap grade (EM-Omnisolv EM-0482-6 or equivalent)

10.4 Methanol - suitable for use in gas chromatography (B&J Omnisolv MX0484- 1, or equivalent)

10.5 Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).

10.6 Stock standards are purchased in mixtures from reputable vendors. The date they are received is noted on the label. The date they are opened is noted on the label and recorded in the LIMS system along with their lot number and vendor and given a sequential number. Each standard label is completed with the standard number, name, preparation date, expiration date, solvent and analyst initials. Stock standards, when opened, have an expiration date of 6 months, **except for gas standards for South Carolina samples which have a one week expiration date**. All stocks and standards are stored in the freezer at a temperature of -15°C $\pm$ 5°C or less from the date they are received/prepared. The freezer temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. Makeup of common standards is detailed below. See standard ID in LIMS system for makeup of other standards.

10.6.1 The Bromofluorobenzene (BFB) tuning standard is prepared as follows: Using a 50µL syringe, 40µL of standard (BFB @ 2500ng/µL) is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same making a 50ng/µL standard. After capping and inverting 3 times, the solution is transferred to a labeled 2ml, teflon-lined, screw-capped vial and stored in the freezer at -15°C $\pm$ 5°C or less for up to 6 months **(1 week for South Carolina samples)**. A direct injection of 1µL (or equivalent purge) is used to tune the instrument.

10.6.2 The internal and surrogate standards are prepared as follows: Using the indicated syringe, the indicated amount of standard is injected into a 50 mL volumetric flask containing P&T methanol (Vendor,Lot) and diluted to volume with same making a 150ng/µL standard. After capping and inverting 3 times, the solution is transferred to the Archon standard vial and stored under helium for 1 month or less. Each 8260/624 sample is automatically injected with 1µL of this standard. (The internal standard/surrogate solution may be replaced if the –50%-200% criteria fails in the CCV when calculated against the previous CCV.)

Standard	Conc. (ng/µL)	Syringe (µL)	Amount (µL)
8260 ISTD Mix	2500	1000	3000
Surr. Mix	2500	1000	3000

10.6.3 Calibration standards are prepared from the vendor stock standards at appropriate concentrations as follows. Occasionally unusual compounds are added to the mix so it is best to check the LIMS for exact standard makeup. Note: for laboratory control spikes (LCS), alternate sources or lot numbers from the main calibration standard are used to make the LCS standard.

10.6.3.1 Primary Standard: Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 100µg/L (5mL purge) standard is made using 50µL of this standard to 50mLof reagent water.

Stock Standard(CCV)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE **(Cat#30265)**	20000	25	20	200
Vinyl Acetate **(#3766)**	5000	100	80	200
Ketones **(cat#30006)**	5000	100	80	200
Liquid mix **(C-349H-07)**	2000	100	100	100
Custom mix **(CCS-1037)**	5000	50	40	100
Gases **(cat#30042)**	2000	100	100	100
Acrolein/Acrylonitrile **(CC2098.10)**	20,000	50	50	500

Additional compounds may be added such as Appendix IX. Refer to standard ID in LIMS system.

10.6.4 ICV/LCS/Matrix Spike Mix: A second source standard is used to check the validity of the gas and primary calibration standards used in analyzing the calibration curve. Using the indicated syringe, the indicated amount of standard is injected into a 2mL volumetric flask containing approximately 1.0mL P&T methanol (Vendor,Lot) and diluted to volume with same to make a 100-500ng/µL standard. After capping and inverting 3 times, the solution is transferred into 2ml amber vial w/mini-inert valve and stored in the freezer at -15°C ± 5°C for 1 week. A 50µg/L ICV/LCS/Matrix Spike is made using 25µL of this standard to 50mL of reagent water/Sample Matrix.

Stock Standard(ICV/LCS)	Conc (ng/µL)	Syringe(µL)	Amount(µL)	Final Conc (ng/µL)
2-CEVE	20,000	25	20	200
Vinyl Acetate	5000	100	80	200
Ketones	5000	100	80	200
Liquid mix	2000	100	100	100
Custom Mix	5000	50	40	100
Gases	2000	100	100	100
Acrolein/Acrylonitrile	50,000	50	50	500

11. 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 All water samples are stored in the "True" refrigerator in the VOA lab at a temperature of 4°C. All unpreserved soil samples in TerraCore or encores are stored in the freezer in the VOA lab. All soil samples in bulk jars or chemically preserved TerraCore are stored in the soil walk-in refrigerator at a temperature of 4°C. Non-preserved water volatile samples have a holding time of 7 days from date of sampling. Preserved water samples and soil volatile samples have a holding time of 14 days from date of sampling (unless otherwise specified for the project). The temperature is monitored daily with a calibrated thermometer (annual calibration for liquid in glass and quarterly calibration for digital) and recorded with calibration correction in the VOA refrigerator/freezer logbook. The weekend temperature is monitored with a Min/Max thermometer and recorded upon arrival next business day.

12. 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.

12.1 Internal Standards - All samples and QC are spiked with internals. See **Table 2** for acceptance criteria and corrective action.

12.2 Surrogates - All samples and QC are spiked with surrogates. See **Table 2** of this SOP for acceptance criteria and corrective action.

12.3 LCS Sample - An LCS is analyzed every 12 hour tune. To prepare the LCS, a blank is spiked with standards prepared from an alternate vendor or lot number from the calibration standards. Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample). See **Table 2** of this SOP for acceptance criteria and corrective action. **When analyzing samples for South Carolina the limits are 70-130% except for poor purgers which are 60-140%.**

12.4 Method Blanks - A method blank is analyzed every 12 hour tune. See **Table 2** of this SOP for acceptance criteria and corrective action..

12.5 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample - 1 in 20 samples are spiked for an MS/MSD with the LCS standard. See **Table 2** of this SOP for acceptance

criteria and corrective action. MS data evaluation must include the consideration of the following factors.

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. A water sample which was taken from the same VOA vial for the original sample and the MS/MSD should have very good RPDs unless there has been a method problem. 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 problem in both the MS and MSD, review the LCS and if acceptable no further action may be necessary since it is attributable to matrix effect.

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.

13. 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 Chromatographic conditions – Refer to corresponding instrument maintenance log for current gas chromatograph, mass spectrometer, and concentrator conditions.

13.3 System Bakeout - Prior to analysis an instrument blank is analyzed.

NOTE: Further cleaning may be accomplished by backflushing the lines with methanol and then analyzing blanks overnight.

13.4 Tuning - Prior to any calibration or analysis, BFB tuning criteria must be met for a 1.0µL injection of the tuning standard. See **Table 5** of this SOP for acceptance criteria. Tune must be met every 12 hours sample analysis is to be performed **(every 24 hours for *Federal Register* Method 624 except for South Carolina which only allows 12 hours).** The mass spectrum of BFB is acquired as follows: by using the BFB method in Target (which uses three scans with backround subtraction) to process the BFB data file. If the BFB tune does not pass criteria corrective action should be taken

13.5 **Calibration:** Calibration standards are made up in water using the appropriate amount of the methanol standard. See the LIMS for preparation of standards. **Calibration for soils for South Carolina requires that 5mL of sodium bisulfate**

solution is added to each calibration standard made if the samples will be preserved with sodium bisulfate. All manual calibration integrations must be approved by the section manager or designated peer reviewer.

13.5.1 Initial Calibration - An initial calibration curve at no less than five (six if using a quadratic curve fit) concentration levels must be analyzed and shown to meet the initial calibration criteria before any sample analysis may be performed. **For Arizona samples the surrogates must also be calibrated at a minimum of five concentrations.** See **Table 2** of this SOP for acceptance 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. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual calibration integrations must be approved by the section manager or designated peer reviewer Any response factors less than 0.050 must be supported by the mass spectrum of the lowest standard. **No quadratic curves for South Carolina.**

CCCs:	1,1-Dichloroethene	Toluene
	Chloroform	Ethylbenzene
	1,2-Dichloropropane	Vinyl chloride

SPCCs:	Chloromethane	0.10
	1,1-Dichloroethane	0.10
	Bromoform	0.10
	Chlorobenzene	0.30
	1,1,2,2-Tetrachloroethane	0.30

13.5.2 Initial Calibration Verification (ICV) - A second source standard is prepared at or near the CCV concentration and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual ICV integrations must be approved by the section manager or designated peer reviewer.

13.5.3 Continuing Calibration Verification (CCV) - A CCV is analyzed every 12 hour tune and calculated against the initial calibration curve, then shown to meet the calibration check criteria before any sample analysis may be performed. See **Table 2** of this SOP for acceptance criteria and corrective action. Any manual integrations are documented by inclusion of the integrated signals (**before and after manual integration**) initialed, dated, and reason with the quantitation report and chromatograms. All manual CCV integrations must be approved by the section manager or designated peer reviewer. **.**

NOTE: Acceptance criteria for method 624 consists of meeting recovery limits found in table 5 of the method for a QC check sample. This QC check

sample is made from a separate source or lot number than the calibration standard at a concentration of 20 µg/L.

14. **Procedure**

14.1 LCS - An LCS is analyzed every 12 hour tune.Using standards prepared from an alternate vendor or lot number, blank water is spiked at the 50 µg/L (5mL/soil) or 10 µg/L (25mL) level. See **Table 2** of this SOP for acceptance criteria and corrective action. **Note: the concentration of the LCS will be 20 µg/L when analyzing 624 samples (QC Check Sample).**

14.2 Method Blank - Prior to sample analysis, the system must be shown to be free of contamination through analysis of a method blank. See **Table 2** of this SOP for acceptance criteria and corrective action.

14.3 Sample Analysis - Prior to analysis, the samples are prepared for chromatography using the appropriate sample preparation method (5mL water, 25mL water, low soil, high soil, etc.) See SOP 225 for preparation of a 5035 soil sample. For a 5mL/25mL water sample, use the following procedure:

14.3.1 Load the vial into the Archon autosampler in the expected position.

14.3.2 Program the Archon for the loaded vial range and necessary dilutions, making sure the programmed method is set for the same volume as the purge vessel on the front of the LSC 2000 or 3000/3100 and that the Chemstation sequence matches the Archon sequence. Note: TCLP samples are analyzed at a 10x dilution. One TCLP sample is spiked per batch at receipt of leachates.

14.3.3 After analysis of the sample has been completed, check the pH of the sample using pH paper and verify it to be less than a pH of 2 (recorded on the sequence log). If it is not, record the pH on the sequence log and generate a non-conformance report. The sample report will have to be qualified for preservation if the analysis is being performed more than 7 days after sampling. [Note: TCLP samples do not require a pH check.]

14.4 **Instrument sequence**

An example of a typical instrument sequence log follows:

1-BFB Tune (12:00 am)
2-CCV
3-LCS
4-Method Blank
5-Sample
6-Sample
7-Sample
8-Sample
9-Sample
10-Sample
11-Sample
12-Sample
13-Sample
14-Sample

15-Sample
16-Sample
17-Sample MS
18-Sample MSD
19-BFB (12:00pm - 12 hours since last BFB/CCV)
20-CCV
21-LCS
22-Method Blank
23-Sample
24-Sample

14.5 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 the TargetDB on Windows NT data system. Quantitative measurements are performed using the calculations found in section 15.2 of this SOP. The following must be checked to determine if the sample will need any reanalysis or dilution. See **Table 2** of this SOP for acceptance criteria and corrective action. Formal data evaluation is detailed in SOP QS05. **See SOP QS07 for guidance on manual integrations.**

14.5.1 Internal Standards - Areas counts and retention times.

14.5.2 Surrogates – Recoveries and retention times.
Federal Register **Method 624 contains no criteria for surrogate recovery.**

Surrogate	WATER	SOIL
Dibromofluoromethane	85-120	80-125
1,2-Dichloroethane-d4	85-135	75-140
Toluene-d8	85-115	80-120
Bromofluorobenzene	80-120	80-125

14.5.3 Analyte concentration.

14.5.4 Qualitative identification based on spectrum and retention time.

15. 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 Calculations:

15.2.1 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.

15.2.2 Calibration verification involves the calculation of the percent drift (linear) 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.

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

where CCV RF is the response factor from the analysis of the verification standard and Average RF is the average response factor from the initial calibration. The % difference or % drift calculated for the calibration verification standard must be within ±20% for each CCC analyte, or for all target analytes if the CCCs are not target analytes, before any sample analyses may take place.

15.2.3 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 ug/L.]

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

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 purged in ug/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 = For purge-and-trap analysis, V_i is not applicable and is set at 1.

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

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

15.2.4 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 ug/kg.]

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

where: A_s, A_{is}, C_{is}, D, and $\overline{RF}$ are the same as for aqueous samples.
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.

16. **Method Performance**

16.1 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 is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria.

17. **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. **Data Assessment and Acceptance Criteria for Quality Control Measures**

18.1 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**

19.1 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. **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. **References**

21.1 40 CFR, Part 136; Appendix A
21.2 Test Methods for Evaluating Solid Waste, SW-846, Third Edition and updates
21.3 National Environmental Laboratory Accreditation Conference; CH. 5, 2001
21.4 USACE, EM 200-1-3; Appendix 1; Shell, 2/2001
21.5 DOD Quality Systems Manual for Environmental Laboratories version 3, 3/2005

22. 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, BFB Tuning Criteria
22.6 Table 6, Analyst Checklist
22.7 Table 7, INTERNAL STANDARD ASSOCIATION

Table 1 – DL/LOD/LOQ

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,1,1,2-Tetrachloroethane	1.25	2.50	5.00	ug/Kg
1,1,1-Trichloroethane (1,1,1-TCA)	1.25	2.50	5.00	ug/Kg
1,1,2,2-Tetrachloroethane	1.25	2.50	5.00	ug/Kg
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	2.50	5.00	10.0	ug/Kg
1,1,2-Trichloroethane	1.25	2.50	5.00	ug/Kg
1,1-Dichloroethane (1,1-DCA)	1.25	2.50	5.00	ug/Kg
1,1-Dichloroethene (1,1-DCE)	1.25	2.50	5.00	ug/Kg
1,1-Dichloropropene	1.25	2.50	5.00	ug/Kg
1,2,3-Trichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2,3-Trichloropropane	1.25	2.50	5.00	ug/Kg
1,2,4-Trichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2,4-Trimethylbenzene	1.25	2.50	5.00	ug/Kg
1,2-Dibromo-3-chloropropane (DBCP)	2.50	5.00	10.0	ug/Kg
1,2-Dibromoethane (EDB)	1.25	2.50	5.00	ug/Kg
1,2-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
1,2-Dichloroethane (EDC)	1.25	2.50	5.00	ug/Kg
1,2-Dichloropropane	1.25	2.50	5.00	ug/Kg
1,3,5-Trimethylbenzene	1.25	2.50	5.00	ug/Kg
1,3-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
1,3-Dichloropropane	1.25	2.50	5.00	ug/Kg
1,4-Dichlorobenzene	1.25	2.50	5.00	ug/Kg
2,2-Dichloropropane	1.25	2.50	5.00	ug/Kg
2-Butanone (Methyl ethyl ketone; MEK)	2.50	5.00	10.0	ug/Kg
2-Chlorotoluene	1.25	2.50	5.00	ug/Kg
2-Hexanone (Methyl butyl ketone; MBK)	1.25	2.50	5.00	ug/Kg
4-Chlorotoluene	1.25	2.50	5.00	ug/Kg
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	1.25	2.50	5.00	ug/Kg
Acetone	5.00	10.0	20.0	ug/Kg
Acrolein	5.00	10.0	20.0	ug/Kg
Acrylonitrile	5.00	10.0	20.0	ug/Kg
Benzene	1.25	2.50	5.00	ug/Kg
Bromobenzene	1.25	2.50	5.00	ug/Kg
Bromochloromethane	1.25	2.50	5.00	ug/Kg
Bromodichloromethane	1.25	2.50	5.00	ug/Kg
Bromoform	1.25	2.50	5.00	ug/Kg
Bromomethane	2.50	5.00	10.0	ug/Kg
Carbon Disulfide	1.25	2.50	5.00	ug/Kg
Carbon Tetrachloride	1.25	2.50	5.00	ug/Kg
Chlorobenzene	1.25	2.50	5.00	ug/Kg
Chloroethane	2.50	5.00	10.0	ug/Kg
Chloroform	1.25	2.50	5.00	ug/Kg
Chloromethane	2.50	5.00	10.0	ug/Kg
cis-1,2-Dichloroethene (cis-1,2-DCE)	1.25	2.50	5.00	ug/Kg
cis-1,3-Dichloropropene	1.25	2.50	5.00	ug/Kg
Cyclohexane	1.25	2.50	5.00	ug/Kg
Dibromochloromethane	1.25	2.50	5.00	ug/Kg

Analyte	MDL/DL	LOD	MRL/LOQ	Units
Dibromomethane	1.25	2.50	5.00	ug/Kg
Dichlorodifluoromethane (CFC-12)	2.50	5.00	10.0	ug/Kg
Ethyl methacrylate	1.25	2.50	5.00	ug/Kg
Ethylbenzene	1.25	2.50	5.00	ug/Kg
Hexachlorobutadiene	1.25	2.50	5.00	ug/Kg
Iodomethane	5.00	10.0	20.0	ug/Kg
Isopropylbenzene (Cumene)	1.25	2.50	5.00	ug/Kg
Methyl Acetate	2.50	5.00	10.0	ug/Kg
Methyl methacrylate	1.25	2.50	5.00	ug/Kg
Methyl Tertiary Butyl Ether (MTBE)	1.25	2.50	5.00	ug/Kg
Methylcyclohexane	1.25	2.50	5.00	ug/Kg
Methylene Chloride, or Dichloromethane	2.50	5.00	10.0	ug/Kg
Naphthalene	1.25	2.50	5.00	ug/Kg
n-Butylbenzene	1.25	2.50	5.00	ug/Kg
n-Propylbenzene	1.25	2.50	5.00	ug/Kg
p-Isopropyltoluene	1.25	2.50	5.00	ug/Kg
sec-Butylbenzene	1.25	2.50	5.00	ug/Kg
Styrene	1.25	2.50	5.00	ug/Kg
tert-Butylbenzene	1.25	2.50	5.00	ug/Kg
Tetrachloroethene (PCE; PERC)	1.25	2.50	5.00	ug/Kg
Toluene	1.25	2.50	5.00	ug/Kg
trans-1,2-Dichloroethene (trans-1,2-DCE)	1.25	2.50	5.00	ug/Kg
trans-1,3-Dichloropropene	1.25	2.50	5.00	ug/Kg
Trichloroethene (TCE)	1.25	2.50	5.00	ug/Kg
Trichlorofluoromethane (CFC-11)	2.50	5.00	10.0	ug/Kg
Vinyl acetate	2.50	5.00	10.0	ug/Kg
Vinyl Chloride (VC)	2.50	5.00	10.0	ug/Kg
m,p-Xylene	2.50	5.00	10.0	ug/Kg
o-Xylene	1.25	2.50	5.00	ug/Kg
1,1,1,2-Tetrachloroethane	0.25	0.50	1.00	ug/L
1,1,1-Trichloroethane (1,1,1-TCA)	0.25	0.50	1.00	ug/L
1,1,2,2-Tetrachloroethane	0.25	0.50	1.00	ug/L
1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)	0.50	1.00	2.00	ug/L
1,1,2-Trichloroethane	0.25	0.50	1.00	ug/L
1,1-Dichloroethane (1,1-DCA)	0.25	0.50	1.00	ug/L
1,1-Dichloroethene (1,1-DCE)	0.25	0.50	1.00	ug/L
1,1-Dichloropropene	0.25	0.50	1.00	ug/L
1,2,3-Trichlorobenzene	0.25	0.50	1.00	ug/L
1,2,3-Trichloropropane	0.50	1.00	2.00	ug/L
1,2,4-Trichlorobenzene	0.25	0.50	1.00	ug/L
1,2,4-Trimethylbenzene	0.25	0.50	1.00	ug/L
1,2-Dibromo-3-chloropropane (DBCP)	0.50	1.00	2.00	ug/L
1,2-Dibromoethane (EDB)	0.25	0.50	1.00	ug/L
1,2-Dichlorobenzene	0.25	0.50	1.00	ug/L
1,2-Dichloroethane (EDC)	0.25	0.50	1.00	ug/L
1,2-Dichloropropane	0.25	0.50	1.00	ug/L
1,3,5-Trimethylbenzene	0.25	0.50	1.00	ug/L
1,3-Dichlorobenzene	0.25	0.50	1.00	ug/L
1,3-Dichloropropane	0.25	0.50	1.00	ug/L

