SITE-SPECIFIC QUALITY ASSURANCE PROJECT PLAN: PHASE II ENVIRONMENTAL SITE ASSESSMENT OF THE URBAN RESERVE

SMS # 94-1585

BURLINGTON, VERMONT

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STONE ENVIRONMENTAL INC

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1. FORM A: TITLE AND APPROVAL PAGE

SSQAPP Site-Specific Quality Assurance Project Plan: Phase II Environmental Site Assessment of the Urban Reserve, Burlington, Vermont

Document title

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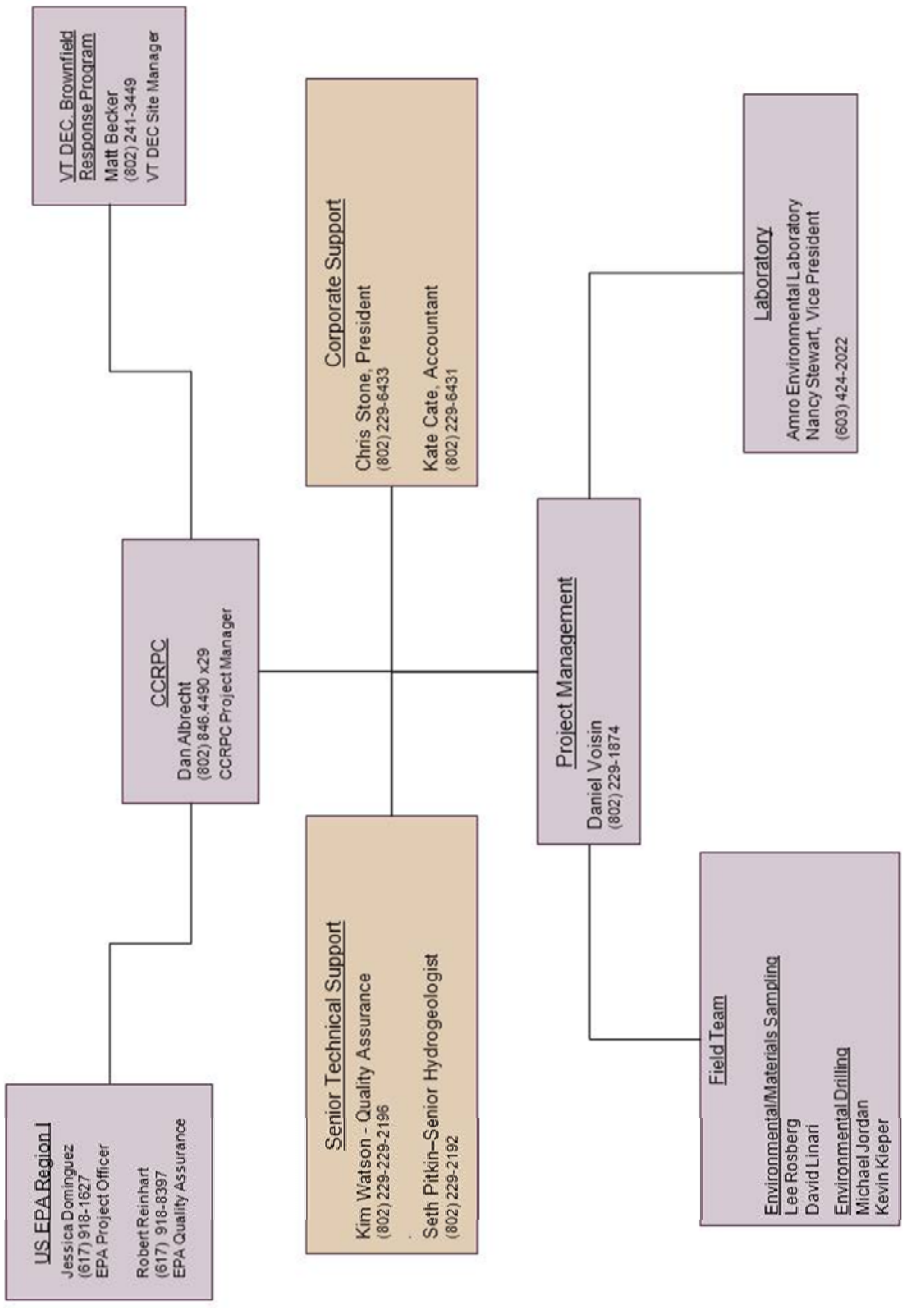
15/February/2013

Day/month/year

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2. FORM B: PROJECT ORGANIZATION AND RESPONSIBILITIES

Figure 1: Project Organizational Chart



3. FORM C: PROBLEM DEFINITION

This Site-Specific Quality Assurance Project Plan (SSQAPP) along with the Brownfields Assessment (BA) Generic QAPP on file with the Vermont Department of Environmental Conservation (VT DEC) and United States Environmental Protection Agency (US EPA) Region I (RFA#07166) constitute the work plan and field and analytical quality assurance plan for a Phase II Environmental Site Assessment (ESA) at the 40-acre lakefront property owned by the City of Burlington known as the Urban Reserve (Site), in Burlington, Vermont (the Site or Property; Figure 2). This SSQAPP was prepared by Stone Environmental, Inc. (Stone) for the Chittenden County Regional Planning Commission (CCRPC) with funding provided through a Petroleum Assessment Grant from the US EPA.

Although currently the Site is largely undeveloped, the Site formerly was used for various industrial purposes, including lumber yards, rail transport, and bulk petroleum storage. Environmental investigation of the Site began in 1986 and extends to present. On November 5, 2012, Stone presented the findings of a Data Gap Analysis and preliminary Conceptual Site Model (CSM) to CCRPC, the Vermont Department of Environmental Conservation (VTDEC), and the City of Burlington's Community and Economic Development Office (CEDO). Data gaps or limitations identified in the 2012 Data Gap Analysis are presented in Table 3-1, below.

Table 3-1: Summary of Data Gaps

Media of Interest	Data Gap
Soil	1. There are insufficient data on the identity, concentration and distribution of polycyclic aromatic hydrocarbons (PAH) to adequately assess current and future risk of exposure at the Site.
	2. There have been no analyses for herbicides, PAHs, heavy metals, or polychlorinated biphenyls (PCBs) as Aroclors along the bike path to assess future risk of exposure if the soils beneath the paved area are exposed.
	3. There are no analytical data which characterize imported fill materials.
	4. There are no shallow chlorinated volatile organic compound (VOC) data at the Tidewater parcel where a previous investigation found 1,1,2,2-tetrachloroethane (1,1,2,2-TCA) at 14 ft bgs.
Groundwater	1. There are an insufficient number of groundwater sampling locations to reasonably characterize impact to groundwater from contaminants of concern (COCs) across the Site.
	2. There are insufficient groundwater quality data to evaluate whether contaminant concentrations are stable, increasing, or decreasing over time, and if natural attenuation processes are occurring at an acceptable rate.
	3. There has been no assessment of the deep overburden aquifer for chlorinated VOCs.
	4. The risk of impact to Lake Champlain in the absence of the retaining wall is unclear.
	5. Previous groundwater results may be biased (low for VOCs; high for metals) due to sampling methodology.
Soil Gas	1. The potential impact to any future occupants of inhabited structures from vapor intrusion of contaminated soil gas is unknown. Previous investigations of soil gas contamination were limited in their spatial coverage and analytical suite (BTEX/TPH only).

Redevelopment plans for the Site are currently being established. Near term goals for the Site include improvements to the Burlington Bike Path, which traverses the Site and is an actively used recreational destination for the City of Burlington. In the absence of a redevelopment plan, and due to current funding limitations, this portion of the Phase II ESA will be focused on near term objectives, specifically risk to workers reconstructing the bike path and users of the bike path and whether there should be special considerations for soil management.

3.1. Site Description:

The Urban Reserve, or "North 40", is a 40-acre lakefront property owned by the City of Burlington (Figure 2). Currently the Site is transected by a portion of the Burlington Bike Path. Other portions of the Site are used by the local populace for dog walking and other recreation. The Site was purchased by the City along with several parcels to the south in the late 1980s and early 1990s as part of the renewal of Burlington's Lake Champlain waterfront. It was decided at this time that the Urban Reserve would be set aside for future generations to decide on its use: 2012 marks the first year of the planning process for that use.

The Urban Reserve is approximately ½ mile long and 500 to 1,200 feet wide. The eastern boundary of the Property is along a steeply wooded slope rising about 100 feet to the residential neighborhoods above. The former Moran Generating Plant (SMS #2005-3357) is located immediately south of the Site, and North Beach abuts the Property to the north. The land west of the Bike Path is relatively flat, and extends westward to the Lake Champlain shoreline. Most of the Property west of the current Bike Path was in-filled beginning in the mid-1800s for the expansion of the railroads; the source of the fill apparently was the sandy bluffs along the eastern Property boundary.

3.2. Site History

Prior to 1980, the Site was in industrial use with the earliest development beginning in the middle of the nineteenth century. At this time the Site was purchased from the State of Vermont by the Central Vermont Railroad (CVR), which leased parcels to different industries. Confirmed past identified uses of the Site include lumber yards, rail transport, and bulk petroleum storage. Bulk petroleum storage occurred in aboveground storage tanks (ASTs) ranging in size from 400,000 to 4,000,000 gallons; petroleum products included gasoline, kerosene, fuel oil, an unknown oil, and JP-4 jet fuel. Some elements of past use, including building foundations and slabs, several small pipelines related to the former petroleum bulk storage operations, and rail spurs remain on Site; however, bulk petroleum ASTs, the heating oil underground storage tanks (USTs) used for heating Site buildings, and the railroad tracks along the current bile path corridor, have been removed (Figure A1).

Historically, the Property was comprised of multiple parcels, the names of which were based on the petroleum storage / transfer businesses that occupied the respective parcel. These parcels included: Astroline, Astroline West, Tidewater, Astroline East / Flying A Site, and Texaco Beach (Figure A2).

Most of the Site is vegetated. Four areas of the Property have surficial asphalt or concrete cover, including the central portion of the Astroline site, the Tidewater site, the southern part of Texaco Beach, and the Bike Path. A sheet pile retaining wall forms the shoreline along the northern portion of the Astroline parcel. The balance of the shoreline generally consists of riprap. A wetland is present in the southern portion of the Astroline parcel. Imported fill has been placed over much of the central and part of the western portion of the Astroline site. The source of imported fill which covers the dog park is believed to be from construction activities at the

corner of College and Battery Street, while the source of fill placed over the western portion of the Astroline parcel is unknown.

A brief history of each parcel is summarized in Table 3-2, below. Rail service was provided to each parcel by the CVR throughout the Site's industrialized history.

Table 3-2: Summary of Site History

Area	History
Astroline	1850s to 1890s – Property used for lumber milling and storage. 1894 - Massive fire consumed 15 acres of the lumber yard (including several rail cars full of coal and oil). 1930s to late 1980s - Bulk petroleum storage and distribution. 1962 - 130,000 gallons of gasoline released from tank 104 (release reportedly contained and returned to AST). 1994 - Last petroleum AST removed. 1996 - Imported fill (about 5 feet) placed in bermed areas of former ASTs (except tanks 102 and 103). 1998 - USTs and oil-water separator removed.
Tidewater Site	1910 to 1930 - Used by refrigerator and venetian blind manufacturing companies. 1935 to 1974 - Bulk petroleum storage and distribution. 1974 to mid-1980s – Burlington Electric 1988 - ASTs decommissioned.
Astroline East / Flying A Site	1935 to 1980s - Bulk petroleum storage and distribution. 1980s - ASTs decommissioned. 1998 - Soils contaminated with PAHs stockpiled in area and treated via bioventing. Soils thinspread in area following treatment.
Texaco Beach	1886 - Filled. Lumber mill and storage. 1920s to 1980s - Bulk petroleum storage and distribution. 1980s - ASTs decommissioned.


NORTH AV
CONVENT SQ
MANHATTAN DR
SUNSET CT
WASHINGTON
VOLZ
BERRY
LAKEVIEW TERR
CROWLEY
WARD
CANFIELD
Lake Champlain
Colchester
Burlington
Winooski
N
0 100 200 400
Feet
Urban Reserve
Parcel Boundaries 2002
Sources: Urban Reserve, digitized by Stone; Parcels (2002), VCGI; Road labels, VCGI; Bing Imagery, ESRI
Urban Reserve Area Map
Burlington, VT
Data Analysis
for Chittenden County RPC
STONE ENVIRONMENTAL INC
Figure 2
O:\Proj-10\IR\2417-R CCRPC Brownfields Assessments\01 Urban Reserve\F-Data\GIS\Data\MapDocuments\SiteMap_Indscp8x11.mxd created on 3/2/2012 by ced



Legend

Urban Reserve Study Area Boundary Line

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

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TITLE: Site Map

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: 3

DATE: 11/5/12

DRAWN BY: DJL

CHECKED BY: EJS

3.3. Previous Environmental Investigations

The following studies of the Site have previously been completed.:

- 1986 - Initial Screening Investigation for Petroleum Contamination (Roy F. Weston, Inc.)
- 1990 - Preliminary Remediation Assessment CVR Option Lands (Roy F. Weston, Inc.)
- 1997 - Phase I Environmental Site Assessment (ESA) (Heindel & Noyes, Inc.)
- 1998 - Underground Storage Tank (UST) Removal Assessment (Griffin International)
- 2000 - Phase II ESA (Lamoureux & Dickinson, Inc.)

In 2008 an Archaeological Resource Assessment report was completed by the University of Vermont Consulting Archaeology Program. This report did not have any environmental investigation components.

A brief summary of the field investigation components of relevant previous assessments is provided in Table 3-3, below. Note that some of the sampling locations conducted during L&D's Phase II ESA were positioned immediately south of the current southern Property boundary; these locations are not included in the sample quantities summarized in Table 3-3.

Table 3-3: Field Investigation Components of Previous Environmental Investigations

Study	Year	Firm	Field Environmental Investigation Components
Initial Screening Investigation for Petroleum Contamination	1986	Roy F. Weston	• 10 test pits, depths ranging from 3.5 to 14 feet bgs. • Seven temporary monitoring points (five in groundwater and two in unsaturated zone). • Field screening of soil gas in temporary monitoring points for total organic vapors. • 18 soil samples for on-Site screening with an organic vapor analyzer (OVA) (in GC mode). Laboratory analysis for VOCs of nine soil samples. • Two soil samples for laboratory analysis of SVOCs, metals, cyanide, and phenols.
Phase I ESA	1997	H&N	• Six hand-auger soil borings with PID screening.
Underground Storage Tank (UST) Removal Assessment	1998	Griffin International	• Removal of USTs including: 1. One 1,000 gallon No. 2 fuel oil UST 2. One 2,000 gallon diesel UST 3. One 500 gallon No. 2 fuel oil UST 4. One 2,000 gallon oil/water separator (not UST) • Removal and transport offsite of 5,090 gallons of liquid and 100 gallons of sludge from the tanks

Study	Year	Firm	Field Environmental Investigation Components
			• Field screening of excavation pits for total volatile organic vapors with PID
Phase II ESA	2000	L&D	• Passive soil gas survey at 18 points (BTEX and TPH analysis). • 33 surface soil samples with PID field screening for VOCs and Hanby test kit screening for PAHs. • Seven surface soil samples for laboratory analysis of VOCs, TPH, and PAHs. • 23 soil borings with continuous PID field screening for VOCs and Hanby test kit screening for PAHs on select samples. • 13 water table monitoring wells. Three surface water and 13 groundwater samples analyzed for VOCs, PAHs, and PP metals.