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,4-Dichlorobenzene	0.25	0.50	1.00	ug/L
1-Chlorohexane	0.50	1.00	2.00	ug/L
2,2-Dichloropropane	0.25	0.50	1.00	ug/L
2-Butanone (Methyl ethyl ketone; MEK)	2.50	5.00	10.0	ug/L
2-Chloroethyl vinyl ether	1.25	2.50	5.00	ug/L
2-Chlorotoluene	0.25	0.50	1.00	ug/L
2-Hexanone (Methyl butyl ketone; MBK)	1.25	2.50	5.00	ug/L
4-Chlorotoluene	0.25	0.50	1.00	ug/L
4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)	1.25	2.50	5.00	ug/L
Acetone	2.50	5.00	10.0	ug/L
Acrolein	1.25	2.50	5.00	ug/L
Acrylonitrile	2.50	5.00	10.0	ug/L
Benzene	0.25	0.50	1.00	ug/L
Bromobenzene	0.25	0.50	1.00	ug/L
Bromochloromethane	0.25	0.50	1.00	ug/L
Bromodichloromethane	0.25	0.50	1.00	ug/L
Bromoform	0.25	0.50	1.00	ug/L
Bromomethane	0.50	1.00	2.00	ug/L
Carbon Disulfide	0.25	0.50	1.00	ug/L
Carbon Tetrachloride	0.25	0.50	1.00	ug/L
Chlorobenzene	0.25	0.50	1.00	ug/L
Chloroethane	0.50	1.00	2.00	ug/L
Chloroform	0.25	0.50	1.00	ug/L
Chloromethane	0.25	0.50	1.00	ug/L
cis-1,2-Dichloroethene (cis-1,2-DCE)	0.25	0.50	1.00	ug/L
cis-1,3-Dichloropropene	0.25	0.50	1.00	ug/L
Cyclohexane	0.25	0.50	1.00	ug/L
Dibromochloromethane	0.25	0.50	1.00	ug/L
Dibromomethane	0.25	0.50	1.00	ug/L
Dichlorodifluoromethane (CFC-12)	0.50	1.00	2.00	ug/L
Di-isopropyl ether	0.25	0.50	1.00	ug/L
ETBE	0.25	0.50	1.00	ug/L
Ethyl methacrylate	0.25	0.50	1.00	ug/L
Ethylbenzene	0.25	0.50	1.00	ug/L
Hexachlorobutadiene	0.25	0.50	1.00	ug/L
Iodomethane	0.25	0.50	1.00	ug/L
Isopropylbenzene (Cumene)	0.25	0.50	1.00	ug/L
Methyl Acetate	0.50	1.00	2.00	ug/L
Methyl methacrylate	0.25	0.50	1.00	ug/L
Methyl Tertiary Butyl Ether (MTBE)	0.25	0.50	1.00	ug/L
Methylcyclohexane	0.25	0.50	1.00	ug/L
Methylene Chloride, or Dichloromethane	0.50	1.00	2.00	ug/L
Naphthalene	0.25	0.50	1.00	ug/L
n-Butylbenzene	0.25	0.50	1.00	ug/L
n-Propylbenzene	0.25	0.50	1.00	ug/L
p-Isopropyltoluene	0.25	0.50	1.00	ug/L
sec-Butylbenzene	0.25	0.50	1.00	ug/L
Styrene	0.25	0.50	1.00	ug/L
t-Butyl alcohol	1.25	2.50	5.00	ug/L

Analyte	MDL/DL	LOD	MRL/LOQ	Units
tert-Amyl methyl ether	2.50	5.00	10.0	ug/L
tert-Butylbenzene	0.25	0.50	1.00	ug/L
Tetrachloroethene (PCE; PERC)	0.25	0.50	1.00	ug/L
Tetrahydrofuran	1.25	2.50	5.00	ug/L
Toluene	0.25	0.50	1.00	ug/L
trans-1,2-Dichloroethene (trans-1,2-DCE)	0.25	0.50	1.00	ug/L
trans-1,3-Dichloropropene	0.25	0.50	1.00	ug/L
Trichloroethene (TCE)	0.25	0.50	1.00	ug/L
Trichlorofluoromethane (CFC-11)	0.50	1.00	2.00	ug/L
Vinyl acetate	1.25	2.50	5.00	ug/L
Vinyl Chloride (VC)	0.50	1.00	2.00	ug/L
m,p-Xylene	0.50	1.00	2.00	ug/L
o-Xylene	0.25	0.50	1.00	ug/L

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Method 8260B)					
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. 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 5 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.
Minimum five-point initial calibration (ICAL) for all analytes	ICAL prior to sample analysis.	1. Average response factor (RF) for SPCCs: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. RSD for RFs for CCCs: VOCs ≤ 30% and one option below: **Option 1:** RSD for each analyte ≤ 15%; **Option 2:** linear least squares regression r ≥ 0.995; **Option 3**: non-linear regression–coefficient of determination (COD) r2 ≥ 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.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Second source calibration verification (ICV)	Once after each ICAL.	All project analytes within ± 20% of true value. [± 25% for non-DoD 8260B;]	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 may 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.	
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: VOCs ≥ 0.30 for chlorobenzene and 1,1,2,2-tetrachlorolethane; ≥ 0.1 for chloromethane, bromoform, and 1,1-dichloroethane. 2. %Difference/Drift for all target compounds and surrogates: VOCs ≤ 20%D (Note: D = difference when using RFs or drift when using least squares regression or non-linear calibration). [± 20% for CCCs only non-DoD 8260B]	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 must 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 8260B, if CCCs exceed, evaluate all analytes for 20%D and qualify as above]	Problem must be corrected. Results may 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.

Table 2. Organic Analysis by Gas Chromatography/Mass Spectrometry (Methods 8260B) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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 qulaifier 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.
Method blank	One per preparatory batch.	No analytes detected > ½ 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. 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. 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, 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 (see Box D-7).	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 (see Box D-7).	MSD: For matrix evaluation, use LCS acceptance criteria above. MSD or sample duplicate: RPD ≤ 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 8260B) (continued)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Surrogate spike	All field and QC samples.	<u>**Surrogate**</u> / **WATER** / **SOIL** Dibromofluoromethane: 85-120 / 80-125 1,2-Dichloroethane-d4: 85-135 / 75-140 Toluene-d8: 85-115 / 80-120 Bromofluorobenzene: 80-120 / 80-125 QC acceptance criteria specified by DoD (above) or Client. Otherwise, in-house control limits may be used. No limits specified for Method 624.	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.
Results reported between DL and LOQ	NA.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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 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 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

Table 5, Tuning Criteria

m/z	Required Intensity (relative abundance)
50	15 to 40% of m/z 95
75	30 to 60% of m/z 95
95	Base peak, 100% relative abundance
96	5 to 9% of m/z 95
173	Less than 2% of m/z 174
174	Greater than 50% of m/z 95
175	5 to 9% of m/z 174
176	Greater than 95% but less than 101% of m/z 174
177	5 to 9% of m/z 176

Table 6, 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 7, Internal Standard Association

Analyte	Internal Standard	Analyte	Internal Standard
1,1,1-Trichloroethane	Fluorobenzene	1,1,1,2-Tetrachloroethane	d5-Chlorobenzene
1,1,2-Trichloro-1,2,2-trifluoroethane	Fluorobenzene	1,1,2-Trichloroethane	d5-Chlorobenzene
1,1-Dichloroethane	Fluorobenzene	1,2,3-Trichloropropane	d5-Chlorobenzene
1,1-Dichloroethene	Fluorobenzene	1,2-Dibromoethane (EDB)	d5-Chlorobenzene
1,1-Dichloropropene	Fluorobenzene	1,3-Dichloropropane	d5-Chlorobenzene
1,2-Dichloroethane	Fluorobenzene	1-Chlorohexane	d5-Chlorobenzene
1,2-Dichloroethane-d4	Fluorobenzene	2-Hexanone	d5-Chlorobenzene
1,2-Dichloroethene (total)	Fluorobenzene	Bromofluorobenzene	d5-Chlorobenzene
1,2-Dichloropropane	Fluorobenzene	Bromoform	d5-Chlorobenzene
1,4-Dioxane	Fluorobenzene	Chlorobenzene	d5-Chlorobenzene
2,2-Dichloropropane	Fluorobenzene	Chlorobenzene-d5	d5-Chlorobenzene
2-Butanone	Fluorobenzene	Dibromochloromethane	d5-Chlorobenzene
2-Chloroethyl vinyl ether	Fluorobenzene	Ethyl Methacrylate	d5-Chlorobenzene
4-Methyl-2-pentanone	Fluorobenzene	Ethylbenzene	d5-Chlorobenzene
Acetaldehyde	Fluorobenzene	m,p-Xylene	d5-Chlorobenzene
Acetone	Fluorobenzene	Methacrylonitrile	d5-Chlorobenzene
Acetonitrile	Fluorobenzene	o-Xylene	d5-Chlorobenzene
Acrolein	Fluorobenzene	Styrene	d5-Chlorobenzene
Acrylonitrile	Fluorobenzene	Tetrachloroethene	d5-Chlorobenzene
Allyl chloride	Fluorobenzene	Toluene	d5-Chlorobenzene
Benzene	Fluorobenzene	Toluene-d8	d5-Chlorobenzene
Bromochloromethane	Fluorobenzene	trans-1,3-Dichloropropene	d5-Chlorobenzene
Bromodichloromethane	Fluorobenzene	Xylenes (total)	d5-Chlorobenzene
Bromomethane	Fluorobenzene	1,1,2,2-Tetrachloroethane	1,4-dichlorobenzene-d4
Carbon disulfide	Fluorobenzene	1,2,3-Trichlorobenzene	1,4-dichlorobenzene-d4
Carbon tetrachloride	Fluorobenzene	1,2,4-Trichlorobenzene	1,4-dichlorobenzene-d4
Chloroethane	Fluorobenzene	1,2,4-Trimethylbenzene	1,4-dichlorobenzene-d4
Chloroform	Fluorobenzene	1,2-Dibromo-3-chloropropane	1,4-dichlorobenzene-d4
Chloromethane	Fluorobenzene	1,2-Dichlorobenzene	1,4-dichlorobenzene-d4
Chloroprene	Fluorobenzene	1,3,5-Trimethylbenzene	1,4-dichlorobenzene-d4
cis-1,2-Dichloroethene	Fluorobenzene	1,3-Dichlorobenzene	1,4-dichlorobenzene-d4
cis-1,3-Dichloropropene	Fluorobenzene	1,4-Dichlorobenzene	1,4-dichlorobenzene-d4
Cyclohexane	Fluorobenzene	1,4-Dichlorobenzene-d4	1,4-dichlorobenzene-d4
Dibromofluoromethane	Fluorobenzene	2-Chlorotoluene	1,4-dichlorobenzene-d4
Dibromomethane	Fluorobenzene	4-Chlorotoluene	1,4-dichlorobenzene-d4
Dichlorodifluoromethane	Fluorobenzene	Bromobenzene	1,4-dichlorobenzene-d4
Diisopropyl Ether	Fluorobenzene	cis-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Ethyl tert-Butyl Ether	Fluorobenzene	Hexachlorobutadiene	1,4-dichlorobenzene-d4
Fluorobenzene	Fluorobenzene	Naphthalene	1,4-dichlorobenzene-d4
Hexane	Fluorobenzene	n-Butylbenzene	1,4-dichlorobenzene-d4
Iodomethane	Fluorobenzene	n-Propylbenzene	1,4-dichlorobenzene-d4
Isobutyl alcohol	Fluorobenzene	p-Isopropyltoluene	1,4-dichlorobenzene-d4
Isopropylbenzene	Fluorobenzene	sec-Butylbenzene	1,4-dichlorobenzene-d4
Methyl Acetate	Fluorobenzene	tert-Butylbenzene	1,4-dichlorobenzene-d4
Methyl Methacrylate	Fluorobenzene	trans-1,4-Dichloro-2-butene	1,4-dichlorobenzene-d4
Methyl t-Butyl Ether	Fluorobenzene		
Methylcyclohexane	Fluorobenzene		
Methylene chloride	Fluorobenzene		
Propionitrile	Fluorobenzene		
t-Butyl alcohol	Fluorobenzene		
Tert-Amyl Methyl Ether	Fluorobenzene		
Tetrahydrofuran	Fluorobenzene		
trans-1,2-Dichloroethene	Fluorobenzene		
Trichloroethene	Fluorobenzene		
Trichlorofluoromethane	Fluorobenzene		
Vinyl acetate	Fluorobenzene		
Vinyl chloride	Fluorobenzene		

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP221 REVISION #: 09 EFFECTIVE DATE: 071210

TOTAL ORGANIC CARBON (TOC)
BY SM5310C, SW846 METHOD 9060/9060A AND Lloyd KAHN
METHOD "*DETERMINATION OF TOC IN SEDIMENT*"

APPROVALS:

Lab Director: _____ Date: 7/12/10

Data Quality Manager: _____ Date: 7/12/10

Section Supervisor: _____ Date: 7/12/10

Changes Summary

Revision 09, 07/12/10

- The SOP is an update from Revision 08 dated 04/28/09
- The SOP has been reviewed and confirmed to be accurate.
- The soil TOC method calibration concentrations were updated.
- The LCS reference was changed to BS.

TOTAL ORGANIC CARBON (TOC)
BY SM5310C, SW846 METHOD 9060/9060A AND Lloyd KAHN
METHOD "*DETERMINATION OF TOC IN SEDIMENT*"

I. SCOPE AND APPLICATION

This SOP describes the measurement of TOC by SM5310C, SW-846 Method 9060/9060A and Lloyd Kahn Method for determination in soil /sediment matrix.

SM5310C is used to determine the concentration of organic carbon in source and drinking water, SW-846 Method 9060/9060A is used to determine concentrations of carbon in saline waters, domestic and industrial wastes and SW846 Method 9060 is modified for soil determination and the L1oyd Kahn Method is used for determination of TOC in soil/sediment and solid matrices.SW846 Method 9060/9060A and the Lloyd Kahn Method require quadruplicate analysis of samples, where as SM5310C requires a minimum of two analyses. These methods should be read over carefully by the analyst and any restrictions should be noted.

II. SUMMARY OF METHOD

The organic carbon is measured using an Shimadzu Total Organic Carbon Analyzer (aqueous samples) and an OI Analytical Solids TOC Analyzer model 1010 (soil/sediment samples). The Shimadzu instrument converts the organic carbon in a sample using wet chemical oxidation. The CO_2 formed is then measured by an infrared detector (replaces ultraviolet detector in SM 5310C). With the model 1010 Solids TOC analyzer, TOC is determined by acidifying a sample and heating it to 250°C to remove the TIC. The sample is then heated to 900°C to combust the remaining TOC. The resulting carbon dioxide from the TOC is detected by a non-disperse infrared (NDIR) detector that has been calibrated to directly display the mass of carbon dioxide detected. This mass is proportional to the mass of TOC in the sample.

The limit of detection for the water method is 0.50 mg carbon/L and the Limit of quantitation is 1.0 mg carbon/L. The limits of detection and quantitation with the soil method depends on the how many grams of sample is used for the analysis. For a 250 mg sample the limit of detection is 460 mg/kg and the limit of quantitation is 1600 mg/kg.

III. SAMPLING HANDLING AND PRESERVATION

3.1 Sampling and storage in glass bottles is preferable. Sampling and storage in plastic bottles such as conventional polyethylene and cubitainers is permissible if it is established that the containers do not contribute contaminating organics to the samples. NOTE 1: A brief study performed in the EPA Laboratory indicated that distilled water stored in new, one quart cubitainers did not show any increase in organic carbon after two weeks exposure.

3.2 Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, the lapse of time between collection of samples and start of analysis should be kept to a minimum. The holding time is 28 days for waters and soils with the exception of

the Lloyd Kahn method soils, which requires a 14 day holding time. Also, samples must be kept cool (4°C) and protected from sunlight and atmospheric oxygen.

3.3 When water samples cannot be analyzed immediately, the sample is preserved by acidification to (pH </= 2) with HCl or H_2SO_4. Both water and soil samples are stored at 4°C.

IV. INTERFERENCES

4.1 WATER METHOD

4.1.1 Removal or carbonate and bicarbonate carbon by acidification and purging with purified gas results in the loss of volatile organic substances. The volatiles also can be lost during sample blending, particularly if the temperature is allowed to rise. Another important loss can occur if large carbon-containing particles fail to enter the needle used for injection. Filtration although necessary to eliminate particulate organic matter when only DOC is to be determined, can result in loss or gain of DOC, depending on the physical properties of the carbon-containing compounds and the adsorption of carbonaceous material on the filter, or its desorption from it. Check filters for their contribution to DOC by analyzing a filtered blank. Note that any contact with organic material may contaminate a sample. Avoid contaminated glassware, plastic containers, and rubber tubing. Analyze treatment, system, and reagent blanks.

4.1.2 This procedure is applicable only to homogenous samples which can be injected into the apparatus reproducibly by means of a pipette. The openings of the pipette limit the maximum size of particles which may be included in the sample.

4.2 SOIL METHOD

4.2.1 All materials must be routinely demonstrated to be interference –free under the analysis conditions by running blanks. Use high purity or purified reagents and gases to help minimize interference problems.

4.2.2 The infrared detector is sensitized to CO_2 and accomplishes virtually complete rejection of response from other gases that absorb energy in the infrared region.

V. DEFINITIONS

5.1 ANALYTICAL BATCH-The set of samples extracted /distilled/ or digested at the same time to a maximum of 20 samples.

5.2 CALIBRATION BLANK (CB)- A volume of reagent water in the same matrix as the calibration standards, but without the analyte.

5.3 CALIBRATION STANDARD (CAL)- A solution prepared from the primary dilution standard solution or stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.

5.4 FIELD BLANK (FMB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, including exposure to a sample bottle holding time, preservatives, and all preanalysis treatments. The purpose is to determine if the field or sample transporting procedures and environments have contaminated the sample.

5.5 FIELD DUPLICATE (FD)- Two samples taken at the same time and place under identical circumstances which are treated identically throughout field and laboratory procedures. Analysis of

field duplicates indicates the precision associated with sample collection, preservation, and storage, as well as with laboratory procedures.

5.6 LABORATORY BLANK (LRB)- An aliquot of reagent water or equivalent neutral reference material treated as a sample in all aspects, except that it is not taken to the sampling site. The purpose is to determine if the analytes or interferences are present in the laboratory environment, the reagents, or the apparatus.

5.7 LABORATORY CONTROL SAMPLE (BS)- A solution prepared in the laboratory by dissolving a known amount of one or more pure compounds in a known amount of reagent water. Its purpose is to assure that the results produced by the laboratory remain within the acceptable limits for precision and accuracy. (This should not be confused with a calibrating standard, it must be prepared from a source other than the same source as the calibration standards).

5.8 LABORATORY DUPLICATE (LD)- Two aliquots of the same environmental sample treated identically throughout a laboratory analytical procedure. Analysis of laboratory duplicates indicates precision associated with laboratory procedures but not with sample collection, preservation, or storage procedures.

5.9 QUALITY CONTROL CHECK SAMPLE (QCS)- A sample containing analytes of interest at known concentrations (true value) of analytes. The QCS is obtained from a source external to the laboratory or is prepared from standards obtained from a different source than the calibration standards. The purpose is to check laboratory performance using test materials that have been prepared independently from the normal preparation process.

5.10 METHOD DETECTION LIMIT (MDL)- The lowest level at which an analyte can be detected with 99 percent confidence that the analyte concentration is greater than zero.

VI. REAGENTS/STANDARDS

Store all reagents and standards according to recommendations. All standards should be stored away from light and at 4°C (± 2°C).

6.1 The laboratory reagent blank water used for TOC analysis is obtained from the Modulab Analytical water purification system in the analytical laboratory. **Boiling the water is not necessary as the method states.**

6.2 Potassium hydrogen phthalate, primary stock solution, 1000 mg/L: Dissolve 0.2128g of potassium hydrogen phthalate (primary standard grade) in 100.0 mL water.

6.3. Potassium hydrogen phthalate, standard solutions : A 100 mg/L standard is prepared by transferring 10 mL of the stock solution to a 100 mL volumetric flask and diluting to the mark with water. This solution is prepared on a daily basis.

6.4. The carbonate-bicarbonate solutions are not needed for this instrument.

6.5 Calibration Standards

1. For the water method, calibration standard is Potassium Hydrogen Phthalate. Standards are made from dilutions of the stock 1000 mg/L standard as follows:

1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL
25.0 mg/L = 5.0 mL of 1000 mg/L -> 200 mL
50.0 mg/L = 10.0 mL of 1000 mg/L -> 200 mL
100 mg/L = 10.0 mL of 1000 mg/L -> 100 mL

A low level standard curve must be run for drinking water samples with the standards made as follows:

0.25 mg/L = 0.025 mL of 1000 mg/L -> 100 mL
0.50 mg/L = 0.050 mL of 1000 mg/L -> 100 mL
1.0 mg/L = 0.10 mL of 1000 mg/L -> 100 mL
1.5 mg/L = 0.15 mL of 1000 mg/L -> 100 mL
2.5 mg/L = 0.25 mL of 1000 mg/L -> 100 mL
5.0 mg/L = 0.50 mL of 1000 mg/L -> 100 mL
10.0 mg/L = 1.0 mL of 1000 mg/L -> 100 mL

2. The soil method the calibration standard is prepared by using an OI commercially prepared 30% carbon sucrose solution.

6.6 Laboratory Control Sample:

1. For the water method, the Laboratory Control Sample is normally made from a performance evaluation solution of which the true value is known. This solution is given a unique identifier.