Notes:
bgs= below ground surface
PID = photoionization detector
OVA = organic vapor analyzer
GC = Gas Chromatograph
VOCs = volatile organic compounds
SVOCs = semi-volatile organic compounds
BTEX = benzene, toluene, ethylbenzene, and total xylenes
PAHs = polycyclic aromatic hydrocarbons
TPH = total petroleum hydrocarbons

3.4. Nearby Active Hazardous Waste Sites

The Site is abutted by one active State of Vermont Hazardous Waste Site: Moran Plant (SMS# 20053357), located immediately south of the Site (Figure 4). Other nearby active State of Vermont Hazardous Waste Sites include: Burlington Public Works (SMS# 90-0540) located immediately south of the Moran Plant, Burlington College (SMS# 96-2048) located approximately 500 feet west of the Site, and Mcgaffrey's Sunoco (SMS # 94-1698) located immediately west of the Burlington College Site (Figure 4). Sections 3.4.1 through 3.4.4 present a summary of each of these sites and their current status.

3.4.1. Moran Plant (SMS# 20053357)

The Moran Plant property hosted a coal-fired electrical power generation plant from 1954 to 1986 when it was officially decommissioned. Prior to 1953, this portion of Burlington was used for a wide range of industrial purposes, including railroad, lumber yard and mill, and storage/transportation of petroleum products. The Moran Plant parcel encompasses an area approximately 600 ft by 350 ft at the edge of Lake Champlain. The Plant building, a 120 ft by 140 ft steel, brick, and concrete structure on a mat foundation is the only building on Site. While most of the building is vacant, an area in the western end is currently used as the Lake Champlain Community Sailing Center (LCCSC) under agreement with the City of Burlington.

Several investigations have been carried out at the Moran Plant since its decommissioning in 1986. The most recent data (Phase II ESA conducted by Waite Environmental Management, LLC. (WEM), 2005) concluded that several PAHs were detected in soil at concentrations in excess of the soil guidance levels in the shallow soil at locations immediately south, north and northeast of the Moran Plant. Trichloroethene (TCE) was detected in excess of the guidance level for soil at one deep sample in the area immediately north of the Moran

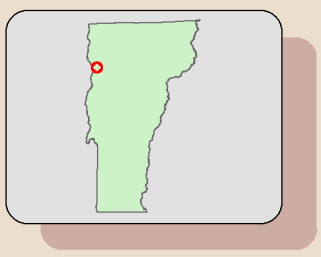
Plant. Based on discussion with a Burlington Electric Department (BED) representative, which formerly operated the facility, it is possible the source of chlorinated VOCs detected in in soils at the Moran Plant is drums that were formerly stored in this area for a brief time in 1994, after the plant was decommissioned.

Arsenic was also detected at concentrations in excess of the soil guidance levels in samples collected throughout the Site; however concentrations appear to be of the same order of magnitude as naturally occurring levels in this area. Levels of PCBs below the guidance criteria at three locations were reported.

Several chlorinated VOCs have been detected in groundwater monitoring wells at the Moran Plant. TCE is the only compound that has been detected above the Vermont Groundwater Enforcement Standard (VGES) in one sample in the area immediately north of the Moran Plant. No PAHs, PCBs, total metals, nor petroleum VOCs were detected in groundwater during the 2005 sampling event. Groundwater flow at the Moran Plant is parallel to groundwater flow at the Urban Reserve (to the west towards Lake Champlain) and is not likely to impact Site groundwater.

The 2005 Phase II ESA conducted by WEM concluded asbestos was present at the Moran Plant in exterior window caulk, corrugated window panels, and exterior roof flashing. In addition it was noted asbestos dust was found in dust inside the Plant in 1987 and there has been no further mitigation since that time, there continues to be an asbestos hazard to future construction workers during any demolition or cleanup activities. Lead paint and bird droppings were noted contaminants of concern during the 2005 Phase II ESA, but are not likely to migrate to the Urban Reserve Site.





Nearby Hazardous Waste Sites

Study Area

0 75 150 300 Feet

N

Sources. Study Area: Stone Environmental; Hazardous Waste Sites: Vermont ANR Environmental Interest Locator.

Study Area and Nearby Managed Environmental Sites

Figure 4

Phase II Environmental Site Assessment of the Urban Reserve.

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3.4.2. Burlington Public Works (SMS# 90-0540)

Two underground storage tanks were removed from the Burlington Public Works Site. The first, a 3,000 gallon gasoline UST, was removed in 1990. The second, a 2,000-gallon diesel UST, was removed in 1992. The tank graves were backfilled with soils removed during the excavations. The degree of soil contamination was defined as a maximum of 110 parts per million (June 21, 1990) in field screening the excavated soil by handheld PID during the tank pulls. On September 10, 1993The VT DEC Sites Management Section (SMS) issued the Burlington Public Works Site a closure letter. Analytical results from a monitoring well in the area of the former USTs detected MTBE at less than 10 parts per billion, and total volatile hydrocarbons at 162 parts per billion. The SMS concluded that contaminant levels in groundwater downgradient from the former USTs are below the State's groundwater enforcement standards, natural biodegradation processes have likely substantially reduced contaminant levels, and the potential for the contamination to adversely affect any sensitive receptors such as adjacent basements or Lake Champlain is minimal. The migration of contaminants from the Burlington Public Works Site to the Urban Reserve Site is unlikely.

3.4.3. Burlington College (SMS # 96-2048)

On August 19, 1996 a 1,000 gallon heating oil UST was removed from the Burlington College Site. Twin State Environmental Corporation (TSEC) oversaw the removal of the UST and monitored VOC concentrations of excavated soils with a photoionization detector (PID). The highest reading (26 parts per million volume) was detected beneath the UST fill port. A PID reading of 19 ppm v/v was measured below the tank bottom, at a depth of 6 ft below ground surface. The most contaminated soils were removed during excavation, the rest of the soils were used s backfill.

In May, 1997 TSEC advanced three soil borings in the vicinity of the former UST. Soils were found to be fine to coarse sand with gravel and some burned material. Borings were advanced to depths ranging from 16 to 20 ft bgs, groundwater was not encountered in any of the borings. Headspace concentrations of VOCs were not detectable with a PID (<0.1 ppmv)

The Burlington College site received a Sites Management Activity Completed (SMAC) designation by the VT DEC WMD on March 24, 1998. The risk of contaminants migrating from the Burlington College Site to the Urban Reserve Site appears to be minimal.

3.4.4. Mcgaffrey's Sunoco (SMS # 94-1698)

A leaking UST system existed at the Mcgaffrey's Sunoco site. Stone was unable to find details concerning when the leaky UST system was removed. However as a result of the UST removal a stockpile of approximately 35 cubic yard of contaminated soil was poly encapsulated on the Sunoco Site, and was monitored by Griffin International, Inc. on a semi-annual basis until the VT DEC SMS assigned the site a SMAC designation. The SMS determined that the source of contamination has been adequately characterized and remediated, the leaking UST system had been removed, and the soil stockpile has been adequately treated and disposed. The VTDEC concluded that the Sunoco Site does not pose an unreasonable risk to human health or the environment. The risk of contaminants migrating from the Sunoco Site to the Urban Reserve Site is minimal.

3.5. Conceptual Site Model

Stone prepared this CSM based on available data for the Site. A CSM is a set of working hypotheses which describe key aspects of the problem(s) at a site. As with any hypothesis, the CSM is not conclusive and may require testing to arrive at desired levels of certainty. The CSM includes discussions of Site geology/hydrogeology, contaminants of concern based on historic Property use and previous investigation results, how contaminants were possibly released at the Site, their transport pathways and fate mechanisms, as well as exposure routes for both ecological and human receptors based on current use and the proposed redevelopment plans for the Site.

3.5.1. Geology / Hydrogeology

Overburden across the Site generally consists of fine to coarse sand, with little fines. This type of material is typified by relatively high hydraulic conductivity values which can result in high groundwater flow velocities beneath the site. Non-native fill is present across much of the Site. Underlying these soils, Stone expects that native soils are consistent with lake-bottom sediments, specifically silts and clays. The silts and clays are likely to limit vertical groundwater movement and to cause flow to be largely horizontal toward, and into the Lake. Bedrock beneath the Site is mapped as the Dunham Dolomite. Bedrock was not encountered to maximum exploratory depths of 15 feet in areas west of the bike path, and 45 feet in areas east of the bike path.