2. For the soil method, the Laboratory Control Sample is made from a 30% sucrose solution which is made by weighing up 7.125 grams of EM Reagent Grade Sucrose and diluting to 10 mL with deionized water volumetrically.

6.7. Persulfate oxidation solution: This solution is made by dissolving 60g of sodium persulfate in DI water, adding 15 ml of phosphoric acid and diluting to 500 ml.

6.8 Phosphoric acid solution: Dilute 100 mL of concentrated 85% phosphoric acid in 500 mL of water. This is used for water.

6.9 Phosphoric acid solution 5%: Dilute 59 mL of concentrated 85% phosphoric acid in 1000 mL of water. This is used for soil.

VII. INSTRUMENTATION

7.1 The instrument used for the Water TOC analysis is a Shimadzu Total Carbon Analyzer. An OIC 1010 soil/sediment carbon analyzer is used for soil samples.

7.2 There is a Shimadzu autosampler which will hold 68 samples.

7.3 The corresponding data for each sample is obtained from the Shimadzu software for the water samples. The soil/sediment data are printed out at the organic GC printer.

VIII. AQUEOUS SAMPLE PROCEDURE

8.1 Wearing labcoat, gloves and safety glasses, the standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. Also, remove samples from sample storage signing them out appropriately on the internal chain of custody form. Fresh acid and oxidation solutions should be poured into the appropriate containers on the front of the instrument.

8.2 Follow the instructions for operation of the instrument in Chapter 4, section 4.3 of the Shimadzu Model TOC-VWS User Manual. **See Appendix I. for Basic TOC start-up notes for analysis.**

8.3 **Following is a list outlining the order in which the samples should be run.** Each sample VOA vial should be numbered and its identity entered into the TOC schedule. Note: All blanks should be acidified to pH 2 to match the matrix of the samples analyzed.

1. 100 ppm
2. 50 ppm
3. 25 ppm
4. 10 ppm
5. 5.0 ppm
6. 2.5 ppm
7. 1.0 ppm
8. Method blank
9. BS + 9 samples (including any sample QC
10. 25 ppm
11. 10 samples (including any sample QC)
12. 50 ppm

8.8 Insrument printouts are generated from the software. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

IX. SOIL/SEDIMENT SAMPLE PROCEDURE

A sample is introduced into the Solid Module via a conditioned sample cup. Once the sample has been introduced the entire analysis sequence is automatic. Please reference Chapter 4 of the OI 1010 Solid Module instrument manual for instrument states and configuration when initially setting the instrument methods up.

TC Mode Instrument Settings:

Analysis Temp:900°C
Analysis Time: 6.5 minutes
Nitrogen Gas Flow: 60-100 psi (external regulator regulator)
Oxygen Gas Flow: 40-60 psi (external regulator)

This is a step by step description of a routine soil TOC analysis.

9.1 The standards and check solutions should be taken out of the refrigerator and allowed to warm to room temperature. The nitrogen and oxygen (internal regulator should be set at 50-60 psi) turned on allowing a nitrogen flow of 350-400mL/minute and an oxygen flow of 180 mL/minute (± 3 mL/minute).

NOTE: DO NOT TURN THE ANALYZER ON BEFORE TURNING THE GAS ON!

9.2 Let the gas flow through the instrument for a few minutes. The instrument should now be turned on and let to stabilize for 30 minutes.

9.3 Condition the cups (with quartz wool in them) using Diagnostics under Instrument Menu commands, (don't condition too many cups at a time since setting in contact with the air can cause contamination).

9.4 Set up the subdirectory (using the current date to ID it) under WinTOC output.

9.5 If doing an initial calibration curve use an appropriate µL syringe to make the following measurements of the sucrose standard in order to achieve the indicated concentrations. Make sure that there are no air bubbles in the syringe. Turn the syringe with the needle pointed up and vibrate the barrel and disperse any air from the syringe. To enter the calibration information on the instrument go to Instrument Cal Menu, type in the calibration standard values and save the file as the cal.. date analyzed.

µL 30% Sucrose STD	Concentration (mg)
0	0
2.0 (1:6 solution)	0.10
3.0	0.90
25	7.5
50	15

Note: The 1:6 solution of the 30% Sucrose standard is prepared by mixing 100µL of the 30% Sucrose standard with 500µL of water.

9.6 Enter the sequence to be analyzed as listed below:

1. CCV(CCV1+ date analyzed for ID) or Initial calibration – single analyses
2. Method Blank(MB + date analyzed for ID) – single analyses
3. BS, 15 mg dextrose (BS + date analyzed for ID) – single analyses
4. Sample – 4 replicates
5. Sample – 4 replicates
6. Sample – 4 replicates
7. Sample – 4 replicates
8. Sample – 4 replicates
9. CCV(CCV1+ date analyzed for ID)2 – single analyses
10. Sample – 4 replicates
11. Sample – 4 replicates
12. Sample – 4 replicates
13. Sample – 4 replicates
14. Sample – 4 replicates
15. CCV (CCV2+ date analyzed for ID) – single analyses

16. Sample – 4 replicates
17. Sample – 4 replicates
18. Sample – 4 replicates
19. Sample – 4 replicates
20. Sample – 4 replicates
21. CCV(CCV3+ date analyzed for ID) – single analyses
22. Sample – 4 replicates
23. Sample – 4 replicates
24. Sample – 4 replicates
25. Sample – 4 replicates
26. Sample – 4 replicates
27. CCV(CCV4+ date analyzed for ID) – single analyses
28. SampleMS – 4 replicates
29. SampleDUP – 4 replicates
30. FCV(CCV4+ date analyzed for ID) – single analyses
31. FCB(FCB4+ date analyzed for ID) – single analyses

9.7 Samples should be stored away from light and at 4°C (± 2°C). Wearing labcoat, gloves and safety glasses remove samples from sample storage signing them out appropriately on the internal chain of custody form.

9.8 Transfer a homogeneous aliquot(~5 g) of the sample into a small pre-labeled aluminum weighing pan. Label each pan with the appropriate sample ID then add enough phosphoric acid (1-2 ml) to remove the Total inorganic carbon (TIC) when the sample is placed in an oven at 250°C. Place the samples in the 250°C oven for 10 minute and begin prepping the sample cups to weigh 0.2g-1.0g of each sample(in quadruplicate). Limit the time that the cups are exposed to the atmosphere as to reduce potential contamination. **Note: Since the samples are dried in this manner, before the sample aliquot it taken, a % solids determination and calculation is NOT necessary to report the sample concentrations in dry weight.** After samples are dried crush samples using a clean mortar and pestle.

9.9 Set the OI 1010 to the TC Mode and start running the sequence beginning with the initial calibration or calibration verification standard as illustrated above. Weigh each sample in quadruplicate making sure to limit the time that samples are exposed to the atmosphere.

9.10 The Excel file for calculations is located in "V:\WCM\TESTS\TOC soil\". The sample identity, its corresponding mgC reading, and the sample weight are entered into the appropriate columns. The Excel worksheet is self explanatory. Normal procedure is followed for preparing reports and the data is second checked before being given to the supervisor.

X. QC REQUIREMENTS

10.1 Analyze a laboratory control sample (BS) for each batch of samples **(maximum of 10 samples per day).** If the BS does not fall within the control limits of 80 to 120%, corrective action must be taken to find and correct the problem.

10.2 Run a method blank (PB) for each batch of samples (maximum of 20 samples per day). The PB should be less than 1/2 the reporting limit.

10.3 One matrix spike and matrix spike duplicate must be run per set of 20 samples. For water analysis, a spike and spike duplicate are made by mixing 20 mLs of sample with 0.30 mLs of stock 1000 mg/L standard using an ependorf pipette. The true value is 15 mg/L. The percent recoveries on a MS and a MSD should be within 75 and 125%. Relative percent difference (RPD) on duplicates should be less than 20%. If not, a corrective action (CAR) must be approved by your supervisor.

10.4 Analyze an initial calibration verification (ICV) immediately after the calibration curve. Analyze a calibration check verification (CCV) standard every tenth sample and at the end or after every fifth sample when analyzing samples in quadruplicate. Analyze a CCV after every 5th sample when analyzing soil/sediment samples. The percent recoveries should be in the range of 90 to 110%. The CCV %RSD warning limits are $\leq$15% for aqueous samples and $\leq$20% for soil/sediment samples. If the CCV % RSD exceeds 15%(aqueous) or $\leq$30% (soil/sediment) and the correlation coefficient is less than 0.990 correct the problem and re-analyze the CCV.

10.5 When analyzing water samples, all water blanks before samples and standards must be below the detection limit, otherwise the samples must be rerun.

10.6 Analyze an initial calibration blank (ICB) following the ICV. Analyze a continuing calibration blank (CCB) following each CCV. The ICB and CCB should be less than $\pm$ the MDL.

10.7 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

10.8 Calculate all percent recoveries and relative percent differences on duplicates and show calculations on data.

Calculate spikes as follows where everything is in concentration.

$$\% \text{ Recovery} = \frac{\text{Spike} - \text{Sample}}{\text{True Value}} \times 100$$

. Relative percent difference is calculated as follows, with everything in concentration:

$$\text{RPD} = \frac{\text{Higher Concentration} - \text{Lower Concentration}}{\text{Average of Concentrations}} \times 100$$

10.9 SM5310B requires that the analyst repeat injection until consecutive measurements are obtained that are reproducible to within $\pm$10%. A minimum of two injections is required for water samples with three replicates preferred. SW-846 Method 9060/9060A requires quadruplicate analysis of each sample. The Loyd Kahn soil method suggests 1 sample per 20 be run in quadruplicate. Some clients may request that all samples to be done in quadruplicate. Please check with your supervisor if you have any questions about the required numbered of sample replications.

10.10 **For aqueous samples check an acidified 20mg/L inorganic carbon standard quarterly, to assure that purge gas flow is adequate to remove inorganic carbon. The result should be below the reported quantitation limit.**

XI. CORRECTIVE ACTIONS

11.1 INSTRUMENT RELATED

1. ICV not within ± 20% (Soil) or ± 10% (SM 5310C0
   a. If the problem is with the solution.
      i. Re-prepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate through analysis of appropriate standards and recheck ICV.
2. CCV not within ± 30% (Soil) or ±15% (SM 5310C)
   a. If the problem is with the solution.
      i. Re-prepare, obtain new stock if necessary.
   b. If the problem is with the calibration.
      i. Recalibrate through analysis of appropriate standards and re-prepare /reanalyze the previous ten sample according the following guidelines.
         a. If the CCV was biased high, any of the previous ten samples which were below the minimum detection limit do not require reanalysis.
         b. If the CCV was biased low, the previous ten samples must be reanalyzed.

*** Incorrectly set gas flow is a common instrument related problem which requires corrective action. Verify that all gas flows are adjusted properly.**

11.2 SAMPLE MATRIX RELATED

1. Replicate analysis RPD not within ±20% aqueous or ±50% soil/sediment
   i. The associated sample data must be qualified on the final report.
2. Spike analysis recovery not within ±25% aqueous or ±50% soil/sediment
   i. If the analyte level in the sample is greater than 4X the spiking level, the %recovery can not be evaluated and no action is taken.
   ii. If the analyte level in the sample is not greater than 4X the spiking level, the associated sample data must be qualified on the final report. A corrective action report must accompany the data and be emailed or given to the supervisor.

XII. HEALTH AND SAFETY

A. 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.

B. 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.

C. MSDS are available for all reagents and standards, which have been purchased. These are located in the administrative section next to the break room.

D. Please see *Waste Disposal; SOP-405* for proper disposal of the waste generated from this area.

XIII. WASTE DISPOSAL and POLLUTION PREVENTION

Please see Waste Disposal SOP-405, for instruction of proper 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.

XIV. METHOD PERFORMANCE

14.1 Precision and Bias for Total Organic Carbon (TOC) by Persulfate-Ultraviolet Oxidation. (Water samples)

Characteristic Of Analysis Concentration determined, mg/L:	Spring Water	Spring Water +0.15 mg/L KHP*	Tap Water	Tap Water +10 mg/L KHP*	Municipal Wastewater Effluent
Replicate 1	0.402	0.559	2.47	11.70	5.88
Replicate 2	0.336	0.491	2.49	11.53	5.31
Replicate 3	0.340	0.505	2.47	11.70	5.21
Replicate 4	0.341	0.523	2.47	11.64	5.17
Replicate 5	0.355	0.542	2.46	11.55	5.10
Replicate 6	0.366	0.546	2.46	11.68	5.33
Replicate 7	0.361	0.548	2.42	11.55	5.35
Mean, mg/L	0.35	0.53	2.46	11.53	5.32
Std. Deviation: mg/L	0.02	0.03	0.02	0.21	0.23
%	6	6	1	2	4
Actual Value, mg/L	-	0.50	-	12.46	-
Recovery, %	-	106	-	93	-
Error, %	-	6	-	7	-

***KHP = potassium acid phthalate.**

14.2 There was no method performance data available for the soil procedure.

XV. REFERENCES

1. Annual Book of ASTM Standards, Part 31, "Water," Standard D 2574-79, p. 469 (1976).

2. Standard Methods for the Examination of Water and Wastewater, 19th ED., Method 5310C (1999).

3. EPA SW-846, Method 9060/9060A.

4. Lloyd Kahn Method, *"Determination of Total Organic Carbon in Sediment"*

APPENDIX I.

1. Power up the lamp for warm –up, check reagents inside instrument cavity to make sure all are filled before starting the run.
2. Fill Fresh DI water in 1 gallon jug; DI squirt bottle and 1 L plastic
3. Label and load VOA vials with standards and samples into round tray.
4. Place round tray onto autosampler, get a final sample count for end point and replace lid.

5. Make sure that round tray fits down flush onto the autosampler.
6. On computer screen, select "TOC-Control V" icon.
7. Then select "Sample Table Editor"
8. Enter user name: "analyst initials" select OK.
9. Under "File" select "calibration curve" "OK".
10. Under system select Shimadzu TOC-BWS Enter/next
11. Select Edit Calibration points manually Enter/next
12. Under "Analysis" select "NPOC" then make up your file name (use today's date) Enter/next.
13. Calibration Measurement Parameters are default: Just hit "next"
14. Select "ADD" and enter calibration points starting at (1) 100 mg/L (2) 50 mg/L (3) 25 mg/L (4) 10 mg/L (5) 5.0 mg/L (6) 2.5 mg/L (7) 1.0 mg/L (8) 0.0 mg/L. After 8 points it should show 0.00 mg/L first and 100 mg/L eigth if so "next"
15. Put a check mark in "Correlation Coefficient" check box "next"
16. "next"
17. "finish"
18. Go to file and select "new", "sample run" "ok" "ok" enter file name: user date "save"
19. Now go to insert and select "calibration curve" then scroll till you find your file name/date should have .cal after date "select" the "open"
20. You should now see the sparging /acid addition page which shows a picture of the round sample tray. Under vial manually enter "1"beside 0.00 mg/L.
21. manually enter "2" beside 1.0 mg/L and "3" beside 2.5 mg/L and so on and so forth all the way to "8" this shows what order they ar loaded on the tray. "Enter/OK"
22. Then a screen with your filename/date and all info should be in row 1 only with vial column showing. 1,2,3,4, etc.
23. Select the lightening bolt symbol then enter "use PC settings" this will start initializing wait till screen goes away then you will see the stop light symbol appear with green light showing, select that icon select "keep running" select "standby"
24. Sparging/acid addition page will re-appear just hit "OK"
25. Start ASI tray screen will appear hit "Start"
26. The instrument should start establishing the baseline and move auto tray into position – Lid must be on and samples loaded into corret position will take almost 3 hours to finish. Can view data as its coming off by selecting "view" "sample window". After calibration is done review.
27. Select "File" then "New" then "sample run" "ok"
28. General information screen: No change select "ok"
29. Save as screen: Select today's date for file name example 00month/00day/00year
30. Select "save"
31. Sample Table Screen: Select "insert" then select " auto generate" enter
32. **Page 1** sample group wizard sample source: select "calibration curve" then double click on box with 3 dots …
33. Open latest curve from calibration curves file
34. Highlight latest curve and select "open"
35. Should send you back to page 1 with calibration curve info submitted. Select "next"
36. **Page 2** Sample Parameter: Enter final sample count for "number of samples" select "next"
37. **Page 3** Calibration Curves: No changes Select "Next"
38. **Page 4** Calibration Checks: No changes Select "Next"
39. **Page 5** Controls: No changes select "finish", Select "ok" on "Sparging/ Acid page.
40. Type sequence as they are loaded on tray: ICV, ICB, BSW, Sample #, client,etc.
41. Once everything is typed in double check that it matches the way samples and QC are loaded..
42. Click or select the lightening bolt symbol then select "use settings on PC". Wait for initializing. When screen goes awy the traffic light symbol should appear next to the lightning bolt symbol. Click on the traffic light symbol.

43. Click or select "shut down Instructions". Then select "standby" Sparging/ Acid addition screen will appear so you can confirm your tray is loaded the wax things are highlighted in blue. Select "OK" if it looks the same.

44. Start ASI measurement: External acid addition should have a check mark click on "start" analysis should begin to start.

45. Click on view and chose "sample window" to watch curves come off and to see beginning values.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 225 REVISION #: 09 EFFECTIVE DATE: 20100907

GC/MS VOLATILE NON-AQUEOUS MATRIX EXTRACTION USING SW-846 METHOD 5035/5035A FOR 8260B ANALYSIS

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/8/10

Changes Summary

Revision 09, 09/07/10

- The SOP is an update from Revision 08 dated 09/24/08
- The SOP has been updated to include reference to 5035A and preservation by freezing for unpreserved Terracores and Encores.

GC/MS - VOLATILE
NON - AQUEOUS MATRIX EXTRACTION
USING SW-846 METHOD 5035/A

1.0 SCOPE AND APPLICATION

The purpose of this SOP is to detail soil sample preparation for EPA method SW-846 5035 and 5035A. Soil samples should be sampled in the field using the EnCore™ sampler or prepared VOA vials (sometimes referred to as Terracore samplers) then shipped to the lab within 24 hours for preservation, storage and analysis. This SOP should be used in conjunction SOP-202, which details the analytical technique.

2.0 SUMMARY

Samples are collected in EnCores or prepared VOA vials and submitted to the laboratory for preparation/analysis.. EnCore samplers have to be frozen or prepared within 48 hrs of collection. Prepared VOA vials (sometimes referred to as Terracores) are shipped already prepared in water, methanol or preservative solution. If prepared in water, freezing is required within 48 hours. If preservative is used, refrigeration is the only requirement.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

EnCores are prepped within 48 hrs of collection or frozen until preparation can be completed. Preparation can be in sodium bisulfate with refrigeration at 4°C or in reagent water with freezing. Prepared VOA vials are received already prepared in water, methanol or sodium bisulfate solution. If prepared in water, freezing is required within 48 hours of collection. If preservative is used, refrigeration at 4°C is the only requirement. Holding Time is 14 days from collection once preserved.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Sample vials can be a source of contamination. Vials should be checked for contamination before use. Samples can be contaminated during sample prep. Prep blanks should be prepared at the same time as the samples to check for contamination.

5.0 EQUIPMENT AND MATERIALS

- Sample Containers – 40mL VOA vials with low bleed septa. Available from ESS (Part No. PC0040-0300 pack of 72), alternate sources are possible but must be checked for contaminants before use. ESS also supplies pre-prepped vials with the preservative and stirbar (Part No. PC4039-5035 pack of 72).
- Varian Archon 51 position programmable autosampler, or equiv.
- Top-loading balance – capable of accurately weighing to 0.01g.

- 1-10 mL Adjustable Dispenser, Model 400 Series, Oxford pipettor. Available from Oxford (Part No. 8885-040009).
- Spatula, stainless steel – narrow enough to fit into a sample vial.
- Magnetic stirring bars – PTFE- or glass-coated, of the appropriate size to fit the sample vials. Available from A. Daigger (Part No. WX22782A, case of 50).
- EnCore™ sampler – (En Chem, Inc., 1795 Industrial Drive, Green Bay, WI 54302), or equivalent. Necessary for field sampling crew.
- Terracore Vials- Available from QEC.
- Balance weights – used to calibrate the balance.
- Labels.

6.0 REAGENTS

- Reagent Water - Reagent water is NANO PURE WATER from source in the instrument lab, which is then purged with helium before use.
- Methanol, CH_3OH – purge-and-trap quality, or equivalent. Store away from other solvents.
- Sodium bisulfate, $NaHSO_4$ – ACS reagent grade, or equivalent. Available from Aldrich (Part No. 30,782-3).
- Sodium bisulfate solution – Prepare by adding 200 grams of $NaHSO_4$ (ACS reagent grade, or equivalent) to 1000 milliliters of helium-purged reagent water. Record the vendor and lot number of the $NaHSO_4$ in the Standards and Reagents Logbook. Each standard/reagent that is prepared is recorded in the logbook and given a sequential number. The label is completed with the standard/reagent number, name, preparation date, expiration date, solvent and analyst initials. The solution should be discarded after six months or sooner if it shows signs of contamination.

7.0 SAMPLE COLLECTION

As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile compounds. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the EnCore™ sampler, a cut off disposable plastic syringe, or a stainless steel spatula. We prefer to use the EnCore™ sampler.