Water levels in 13 overburden monitoring wells were measured by L&D on three dates in 1999: August 5th, August 19th, and September 28th. Depth to groundwater beneath the relatively flat tract of land west of the bike path ranged from about 3 to 10 feet bgs; depth to groundwater on the slope east of the bike path was measured at about 37 feet bgs at monitoring well MW-11.

Groundwater flow within the shallow unconfined overburden aquifer is to the southwest, discharging to Lake Champlain (Figure A3). The hydraulic influence of the sheet pile retaining wall is unclear, however we expect that the sheet pile retaining wall impedes flow of groundwater and contaminants from the Site to Lake Champlain. The wall would cause flow paths to diverge horizontally around the wall and perhaps vertically under the wall unless it is keyed into the silts and clays at the base of the shallow unconfined aquifer.

3.5.2. Areas and Contaminants of Concern

Areas and COCs, based on historic Property use and previous investigation results, are summarized in Table 3-4, below. Historic Property uses / features of concern are displayed on Figure A4.

Table 3-4: Areas and Contaminants of Concern

Area	Historic Use or Potential Contaminant Source	COCs
Astroline	• Lumber Mill / Bulk Petroleum Storage • Petroleum USTs / Oil-Water Separator • Rail Spurs	• Petroleum products, Metals • Petroleum products • PAHs, TPH, herbicides, PCBs, metals
Tidewater Site	• Bulk Petroleum Storage	• Petroleum products

Area	Historic Use or Potential Contaminant Source	COCs
	• Refrigeration Company	• Chlorinated VOCs • Chlorofluorocarbons (CFCs):
Astroline East / Flying A Site	• Bulk Petroleum Storage • Former PAH-Contaminated Soil Stockpile	• Petroleum products • PAHs
Texaco Beach	• Lumber Mill / Bulk Petroleum Storage	• Petroleum products, metals
Bike Path	• Former Railroad Tracks	• PAHs, TPH, herbicides, PCBs, heavy metals
Existing Rail Lines	• Railroad Tracks	• PAHs, TPH, herbicides, PCBs, metals
Site Wide	• Moran Plant / Fire of 1894	• PAHs from atmospheric deposition

Notes:
PAHs: polycyclic aromatic hydrocarbons; PCBs; polychlorinated biphenyls; TPH: total petroleum hydrocarbons.

Petroleum products, including gasoline, fuel oil, and/or JP-4 jet fuel appear to have been released to the surface or subsurface via surface spills and/or leaks from conveyance piping associated with the former bulk storage operations at the Site. Fuel oil could have also been released to the subsurface via the former heating oil USTs. These petroleum products include volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs), have a density less than water, a moderate to low aqueous solubility, and biodegrade under both aerobic (faster) and anaerobic (slower; and very limited for methyl tert butyl ether (MTBE) under most conditions) conditions.

Polycyclic aromatic hydrocarbons are a subset of SVOCs. PAHs occur in fossil fuels, and are produced from the oxidation of fossil fuels or biomass through incomplete combustion. They may be present in refined heating oil at concentrations of a few percent. PAHs have a low aqueous solubility and are persistent (i.e. degrade slowly) in the environment. They are a COC due to the past use of heating oil at the Site, occurrence of the fire in 1894, likely use of creosote-treated railroad ties, past storage and transport of coal along the rail, and proximity of the Site to the Moran Plant (due to concerns of atmospheric deposition from the former coal-fired generating plant).

Chlorinated VOCs are COCs for the Site because 1,1,2,2 tetrachloroethane (TeCA) was detected in a deep test-pit soil sample collected during the investigation in 1986; trichloroethene (TCE) was also reported at estimated concentrations (below detection limits) in soil samples collected from test pits TP-2, TP-3, TP-5, and TP-7. The source of 1,1,2,2 TeCA is unknown. Historically, 1,1,2,2 TeCA was widely used in the United States as a solvent, as well as a refrigerant (under the name R-130). It may have been used on-Site for cleaning oily equipment, stripping paint in preparation for re-painting ASTs, or in association with the refrigerator company that leased the Tidewater parcel between 1910 and 1930. 1,1,2,2 TeCA has a density greater than water and is

considered a dense non-aqueous phase liquid (DNAPL); DNAPLs may penetrate the water table and migrate downward through the aquifer if sufficient driving forces (volume of DNAPL) are present. 1,1,2,2 TeCA can degrade under anaerobic conditions to vinyl chloride.

Herbicides are a COC because they were likely historically applied along the rail lines that traverse the Site and transported via rail. Herbicides were arsenic based prior to the 1950s, and were replaced by organochlorines until about 1975. Some herbicides can be persistent in the environment.

Metals, including arsenic, lead, and thallium are COCs at the Site due to the possible historic use of arsenic-based herbicides, lead and thallium-based insecticides, past storage of leaded gasoline, and former coal combustion at the Moran Plant, since coal ash may contain these metals in trace quantities. Historically, wood preservatives, such as those used in rail ties, included arsenic in their formulations to prevent damage by insects. Once deposited, these metals typically have a low mobility, but can leach into groundwater under low pH conditions.

Polychlorinated biphenyls may have been released to the ground surface in the vicinity of the railroad tracks and spurs from leaking train-locomotive transformers that may have contained PCBs or from liquids transported by the rail. PCBs have a low aqueous solubility, are persistent in the environmental, and typically have low mobility.

3.5.3. Contaminant Distribution

Previous investigations have confirmed the presence of VOCs, PAHs, and metals in Site soils and /or groundwater at concentrations above applicable standards or screening levels. Figures presenting cumulative results of previous environmental investigations are provided in Appendix A. Detected VOC, PAH, and TPH contamination appears to be primarily related to surface or near surface releases associated with former petroleum bulk storage and transfer operations at the Site. Elevated arsenic concentrations identified in surface soils are consistent with naturally occurring “background” concentrations in Vermont resulting from arsenic-bearing minerals in parent rock.

The magnitude and distribution of these contaminants are described in greater detail in the following sections.

3.5.3.1. Soil

Soils at the Site have previously been investigated via 10 test pits, 39 hand-auger soil borings, and 23 hollow-stem auger (HSA) soil borings (Figure A5).

3.5.3.1.1. Surface Soil

PID Readings

Of the 62 surface soil samples (i.e. 0-2 feet bgs) collected across the Site and screened in the field with a PID, 10 samples exhibited PID readings greater than 20 parts per million volume/volume (ppm v/v) (Figure A6). As per V T DEC guidance, PID readings greater than 20 ppm v/v generally require additional evaluation for gasoline contaminated soils; PID readings greater than 10 ppm v/v generally require additional evaluation for #2 fuel oil/diesel fuel contaminated soils. Three of the samples were located in the northern portion of the Flying A area, and the balance of the samples were located in the western portion of the Astroline site. Of note, imported fill was placed over a portion of the impacted soils at the Astroline site following the field screening in 1999 (Figure A6).

VOCs

Seven surface soil samples from the Site were analyzed at a laboratory for VOCs via EPA Method 8260. No VOCs were detected at concentrations above the respective Vermont Soil Screening Value SSVs for residential and/or industrial settings (Figure A7).

In the area of the Burlington Bike Path, the potential release to surface soil would have occurred over 25 years ago. As such, VOCs are very unlikely to remain in these soils due to volatilization. This is supported by prior environmental investigation of surface soil that found depleted VOCs in surface soils even when TPH was found to be elevated.

PAHs/TPH (Hanby Field Test Kit)

Surface soils at 33 locations were screened for PAHs / TPH using a Hanby test kit; of these, eight samples exhibited concentrations greater than 200 parts per million (ppm), which exceeds the Vermont Soil Screening Value (SSV) of 200 ppm for TPH. These samples were located in the Flying A area and the central portion of the Astroline site. Of note, imported fill was placed over a portion of the impacted soils at the Astroline site following the field screening in 1999 (Figure A8).

PAHs

Seven surface soil samples from the Site were analyzed at a laboratory for PAHs via EPA Method 8270C. Of these, one soil sample (from HA-15) exhibited several PAHs at concentrations above the respective Vermont Soil Screening Values (SSVs) for residential and/or industrial settings (Figure A9). However, the laboratory detection limits for several PAHs at each of the other five locations were above the respective SSV.

Metals

Arsenic was detected above the SSV of 0.39 mg/Kg (for residential settings) in each of the seven surface soil samples analyzed from the Site, with concentrations ranging from 3.2 to 7.4 milligrams/kilogram (mg/kg) (Figure A10). These concentrations are consistent with naturally occurring “background” concentrations in Vermont resulting from arsenic-bearing minerals in parent rock.

3.5.3.1.2. Subsurface Soil

PID Readings

Soils exhibited PID readings greater than 20 ppm v/v in subsurface soils (i.e. >2 ft bgs) at17 boring locations across the Site (Figure A11), suggesting, in conjunction with visual observations from soil testing logs, that the Flying A parcel and much of the Site west of the bike path bike path are impacted by petroleum contamination at depth.

VOCs

During the 1986 Site investigation, 1,1,2,2- TeCA, a chlorinated VOC, was detected at a concentration of 17 micrograms per kilogram (µg/kg)in a sample collected from test pit TP-7 at a depth of 14 feet bgs (Figure A5). TCE was detected at estimated concentrations (below detection limits) in soil samples collected from test pits TP-2, TP-3, TP-5, and TP-7. No VOCs were detected at concentrations above the respective SSVs. Based on the limited number of detections and low concentrations of these compounds, it does not appear that exceedances of 1,1,2,2-TeCA or TCE in Site media are widespread, however as the number of samples and

spatial distribution of those collected is limited, higher concentrations may exist on Site than what have been previously detected.

3.5.3.2. Groundwater

Groundwater was previously assessed at the Site via 13 water-table monitoring wells installed and sampled during L&D's Phase II ESA in 1999.

VOCs

Petroleum-related VOCs, including benzene, total trimethylbenzenes (TMBs) and/or naphthalene, where detected at concentrations above respective Vermont Groundwater Enforcement Standards (VGESs) in samples collected from monitoring wells MW5, MW-6, MW-7, MW-9, and MW-16, located on the former Astroline and Tidewater parcels (Figure A12). This contamination appears indicative of weathered gasoline and/or heavier molecular weight fuels such as diesel, heating oil, or JP-4. Natural attenuation of the petroleum contamination is likely occurring; however, there are no long-term monitoring data to support the suspected occurrence of this process. No free-phase product was detected in any of the Site monitoring wells.

No chlorinated VOCs were detected in any of the shallow groundwater samples collected at the Site.

PAHs

Other than naphthalene, no PAHs were detected in shallow groundwater samples collected from the Site.

Heavy Metals

Except at monitoring wells MW-1 and MW-4, one or more of the following metals were detected at concentrations above respective VGESs in groundwater samples collected from the Site: antimony, arsenic, lead, and thallium (Figure A13). Due to suspected high sample turbidity given the sample collection method (i.e. bailer), metal concentrations may be biased high.

Leaded gasoline was historically stored at the Site; it is not clear whether possible releases of lead-gasoline at the Site are attributing to the observed impact to groundwater. Thallium was a historic component of insecticides; it is unclear whether these insecticides were previously used at the Site. Arsenic was a historic component of herbicides; it is possible that these herbicides were used previously at the Site along the rail lines; however, it is not clear whether this is a contributing source to the observed impact to groundwater. Antimony is not a COC for the Site based on historic Site use.

3.5.3.3. Surface Water

No VOCs or PAHs were detected in the three surface water samples collected from Lake Champlain. Copper was detected in SW-2 at a concentration of 60 micrograms per Liter (µg/L).

3.5.3.4. Soil Gas

Twenty-two Emflux® passive soil gas collectors were installed as part of the Phase II ESA (L&D, 2000) across the Site and adjacent Moran Plant property for TPH and BTEX analyses. The Emflux® collectors provide total mass of analyte detected without measurement of volume of soil gas. Therefore concentrations are not obtained, but rather relative contaminant mass. The results of this assessment indicated TPH and

BTEX present in soil gas at locations down gradient of and adjacent to the ASTs on the former Flying A parcel. No full spectrum VOC analyses of soil gas has been performed on the Site.

3.5.4. Data Gaps

Stone assessed existing environmental data for the Site and adjacent sites in the context of the CSM to identify data gaps and data limitations associated with the Site. The data gaps/limitations are presented by media type in sections 3.5.4.1 and 3.5.4.3.

3.5.4.1. Soil

Stone has identified the following data gaps / limitations associated with shallow soils at the Site:

1. There are insufficient PAH analytical data for the Site to adequately assess current and future risk of exposure: Only seven surface soil samples have been collected from the Site for laboratory analysis of PAHs via EPA Method 8270C. PAHs were detected above respective SSVs at one location; however, the laboratory detection limits for some PAHs were above the SSVs at the remaining six sample locations. Given the approximately 40-acre area of the Site, the elevated laboratory detection limits for previous samples, and potential sources for PAH contamination based on historic Site use, the existing data set is not sufficient to evaluate current exposure risks across the Site.

2. There have been no analyses for herbicides, PAHs, heavy metals, or PCBs along the bike path to assess future risk of exposure if the soils beneath the paved area are exposed: No analysis for these COCs in surface soil has been completed to date beneath the current bike path, which is built along the corridor formerly occupied by the railroad tracks. Therefore, it is unknown whether these COCs are present in shallow soils in the vicinity of the bike path at concentrations above the SSVs. Since the bike path is currently paved with asphalt, there is no exposure to underlying soils. However, the City of Burlington anticipates renovating the bike path in spring 2013, which will involve removing existing pavement and reworking underlying soils. As such, this data gap should be filled in advance to determine if there will be exposure risks to workers and recreational users during the renovations, and whether there are special considerations for soils management.

3. There are no analytical data that characterize imported fill materials:. There are no documented analytical data on the quality of the imported fill that was used to cover areas in the central and western portion of the Astroline site, and these soils were not analyzed for COCs during the Phase II ESA. The source of imported fill which covers the current dog park is believed to be from construction activities at the corner of College and Battery Street; based on historic use where the fill was derived, these soils may be impacted by PAHs and TPH. The source of fill placed over the western portion of the Astroline parcel is unknown; consequently, potential COCs associated with these materials are unknown.

4. There are no shallow chlorinated VOC data at the Tidewater parcel or near test pit TP-7:. 1,1,2,2-TeCA was detected in a sample collected at a depth of 14 feet bgs from test pit TP-7, located on the former Texaco Beach parcel. No surface soil samples have been collected for chlorinated VOC analysis from the vicinity of TP-7 or at the former Tidewater site, where 1,1,2,2- TeCA may have been used as refrigerant for the former refrigerator manufacturer, to evaluate whether chlorinated VOCs are present at concentrations that present a current or future risk of exposure.

3.5.4.2. Groundwater

Stone has identified the following data gaps / limitations associated with groundwater at the Site:

1. There are an insufficient number of groundwater sampling locations to reasonably characterize impact to groundwater from COCs across the Site: Thirteen monitoring wells have been installed and sampled across the 40-acre Site; given the numerous areas of concern across this relatively large area, this sampling density is inadequate to reasonably characterize impact to groundwater from COCs.

2. There are insufficient water quality data to evaluate whether contaminant concentrations are stable, increasing, or decreasing over time, and if natural attenuation processes are occurring at an acceptable rate. In 1999, groundwater was found to be impacted by petroleum VOCs at several wells across the Site at concentrations above the VGESs. However, there has only been one round of data collected to date. As a result, there are insufficient data to evaluate whether concentrations are stable, increasing, or decreasing over time, and if natural attenuation processes are occurring at an acceptable rate. Due to the presence of a "smear zone" found at many petroleum-impacted Sites, dissolved-phase concentrations and even the occurrence of light non-aqueous phase liquids (LNAPL) can be highly dependent on seasonal fluctuations in the water table elevation. One round of monitoring is insufficient to characterize the impact to groundwater at the Site.