7.1 The EnCore™ sampler is both a sampler and a container for low-level and high level soils. It is designed to collect an average weight with the exact weight to be determined in the lab. It is disposable and is also designed to have zero headspace. The EnCore™ sampler will require the field personnel to get the sample to the laboratory within 24-36 hours of collection. The laboratory needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used.

7.2 All low-level soil samples must be collected in duplicate to allow the laboratory an additional sample for reanalysis. A third sample should be collected for preparation of a high-level sample. This sample would be prepared at the same time as the "low-level" sample. (Some projects may not require the "low-level" detection limits, in this case only the high level sample preparation would be required.). A fourth sample may be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic sodium bisulfate preservative solution in the low concentration sample vial. The additional soil samples must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected. **Additional bulk samples should be collected for screening and dry weight determination without the preservative.** Note: If the low-level sample cannot be preserved with sodium bisulfate, the remaining low-level sample aliquot(s) is(are) transferred to a pre-weighed vial containing 5 mL of reagent water. The sample in the unpreserved vial must either be analyzed immediately (within 48 hours of collection) or frozen within the 48 hour time frame and then analyzed within the 14 day holding time.

8.0 PROCEDURE

8.1 Log-in personnel will log the samples in, place them in the Soil walk-in cooler assigned for volatile sample storage and notify the Organic Lab Manager that samples are in-house for 5035 preparation.

8.2 The Organic Lab Manager or designee will determine the amount of time remaining on the 48 hour EnCore™ holding time and assign the task of preserving the samples.

8.3 Samples received from the field should be designated for low-level, high-level or % solids/screening (this fraction should be in a regular soil jar, if it is not, it will require transfer to a VOA vial). Each low-level and high-level sample must be preserved appropriately as follows:

8.3.1 Organize the VOA vials required and label them with the sample number, date and LOW or HIGH for either low-level or high-level preservation. The LOW level VOA vials should have gray caps and septa if using the ESS brand.

8.3.2 Get the samples from the Hobart assigned for volatile sample storage and log them out.

8.3.3 Enter the sample numbers in the soil sample preparation logbook and add a sample preparation/storage blank to the book for each level being prepared (HIGH/LOW). There must be a line in the logbook for each sample vial being prepared (i.e. if there are 2 low-level samples and 1 high-level sample, the sample number should be listed in the logbook 3 times- use a,b,c to designate each vial associated with the same sample).

8.3.4 Using an adjustable Oxford pipettor, add 5 mL P&T methanol to each of the vials marked HIGH. Then record the vendor & lot number of methanol and the exact volume of methanol added to each sample in the sample preparation logbook. If the vial is not to be used immediately, weigh the vial

to the nearest 0.01g and record the weight on the vial. The vial weight must be verified to be within ±0.01g of this value before using for sample preparation.

8.3.5 For each of the vials marked LOW, add 5 mL of sodium bisulfate or reagent water if frozen and record the reagent number in the sample preparation logbook. Add a magnetic stir bar to each vial. If pre-prepped vials from ESS (or equivalent) are used, this step is unnecessary but the lot number and the pre-prepped status must be recorded in the preparation log.

NOTE: Soil samples that contain carbonate minerals (either from natural sources or applied as an amendment) may effervesce upon contact with the acidic preservative solution in the low concentration sample vial. If the amount of gas generated is very small (i.e., several mL), any loss of volatiles as a result of such effervescence may be minimal if the vial is sealed quickly. However, if larger amounts of gas are generated, not only may the sample lose a significant amount of analyte, but the gas pressure may shatter the vial if the sample vial is sealed. Therefore, when samples are known or suspected to contain high levels of carbonates, a test sample should be collected, added to a vial, and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and place low concentration samples in vials that contain 5ml water and a stir bar. This sample must be frozen in a slanted position until analysis or analyzed within 48 hours of sampling. Notify the Organic Lab Manager if this occurs, note this in the sample preparation logbook and generate an NCR to document the problem.

8.3.6 Place the vial (LOW/HIGH) on the top-loading balance, tare the vial then extrude the sample into the vial and record the weight of the sample in the sample preparation logbook. Make sure the lip of the vial does not have any soil on it, which might cause a leak, cap the vial tightly and mark the weight on the sample label.

8.3.7 Place the preserved samples in a box, return them to the Hobart assigned for volatile sample storage and log them back in.

9.0 ANALYSIS

9.1 Samples are analyzed by USEPA SW-846 methods 5035/8260B (low-level) using the Archon 51 position autosampler in conjunction with the GC/MS or 5030B/8260B (high-level) using any purge and trap instrument in conjunction with the GC/MS. For method 5035, the prepared low-level vials are placed in the Archon autosampler. The autosampler is programmed to add the appropriate internals and surrogates to each sample. Use of the autosampler is covered in the owners manual. Calibration of the analytical instrument with subsequent analysis of the samples is covered under SOP-202.

9.2 Determination of % Dry Weight – Weigh 5-10 grams of the sample from the bulk jar used for dry weight analysis in a tared crucible or aluminum pan. Dry overnight at 105°C. Allow to cool in a dessicator before weighing. Calculate % dry weight as follows:

$$\% \text{ dry weight} = \frac{\text{g of dry sample}}{\text{g of sample}} \times 100$$

9.2 If an extra bulk jar was not received for percent moisture determination, an alternate procedure using the methanol vial can be used with advance notice:

- Weigh the sample in the VOA vial to the necessary degree of accuracy for % solids (recommend a tare weight on the vial to the same degree of accuracy).
- Preserve the vial as normal.
- After we know the methanol extract is not needed or has been analyzed successfully, allow the methanol to evaporate and dry as necessary for % solids determination.
- Weigh the sample in the VOA vial to the necessary degree of accuracy for % solids.

10. HEALTH,SAFETY,WASTE MANAGEMENTAND POLLUTION PREVENTION

10.1 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.

10.2 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.

10.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the Quality Assurance Officers office.

10.4 Please see Waste Disposal SOP QS14 for proper disposal of waste coming from this area within our laboratory. 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.

REFERENCES

1.Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 5035, December 1996.
1.Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Other Methods; Method 5035A, July 2002.

DEFINITIONS

Refer to SOP QS08 for common environmental laboratory definitions.

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 233 **REVISION #: 03** **EFFECTIVE DATE: 022310**

NITROGUANIDINE IN SOIL AND WATER BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

APPROVALS:

Lab Director: _____ Date: 3/17/10

Data Quality Manager: _____ Date: ___/___/___

Section Supervisor: _____ Date: 3/17/10

Changes Summary

Revision Date: 02/23/10

- The SOP is an update from Revision 02 dated 01/20/2010.
- 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.

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1.0 Identification of the Test Method

This SOP is compliant with USEPA SW-846 Method 8330 and 8330A. When analyzing samples for DOD QSM Version 4.1, DOD requirements will be followed.

2.0 Applicable Matrix or Matrices

This SOP is applicable to water, soil, and sediment matrices.

3.0 Detection Limit

3.1 For aqueous samples = 2.5ug/L

3.2 For non-aqueous samples = 25ug/Kg

4.0 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 Reporting Limit (also defined as the Limit of Quantitation) and the lowest calibration level for each analyte.

5.0 Summary of the Test Method

Samples are analyzed after appropriate sample preparation using HPLC with identification at 263nm on a Restek Ultra IBD column (150mm x 4.6mm). Water samples are prepared by filtering through a 0.45um filter. Soils are prepared by shaking an air-dried aliquot of sample for 16-18 hours and filtering through a 0.45um filter. Note – shaker extraction is consistent with the 8330B extraction process.

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

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. Interferences from melamine and guanidine nitrate are minimized since melamine is insoluble in water and the relative absorptivity of guanidine nitrate at 263 nm is small.

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 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.

8.3 MSDS sheets are available for all reagents and standards which have been purchased.

8.4 Please see Laboratory Waste QS14 for instruction of proper disposal of waste generated.

9.0 Equipment & Supplies

9.1 Hewlett Packard Series 1050 HPLC System or Agilent Series 1100 HPLC System with Chemstation and Restek Ultra IBD Column (150x4.6mm)

9.2 Apparatus and Materials

9.3.1 New Brunswick Scientific Model G25 Incubator Shaker
9.3.2 Balance $\pm$ 0.1g
9.3.3 Disposable cartridge filters – 0.45mm PTFE filter
9.3.4 Scintillation Vials – 20mL, glass
9.3.5 Vials – 12mL, glass, Teflon-lined cap; 2.0mL, glass
9.3.6 Disposable syringes – Plastipak, 5mL or 10mL or equivalent
9.3.7 Volumetric flask with ground glass stopper – 10mL, 100mL, and 1000mL
9.3.8 Mortar and pestle
9.3.9 Graduated cylinders – 10mL, 25mL, 50mL
9.3.10 Pasteur pipet - 9”

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 Reagents

10.2.1 Nitroguanidine (Aldrich #N17351-100G or equivalent)
10.2.2 Organic-free reagent water from the Nano Pure Water System located in Wet Chemistry

10.3 Calibration/Method Standards

10.3.1 Nitroguanidine is purchased from Aldrich or equivalent manufacturer. The date it is received is noted on the label and all reagent information is documented within the LIMS once it is opened. The standard that is prepared is recorded in LIMS and given a LIMS-generated standard ID. All standards are stored in the refrigerator in the dark at a temperature of 4˚C $\pm$ 2˚C or less from the date they are 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 refrigerator logbook.

10.3.2 Stock Solution 100mg/L – An adequate amount of neat material is dried in a desiccator to constant weight. After drying, 100mg are transferred to a 1L volumetric flask and brought to volume with reagent grade water. The solution can be placed in an ultrasonic bath momentarily to aid is dissolution. An intermediate standard at 1.0mg/L is prepared by dilution 100uL of the stock standard to reagent water in a 10mL volumetric flask. Expiration date is six months for stock and intermediate standards. Calibration standards are prepared

by adding 5, 10, 50, 100, 250, 400, and 500uL of the intermediate standard to 1mL of reagent water to make standards at 5, 10, 50, 100, 250, 400, and 500ug/L.

10.3.3 Matrix/LCS Spike Standard 100mg/L - A second standard is prepared in the same way as the stock standard. Expiration date is six months. Water spikes are prepared by spiking 50uL of the 100 mg/L spiking standard to 20mL of sample. Filter and discard the first 10mL and add sample to a 2.0mL vial. Soil spikes are prepared by spiking 50uL of the spiking standard to 2g of dried sample in a 40mL VOA or scintillation vial and adding 20mL of water.

11.0 Sample Collection, Preservation, Shipment, and Storage

Quality Systems SOP QS10 related to Sample Receipt, Handling, and Processing provides details for collection, preservation, shipment, and storage. Sample holding times are 14 days for soil sample prep from sampling date and 40 days after sample prep for analysis. Water holding times are 47 days from sampling date until analysis.

12.0 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. See Table 2 for instrument and batch QC acceptance criteria.

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 The instrument should be allowed to pump for 30 minutes while mobile phase is passed through the column.

13.3 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. The calibration curve should be linear. Linearity may be evaluated by calculating the percent relative standard deviation (*%RSD*) and the average response factor used if the *%RSD* is less than 20%. Use of a linear curve is acceptable if *r* (the correlation coefficient) is equal to or greater than 0.995 while use of a quadratic curve (with 6 or more points) is allowable if r^2 (the square correlation coefficient) is equal to or greater than 0.99. Note that calibration may not be forced through the origin for any DoD project.

13.4 Initial Calibration Verification: The standards used to make the calibration curve are verified to be accurate using the spiking standard, initial calibration verification (ICV). The percent recovery for the ICV determined from the initial calibration curve must agree within $\pm$ 15%. If not, the source of the problem must be identified before analysis continues.

13.5 Daily Calibration: Continuing calibration verification (CCV) standard must be analyzed, at a minimum, at the beginning of the day, after every 20 samples and at the end of the sequence. The response factor for the CCV must agree within $\pm$ 15% of the response

factor of the initial calibration. The same criteria are required for subsequent CCV responses compared to the response factor of the CCV beginning the day. If the CCV criteria are not met and a reanalysis does not meet, a new initial calibration curve may be needed.

13.6 RT Windows: Retention time (RT) windows are determined 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. Retention time in each CCV is compared to the established retention time window from the beginning CCV. The analyte should fall within its respective RT window. If this criterion is not met, system maintenance should be performed.

14.0 Procedure

14.1 All samples should be processed as soon as possible. Determine the samples necessary to prepare as follows:

14.1.1 Each day a backlog report will be generated from LIMS

14.1.2 Samples requiring RUSH turn around time may be logged in throughout the day that will require immediate attention. Sample receiving personnel will generally communicate this need.

14.1.3 Check LIMS throughout the day to determine if more applicable samples arrive. If an analysis is ongoing, extra QC may be avoided by preparing those samples on the same day.

14.1.4 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.

14.2 Before sample preparation, 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 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 sample preparation, all pertinent information (amounts, reagent IDs, standards is recorded in LIMS so as to allow reconstruction of the preparation in the future.

14.3 Aqueous Sample Preparation Method: Water samples are prepared by filtering 20mL of sample through a 0.45um filter. Discard the first 10mL. Fill a 2.0mL sample vial for analysis. Prepare a method blank, LCS, MS/MSD to go along with the batch.

14.4 Soil Sample Preparation Method: A representative subsample is air dried overnight and ground with a mortar and pestle prior to extraction. Weigh 1.8-2.2g of dried sample into a 40mLVOA vial or 20mL scintillation vial and add 20mL of reagent grade water. Place in shaker for 16-18 hours. Remove the samples from the shaker and centrifuge at 1500rpm for 5 minutes to aid filtration. A 6mL portion of extract is filtered through a 0.45um filter. Discard the first 3mL and then fill a 2.0mL sample vial for analysis. Prepare a method blank, LCS, MS/MSD to go along with the batch.

15.0 Data Analysis and Calculations

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

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

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

The mean CF is calculated as follows:

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

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

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

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

15.4 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.

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

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

$$\%\ Difference = \frac{\text{(CCV CF – Average CF) x 100}}{\text{Average CF}}$$

15.5 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:

Aqueous Samples**:**

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

where:

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

V_t = Total volume of sample, mL.

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, mL.

Using the units specified here for these terms will result in concentration units of ng/mL, which is µg/L.

Nonaqueous Samples**:**

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

where:

W_s = Weight of dry sample extracted, g.

A_S, V_t, D, $\overline{CF}$ and V_i have the same definition as for aqueous samples.

15.6 Chromatographic Condition

15.6.1 Hewlett Packard Series 1050 or Agilent Series 1100 HPLC System
- 15.6.1.1 Primary Column: Restek Ultra IBD, 150 x 4.6mm
- 15.6.1.2 Mobile Phase: 100% water
- 15.6.1.3 Flow Rate: 1.5mL/min
- 15.6.1.4 Injection Volume: 100uL
- 15.6.6.5 UV Detector: 263nm

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. The DOC

form, as listed within section 2.5 of the Quality is completed by each analyst and then provided to the supervisor for further processing and approval.

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, "Laboratory Non-Conformance/ Corrective Action Procedures, Decision Making Procedures, Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results & Statistical Calculations," 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

19.1 Quality Control SOP QS05, "Laboratory Non-Conformance/ Corrective Action Procedures, Decision Making Procedures, Guidelines for Evaluating Laboratory Analytical Sample and Quality Control Results & Statistical Calculations," 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.

19.2 LCS Sample - The LCS is analyzed at the frequency required by the regulatory agency or client (every batch or 20 samples, whichever comes first, for DoD projects). The recoveries are charted to generate control charts and limits. The default limit is 70-130% until in-house limits are generated. If the LCS compound has a recovery above the upper limit, but the same compound is not detected in any of the batch samples, no corrective action is required (Q qualify for DoD projects). For all other situations, the LCS should be reanalyzed. If the second analysis fails, all associated samples should be reextracted/reanalyzed. All recovery problems need to be addressed and steps taken to locate their source.

19.3 Method Blanks - The concentration of the target analyte must be below ½ of the method reporting limit, MRL/LOQ. The first step of corrective action is to assess the effect on the samples. If the analyte is found only in the method blank, but not in any batch samples, no further corrective action may be necessary. Steps should be taken to find/reduce/eliminate the source of this contamination in the method blank. If the analyte is found in the method blank and some, or all, of the other batch samples, then corrective action is required. The source of contamination must be investigated and appropriate action taken and documented to find/reduce/eliminate the source of this contamination. The method blank, and any samples containing the same contaminant, would likely be reextracted/reanalyzed. If a contaminant is found in the method blank and the samples, the compound concentration must be flagged with a 'B' on the final report unless the concentration is greater than 10x that found in the method blank. In any case, a non-conformance report must be completed and forwarded within the same shift in which the contaminant is found.

19.4 Matrix Spike/Matrix Spike Duplicate (MS/MSD) Sample – The frequency is 1 MS/MSD per batch up to 20 samples. MS/MSD recovery limits are set at 70-130% recovery with a maximum RPD of 30%. Generally, batch control is not based on MS/MSD results unless general method failure is determined to be the problem. MS data evaluation must include the consideration of the following factors.

19.4.1 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.

19.4.2 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 non-conformance report to document the problem.

19.4.3 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.

19.4.4 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.

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

21.1 USEPA SW-846 Method 8330

21.2 USATHAMA (1987) U.S. Army Toxic and Hazardous Materials Agency Installation Restoration Quality Assurance Program. Aberdeen Proving Ground, Maryland 21010

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, for all technical methods, should always be the QA/QC summary table and I am including a format for this at the end.

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

Table 1 - Analytes, Reporting Limit (RL), & Low Calibration Standard

Parameter	DL	LOD	LOQ\RL	LowCal
Nitroguanidine	2.5ug/L	5.0ug/L	10ug/L	5.0ug/L
Nitroguanidine	25ug/Kg	50ug/Kg	100ug/Kg	50ug/Kg

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Usability**
DOC Study	• Initially per analyst prior to reporting data • Annually • Follow specific guidelines from section 16 for the preparation and analysis of DOC samples	• Must meet the criteria of the LCS for average accuracy • Precision criteria is 30% relative standard deviation.	• Re-prep and / or • Re-analysis
LOD Verification	• Every quarter	• Parameter must be detected with response greater than 3-times the noise level	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
LOQ Verification	• Every quarter	• Bias Requirement: Organics 50-150% • The LOQ value must be greater than the LOD value	• Re-prep and / or re-analysis • Follow guidelines from SOP QS05
Calibration Curve	• Prior to analyzing any samples • A minimum of 5-points for linear fits • A minimum of 6-points for quadratic fits • Low standard at the RL/LOQ level	• Average RF if %RSD $\leq$ 20% • Linear fit with correlation coefficient of 0.995 or better • Quadratic fit with squared correlation coefficient of 0.99 or better • Manual integrations on curve standards must have supervisory approval • Must follow curve processing requirements from SOP QS08	• Samples cannot be analyzed until there is a passing calibration • Re-evaluate curve mix and makeup • Re-run curve • Check instrument for maintenance needs • Re-prep the curve standards
Retention Time Established	• Once per ICAL and at the beginning of the analytical shift.	• Set using the midpoint of the ICAL or the initial CCV when ICAL is not performed.	
ICV	• Alternate source standard to be analyzed after every calibration curve	• < 15% drift or difference	• Samples cannot be analyzed until there is a passing ICV • Re-analyze an ICV from a different source • Re-prep and re-analyze the ICV • Re-calibrate and verify standard preps and sources

Table 2 - Method Quality Control Requirements Summary			
QC Check	**Minimum Frequency / Requirements**	**Acceptance Criteria**	**Corrective Action for Failures / Data Usability**
CCV	• At the beginning of every sequence • For every 10-client samples • The concentration must be varied from low to mid range • At the end of every sequence	• < 15% drift or difference	• Re-prep and re-analyze the CCV • Re-calibrate and verify standard preps and sources • Re-analyze all samples analyzed since the last passing CCV
MB	• One per prep batch	• Must be less than ½ the LOQ	• 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-prep of samples associated with the MB • NCR will be required for data reported • Final Report data flagging will be required
LCS	• One per prep batch	• 70-130% recovery	• Re-prep of samples associated with the LCS • NCR will be required for data reported • Final Report data flagging will be required
LCSD	• One per prep batch, when MS/MSD not included.	• 70-130% and 30% RPD	• See LCS Criteria
MS/MSD	• One per prep batch, if sample volume available.	• 70-130% and 30% RPD	• Apply J-flag if sample cannot be rerun or reanalysis does not correct problem.