3. There has been no assessment of the deep overburden aquifer for chlorinated VOCs: 1,1,2,2. TeCA and other chlorinated VOCs, which have a density greater than water, have been detected at relatively low concentrations in deep soils at the Site; the deeper portion of the overburden aquifer has not been assessed to determine if chlorinated VOCs have impacted groundwater at depth.

4. The risk of impact to Lake Champlain in the absence of the sheet piling retaining wall is unclear: The magnitude and extent of COC impact to the overburden aquifer immediately upgradient of the sheet pile retaining wall has not been reasonably characterized, and the role, if any, the sheet pile retaining wall currently plays in preventing contaminant migration to Lake Champlain is uncertain. Should the sheet pile retaining wall be removed in the future, as has been discussed as a possible redevelopment option, the risk of contaminant impact to Lake Champlain is unknown. If only dissolved and immobile (residual) contamination is present upgradient of the sheet pile retaining wall, the risk may be minimal; however, if mobile LNAPL is present behind the wall, the risk may be increased. Even if the LNAPL is depleted of target COCs that could possibly present a risk to ecological receptors, the LNAPL could impart a negative aesthetic on the lake if sheening or iron staining occurs.

5. Previous groundwater results may be biased due to sampling methodology: Previous groundwater samples were collected with a bailer; this sampling technique may have biased VOC results low (due to increased potential for volatilization) and biased heavy metal results high (due to likely hood of high sample turbidity).

3.5.4.3. Soil Gas

Stone has identified the following data gaps / limitations associated with soil gas at the Site:

1. Prior soil gas assessment did not assess non-petroleum COCs. Prior assessment of VOCs in soil gas relied on passive soil gas collection methods (not concentration based). Comparison to criteria to assess potential threat to any future occupied building is not possible.

2. Soil gas investigations were limited in their analytical suite to BTEX and TPH. No analyses for chlorinated solvent or refrigerant COCs have been performed to date.

3. Prior assessments of soil gas contamination in certain portions of the Site, specifically the Astroline West parcel, were inadequate for their spatial coverage. Prior environmental assessment of the Astroline West parcel collected only two soil gas samples.

3.5.5. Sensitive Receptors and Exposure Pathways

3.5.5.1. Soil

Exposure pathways for humans from impacted surface soils include incidental ingestion, inhalation (of dust), and dermal contact.

Currently, due to known exceedances of the SSLs for select PAHs and/or TPH in surface soil, areas B (Flying A) and C (northwest portion of Astroline) designated on Figure A14 present a risk of exposure to human receptors from these COCs.

Currently, areas D, E (including E1 and E2), and F, as indicated on Figure A14, represent areas with possible PAH or TPH exceedances of SSLs either due to elevated detection limits from previous analyses or lack of sufficient spatial data for these COCs. Based on the source of the imported fill used in area E1, PAHs and TPH are potential COCs in this area. Since the source of the fill used to cover area E2 is unknown, additional COCs may be of concern.

There are no analytical data for surface soils beneath the bike path to evaluate whether COCs, including herbicides, PAHs, TPH, heavy metals, and PCBs, are present at concentration above respective SSLs. Currently, the area is paved with asphalt, so the exposure pathway to potentially contaminated soils is incomplete. However, the City of Burlington anticipates renovating the bike path in spring 2013, which will involve removing existing pavement and reworking underlying soils. As such, if there are COCs present beneath the asphalt at sufficient concentrations and quantities, there may be a risk of exposure to workers and recreational users during the renovations. Furthermore, if soils are to be removed from the bike path area, such as to install engineered fill, a soil management plan will need to consider the waste characteristics of the soils to be removed.

Due to the presence of elevated petroleum-related impacted soils present beneath much of the Site at depth, any intrusive activities across the Site could result in exposure to petroleum contaminants.

3.5.5.2. Groundwater

Exposure pathways for humans from impacted groundwater include ingestion, inhalation, and dermal contact. Given current Site use, the exposure pathway for human receptors from contaminated groundwater beneath the Site is not complete. A notice to land record should be filed to ensure that there is no future use of Site groundwater that could result in possible exposure.

Surface water data and observations of the shoreline suggest groundwater is not currently significantly impacting Lake Champlain. However, without data to suggest otherwise, the lake may be at risk of future impact if the sheet pile retaining wall is removed.

As depth to groundwater is shallow (3 to 10 ft bgs), redevelopment plans for the Site may, at some point, include excavations below the water table. Exposure to contaminated groundwater by Site workers will need to be evaluated. Dewatering operations, if required, will need to consider treatment or containment of contaminated groundwater.

3.5.5.3. Soil Gas

Construction of any new buildings at the Site would need to consider the vapor intrusion pathway, *via* contaminated soil and groundwater. Existing data are insufficient to evaluate risk of exposure to Site users or future occupants through contaminated soil gas. At this time, no inhabited structure is planned for the Site.

4. FORM D: PROJECT DESCRIPTION

As stated in Section 3 Form C, the objective of the Phase II ESA is to assess whether shallow soils beneath the bike path, which currently bisects the Site, contain contaminants at concentrations that, if present, would present a risk to recreational users or construction workers during upgrades to the bike path and whether there should be special considerations for soil management. Based on past Site uses and results from previous environmental investigations potential contaminants of concern (COCs) include:

- Herbicides
- Metals
- PCBs
- TPH-DRO
- PAHs

As stated in Section 3.5.3.1.1, due to the length of time since the potential release(s) to shallow soils, VOCs are not a COC for the soils underlying the bike path.

4.1. Utility Clearance and Site Health and Safety

Stone will mark out proposed investigation locations for Digsafe utility clearance activities prior to initiating intrusive Site investigation activities. Stone will coordinate with appropriate Site representatives to ensure that Site-owned utilities are not impacted by drilling activities.

A Health and Safety Plan (HASP) is included in Appendix E. The HASP details roles and responsibilities, Site hazards, Site activities in relation to health and safety, personal protective equipment, emergency contact information, as well serve as a reference to be used by Site personnel in the event of an emergency.

4.2. Surface Soil Assessment

To assess whether past Site activities have resulted in a release of hazardous materials to the environment, twenty six (26) shallow soil borings will be performed at regular intervals along the section of the Burlington Bike Path that crosses the Site. The soil borings will be performed to a target depth of 5 feet. Proposed soil boring locations are presented on Figure 5, below. Soil borings will be logged for texture, moister, color, and VOCs using a hand held PID equipped with a 10.6 eV lamp. Soil samples will be collected from the interval within each soil boring that corresponds to the former, pre-bike path surface horizon as determined by textural disconformity.

Soil samples will be analyzed for RCRA 8 Metals, PAHs, PCBs, TPH-DRO, and Herbicides analyses via EPA Methods SW-846-6010B, 7471A (Hg), 7060 (As),8270C with SIM, 8082 with manual Soxhlet extraction, 8015B DRO, and 8151A respectively.

If, during VOC logging, a soil interval is found to contain VOCs greater than 10 parts per million volume/volume (ppm v/v), a discrete sample for VOC analysis by EPA Method 8260 will be collected. Based on the current CSM, Stone does not anticipate any elevated VOCs.



Legend

- Urban Reserve Study Area Boundary Line
- Former Rail Road/ Bike Path
- Retaining Wall
- Existing Rail Road Tracks
- Bike Path
- MW-1 Previous Monitoring Wells
- Proposed Soil Sample Locations

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

STONE ENVIRONMENTAL INC

TITLE: Soil Sampling Locations

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: 5

DATE: 1/7/12

DRAWN BY: DJL

CHECKED BY: DTV

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4.3. Evaluation of Sampling Results with Action Limits

Tables B.1 through B.5in Appendix B provides Site Contaminants of concern (COCs), their associated laboratory reporting limit and action level. For this Phase II ESA, the Regional Screening Levels (RSLs) for residential soils will be used as action limits.

4.4. Quality Assurance/Quality Control (QA/QC)

To ensure that data produced during field and analytical efforts are of known and acceptable quality, several QA/QC samples will be collected for each medium of interest. Field duplicates will be collected for each analytical method at a 5 percent (1 in 20) frequency. Field quality control requirements are summarized, below, in Table 4-1.

Table 4-1: Field Quality Control Requirements

QC Sample	Frequency	Acceptance Criteria	Corrective Action
Field Duplicates	5% per analyte group	Precision 50% Soil	Assess sampling and lab precision, qualify Data

Sampling apparatus will be decontaminated prior to initiating sample collection and between locations (Stone Standard Operating Procedure (SOP) SEI 5.1.5).

Specific QA/QC-related details, as well as relevant SOPs are presented in Appendix C.

4.5. Report

Following completion of all field investigation work and receipt of all analytical data, Stone will compile all appropriate data and prepare a Phase II ESA Report summarizing field activities, field observation and concentration results. The report will present conclusions based on these results and propose recommendation for further Site activities, as appropriate. The report will include laboratory reports, Site maps, soil boring logs, and field notes. The Phase II ESA Report will be prepared in accordance with the VT DEC SMS guidance document titled “Investigation and Remediation of Contaminated Properties Procedure” dated April, 2012 and ASTM E 1903-02 “Standard Guide for Environmental Site Assessments: Phase II Environmental Site Assessment Process”.

4.6. Project Timeline

Digsafe utility mark-out will occur within one week of acceptance of this SS QAPP by VT DEC and EPA Region I. Field efforts can commence within three business days of utility clearance pending resource availability and weather.

Stone anticipates that environmental drilling activities and soil sample collection will require two field days to perform. Analytical results will be produced by the laboratory within two weeks of sample receipt.

A draft Phase II ESA Report will be presented for stakeholder review within 10 business days of receiving final laboratory results. A final Draft Phase II ESA Report will be submitted to the VT DEC and EPA within five days of receiving comments. Table 4-2, below, presents the timeline for completion of these activities.

Table 4-2: Project Timeline

Tasks	Task Start	Task End
SSQAPP Revisions and Approval		February 1, 2013
Digsafe Utility Mark-Out	February 4, 2013	February 6, 2013
Environmental Drilling	February 7, 2013	February 8, 2013
Soil Sample Collection	February 7, 2013	February 8, 2013
Analytical Results	February 11, 2011	February 25, 2013
Reporting		March 8, 2013

4.7. Cost

The anticipated Costs to perform the scope of services described above is summarized in the Table 4-3 below and detailed in Table D1 of Appendix D.

Table 4-3: Cost Estimate, Urban Reserve Phase II ESA

Task	Subtotal
Task 1 - SS QAPP	$3,045
Task 2 – Surface Soil Assessment (Area A – Bike Path)	$22,825
Task 3 - Report	$1,520
Project Total	**$27,390**

5. FORM E: SAMPLING DESIGN

5.1. Surface Soil Assessment

Stone proposes to utilize a Geoprobe 6610 DT track mounted drill rig, or similar, to perform Site drilling activities. Soil cores will be collected using 2.25-inch outside diameter (OD) direct push casing equipped with a dual tube soil sampling tool (i.e. Geoprobe DT22). Soils are retained in the inner tube within a PVC sleeve. Soils will be collected continuously from the ground surface to five feet below ground surface. Upon retrieval, soils will be logged for texture, color, moisture content, and VOCs by handheld PID. Screening of soils for VOCs will consist of slicing the core longitudinally and inserting the tip of the PID into the soil boring for each one- foot vertical interval. Peak readings will be recorded onto soil boring logs.

Samples will be transported under chain of custody to AMRO Environmental Laboratories Corporation (AMRO) in Merrimack, New Hampshire to be analyzed for RCRA 8 Metals, PAHs, PCBs, TPH-DRO, and Herbicides analyses via EPA Methods SW-846 6010B, 7471A (HG), 7060 (As), 8270C with SIM, 8082 with manual Soxhlet extraction, 8015B DRO, and 8151A respectively.

In the event that VOCs are detected greater than 10 ppm v/v by PID, Stone will collect a discrete soil sample from the interval exhibiting the highest PID response. Soil will be collected from the soil core using a disposal En-Core sampler, or equivalent, directly in to a 40 mL vial containing 10 mL of methanol for field preservation. A separate 10 gram sample will be collected in to a 40 mL vial for percent moisture determination. Soil samples collected for VOC analysis will be transported under chain of custody protocols to AMRO for analysis by EPA Method 8260. Based on the current CSM, Stone does not anticipate collecting any samples for VOC analysis.

5.1.1. Surface Soil Assessment Locations Justification

Locations of each proposed surface soil boring are presented on Figure 5. Soil borings will be performed approximately every 100 feet along the section of the Burlington Bike Path that bisects the Site.

6. FORM F-1: METHOD AND SOP REFERENCE TABLE

Stone Environmental's study-specific SOPs are provided in Appendix C of this document, and Appendix F of the BA QAPP.

Table 6-1: Analytical method reference table

Analytical Method Reference (include document title, method name/number, revision number, date)		Project Analytical SOPs (include document title, date, revision number, and originator's name)	
1a.	Semivolatile Organic Compounds by Gas Chromatograph/Mass Spectrometry by SW-846 Method 8270C	1b.	AMRO Environmental Laboratories, Merrimack, NH
2a.	Mercury in Solid or Semisolid Waste, SW-846 Method 7471A by Perkin Elmer FIMS 100 Automated Mercury Analyzer Analysis of Metals by Inductively Coupled Plasma Optical Emission Spectrometry according to SW-846 Method 6010B.	2b.	AMRO Environmental Laboratories, Merrimack, NH
3a.	Polychlorinated Biphenyls by Gas Chromatograph/Electron Capture Detector by SW-846 Method 8082	3b.	AMRO Environmental Laboratories, Merrimack, NH
4a.	Laboratory Method for the Determination of Herbicides in All Matrices by SW-846 Method 8151A	4b.	Analytics Environmental Laboratory, Portsmouth, NH via subcontract to Amro Environmental Laboratory, Merrimack, NH
5a.	Total Petroleum Hydrocarbon by Gas Chromatography and Diesel Range Organics (DROs): by SW-846 Method 8015B	5b.	AMRO Environmental Laboratories, Merrimack, NH
6a.	Volatile Organics Analysis, SW-846 Method 8260B	6b	AMRO Environmental Laboratories, Merrimack, NH

Table 6-2: Project Sampling Procedures Reference Table

Project Sampling SOPs*
1c. SEI SOP 6.42.2: Geologic Description of Unconsolidated Deposits Using the Wentworth Grain Size Scale
2c. SEI SOP 6.19.2: Use, Maintenance and Calibration of the Ion Science PhoCheck 1000+ Photo Ionization Detector (PID)
3c. SEI SOP 6.45.0: Collection, Handling, and Preservation of Discrete Soil Samples
4c. SEI SOP 5.1.5: Maintenance and Decontamination of Field Equipment

* Project Sampling SOPs include sample collection, sample preservation, equipment decontamination, preventive maintenance, etc.