Table 3 - Technical Completeness / Accuracy Checklist

- Were all the QC check elements analyzed – refer to Table 2 of the SOP
- Were the QC criteria met
- In cases of failures, was there an NCR written
- Were all manual integrations signed
- Were dilution factors applied correctly
- Was there supervisory approval for manual integrations on standards and QC samples
- Was the data uploaded into LIMS via direct upload – if yes, then was a cross check subset of the uploaded values performed
- If the data was entered into LIMS manually, was a check of all entered values performed
- Was the red marked data in LIMS checked for accuracy and the corresponding hard copy data documented appropriately
- Were proper data qualifiers applied to the data in LIMS
- 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

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 236 **REVISION #: 02** **EFFECTIVE DATE: 20100907**

METHANE, ETHANE, ETHENE IN AQUEOUS SAMPLES BY MODIFIED RSK-175 (AUTOMATED HEADSPACE)

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/8/10

Changes Summary

Revision 02, 09/07/2010

- The SOP is an update from Revision 01 dated 04/28/09
- 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.
- Calculations have been added to

Table of Contents

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8. Safety
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20. Waste Management
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1.0 Identification of the Test Method

The GC/FID/Headspace system is used to analyze methane, ethane, and ethene in aqueous samples.

2.0 Applicable Matrix or Matrices

Aqueous samples

3.0 Detection Limit

Analyte	MDL/DL	LOD	LOQ/RL	Units
Ethane	1.00	2.00	4.00	ug/L
Ethene	1.00	2.00	4.00	ug/L
Methane	1.00	2.00	4.00	ug/L

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 above.

4.2 The GC/FID/Headspace system is used to analyze methane, ethane, and ethane in aqueous samples.

5.0 Summary of the Test Method

This test is used to determine the amount of Methane, Ethene and Ethane in an aqueous sample. Samples are collected using 40mL VOA vials without headspace. Each sample is analyzed using a Tekmar 7000 Headspace autosampler and HP 5890 series II GC with FID.

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

Methane found in the lab environment can be a source of contamination.

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 Care should be used in handling all samples.

8.2.1 Safety glasses must be worn in the lab at all times. The use of gloves and lab coats is highly recommended.

8.2.2 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.

8.2.3 MSDS sheets are available for all reagents and standards that have been purchased. These are located in the bookshelves across from the Quality Assurance Officers cube.

8.2.4 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.

9.0 Equipment & Supplies

9.1 Gas Chromatograph:
HP 5890 Series II (temperature programmable).

9.2 Autosampler:
Tekmar 7000 Headspace autosampler

9.3 Columns-Capillary columns:
Carboxen 1006 PLOT column–30 meter x 0.53mm ID(or equivalent)

9.4 Data Acquisition and Processing Software:
9.4.1 HP Chemstation system is interfaced to the HP-GC for data acquisition, processes, and stores acquired data.

9.5 Detector
Flame Ionization Detector

9.6 Glassware
9.5.1 25ml Syringe
9.5.2 20ml headspace vials with crimp tops
9.5.3 Gastight syringes- 10µL, 100µL, 500µL and 1mL

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. 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 Gas standards are purchased from Restek and other vendors depending on their availability. The date they are received is noted on the container they are received in. The standards are given a sequential number the day they are opened and this is noted on the tank. Standards for MEE are Scotty gases purchased in pressurized tanks.

11.0 Sample Collection, Preservation, Shipment, and Storage

Section 3.0 and tables 3-1 and 3-2 of the Empirical Laboratories, LLC Quality Assurance Manual include details concerning sample preservation, containers and handling of volatile

samples. Samples are collected in 40 ml VOA vials and shipped to the lab in coolers with ice. Water samples are stored in the Hobart in the sample storage room at a temperature of 4°C.

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. Four LCSs are analyzed. See **Table 2** for acceptance criteria.

13.0 Calibration and Standardization

13.1 Refer to SW-846 Method 8000B for proper calibration techniques.

13.2 A minimum five point calibration curve must be introduced into the GC and analyzed for each analyte of interest using the appropriate instrument parameters prior to running samples. If the percent relative standard deviation (% RSD) of the calibration factor is more than 20 percent over the working range, linearity through the origin can be assumed and the average calibration factor can be used in place of a calibration curve. If % RSD is over 20 percent Linear or Quadratic ranges may be used (linear curve corr. >0.995, quadratic >0.99 with six points). The curve is then verified using a second source standard (80-120% criteria).

13.3 Calibration standards at a minimum of five levels are prepared by injecting the gas from a Scotty gas standard tank into capped 20 ml headspace vials with 15 ml of D.I. water using a gas-tight syringe.

13.3 The calibration curve must be verified every day samples are run through the analysis of a mid-level standard at the beginning of the sequence, after every 10 field samples and at the end of the sequence. The percent difference back to the curve must not exceed 20 percent. If this criteria is not met, corrective action must be taken before sample analyses continues. Usually this involves recalibration or checking the gastight syringes.

14.0 Procedure

The following information describes the instrument and QC requirements to analyze the compounds that we do by this method.

14.1 Instrumentation

14.1.1 GC

a. Initial Temperature: 35 º C hold for 3.0 minutes.
b. Ramp: 25 ºC/minute to 225 º C.
c. Final Temperature 225 º C hold for 3.08 minutes.
d. Detector Temperature 230 º C.

14.1.2 Headspace Autosampler

a. Platen: 80 º C/Platen Equilibration: 0.50 min.
b. Sample Equilibration: 2.0 min.
c. Pressurization: 0.50 min/Pressure Equilibration: 0.25 min.

d. Loop Equilibration: 0.20 min.
e. Injection Time: 0.75 min.
f. Valve and Line Temp.: 90ºC.

14.2 Retention Time (RT) Windows - RT criteria set forth in SW-846 method 8000C are used to set retention time windows. New in-house retention time windows are established after every major change to the system (new column or temperature program) and at initial calibration with midpoint standard. If the established retention time window is less than +/ 0.03 minutes, the window defaults to +/ 0.03 minutes. Retention times are updated with the first CCV of the day or the mid-level standard of the curve if samples are analyzed directly after a curve.

14.3 Quality control for this method can be referenced in SW-846 Method 8000C.

14.3.1 A method blank is required before analyzing samples. The contamination level should not exceed the CRDL.

14.3.2 A Laboratory Control Sample (LCS) is required every 20 samples. A Laboratory Control Sample Duplicate (LCSD) is used if sample volume is insufficient for running MS/MSD. Limits 80-120 %.

14.3.3 An MS/MSD pair are run upon client request every 20 samples per matrix. Limits 80-120 %.(Due to limited sample, MS/MSD maybe omitted and an LCSD is used instead.)

14.4 Sample analysis includes the following steps: 15 ml of sample are transferred to 20ml headspace using a 25mL syringe. Vials capped and loaded onto the autosampler along with a method blank with 15 ml of D.I. water, an LCS and LCSD.

14.4.1 A mid-level standard must be run at the beginning, after every 10 field sample, and end of the sequence. The mid-level standard cannot exceed 20 percent difference from the initial calibration.

14.4.2 The retention times are updated with the first midpoint check of the day or from the midpoint of the calibration curve if analyzed before the samples.

14.5 Following sample analysis, the data is reduced using the Chemstation data system. The following must be checked to see if the samples will require re-analyses or dilution.

14.5.1 The analyte concentration must be within the range of the calibration curve. If an analyte exceeds the curve, a dilution must be performed and the next sample must be checked for carryover. Any dilution should keep the concentration of the analyte in question within the top half of the curve.

14.6 Demonstration of Capability (DOC) – Each analyst must perform a DOC to demonstrate proficiency with these methods. See Table 2.

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 Calculations:

$$\text{Calibration Factor (CF)} = \frac{\text{Response}}{\text{Dec Equivx1000}}$$

Sample	Example	Example Calculated
Dilution	1	1
Methane Instrument Reading (DE*1000)	0	0.592438
Ethene Instrument Reading (DE*1000)	0	0.0297104
Ethane Instrument Reading (DE*1000)	0	0
Methane (µg/L)	=B26	150.76
Ethene (µg/L)	=B37	15.81
Ethane (µg/L)	=B48	ND
Samples	**Methane**	**Methane**
Dec. equiv. Calc.	=B3/1000	0.000592438
x(g)	=B18/41300	1.43447457627119E-08
n(g)	=55.5*B19/1.137	7.00205268100711E-07
C (mg cpd/L water)	=B20*16000	0.0112032842896114
D (g/L)	=16/(22.4*(298/273))	0.654362416107383
A(h) mL cpd	=5.4*B18	0.0031991652
A(l) (mg cpd/L water)	=(B23/0.015)*(B22)	0.139560897986577
TC (mg cpd/L water)	=B24+B21	0.150764182276189
TC in µg/L	=B25*1000	150.764182276189
Samples	**Ethene**	**Ethene**
Dec. equiv. Calc.	=B7/1000	0.0000297104
x(g)	=B29/11400	2.60617543859649E-09
n(g)	=55.5*B30/1.137	1.27214368374763E-07
C (mg cpd/L water)	=B31*28000	0.00356200231449336
D (g/L)	=28/(22.4*(298/273))	1.14513422818792
A(h) mL cpd	=5.4*B29	0.00016043616
A(l) (mg cpd/L water)	=(B34/0.015)*(B33)	0.0122480625503356
TC (mg cpd/L water)	=B35+B32	0.0158100648648289
TC in µg/L	=B36*1000	15.8100648648289
Samples	**Ethane**	**Ethane**
Dec. equiv. Calc.	=B8/1000	=M8/1000
x(g)	=B40/30200	=M40/30200
n(g)	=55.5*B41/1.137	=55.5*M41/1.137
C (mg cpd/L water)	=B42*30000	=M42*30000
D (g/L)	=30/(22.4*(298/273))	=30/(22.4*(298/273))
A(h) mL cpd	=5.4*B40	=5.4*M40
A(l) (mg cpd/L water)	=(B45/0.015)*(B44)	=(M45/0.015)*(M44)
TC (mg cpd/L water)	=B46+B43	=M46+M43
TC in µg/L	=B47*1000	=M47*1000

16.0 Method Performance

16.1 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 is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria.

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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.
Please see Waste Disposal, SOP-210 and SOP-405 for proper disposal of waste coming from this area within our laboratory.

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.

21.0 References

21.1 Newell, Bryan, RSKSOP-175, Rev.0, August 1994.

21.2 Newell, Bryan, RSKSOP-147, Rev.0, January 1993.

21.3 Test Methods for Evaluating Solid Waste Physical/Chemical Methods, SW-846; Third Edition (Update III); Method 8000C.

Acronyms

°C - degrees centigrade
CF - calibration factor
CRDL - contract required detection limit
%D - percent difference
FID - flame ionization detector

GC - gas chromatograph
LCS - laboratory control sample
MDL - method detection limit
µL - microliter
µm - micrometer
mL - milliliter
mm - millimeter
MS - matrix spike
MSD - matrix spike duplicate
%RSD - percent relative standard deviation
RT - retention time
SOP - standard operating procedure

Refer to SOP QS08 for further definitions.

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1 included in section 3.0 above

22.2 Table 2, quality control criteria

22.3 Table 3, analyst review checklist

Table 1. Organic Analysis by Gas Chromatography (Method RSK-175)					
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 or 0.03min., whichever is greater.	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 ≤ 20% Option 2: linear least squares regression: r ≥ 0.995 Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 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.

Table 2. Organic Analysis by Gas Chromatography (Method RSK-175)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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.	
Second source calibration verification (ICV)	Immediately following ICAL.	All project analytes within established retention time windows. GC methods: All project analytes within ± 20% 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. GC methods: All project analytes within ± 20% 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 > ½ 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.

Table 2. Organic Analysis by Gas Chromatography (Method RSK-175)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
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, 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, 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.
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 ≤ 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.
Results reported between DL and LOQ	NA	NA	NA	Apply J-flag to all results between DL and LOQ.	

ANALYST DATA REVIEW CHECKLIST

Sample Number(s):
Batch Number(s):
Method: Methane, Ethane, Ethene Modified RSK-175

QA/QC Item	Yes	No	NA	Second Level Review
A. Initial Calibration				
1. Does the curve consist of five Calibration Standards?				
2. Is the low standard near, but above the MDL?				
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 criteria been met?				
C. Continuing Calibration				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 10 samples and at the end of the sequence?				
2. Are the % differences within QC limits for all analytes?				
D. QC Samples				
1. Is the Method Blank analyzed at the desired frequency and is its concentration for target analytes less than ½ RL?				
2. Is the Laboratory Control Sample and its percent recovery within QC limits?				
3. Is the Matrix Spike/Matrix Spike Duplicate analyzed at the desired frequency and is the percent recovery/RPD within QC limits?				

Comments on any "No" response:

Primary Review: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 239 **REVISION #: 04** **EFFECTIVE DATE: 20100907**

PERCHLORATE IN WATER, SOIL, AND SOLID WASTE USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/ ELECTROSPRAY IONIZATION/ MASS SPECTROMETRY BY SW846 METHOD 6850

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/8/10

Changes Summary

Revision 04, 09/07/2010

- The SOP is an update from Revision 03 dated 08/06/2010
- DL/LOD/LOQ have been increased for perchlorate in section 3.0.
- DoD F-tables have been reviewed/updated for tuning criteria.

Revision 03, 08/06/2010

- The SOP is an update from Revision 02 dated 07/21/2009
- 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.
- DL/LOD/LOQ information and F-tables from DoD QSM added.

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PERCHLORATE IN WATER, SOIL, AND SOLID WASTE USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY/ ELECTROSPRAY IONIZATION/ MASS SPECTROMETRY BY SW846 METHOD 6850

1.0 Identification of the Test Method

1.1 This SOP is used for the Analysis of Perchlorate in Water, Soils, and Solid Wastes using High Performance Liquid Chromatography / Electrospray Ionization/Mass Spectrometry (HPLC / ESI / MS).

2.0 Applicable Matrix or Matrices

2.1 This method is for the determination of perchlorate in groundwater, surface water, soil, and sediment using high performance liquid chromatography (HPLC) coupled with electrospray ionization (ESI) mass spectrometry (MS).

3.0 Detection Limit

3.1 Detection Limits are highly matrix-dependent. Routine limits are listed below:

Analyte	MDL/DL	LOD	LOQ/RL/LowCal	Units
Perchlorate (water)	0.0620	0.200	0.500	ug/L
Perchlorate (soil)	0.620	2.00	5.00	ug/Kg

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 the table above. This table also lists the associated Reporting Limit (also defined as the LOD) and the lowest Calibration level for each analyte.

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 Solids are first extracted prior to analysis with reagent water using an ultrasonic bath. Water samples are not extracted. Extracts and water samples are filtered, if needed. An appropriate volume of the sample or sample extract is introduced into a HPLC/MS instrument. Perchlorate is separated by HPLC from the sample matrix, using negative electrospray ionization and detected by MS using mass-to-charge (m/z) using mass-to-charge ratios 99, 101, and 107. Quantitation is performed using m/z 99 and internal standard calibration. Isotopically-labeled perchlorate (Cl18O4-), m/z 107, serves as the internal standard. The 99/101 isotopic ratio reflects the isotopic ratio of naturally occurring 35Cl/37Cl and is used for additional confirmation of perchlorate identification.

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 artifacts and/or interferences to sample analysis. All of these materials must be demonstrated to be free from interferences under the conditions of the analysis by analyzing method blanks.

7.2 Sample extracts with any visible particulates should be filtered through a minimum 0.45-µm nominal pore size membrane or frit to remove particulates and prevent damage to the instrument, columns and flow systems. Filters specifically designed for HPLC applications should be used.

7.3 Hydrogen sulfate ion H34SO4-, formed from a minor sulfur isotope, is commonly present in samples. H34SO4- elutes before perchlorate but at high concentrations can tail into the retention time of the perchlorate peak and elevate its baseline at m/z 99. Quantitation of perchlorate based on m/z 83 and 89 avoids this potential interference from H34SO4-.

7.4 Glassware must be scrupulously cleaned. Glassware should be rinsed as soon as possible after use with the last solvent used. This should then be followed by detergent washing with hot water and rinses with tap water and organic-free reagent water. After washing, the glassware should then be drained, dried, and heated in a laboratory oven at 130°C for two to three hours before reuse. Solvent rinses with acetonitrile may be substituted for the oven heating. After drying and cooling, glassware should then be stored in a clean environment to prevent accumulation of dust or other contaminants.

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 & Supplies

9.1 Volumetric flasks, 100-mL and other sizes as needed.

9.2 Automatic precision pipetters and disposable tips - 10 mL capacity and gas tight syringes.Disposable autosampler vials, 2mL capacity.

9.3 Disposable plastic centrifuge tubes - 15 mL.

9.4 Disposable plastic micro-beakers.

9.5 Sample bottles - polyethylene or glass of sufficient volume to allow replicate analyses.

9.6 Disposable PTFE 0.45-µm, surfactant-free, membrane syringe filters.

9.7 SPE Cartridges – C18 (2000 mg) chromatography columns or equivalent for extract solution cleanup.

9.8 High Performance Liquid Chromatograph/Mass Spectrometer - An analytical system combining a high performance liquid chromatograph for separation of the sample components, electrospray ionization interface and mass spectrometer for fragmentation and detection of sample components.

9.8.1 High Performance Liquid Chromatograph (Agilent 1200) – The instrument contains a programmable solvent delivery system and all required accessories including injection loop, pump, analytical columns, autosampler and UV detector.

9.8.2 Electrospray ionization (ESI) source – The ESI source generates gas phase ions of perchlorate from the liquid phase.

9.8.3 Mass Spectrometer – A quadrupole mass analyzer, Agilent 6140 LC/MS quadrupole mass spectrometer. Instrument tuning and instrument conditions are based on the manufacturer's instructions. Tuning is not required prior to daily analyses. Tuning parameters are available from the instrument manufacturer,

along with mass tuning solutions and instructions on how to optimize the mass spectrometer.

9.9 Analytical column – 4 mm x 250 mm Metrohm Metrosep A Supp 5 column (cat# 6.1006.530) or equivalent. Guard column- Metrohm Metrosep A Supp 4/5 S-Guard(cat# 6.1006.540).

9.10 Data system capable of performing analyte signal acquisition, peak integration, instrument calibration and analyte quantification.

9.11 Analytical balance, capable of ±0.0001 g accuracy.

9.12 Centrifuge - capable of 5000 rpm and/or generating g-force of such strength as needed for providing adequate separation of a solid suspension and thus yielding a clear supernatant for sample injection and analysis.

9.13 Ultrasonic Bath

9.14 Vortex Mixer

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:

Reagent 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.

10.2 Reagents

- 10.2.1 Acetonitrile – HPLC grade or equivalent
- 10.2.2 Ammonium Formate – Reagent grade
- 10.2.3 Ammonium Carbonate – Reagent grade
- 10.2.4 Ammonium Hydroxide – Reagent grade
- 10.2.5 DI water – MODULAB Research Grade Water System

10.3 Standards:

- 10.3.1 All Standards for this analysis are prepared in water from the MODULAB system.
- 10.3.2 The initial calibration curve given is subject to variation due to detection levels needed. A minimum of five calibration standards is recommended as well as a blank standard. The lowest concentration level shall be at the reporting level and at least three times the MDL. The remaining levels should define the working linear range of the analytical system.
- 10.3.3 Vendors and vendors' products are sometimes listed for the ease of the analyst using this SOP, but products and purchased concentrations are examples only and subject to change at any time. All purchased standards are certified by the vendor.

Certificates of Analysis are kept in the department. Purchased standards are routinely checked against an independent source for both analyte identification and analyte concentration.

10.3.4 Stock standard solution (1000 mg/L ClO_4^-) - This solution is purchased commercially as a certified standard. Stock standards may be stored at room temperature for a period of up to 12 months. Expiration dates should be clearly specified on the label.

10.3.5 Intermediate Standard (10 mg/L ClO_4^-) – Dilute 1.0 ml of 1000 mg/L standard solution to 100 mL with DI water. Working standard may be stored at room temperature for up to 12 months. Expiration dates should be clearly specified on the label.

10.3.6 Intermediate Standard (0.1 mg/L ClO_4^-) – Dilute 1.0ml of 10 mg/L standard solution to 100 mL with DI water. Working standard may be stored at room temperature for up to 12 months. Expiration dates should be clearly specified on the label.

10.3.7 Calibration standards – Prepare by adding various amounts of the 0.1 mg/L intermediate standard solution to DI water and dilute to 10 ml. Spike each calibration standard with 50uL of internal standard spiking solution (1.0 mg/L). The final internal standard concentration is exactly the same (5.0 µg/L $Cl^{18}O_4^-$) for each calibration standard.

Calibration Concentration(ug/L)	Volume (uL) of 0.1 mg/L ClO_4^- working std	Final Volume (mL)
0.20	20	10
0.50	50	10
1.00	100	10
2.00	200	10
5.00	500	10
10.0	1000	10

10.3.8 Internal standard spiking solution (1000 µg/L $Cl^{18}O_4^-$) –Purchased commercially. Refrigerate. Expires per manufacturer's indications or 12 months if no other indication is given.

10.3.9 Each standard and sample is spiked with 50 µL of internal standard spiking solution per 10 mL of sample or standard to obtain a final internal standard concentration of 5 µg/L $Cl^{18}O_4^-$.

10.3.10 HPLC Mobile Phase – Prepare by dissolving 0.946 grams of ammonium formate and 0.721 grams of ammonium carbonate in 250 ml of DI water, add 700 mL acetonitrile, 10 ml of ammonium hydroxide, and bring up to 1 liter with DI water.

10.3.11 Second Source Standard – The second source standard is purchased at 1000 mg/L from a different manufacturer than the Stock standard solution. Make a 10 mg/L standard by diluting 1 ml to 100 ml of DI water. Expires per manufacturer's indications or 12 months if no other indication is given.

10.3.12 Independent Calibration Verification Standard (ICV): The ICV is prepared from the second source standard at a final concentration of 5.0 ug/L.

10.3.13 Continuing Calibration Verification Standard (CCV): The CCV is prepared from the 0.1 mg/L intermediate standard solution at a final concentration of 5.0 ug/L.

10.3.14 LODV(Limit of Detection Verification, 0.2 ug/L) – A standard prepared at a concentration approximately 2 times the MDL.

10.3.15 LCS/MS Spiking Solution (0.1 mg/L) – Dilute 1.0 mL of a 10 mg/L Second Source Perchlorate Standard to 100 mL with DI water. Use this to spike the MS and the LCS. Store at room temperature. Expires in 6 months.

10.3.16 Laboratory Control Sample (LCS) (0.2 ug/L) – Dilute 20 uL of 0.1 mg/L LCS/MS solution to 10 mL DI water. Process as a sample.

10.3.17 Interference Check Sample (ICS) (0.2 ug/L):

10.3.17.1 ICS Matrix preparation - 5000 mg/L each of chloride, sulfate, carbonate by dissolving 8.240g NaCl +7.395g Na2SO4 +5.294g Na2CO3 in DI and diluting to 1000ml.

10.3.17.2 ICS – prepared upon use – add 1.0 mL of ICS matrix to 8.0 mL HPLC water. Add 20 uL of 0.1 mg/L perchlorate standard (second source = LCS/MS Spiking Solution) and 50 uL internal standard. Prepare and process as a sample.

10.3.18 MS/MSD (0.2 ug/L) – Add 20 uL of 0.1 mg/L spiking solution to 10 mL of water sample. Process as a sample.

10.3.19 The LCS for Soil (+2.0 ug/kg) - Add 20 uL of 0.1 mg/L spiking solution to 1 g of Ottawa sand or soil sample. Process as a sample.

10.3.20 Mass Calibration Standard – a proprietary mix from Agilent. Use, store, and discard as per manufacturer's instructions.

10.3.21 Tune Verification Standard – 100 ug/L of perchlorate.

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 Water samples should be collected in purchased, pre-cleaned 125-mL polyethylene bottles.

11.3 For solids, collect samples in purchased clean, 4-oz amber glass jars.

11.4 Prepare and analyze samples and extracts within 28 days of collection.

11.5 Care should be taken to avoid temperature extremes during shipment and storage.

11.6 Samples will be refrigerated to 0-6°C upon sample login.

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. Four LCSs are analyzed at a concentration up to 4x the LOQ. See **Table 2** for acceptance criteria.

12.3 Tune verification – Verification is performed after Mass Calibration and before a new calibration curve. The quantitation ions must be within ±0.3 *m/z* of actual.

12.4 Initial Calibration Criteria: Calibration criteria are given in section 13.

12.5 Method Blank:

12.5.1 Frequency - Process one MB with each preparation batch of 20 or fewer samples.
12.5.2 The MB should be carried through all stages of sample preparation and analysis.
12.5.3 Acceptance Criteria - The result of the MB must be less than ½ the reporting limit.
12.5.4 Corrective Action - If it is not, determine the source of the contamination and reprep and reanalyze the associated samples.

12.6 Reagent blanks:

12.6.1 Frequency – prior to calibration and after each batch is analyzed. When perchlorate concentrations in the samples are over the linear range, any samples which follow the overrange sample will be repeated if the sample concentrations were greater than ½ the RL.
12.6.2 Acceptance Criteria – must be less than ½ RL.
12.6.3 Corrective Action – reanalyze reagent blanks until no carryover is observed (<1/2 RL). Reanalyze all samples processed since the contaminated blank. If the closing reagent blank fails, reanalyze all samples since the last clean reagent blank.

12.7 MS:

12.7.1 Frequency - Prepare one MS (or MS/MSD pair) with each batch of 20 or fewer
12.7.2 samples of the same matrix.
12.7.3 Acceptance Criteria - The limit for the water and soil is 80-120% of the true value.
12.7.4 Corrective Action - If recovery of MS fails on a sample batch the samples are
12.7.5 acceptable only if the LCS meets acceptance criteria. If the LCS passes, but the MS fails, report the MS recoveries and note outliers in the case narrative.

12.8 LCS:

12.8.1 Frequency - Prepare one LCS with each batch of 20 or fewer samples of the same matrix.
12.8.2 Acceptance Criteria (recovery) - The limit for the water and soil LCS is 80-120%.
12.8.3 Corrective Action - If the LCS fails on a sample batch, reprep and reanalyze the
12.8.4 sample batch. If a duplicate LCS was run with the batch and one LCS was slightly out, but the other LCS was in, the data is accepted.

12.9 DUP/MSD Precision:

12.9.1 Frequency - Precision for each prep batch of 20 or fewer samples of the same matrix must be established by either analyzing a sample in duplicate (DUP) or analyzing a MS in duplicate (MSD).
12.9.2 Acceptance Criteria – (for all samples above the reporting limit), the RPD value between samples or MS/MSD results must not exceed 15%.
12.9.3 Corrective Action - examine the chromatograms and benchsheets for potential matrix interferences. Reanalyze the pair if deemed appropriate. The outlying RPD should be mentioned in the Case Narrative so that data may be flagged appropriately.

12.10 The ICV is discussed in Section 13.

12.11 CCV:

12.11.1 Frequency – bracketing every 10 or fewer samples
12.11.2 Acceptance Criteria - All samples must be bracketed by an acceptable CCV. The acceptance limit is 15% difference relative to the true value.

12.11.3 Corrective Action - If the CCV does not meet acceptance criteria, correct the problem and achieve an acceptable CCV. Recalibration may be necessary. All samples bound by the unacceptable CCV must be reanalyzed with acceptable QC.

12.12 Internal standard area response verification:

12.12.1 Frequency – The IS is added to every sample, spiked sample, standard, instrument blank, and method blank

12.12.2 Acceptance Criteria - The internal standard area counts for each sample and QC standard must be monitored throughout the analysis and the IS area must be within ±50% of the average of the internal standard area counts of the ICAL standards.

12.12.3 Corrective Action - If the internal standard area counts exceed ± 50% of the average of the internal standard area counts of the ICAL standards, rerun the sample at increasing dilutions until the acceptance criteria are met. If the criteria cannot be met with dilution, interference is suspected and the sample must be reprepared using additional pretreatment steps. Thoroughly document corrective action in the case narrative.

12.13 Internal standard and Target retention times:

12.13.1 Frequency – IS is added to every sample, spiked sample, standard, instrument blank, and method blank.

12.13.2 Acceptance Criteria – The RRT (relative retention time) must be 0.98-1.02. Calculate the RRT in a sample by dividing the retention time of the perchlorate ion by the retention time of the internal standard.

12.13.3 Corrective Action – Thoroughly document in the case narrative.

12.14 LODV(Limit of Detection Verification 0.2 ug/L)

12.14.1 Frequency – before and directly after every analytical batch of samples. It may be run more frequently to reduce reanalysis rate.

12.14.2 Acceptance criteria – the recovery must be within 30% of the true value. Exception – any samples with concentrations above the RL may be reported.

12.14.3 Corrective Action – if there are samples below the RL, correct the problem, obtain a compliant LODV and reanalyze affected samples.

12.15 ICS (Interference Check Standard)

12.15.1 Frequency – one ICS is extracted with every batch of 20 or fewer samples. At least one ICS must be analyzed daily.

12.15.2 Acceptance Criteria – the recovery must be within 30% of the true value.

12.15.3 Corrective action – Correct the problem and reanalyze all samples associated with a failed ICS.

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

13.2.1 Start up entire HPLC system and allow baseline response to settle.

13.2.2 All injections of standards and samples and associated QC are to be the same. 100 uL injections are used.

13.2.3 The first sample through the system should be an instrument blank. This blank is simply an injection of DI water and internal standard. As the first injection of the day, this helps to further settle the baseline and equilibrate the system.

13.2.4 A pre-curve blank is run to check for system and reagent cleanliness. Any potentially interfering peaks should be noted. This blank is prepared alongside the calibration standards.

13.2.5 Process a minimum of 6 calibration standards and a blank (as described in Section 10). The lowest calibration standard must be at (or below) the reporting limit and the standard at the reporting limit must be at least three times the MDL.

13.2.6 Analyte responses are tabulated and recorded in the integration software.

13.2.7 Calibration Curve –

13.2.8 Response Factor - To obtain the average response factor, calculate the RF (as below) for each calibration point. Calculate the mean of the RFs to use for sample calculation. Calculate the Standard Deviation of the RFs and the Percent Relative Standard Deviation of the RFs to evaluate the linearity. The limit for the calibration to use average response factor for quantitation is 20% RSD. For a linear curve the minimum correlation is 0.995.

$$RF = \frac{(As)(Cis)}{(Ais)(Cs)}$$

Where:
As = Area of the characteristic quantitation ion for compound .
Ais = Area of the characteristic quantitation ion for the specified internal standard.
Cs = The concentration of the compound added (ppb).
Cis = The concentration of the specified internal standard (ppb).
Calculate standard deviation (SD) and the percent relative standard deviations (%RSD) for each analyte from the mean with:

$$SD = \sqrt{\frac{\sum_{I=1}^{N} (RFi - \overline{RF})^2}{N - 1}}$$

where:
RFi = RF for each of the calibration levels
$\overline{RF}$ = mean of initial RFs
N = Number of RF values (i.e., 6)

$$\%RSD = \frac{(SD)}{(Avg\ RF)X\ 100.}$$

where:
RSD = relative standard deviation.
RF = mean of 6 initial RFs for a compound.
SD = standard deviation of average RFs for a compound

13.2.9 The concentration corresponding to the absolute value of the calibration curve's Y-intercept must be ≤ LOD.

13.2.10 All of the ICAL criteria must be acceptable before samples may be analyzed.

13.2.11 Routinely, after the high point of the curve, a blank is analyzed to prove that there is no carry-over from the autosampler. It should be noted if there are any target hits above ½ of the RL.

13.2.12 Verify the calibration by analyzing an independent calibration verification standard. Acceptance criteria for the ICV is 85-115% recovery.

13.2.13 Samples immediately following an initial calibration and ICV may skip the system blank and the opening CCV. Set up the rest of the sequence to comply with the frequency requirements of the QC listed in section 12.

14.0 Procedure

14.1 Start up entire HPLC system and allow baseline response to settle.

14.2 All injections of standards and samples and associated QC are to be the same. 100 uL injections are used.

14.3 The first sample through the system should be an instrument blank. This blank is simply an injection of DI water and internal standard. As the first injection of the day, this helps to further settle the baseline and equilibrate the system.

14.4 Instrument conditions for analysis are set to optimize response and separation of the analytes. Analysts are advised to adjust the HPLC systems to particular analytical needs. Once optimized, the same conditions are to be used for analysis of all standards, client samples, and QC samples. The current system uses the conditions in Table 1. Actual instrument conditions are optimized and recorded in the instrument daily run log.

14.5 Mass Calibration:

14.5.1 Frequency – Upon instrument setup and as needed (QC failures, large ion

14.5.2 mass deviations, major instrument maintenance, tune won't meet criteria)

14.5.3 Acceptance Criteria – the calibration range must bracket the ion masses of interest without greatly exceeding the range. The most recent mass calibration must be used for an analytical run and the same mass calibration must be used for all data files in an analytical run. Mass calibration must be verified by acquiring a full scan mass spectrum of a perchlorate standard (200ug/L). Perchlorate ions should be within ±0.3 *m/z* of mass 99 and 101.

14.5.4 Corrective action – Correct the problem and recalibrate if the mass calibration fails.

14.6 Retention Time - Due to the use of the internal standard, formal retention time studies are not needed. The retention time varies due to sample matrix conductivity, however the internal standard retention time varies at the same rate. It is the relative retention time between the sample and the internal standard which is most critical and must be monitored (see section 12). The retention times of the sample and internal standard are summarized and included in the report.

14.7 Interference Threshold study:

14.7.1 Frequency – at initial setup and when major changes occur in the method's operating procedures (addition of cleanup procedure, column changes, mobile phase changes).

14.7.2 Acceptance Criteria – Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the concentration of the common suppressors where perchlorate recovery of a 0.5 ug/L standard falls outside an 85-115% window. Analyze successively higher conductivity standards with the perchlorate concentration at 0.5ug/L. Presently, samples are diluted starting are a conductivity of 500µs.

14.8 Initial Calibration – see section 13.

14.9 Daily Sample Analysis- When an ICAL is not run on the day of sample analysis, run blanks initially to warm up the system. Do not begin the sequence until the blanks are less than ½ the RL. Analyze the CCV. Set up the rest of the sequence to comply with the frequency requirements of the QC listed in section 12.

14.10 Evaluation of Sample Analysis

14.10.1 Examine solution concentrations of target analytes in the samples. If the concentration is greater than the high calibration standard, reanalyze the sample at a dilution. As a general rule, dilutions should be performed to keep target analytes in the upper half of the calibration range.

14.10.2 Check for possible carryover. Pay particular attention to situations where samples containing low levels of target analyte were analyzed one or two injections after samples containing levels of target analyte near or over the high end of the calibration range. Reanalyze as needed to confirm.

14.10.3 Check peak integrations. Where possible, all integrations should be performed consistent with integration of the corresponding calibration standards.

14.10.4 Monitor the retention times of the ions in samples and QC. The retention times of the ClO3- m/z 99 peak and the IS Cl18O3- m/z 107 peak should not vary by more than 0.2 min from each other.

14.10.5 Monitor the mass ratio between ions 99/101. The ion ratio should fall within the established window of 2.3-3.8. If criteria not met, the sample(s) must be repeated. Reextract using cleanup procedures or alternative techniques to confirm the presence of perchlorate such as post spikes or dilutions to reduce the interference. Qualify the data and discuss in the case narrative as necessary.

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 GC oven conditions – see table 1.

15.3 Identify perchlorate in the sample chromatograms

15.3.1 Compare the retention times of the ClO4-, m/z 99, peak and the internal standard Cl18O4-, m/z 107, peak. The retention times should not vary by more than 0.2 minutes from each other. The relative retention time (Retention time of perchlorate ion divided by the retention time of the internal standard) must be 0.98 – 1.02.

15.3.2 Evaluate the relative abundances of the m/z 99 and 101 ions in the chromatogram. To confirm the presence of perchlorate, the 99/101 peak area counts ratio should be 2.3-3.8. Repeat the samples if ratio criteria not met or flag the data as estimated and discuss in the case narrative.

15.4 Calculate the perchlorate concentration for each sample using the internal standard calibration. Use m/z 99 and 107 from the internal standard for quantitation. Calculate the concentration of each of the sample matrices as follows:

15.4.1 Water Samples

Concentration (µg/L) = $(C)(D)$

Where:
C = Concentration from the calibration curve in µg/L.
D = Dilution factor, if needed

15.4.2 Soil Samples

Concentration (µg/kg ClO4-) = $\frac{(C)(V)(D)}{M}$

Where:
C = Concentration in extract (µg/L ClO4-)
V = Volume of sample extract used for analysis (mL)
D = Dilution factor (if needed)
M = Mass of initial sample extracted (g)
Results are adjusted for soil samples based on Dry Weight.

15.5 Calculate the perchlorate concentration for each sample using the internal standard Adjustments to the Reporting Limit - RLs are elevated for insufficient sample volume, matrix interference, and dilutions.

15.6 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, and/or Technical Director or Quality Assurance Manager.

16.0 Method Performance

16.1 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 is completed by each analyst and then provided to the supervisor for further processing and approval. See **Table 2** for acceptance criteria. **When analyzing DOCs for DOD QSM Version 4.1, DOD limits will be used.**

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

18.1 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

19.1 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

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 Physical/Chemical Methods, SW-846; Third Edition (Update III);* Method 6850.

21.2 DoD Perchlorate Handbook, Revision 1, Change 1, August 2007.

22.0 Tables, Diagrams, Flowcharts and Validation Data

22.1 Table 1, Instrument Settings.

22.2 Table 2, QA/QC summary table

22.3 Table 3, Technical Completeness / Accuracy Checklist

22.4 Table 4, Data Reviewers Checklist

TABLE 1 INSTRUMENT SETTINGS

Autosampler: Injection Volume: 100 µL

Analytical Column: Column Temperature: 35 ºC

HPLC Pump
Flow Rate: 0.5 mL/min
Run Time: 15.0 min
Mobile Phase:
0.946g Ammonium Formate, 0.721g Ammonium Carbonate dissolved in 250 ml DI water, 700 ml Acetonitrile, 10 ml Ammonium Hydroxide, bring to one liter with DI water.

Mass Spectrometer
Ionization Mode: Electrospray
Polarity: Negative

SIM Parameters:

SIM Ion	Fragmentor	Gain (EMV)	Dwell
99	120 V	10	995 msec
101			995 msec
107			995 msec

Spray Chamber:

Gas Temperature	Drying Gas (N2)	Nebulizer Pressure
350 ºC	12.0 L/min	50 psig

Capillary voltage:
1700V Negative

Table 2. Perchlorate Analysis (Method 6850)					
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 the analyte that did not meet criteria (see Section C.1.f).	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.
Initial calibration (ICAL)	Minimum of 5 calibration standards to establish linearity at method set-up and after major maintenance.	$r \geq 0.995$ or RSD $\leq 20\%$. The concentration corresponding to the absolute value of the calibration curve's Y-intercept must be $\leq$ LOD.	Correct problem, then repeat ICAL.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be run until ICAL has passed. The calibration is linear and shall not be forced through the origin.
Initial calibration verification (ICV)	Once after each ICAL, analysis of a second source standard at the midpoint of the calibration.	Within ± 15% 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 may be run until calibration has been verified.
Continuing calibration verification (CCV)	Analysis of mid-level standard after every 10 field samples. All samples must be bracketed by the analysis of a standard demonstrating that the system was capable of accurately detecting and quantifying perchlorate.	Within ± 15% of true value.	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 and discuss in the case narrative for all results for the specific analyte(s) in all samples since the last acceptable calibration verification.	Problem must be corrected. Results may not be reported without a valid CCV. Flagging is only appropriate in cases where the samples cannot be reanalyzed.

Table 2. Perchlorate Analysis (Method 6850) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Limit of detection verification (LODV) (per batch)	Prior to sample analysis and at the end of the analysis sequence. It can be analyzed after every 10 samples in order to reduce the reanalysis rate.	Within ± 30% of true value.	Correct problem and rerun LODV and all samples analyzed since last successful LODV. If a sample with perchlorate concentration at or between the LOD and RL is bracketed by a failing LODV, it must be reanalyzed. A sample with concentration above the RL can be reported.	If reanalysis cannot be performed, data must be qualified and explained in the case narrative. Apply qualifier and discuss in the case narrative for all results for the specific analyte(s) in all samples since the last acceptable LODV.	Problem must be corrected. Results may not be reported without a valid LODV. Flagging is only appropriate in cases where the samples cannot be reanalyzed. Perchlorate spike concentration is approximately 2 times the limit of detection.
Isotope ratio $^{35}Cl/^{37}Cl$	Every sample, batch QC sample, and standard.	Monitor for either the parent ion at masses 99/101 or the daughter ion at masses 83/85 depending on which ions are quantitated. Theoretical ratio ~ 3.06. Must fall within 2.3 to 3.8.	If criteria are not met, the sample must be rerun. If the sample was not pretreated, the sample should be reextracted using cleanup procedures. If, after cleanup, the ratio still fails, use alternative techniques to confirm presence of perchlorate (i.e., a post spike sample, dilution to reduce any interference, etc.).	Apply qualifier and discuss in the case narrative, if acceptance criteria are not met.	Decision to report data failing ratio check should be thoroughly documented in case narrative.
Internal standard (IS)	Addition of ^{18}O-labeled perchlorate to every sample, batch QC sample, standard, instrument blank, and method blank.	Measured ^{18}O IS area within ± 50% of the value from the average of the IS area counts of the ICAL. RRT of the perchlorate ion must be 1.0 ± 2% (0.98 – 1.02).	Rerun the sample at increasing dilutions until the ± 50% acceptance criteria are met. If criteria cannot be met with dilution, the interference are suspected and the sample must be reprepped using additional pretreatment steps.	Apply qualifier and discuss in the case narrative.	If peak is not within retention time window, presence is not confirmed. Use for quantitation and to ensure identification. Failing internal standard should be thoroughly documented in the case narrative.

Table 2. Perchlorate Analysis (Method 6850) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference check sample (ICS)	One ICS is prepared with every batch of 20 samples and must undergo the same preparation and pretreatment steps as the samples in the batch. It verifies the method performance at the matrix conductivity threshold (MCT). At least one ICS must be analyzed daily.	Within ± 30% of true value.	Correct problem and then reanalyze all samples in that batch. If poor recovery from the cleanup filters is suspected, a different lot of filters must be used to reextract all samples in the batch. If column degradation is suspected, a new column must be calibrated before the samples can be reanalyzed.	Flagging criteria are not appropriate.	Analysis of a standard containing perchlorate at the RL and interfering anions at the concentration determined by the interference threshold study. Monitor recovery of perchlorate and retention time. No samples may be reported that are associated with a failing ICS.
Laboratory reagent blank	Prior to calibration, after samples with overrange concentration of perchlorate, and at the end of the analytical sequence.	No perchlorate detected > ½ RL.	Reanalyze reagent blank (until no carryover is observed) and all samples processed since 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 batch.	Problem must be corrected. Results may not be reported without a valid reagent blank. Flagging is only appropriate in cases where the samples cannot be reanalyzed.
Mass calibration	Instrument must have a valid mass calibration prior to any sample analysis. The mass calibration is updated on an as-needed basis (e.g., QC failures, ion masses show large deviations from known masses, major instrument maintenance is performed, or the instrument is moved).	Mass calibration range must bracket the ion masses of interest without greatly exceeding the range. The most recent mass calibration must be used for an analytical run, and the same mass calibration must be used for all data files in an analytical run. Mass calibration is verified by the tuning standard above.	If the mass calibration fails, recalibrate. If it still fails, consult manufacturer instructions on corrective maintenance.	Flagging criteria are not appropriate.	Problem must be corrected. No samples may be analyzed under a failing mass calibration.
Tuning	Prior to ICAL, initial CCV and after any mass calibration or maintenance is performed.	Mass calibration is verified by acquiring a full scan continuum mass spectrum of a perchlorate stock standard at 100ppb. Perchlorate ions should be within ± 0.3 *m/z* of mass 99 and 101.	Retune instrument. If the tuning will not meet acceptance criteria, an instrument mass calibration must be performed and the tuning redone.	Flagging criteria are not appropriate.	Problem must be corrected. Sample analysis shall not proceed without acceptable tuning.

Table 2. Perchlorate Analysis (Method 6850) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Interference threshold study	At initial setup and when major changes occur in the method's operating procedures (e.g., addition of cleanup procedures, column changes, mobile phase changes).	Measure the threshold of common suppressors (chloride, sulfate, carbonate, bicarbonate) that can be present in the system without affecting the quantitation of perchlorate. The threshold is the concentration of the common suppressors where perchlorate recovery falls outside an 85-115% window.	NA.	Flagging criteria are not appropriate.	This study and site history will determine the concentration at which the ICS suppressors should be set.
Method blank (MB)	One per preparatory batch.	No perchlorate detected > ½ RL and greater than 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, then see criteria in Box D-1. 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. Method blank must undergo the same preparation and pretreatment steps as the samples in the batch.
Laboratory control sample (LCS)	One per preparatory batch. LCS must be spiked at the RL.	Recovery within method requirements, laboratory-generated limits, or 80-120% (whichever is more stringent) to verify calibration and to check method performance.	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 (see full explanation in Appendix G).	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. LCS must undergo the same preparation and pretreatment steps as the samples in the batch.

Table 2. Perchlorate Analysis (Method 6850) (continued)					
QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action	Flagging Criteria	Comments
Matrix spike (MS)	One per preparatory batch per matrix (see Box D-7). The MS must be spiked at the RL.	Recovery within 80-120% or within laboratory generated limits, whichever is more stringent.	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 limits, the data must be evaluated to determine the source of the difference and to determine if there is a matrix effect or analytical error. MS must undergo the same preparation and pretreatment steps as the samples in the batch.
Matrix spike duplicate (MSD) or laboratory duplicate (LD)	One per preparatory batch per matrix (see Box D-7). The MSD must be spiked at the RL.	MSD: Recovery within 80-120% or within laboratory generated limits, whichever is more stringent. MSD or laboratory duplicate: RPD < 15%.	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.
Results reported between DL and LOQ	Positive detections calculated per method.	NA.	NA.	Apply J-flag to all results between DL and LOQ.	

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 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 4, Data Reviewers Checklist (Prior to approving data)
ANALYST DATA REVIEW CHECKLIST

Instrument: Run Date:

Sample Number(s):
Sequence/Batch ID:
Method: Perchlorate 6850

QA/QC Item	Yes	No	NA	Second Level Review
C. Continuing Calibration and Tune Verification Check				
1. Are the Continuing Calibration Verification (CCV) standards analyzed every 10 samples and at the end of the sequence?				
2. Was the tune verification check analyzed at the beginning of the sequence and were the masses 99/101 within 0.3 m/z units?				
3. Are the % differences within 15% difference?				
D. Interference Check and Limit of Detection Verification				
1. Is the ICS within 30% recovery?				
2. Are the LODVs within 30% recovery?				
E. Sample Analysis				
1. Are all sample holding times met?				
2. Are all samples with concentrations > the highest standard used for initial calibration diluted and reanalyzed?				
3. Are 99/101 ratios within 2.3- 3.8?				
4. Are the IS recoveries within 50% of the average of the curve?				
F. QC Samples				
1. Is the Method Blank extracted at the desired frequency(at least every 20 samples) and is its concentration for target analytes less than 1/2 RLs?				
2. Is the LCS extracted per batch(at least every 20 samples) and are the percent recoveries within 80-120%?				
3. Is the Matrix Spike/Matrix Spike Duplicate extracted at the desired frequency and are the percent recoveries within 20% and RPD within 15%?				

G. Show calculation verified against the Element calibration curve below: ___ ___ ___ _____

Comments on any "No" response:

Analyst: _____ Date: _____

Second-Level Review: _____ Date: _____

EMPIRICAL LABORATORIES, LLC
STANDARD OPERATING PROCEDURE

ORGANICS: SOP 327 **REVISION #: 18** **EFFECTIVE DATE: 20100907**

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

APPROVALS:

Lab Director: _____ Date: 9/8/10

Data Quality Manager: _____ Date: 9/8/10

Section Supervisor: _____ Date: 9/8/10

Changes Summary

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.**

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1.0 Identification of the Test Method

1.1 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

2.1 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 Branson ultrasonic bath cooled by a chiller to 15-20°C
- 9.3.6 Balance $\pm$ 0.01 g.
- 9.3.7 Vortex mixer.

9.4 Other Components:

- 9.4.1 Shaker
- 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 - 30 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 Clippers for cutting vegetation

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, CH_3CN - HPLC grade.

Methanol, CH_3OH - HPLC grade.

Calcium chloride, $CaCl_2$ - 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, preparation date, expiration date,and analyst initials. All standards are stored in the refrigerator in the dark at a temperature of 4°C $\pm$ 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.

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 100µL syringe, 100µL of Mix # 1, Mix # 2, and Surrogate (Restek at 1000 µg/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 µg/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 µg/mL. The lowest calibration standard can be extended down to 0.010µg/ml, if required.

10.5.2 Mix # 3 Calibration Stock Solution: Using a 500µL syringe, 500µL of Mix # 3 (Restek PETN & Nitroglycerin, NG, at 1000 µg/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 µg/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 µg/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 µg/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 100µL syringe, 100µL of Combined Stock Solution (Ultra at 1000 µg/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 µg/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 µg/mL.

Using a 500µL syringe, 500µL of Mix #3(Accustandard at 1000 µg/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 µg/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 µg/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 250µL syringe, 200µL of 1,2-dinitrobenzene, (or 1-Chloro-3-Nitrobenzene), (Restek at 1000 µg/mL) is injected into a 100mL volumetric flask containing approximately 95mL acetonitrile and diluted to volume with same to make a 2.0 µg/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.

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 ± 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 it's 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** for acceptance criteria.

12.3 Surrogate - All samples and QC are spiked with the surrogate. See **Table 2** 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** 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** 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** 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** 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 eluent 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** for acceptance criteria and corrective action.

14.0 Procedure

14.1 All waters have a seven-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 prioe 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 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 (+/-1%), weigh the entire sample and record in LIMS then sieve the entire sample with a 10 mesh sieve. Breakup 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.

14.6.2 Collect and weigh any portion unable to pass through the sieve. Record in LIMS.

14.6.3 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 sieve 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 subsampled) 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, Subsampling 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.

14.8 Grinding of Vegetation Samples

14.8.1 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.

14.8.2 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 (2.0 µg/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 a shaker for 16-18 hours.

14.8.3 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.

14.9 Sample Analysis

14.9.1 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).

14.9.2 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 exceedence 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.

14.9.3 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. All calculations are performed as covered in section 8.6 of this SOP. 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 ±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:

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

15.3.2 The mean CF is calculated as follows:

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

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

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

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

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**:**

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

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 µg/L.

B. Nonaqueous Samples**:**

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

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** 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

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

TABLE 1 (DL/LOD/LOQ).

Analyte	MDL/DL	LOD	MRL/LOQ	Units
1,3,5-Trinitrobenzene	0.100	0.200	0.400	mg/Kg
1,3-Dinitrobenzene	0.100	0.200	0.400	mg/Kg
2,4,6-Trinitrophenylmethylnitramine (Tetryl)	0.100	0.200	0.400	mg/Kg
2,4,6-Trinitrotoluene (TNT)	0.100	0.200	0.400	mg/Kg
2,4-Dinitrotoluene (DNT)	0.100	0.200	0.400	mg/Kg
2,6-Dinitrotoluene	0.100	0.200	0.400	mg/Kg
2-Amino-4,6-dinitrotoluene	0.100	0.200	0.400	mg/Kg
2-Nitrotoluene (ONT)	0.100	0.200	0.400	mg/Kg
3,5-Dinitroaniline	0.100	0.200	0.400	mg/Kg
3-Nitrotoluene	0.100	0.200	0.400	mg/Kg
4-Amino-2,6-dinitrotoluene	0.100	0.200	0.400	mg/Kg
4-Nitrotoluene (PNT)	0.100	0.200	0.400	mg/Kg
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	0.100	0.200	0.400	mg/Kg
Nitrobenzene	0.100	0.200	0.400	mg/Kg
Nitroglycerin	0.250	0.500	1.00	mg/Kg
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	0.100	0.200	0.400	mg/Kg
PETN	0.250	0.500	1.00	mg/Kg
Nitroguanidine	0.025	0.05	0.1	mg/Kg
1,3,5-Trinitrobenzene	0.0800	0.160	0.320	ug/L
1,3-Dinitrobenzene	0.0800	0.160	0.320	ug/L
2,4,6-Trinitrophenylmethylnitramine (Tetryl)	0.0800	0.160	0.320	ug/L
2,4,6-Trinitrotoluene (TNT)	0.0800	0.160	0.320	ug/L
2,4-Dinitrotoluene (DNT)	0.0800	0.160	0.320	ug/L
2,6-Dinitrotoluene	0.0800	0.160	0.320	ug/L
2-Amino-4,6-dinitrotoluene	0.0800	0.160	0.320	ug/L
2-Nitrotoluene (ONT)	0.0800	0.160	0.320	ug/L
3,5-Dinitroaniline	0.0800	0.160	0.320	ug/L
3-Nitrotoluene	0.0800	0.160	0.320	ug/L
4-Amino-2,6-dinitrotoluene	0.0800	0.160	0.320	ug/L
4-Nitrotoluene (PNT)	0.0800	0.160	0.320	ug/L
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	0.0800	0.160	0.320	ug/L
Nitrobenzene	0.0800	0.160	0.320	ug/L
Nitroglycerin	0.200	0.400	0.800	ug/L
Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	0.0800	0.160	0.320	ug/L
PETN	0.200	0.400	0.800	ug/L
Nitroguanidine	2.5	5	10	ug/L

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 ≤ 20% Option 2: linear least squares regression: r ≥ 0.995 Option 3: non-linear regression: coefficient of determination (COD) r2 ≥ 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. HPLC methods: All project analytes within ± 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. HPLC methods: All project analytes within ± 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 > ½ 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 ≤ 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 ≤ 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 1. 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.	

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

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)					
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 ≤ 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 ± 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 ± 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 > ½ RL and greater than 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.

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

Table F-3. Nitroaromatics, Nitramines, and Nitrate Esters Analysis by High-Performance Liquid Chromatography (Method 8330B) (continued)					
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 ≤ 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 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 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 ½ 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: _____ _____

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: _____ _____

CERTIFICATE OF ACCREDITATION

ANSI-ASQ National Accreditation Board/ACLASS
500 Montgomery Street, Suite 625, Alexandria, VA 22314, 877-344-3044

This is to certify that

Air Toxics, Ltd.
180 Blue Ravine Rd. Suite B
Folsom, CA 95630

has been assessed by ACLASS
and meets the requirements of

DoD-ELAP

while demonstrating technical competence in the field(s) of

TESTING

Refer to the accompanying Scope(s) of Accreditation for information regarding the types of tests to which this accreditation applies.

ADE-1451
Certificate Number

ACLASS Approval

Certificate Valid: 04/27/2010-04/27/2012
Version No. 001

SCOPE OF DoD-ELAP ACCREDITATION

Air Toxics, Ltd.

180 Blue Ravine Rd. Suite B, Folsom, CA 95630
Melanie Levesque Phone: 916-985-1000

TESTING

Valid to: April 27, 2012 Certificate Number: ADE- 1451

I. Environmental

MATRIX	SPECIFIC TEST or GROUP of ANALYTES	SPECIFICATION OR STANDARD METHOD (all SW846 unless specified)	* KEY EQUIPMENT OR TECHNOLOGY USED
Air and Emissions	BTEX / TPH	Modified EPA TO-3	GC/PID/FID
Air and Emissions	VOCs	Modified EPA TO-14A	GC/MS
Air and Emissions	VOCs	Modified EPA TO-15	GC/MS
Air and Emissions	SVOCs and VOCs	Modified EPA TO-17	GC/MS
Air and Emissions	Natural Gases and NMOC	Modified ASTM D-1945	GC/FID/dual TCD

Notes:

1. ** = As Applicable*
2. *This scope is part of and must be included with the Certificate of Accreditation No. ADE- 1451*

Vice President

DoD ELAP -- PT Performance Summary Review				Air ONLY	
Lab Name :	Air Toxics				
City/State :	Folsom, CA				
PT Provider Used :	ERA				
PartName	PartNumber	NELACCode	AnalyteName	EPAmethod#	Analyte Approved
Volatiles	PT-VOA-AIR	4375	Benzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4395	Bromodichloromethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4400	Bromoform	TO-15	Approved
Volatiles	PT-VOA-AIR	4950	Bromomethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4410	2-Butanone (MEK)	TO-15	Approved
Volatiles	PT-VOA-AIR	5000	tert-Butyl methyl ether (MTBE)	TO-15	Approved
Volatiles	PT-VOA-AIR	4455	Carbon tetrachloride	TO-15	Approved
Volatiles	PT-VOA-AIR	4475	Chlorobenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4575	Chlorodibromomethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4485	Chloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4505	Chloroform	TO-15	Approved
Volatiles	PT-VOA-AIR	4960	Chloromethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4555	Cyclohexane	TO-15	Approved
Volatiles	PT-VOA-AIR	4585	1,2-Dibromoethane (EDB)	TO-15	Approved
Volatiles	PT-VOA-AIR	4610	1,2-Dichlorobenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4620	1,4-Dichlorobenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4625	Dichlorodifluoromethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4630	1,1-Dichloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4635	1,2-Dichloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4640	1,1-Dichloroethene	TO-15	Approved
Volatiles	PT-VOA-AIR	4645	cis-1,2-Dichloroethene	TO-15	Approved
Volatiles	PT-VOA-AIR	4655	1,2-Dichloropropane	TO-15	Approved
Volatiles	PT-VOA-AIR	4680	cis-1,3-Dichloropropene	TO-15	Approved
Volatiles	PT-VOA-AIR	4685	trans-1,3-Dichloropropene	TO-15	Approved
Volatiles	PT-VOA-AIR	4695	1,2-Dichlorotetrafluoroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	4765	Ethylbenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	4768	p-Ethyltoluene	TO-15	Approved
Volatiles	PT-VOA-AIR	4825	n-Heptane	TO-15	Approved
Volatiles	PT-VOA-AIR	4855	n-Hexane	TO-15	Approved
Volatiles	PT-VOA-AIR	4860	2-Hexanone	TO-15	Approved
Volatiles	PT-VOA-AIR	4995	4-Methyl-2-pentanone (MIBK)	TO-15	Approved
Volatiles	PT-VOA-AIR	5067	Propylene	TO-15	Approved
Volatiles	PT-VOA-AIR	5110	1,1,2,2-Tetrachloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5115	Tetrachloroethylene	TO-15	Approved
Volatiles	PT-VOA-AIR	5140	Toluene	TO-15	Approved
Volatiles	PT-VOA-AIR	5160	1,1,1-Trichloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5165	1,1,2-Trichloroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5175	Trichlorofluoromethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5195	Trichlorotrifluoroethane	TO-15	Approved
Volatiles	PT-VOA-AIR	5210	1,2,4-Trimethylbenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	5215	1,3,5-Trimethylbenzene	TO-15	Approved
Volatiles	PT-VOA-AIR	5235	Vinyl chloride	TO-15	Approved
Volatiles	PT-VOA-AIR	5260	Xylenes, total	TO-15	Approved

LABORATORY ACCREDITATION BUREAU

Certificate of Accreditation

ISO/IEC 17025:2005 ***Certificate Number L2226***

Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228

has met the requirements set forth in L-A-B's policies and procedures, all requirements of ISO/IEC 17025:2005 "General Requirements for the competence of Testing and Calibration Laboratories" and the U.S. Department of Defense Environmental Laboratory Accreditation Program (DoD ELAP).*

The accredited lab has demonstrated technical competence to a defined "Scope of Accreditation" and the operation of a laboratory quality management system (refer to joint ISO-ILAC-IAF Communiqué dated 8 January 2009).

Accreditation Granted through: November 30, 2012

R. Douglas Leonard, Jr., Managing Director
Laboratory Accreditation Bureau
Presented the 30th of November 2009

*See the laboratory's Scope of Accreditation for details of the DoD ELAP requirements
Laboratory Accreditation Bureau is found to be in compliance with ISO/IEC 17011:2004 and recognized by ILAC (International Laboratory Accreditation Cooperation) and NACLA (National Cooperation for Laboratory Accreditation).

Scope of Accreditation
For
Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228
Marcia K. McGinnity
1-877-345-1113

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Empirical Laboratories, LLC to perform the following tests:

Accreditation granted through: **November 30, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Di-isopropyl ether

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	ETBE
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	t-Butyl alcohol
GC/MS	EPA 8260B	tert-Amyl methyl ether
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)
HPLC/UV	EPA 8330A/B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A/B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A/B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	3-Nitrotoluene

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A/B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A/B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Nitroguanidine
HPLC/UV	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	EPA 6850	Perchlorate
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAA	EPA 6010B/C	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
IC	EPA 300.0	Chloride
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 300.0	Sulfate
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
Titration	SM 2320B $20^{th}/21^{st}$ edition	Alkalinity
Colorimetric	SM 4500 B, G, $20^{th}/21^{st}$ edition	Ammonia
UV/Vis	EPA 7196A	Hexavalent Chromium
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, $20^{th}/21^{st}$ edition	Sulfide
UV/Vis	SM 4500 P B5, E, $20^{th}/21^{st}$ edition	Total Phosphorus (as P)

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/Vis	SM 4500 PE, 20th/21st edition	Ortho-Phosphate (as P)
TOC	9060A/SM5310C, 20th/21st edition	Total Organic Carbon
Gravimetric	SM 2540C, 20th/21st edition	TDS
Colorimetric	EPA 9012A/B	Cyanide
Physical	EPA 1010A	Ignitability
Physical	EPA 9095B	Paint Filter
Probe	EPA 9040B/C	pH
Preparation	**Method**	**Type**
Preparation	EPA 1311	TCLP
Preparation	EPA 3005A	Metals digestion
Preparation	EPA 3010A	Metals digestion
Preparation	EPA 3510C	Organics Liquid Extraction
Preparation	EPA 5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)
HPLC/UV	EPA 8330A	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene (ONT)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A	Nitroguanidine
HPLC/UV	EPA 8330A	PETN
HPLC/UV	EPA 8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330B	3-Nitrotoluene
HPLC/UV	EPA 8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330B	Nitrobenzene
HPLC/UV	EPA 8330B	Nitroguanidine
HPLC/UV	EPA 8330B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
HPLC/MS	EPA 6850	Perchlorate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese
CVAA	EPA 7471A/B	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
UV/Vis	EPA 7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 9012A/B	Cyanide

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	EPA 9034	Sulfide
Probe	EPA 9045C/D	pH
Preparation	**Method**	**Type**
Preparation	EPA 1311	TCLP
Preparation	EPA 1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	EPA 3050B	Metals Digestion
Preparation	EPA 3546	Organics Microwave Extraction
Preparation	EPA 3550B/C	Organics Sonication
Preparation	SM 2540B 20th/21st edition	Percent Solids (Percent Moisture)
Preparation	EPA 5035 /A	Purge and Trap Solid

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____ Date: January 25, 2011

R. Douglas Leonard
Chief Technical Officer

Issued: 11/30/09 Revised: 2/9/10 Revised: 3/31/10 Revised: 10/8/10 Revised: 1/25/11

Scope of Accreditation For Empirical Laboratories, LLC

621 Mainstream Drive, Suite 270
Nashville, TN 37228
Marcia K. McGinnity
1-877-345-1113

In recognition of a successful assessment to ISO/IEC 17025:2005 and the requirements of the DoD Environmental Laboratory Accreditation Program (DoD ELAP) as detailed in the DoD Quality Systems Manual for Environmental Laboratories (DoD QSM v4.1) based on the National Environmental Laboratory Accreditation Conference Chapter 5 Quality Systems Standard (NELAC Voted Revision June 5, 2003), accreditation is granted to Empirical Laboratories, LLC to perform the following tests:

Accreditation granted through: **November 30, 2012**

Testing - Environmental

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	1-Chlorohexane
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chloroethyl vinyl ether
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Di-isopropyl ether

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	ETBE
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	t-Butyl alcohol
GC/MS	EPA 8260B	tert-Amyl methyl ether
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Tetrahydrofuran
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)
GC/MS	EPA 8270C/D	7,12-Dimethylbenz(a)anthracene

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor

Non-Potable Water		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)
HPLC/UV	EPA 8330A/B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A/B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A/B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A/B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A/B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	3-Nitrotoluene

Non-Potable Water		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A/B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A/B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A/B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A/B	Nitrobenzene
HPLC/UV	EPA 8330A/B	Nitroglycerin
HPLC/UV	EPA 8330A/B	Nitroguanidine
HPLC/UV	EPA 8330A/B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A/B	3,5-Dinitroaniline
HPLC/UV	EPA 8330A/B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
GC/FID	RSK-175	Methane
GC/FID	RSK-175	Ethane
GC/FID	RSK-175	Ethene
GC/ECD	EPA 8011	1,2-Dibromoethane (EDB)
GC/ECD	EPA 8011	1,2-Dibromo-3-chloropropane (DBCP)
HPLC/MS	EPA 6850	Perchlorate
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese

Non-Potable Water		
Technology	**Method**	**Analyte**
CVAA	EPA 6010B/C	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
IC	EPA 300.0	Chloride
IC	EPA 300.0	Fluoride
IC	EPA 300.0	Nitrate
IC	EPA 300.0	Nitrite
IC	EPA 300.0	Sulfate
IC	EPA 9056A	Chloride
IC	EPA 9056A	Fluoride
IC	EPA 9056A	Nitrate
IC	EPA 9056A	Nitrite
IC	EPA 9056A	Sulfate
Titration	SM 2320B 20th/21st edition	Alkalinity
Colorimetric	SM 4500 B, G, 20th/21st edition	Ammonia
UV/Vis	EPA 7196A	Hexavalent Chromium
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 353.2	Nitrate/Nitrite
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	SM 4500 S-2CF, 20th/21st edition	Sulfide
UV/Vis	SM 4500 P B5, E, 20th/21st edition	Total Phosphorus (as P)

Non-Potable Water		
Technology	**Method**	**Analyte**
UV/Vis	SM 4500 PE, 20th/21st edition	Ortho-Phosphate (as P)
TOC	9060A/SM5310C, 20th/21st edition	Total Organic Carbon
Gravimetric	SM 2540C, 20th/21st edition	TDS
Colorimetric	EPA 9012A/B	Cyanide
Physical	EPA 1010A	Ignitability
Physical	EPA 9095B	Paint Filter
Probe	EPA 9040B/C	pH
Preparation	**Method**	**Type**
Preparation	EPA 1311	TCLP
Preparation	EPA 3005A	Metals digestion
Preparation	EPA 3010A	Metals digestion
Preparation	EPA 3510C	Organics Liquid Extraction
Preparation	EPA 5030A/B	Purge and Trap Water

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,1,1-Trichloroethane (1,1,1-TCA)
GC/MS	EPA 8260B	1,1,1,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2,2-Tetrachloroethane
GC/MS	EPA 8260B	1,1,2-Trichloro-1,2,2-trifluoroethane (CFC-113; Freon 113)
GC/MS	EPA 8260B	1,1,2-Trichloroethane
GC/MS	EPA 8260B	1,1-Dichloroethane (1,1-DCA)
GC/MS	EPA 8260B	1,1-Dichloroethene (1,1-DCE)
GC/MS	EPA 8260B	1,1-Dichloropropene
GC/MS	EPA 8260B	1,2,3-Trichlorobenzene
GC/MS	EPA 8260B	1,2,3-Trichloropropane
GC/MS	EPA 8260B	1,2,4-Trichlorobenzene
GC/MS	EPA 8260B	1,2,4-Trimethylbenzene
GC/MS	EPA 8260B	1,2-Dibromo-3-chloropropane (DBCP)
GC/MS	EPA 8260B	1,2-Dibromoethane (EDB)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	1,2-Dichlorobenzene
GC/MS	EPA 8260B	1,2-Dichloroethane (EDC)
GC/MS	EPA 8260B	1,2-Dichloropropane
GC/MS	EPA 8260B	1,3,5-Trimethylbenzene
GC/MS	EPA 8260B	1,3-Dichlorobenzene
GC/MS	EPA 8260B	1,3-Dichloropropane
GC/MS	EPA 8260B	1,4-Dichlorobenzene
GC/MS	EPA 8260B	2,2-Dichloropropane
GC/MS	EPA 8260B	2-Butanone (Methyl ethyl ketone; MEK)
GC/MS	EPA 8260B	2-Chlorotoluene
GC/MS	EPA 8260B	2-Hexanone (Methyl butyl ketone; MBK)
GC/MS	EPA 8260B	4-Chlorotoluene
GC/MS	EPA 8260B	4-Methyl-2-pentanone (Methyl isobutyl ketone; MIBK)
GC/MS	EPA 8260B	Acetone
GC/MS	EPA 8260B	Acrolein
GC/MS	EPA 8260B	Acrylonitrile
GC/MS	EPA 8260B	Benzene
GC/MS	EPA 8260B	Bromobenzene
GC/MS	EPA 8260B	Bromochloromethane
GC/MS	EPA 8260B	Bromodichloromethane
GC/MS	EPA 8260B	Bromoform
GC/MS	EPA 8260B	Bromomethane
GC/MS	EPA 8260B	Carbon Disulfide
GC/MS	EPA 8260B	Carbon Tetrachloride
GC/MS	EPA 8260B	Chlorobenzene
GC/MS	EPA 8260B	Chloroethane
GC/MS	EPA 8260B	Chloroform
GC/MS	EPA 8260B	Chloromethane
GC/MS	EPA 8260B	cis-1,2-Dichloroethene (cis-1,2-DCE)
GC/MS	EPA 8260B	cis-1,3-Dichloropropene
GC/MS	EPA 8260B	Cyclohexane
GC/MS	EPA 8260B	Dibromochloromethane

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8260B	Dibromomethane
GC/MS	EPA 8260B	Dichlorodifluoromethane (CFC-12)
GC/MS	EPA 8260B	Ethyl methacrylate
GC/MS	EPA 8260B	Ethylbenzene
GC/MS	EPA 8260B	Hexachlorobutadiene
GC/MS	EPA 8260B	Iodomethane
GC/MS	EPA 8260B	Isopropylbenzene (Cumene)
GC/MS	EPA 8260B	Methyl Acetate
GC/MS	EPA 8260B	Methyl methacrylate
GC/MS	EPA 8260B	Methyl Tertiary Butyl Ether (MTBE)
GC/MS	EPA 8260B	Methylcyclohexane
GC/MS	EPA 8260B	Methylene Chloride, or Dichloromethane
GC/MS	EPA 8260B	Naphthalene
GC/MS	EPA 8260B	n-Butylbenzene
GC/MS	EPA 8260B	n-Propylbenzene
GC/MS	EPA 8260B	p-Isopropyltoluene
GC/MS	EPA 8260B	sec-Butylbenzene
GC/MS	EPA 8260B	Styrene
GC/MS	EPA 8260B	tert-Butylbenzene
GC/MS	EPA 8260B	Tetrachloroethene (PCE; PERC)
GC/MS	EPA 8260B	Toluene
GC/MS	EPA 8260B	trans-1,2-Dichloroethene (trans-1,2-DCE)
GC/MS	EPA 8260B	trans-1,3-Dichloropropene
GC/MS	EPA 8260B	Trichloroethene (TCE)
GC/MS	EPA 8260B	Trichlorofluoromethane (CFC-11)
GC/MS	EPA 8260B	Vinyl acetate
GC/MS	EPA 8260B	Vinyl Chloride (VC)
GC/MS	EPA 8260B	Xylenes (Total)
GC/MS	EPA 8270C/D	Bis(2-chloroisopropyl)ether, or 2,2'-oxybis (1-Chloropropane)
GC/MS	EPA 8270C/D	1,1'-Biphenyl
GC/MS	EPA 8270C/D	1,2,4,5-Tetrachlorobenzene
GC/MS	EPA 8270C/D	1,2,4-Trichlorobenzene

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	1,2-Dichlorobenzene
GC/MS	EPA 8270C/D	1,2-Diphenylhydrazine
GC/MS	EPA 8270C/D	1,3-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dichlorobenzene
GC/MS	EPA 8270C/D	1,4-Dioxane
GC/MS	EPA 8270C/D	1-Methylnaphthalene
GC/MS	EPA 8270C/D	2,3,4,6-Tetrachlorophenol
GC/MS	EPA 8270C/D	2,4,5-Trichlorophenol
GC/MS	EPA 8270C/D	2,4,6-Trichlorophenol (TCP)
GC/MS	EPA 8270C/D	2,4-Dichlorophenol (DCP)
GC/MS	EPA 8270C/D	2,4-Dimethylphenol
GC/MS	EPA 8270C/D	2,4-Dinitrophenol
GC/MS	EPA 8270C/D	2,4-Dinitrotoluene (DNT)
GC/MS	EPA 8270C/D	2,6-Dichlorophenol
GC/MS	EPA 8270C/D	2,6-Dinitrotoluene
GC/MS	EPA 8270C/D	2-Chloronaphthalene
GC/MS	EPA 8270C/D	2-Chlorophenol
GC/MS	EPA 8270C/D	2-Methylnaphthalene
GC/MS	EPA 8270C/D	2-Methylphenol (o-Cresol)
GC/MS	EPA 8270C/D	2-Nitroaniline
GC/MS	EPA 8270C/D	2-Nitrophenol (ONP)
GC/MS	EPA 8270C/D	3,3'-Dichlorobenzidine (DCB)
GC/MS	EPA 8270C/D	3-Methylphenol
GC/MS	EPA 8270C/D	3-Nitroaniline
GC/MS	EPA 8270C/D	4,6-Dinitro-2-methylphenol (DNOC)
GC/MS	EPA 8270C/D	4-Bromophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Chloro-3-methylphenol
GC/MS	EPA 8270C/D	4-Chloroaniline
GC/MS	EPA 8270C/D	4-Chlorophenyl phenyl ether
GC/MS	EPA 8270C/D	4-Methylphenol (p-Cresol)
GC/MS	EPA 8270C/D	4-Nitroaniline (PNA)
GC/MS	EPA 8270C/D	4-Nitrophenol (PNP)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Acenaphthene
GC/MS	EPA 8270C/D	Acenaphthylene
GC/MS	EPA 8270C/D	Acetaphenone
GC/MS	EPA 8270C/D	Aniline
GC/MS	EPA 8270C/D	Anthracene
GC/MS	EPA 8270C/D	Atrazine
GC/MS	EPA 8270C/D	Benzaldehyde
GC/MS	EPA 8270C/D	Benzidine
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)anthracene
GC/MS	EPA 8270C/D	Benzo(a)pyrene
GC/MS	EPA 8270C/D	Benzo(b)fluoranthene
GC/MS	EPA 8270C/D	Benzo(g,h,i)perylene
GC/MS	EPA 8270C/D	Benzo(k)fluoranthene
GC/MS	EPA 8270C/D	Benzoic Acid
GC/MS	EPA 8270C/D	Benzyl alcohol
GC/MS	EPA 8270C/D	bis(2-Chloroethoxy)methane
GC/MS	EPA 8270C/D	bis(2-Chloroethyl)ether (BCEE)
GC/MS	EPA 8270C/D	bis(2-Ethylhexyl)phthalate (BEHP)
GC/MS	EPA 8270C/D	Butyl benzyl phthalate (BBP)
GC/MS	EPA 8270C/D	Caprolactam
GC/MS	EPA 8270C/D	Carbazole
GC/MS	EPA 8270C/D	Chrysene
GC/MS	EPA 8270C/D	Dibenz(a,h)anthracene
GC/MS	EPA 8270C/D	Dibenzofuran (DBF)
GC/MS	EPA 8270C/D	Diethyl phthalate (DEP)
GC/MS	EPA 8270C/D	Dimethyl phthalate (DMP)
GC/MS	EPA 8270C/D	Di-n-butyl phthalate (DBP)
GC/MS	EPA 8270C/D	Di-n-octyl phthalate (DNOP)
GC/MS	EPA 8270C/D	Fluoranthene
GC/MS	EPA 8270C/D	Fluorene
GC/MS	EPA 8270C/D	Hexachlorobenzene (HCB)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/MS	EPA 8270C/D	Hexachlorobutadiene (HCBD)
GC/MS	EPA 8270C/D	Hexachlorocyclopentadiene (HCCPD)
GC/MS	EPA 8270C/D	Hexachloroethane (HCE)
GC/MS	EPA 8270C/D	Indeno(1,2,3-cd)pyrene
GC/MS	EPA 8270C/D	Isophorone
GC/MS	EPA 8270C/D	Naphthalene
GC/MS	EPA 8270C/D	Nitrobenzene
GC/MS	EPA 8270C/D	N-Nitrosodimethylamine
GC/MS	EPA 8270C/D	N-Nitroso-di-n-propylamine (NDPA)
GC/MS	EPA 8270C/D	N-nitrosodiphenylamine (NDPHA)
GC/MS	EPA 8270C/D	Pentachlorophenol
GC/MS	EPA 8270C/D	Phenanthrene
GC/MS	EPA 8270C/D	Phenol
GC/MS	EPA 8270C/D	Pyrene
GC/MS	EPA 8270C/D	Pyridine
GC/ECD	EPA 8081A/B	4,4'-DDD
GC/ECD	EPA 8081A/B	4,4'-DDE
GC/ECD	EPA 8081A/B	4,4'-DDT
GC/ECD	EPA 8081A/B	Aldrin
GC/ECD	EPA 8081A/B	alpha-BHC (alpha-HCH)
GC/ECD	EPA 8081A/B	alpha-Chlordane
GC/ECD	EPA 8081A/B	beta-BHC (beta-HCH)
GC/ECD	EPA 8081A/B	delta-BHC (delta-HCH)
GC/ECD	EPA 8081A/B	Chlordane
GC/ECD	EPA 8081A/B	Dieldrin
GC/ECD	EPA 8081A/B	Endosulfan I
GC/ECD	EPA 8081A/B	Endosulfan II
GC/ECD	EPA 8081A/B	Endosulfan sulfate
GC/ECD	EPA 8081A/B	Endrin
GC/ECD	EPA 8081A/B	Endrin aldehyde
GC/ECD	EPA 8081A/B	Endrin ketone
GC/ECD	EPA 8081A/B	gamma-BHC (Lindane; gamma-HCH)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
GC/ECD	EPA 8081A/B	gamma-Chlordane
GC/ECD	EPA 8081A/B	Heptachlor
GC/ECD	EPA 8081A/B	Heptachlor epoxide
GC/ECD	EPA 8081A/B	Methoxychlor
GC/ECD	EPA 8081A/B	Toxaphene
GC/ECD	EPA 8082 /A	Aroclor-1016
GC/ECD	EPA 8082 /A	Aroclor-1221
GC/ECD	EPA 8082 /A	Aroclor-1232
GC/ECD	EPA 8082 /A	Aroclor-1242
GC/ECD	EPA 8082 /A	Aroclor-1248
GC/ECD	EPA 8082 /A	Aroclor-1254
GC/ECD	EPA 8082 /A	Aroclor-1260
GC/ECD	EPA 8082 /A	Aroclor-1262
GC/ECD	EPA 8082 /A	Aroclor-1268
GC/ECD	EPA 8151A	2,4,5-T
GC/ECD	EPA 8151A	2,4,5-TP (Silvex)
GC/ECD	EPA 8151A	2,4-D
GC/ECD	EPA 8151A	2,4-DB
GC/ECD	EPA 8151A	Dalapon
GC/ECD	EPA 8151A	Dicamba
GC/ECD	EPA 8151A	Dichlorprop
GC/ECD	EPA 8151A	Dinoseb
GC/ECD	EPA 8151A	MCPA
GC/ECD	EPA 8151A	MCPP (Mecoprop)
HPLC/UV	EPA 8330A	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330A	1,3-Dinitrobenzene
HPLC/UV	EPA 8330A	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330A	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330A	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330A	2,6-Dinitrotoluene
HPLC/UV	EPA 8330A	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330A	2-Nitrotoluene (ONT)

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
HPLC/UV	EPA 8330A	3-Nitrotoluene
HPLC/UV	EPA 8330A	3,5-Dinitroaniline
HPLC/UV	EPA 8330A	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330A	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330A	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330A	Nitroglycerin
HPLC/UV	EPA 8330A	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330A	Nitrobenzene
HPLC/UV	EPA 8330A	Nitroguanidine
HPLC/UV	EPA 8330A	PETN
HPLC/UV	EPA 8330B	1,3,5-Trinitrobenzene
HPLC/UV	EPA 8330B	1,3-Dinitrobenzene
HPLC/UV	EPA 8330B	2,4,6-Trinitrophenylmethylnitramine (Tetryl)
HPLC/UV	EPA 8330B	2,4,6-Trinitrotoluene (TNT)
HPLC/UV	EPA 8330B	2,4-Dinitrotoluene (DNT)
HPLC/UV	EPA 8330B	2,6-Dinitrotoluene
HPLC/UV	EPA 8330B	2-Amino-4,6-dinitrotoluene
HPLC/UV	EPA 8330B	2-Nitrotoluene (ONT)
HPLC/UV	EPA 8330B	3-Nitrotoluene
HPLC/UV	EPA 8330B	3,5-Dinitroaniline
HPLC/UV	EPA 8330B	4-Amino-2,6-dinitrotoluene
HPLC/UV	EPA 8330B	4-Nitrotoluene (PNT)
HPLC/UV	EPA 8330B	Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)
HPLC/UV	EPA 8330B	Nitroglycerin
HPLC/UV	EPA 8330B	Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)
HPLC/UV	EPA 8330B	Nitrobenzene
HPLC/UV	EPA 8330B	Nitroguanidine
HPLC/UV	EPA 8330B	PETN
GC/FID	FLPRO	Petroleum Range Organics
GC/FID	EPA 8015B	TPH DRO
GC/FID	EPA 8015B	TPH GRO
HPLC/MS	EPA 6850	Perchlorate

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
ICP	EPA 6010B/C	Aluminum
ICP	EPA 6010B/C	Antimony
ICP	EPA 6010B/C	Arsenic
ICP	EPA 6010B/C	Barium
ICP	EPA 6010B/C	Beryllium
ICP	EPA 6010B/C	Boron
ICP	EPA 6010B/C	Cadmium
ICP	EPA 6010B/C	Calcium
ICP	EPA 6010B/C	Chromium, total
ICP	EPA 6010B/C	Cobalt
ICP	EPA 6010B/C	Copper
ICP	EPA 6010B/C	Iron
ICP	EPA 6010B/C	Lead
ICP	EPA 6010B/C	Magnesium
ICP	EPA 6010B/C	Manganese
CVAA	EPA 7471A/B	Mercury
ICP	EPA 6010B/C	Molybdenum
ICP	EPA 6010B/C	Nickel
ICP	EPA 6010B/C	Potassium
ICP	EPA 6010B/C	Selenium
ICP	EPA 6010B/C	Silver
ICP	EPA 6010B/C	Sodium
ICP	EPA 6010B/C	Strontium
ICP	EPA 6010B/C	Tin
ICP	EPA 6010B/C	Titanium
ICP	EPA 6010B/C	Thallium
ICP	EPA 6010B/C	Vanadium
ICP	EPA 6010B/C	Zinc
UV/Vis	EPA 7196A	Hexavalent Chromium
TOC	Lloyd Kahn	Total Organic Carbon
Colorimetric	EPA 353.2	Nitrocellulose
Colorimetric	EPA 9012A/B	Cyanide

Solid and Chemical Materials		
Technology	**Method**	**Analyte**
Titration	Chap.7, Sect. 7.3.4 Mod.	Reactive Sulfide
Titration	EPA 9034	Sulfide
Probe	EPA 9045C/D	pH
Preparation	**Method**	**Type**
Preparation	EPA 1311	TCLP
Preparation	EPA 1312	SPLP
Preparation	NJ Modified 3060A	Hexavalent Chromium
Preparation	EPA 3050B	Metals Digestion
Preparation	EPA 3546	Organics Microwave Extraction
Preparation	EPA 3550B/C	Organics Sonication
Preparation	SM 2540B 20th/21st edition	Percent Solids (Percent Moisture)
Preparation	EPA 5035 /A	Purge and Trap Solid

Notes:

1) This laboratory offers commercial testing service.

Approved By: _____

R. Douglas Leonard
Chief Technical Officer

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