7. FORM F-2: SAMPLING AND ANALYTICAL METHODS REQUIREMENTS

Table 7-1: Sampling and Analytical Methods Requirements

Parameter	Matrix	Number of Samples (total for project)	Field QC Samples (total for project)	Analytical Method	Sampling SOP*	Containers per sample (number, size and type)	Preservation requirements (temperature, light, chemical)	Maximum Holding Time at Lab from Collection (preparation/ analysis)
TPH-DRO	Soil 26 borings, 26 samples.	26	2 FD	5a.	3c.	1, 4 oz. glass jar	Cool at ≤6ºC	14 Days
PAHs	Soil 26 borings, 26 samples.	26	2 FD	1a.	3c.	1, 4 oz. glass jar	Cool at ≤6ºC	14 days
RCRA 8 Metals	Soil 26 borings, 26 samples.	26	2 FD	2a.	3c.	1, 4 oz. glass jar	Cool at ≤6ºC	28 Days, Hg 180 Days others
PCBs as Aroclors	Soil 26 borings, 26 samples.	26	2 FD	3a.	3c.	1, 4 oz. glass jar	Cool at ≤6ºC	14 days
Herbicides	Soil 26 borings, 26 samples.	26	2 FD	4a.	3c.	1, 4 oz. glass jar	Cool at ≤6ºC	7 Days
VOCs	Soil Contingent on PID response >10 ppm	0	10% of # collected	6a.	3c.	2, 40 mL VOA	10 mL MeOH	14 days

[1]FD: Field Duplicate (5%);

8. FORM G: PREVENTIVE MAINTENANCE—FIELD EQUIPMENT

Preventive Maintenance for Field Equipment is presented in the BA QAPP. All maintenance will be documented in the field notes.

Table 8-1: Preventive Maintenance of Field Equipment

Instrument	Activity	Frequency	SOP Ref.*
Ion Science PhoCheck 1000+	Clean lamp and unit	Monthly or as needed when unit shows evidence of problems	2c.

*Form F-1 contains the SOP Reference Table

9. FORM H: CALIBRATION AND CORRECTIVE ACTION—FIELD EQUIPMENT

Calibration and corrective actions for field equipment are presented in the BA QAPP. More specific details are provided in the field equipment standard operating procedures and/or instruction manuals. Calibrations and corrective actions (if necessary) will be well documented in the field notes.

Table 9-1: Calibration and Corrective Action of Field Equipment

Instrument	Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Ref.*
Ion Science PhoCheck 1000+	Calibration for VOCs with 100 ppm isobutylene standard	At least once daily, more often if response is low	±2%	Rezero and recalibrate with 100 ppm isobutylene	2c.

* Form F-1 contains the SOP Reference Table

10. FORM I: PREVENTIVE MAINTENANCE—LABORATORY EQUIPMENT

As summarized in the BA QAPP, each laboratory's maintenance procedures are addressed in its particular Quality Systems Manual (QSM) and/or Quality Assurance Plan. These manuals are provided as appendices in the BA QAPP or as an attachment to this document.

Table 10-1: Preventative Maintenance of Laboratory Equipment

Instrument	Activity	Frequency	SOP Ref.*
GC/MS	Per manufacturer's instructions and laboratory quality assurance documentation	Per manufacturer's instructions and laboratory quality assurance documentation	N/A
GC/ECD	Per manufacturer's instructions and laboratory quality assurance documentation	Per manufacturer's instructions and laboratory quality assurance documentation	N/A
GC/FID	Per manufacturer's instructions and laboratory quality assurance documentation	Per manufacturer's instructions and laboratory quality assurance documentation	N/A
ICP	Per manufacturer's instructions and laboratory quality assurance documentation	Per manufacturer's instructions and laboratory quality assurance documentation	N/A
CVAA	Per manufacturer's instructions and laboratory quality assurance documentation	Per manufacturer's instructions and laboratory quality assurance documentation	N/A

* Form F-1 contains the SOP Reference Table. GC/MS: Gas Chromatograph/Mass Spectrometer; GC/ECD: Gas Chromatograph/Electron Capture Detector; GC/FID: Gas Chromatograph/Flame Ionization Detector; ICP: Inductively Coupled Plasma Spectrometer; CVAA: Cold Vapor Atomic Adsorption Spectrometer.

11. FORM J: CALIBRATION AND CORRECTIVE ACTION— LABORATORY EQUIPMENT

Calibration of instrumentation is required to ensure that the analytical system is operating correctly and functioning at the proper sensitivity to meet established reporting limits. Each instrument is calibrated with standard solutions appropriate to the type of instrument and the linear range established for the analytical method.

Method-specific SOPs, method references, and the Laboratory Quality Manuals discuss in detail how each instrument is calibrated, including frequency for calibration and re-calibration, and the source or grade of the calibration materials. The range of analyses performed and instrumentation utilized is extensive and the calibration procedures are instrument-specific, varying from analysis to analysis. The calibration procedures for organics usually include an initial system performance check and some type of initial calibration (with a minimum of five calibration standards for most methods) with each analytical series. Ongoing and closing calibration checks are also included in most analytical series. For each type of calibration standard or performance check there are specific criteria to meet before sample analyses begin. These criteria are established in the methodologies as they are written in the referenced texts or by contract specifications.

Table 11-1: Calibration and Corrective Action of Laboratory Equipment

Instrument	Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Ref.*
GC/MS GC/ECD GC/FID	Initial calibration with stock standards	After preventive maintenance or when CV compounds outside criteria	5-6 point calibration correlation coefficient must be -.995 or higher using a linear curve fit; second source standard must be within ±20%	Establish new calibration curve or service instrument	1a
GC/MS GC/ECD GC/FID	Continuing calibration verification with stock standards	Every 12 hours (GC/ECD/FID Every 10 Samples)	Confirm retention times and integrations for each compound; all compounds with an Average Response Factor curve fit must have CV response factor within ±20% drift or calculated percent difference from nominal concentration of ±20%	Establish new calibration curve or service instrument	1a
ICP	Initial instrument calibration	Initiation of each run	Initial calibration standards must be within ±10% recovery; recovery of interference check standards must be within ±20%; the linear dynamic range recovery must be within ±10% linearity; low level calibration checks must be within ±20%	Establish new calibration curve or service instrument	2a
ICP	Continuing calibration verification	Every 10 samples and at the end of each run	Results must agree within ±10% of the expected value; results of calibration blank must be less than reporting limit	Terminate analysis, correct problem, recalibrate, reanalyze previous 10 samples	2a

Instrument	Activity	Frequency	Acceptance Criteria	Corrective Action	SOP Ref.*
CVAA	Initial instrument calibration	Initiation of each run	Initial calibration standards must be within ±10% recovery; low level calibration checks must be within ±20%	Establish new calibration curve or service instrument	2a
CVAA	Continuing calibration verification	Every 10 samples and at the end of each run	Results must agree within ±10% of the expected value; results of calibration blank must be less than reporting limit	Terminate analysis, correct problem, recalibrate, reanalyze previous 10 samples	2a

* Form F-1 contains the Method and SOP Reference Table

12. FORM K: SAMPLE HANDLING AND CUSTODY REQUIREMENTS

Successful analysis depends on the capability to produce valid data and to demonstrate such validity. In addition to proper sample collection and handling, appropriate sample identification and chain of custody procedures are necessary to help support the validity of the data. These standard sample handling and custody requirements are described in detail in the BA QAPP along with a typical chain of custody record. The sample handling and custody will be performed as described in that QAPP and in accordance with Stone Standard Operating Procedures. Sample handling and custody at the receiving laboratory are described in the Laboratory Quality Manual.

Sampling kits will be prepared by the designated laboratory custodian. The sampling kits will be packaged in coolers and will include the appropriate sample containers, preservatives, and trip blanks.

An integral part of field activities will be recordkeeping using Observations and Remarks (O&R) forms.

12.1. Sample Handling and Custody Requirements

Successful analysis depends on the capability to produce valid data and to demonstrate such validity. In addition to proper sample collection and handling, appropriate sample identification and chain of custody procedures are necessary to help support the validity of the data.

Sampling kits will be prepared by the designated laboratory custodian. The sampling kits will be packaged in coolers and will include the appropriate sample containers, preservatives, and trip blanks. Deionized water (or analyte-free water) for field blank preparation shall be obtained from the designated laboratory.

An integral part of field activities will be record keeping using Observations and Remarks (O&R) forms.

Items to be included in the field logbook as applicable are:

- Date and time of site entry
- Description of field activity
- Name of site (facility)
- Weather conditions
- Unusual events
- Personnel at site (e.g., consultants, client, regulatory agencies)
- Description and location of sampling points
- Number and locations of samples collected
- Times of sample collection
- Sample preparation methods and sampling equipment used
- Sample equipment decontamination procedures and when equipment was decontaminated

- Field measurements
- Any deviations from the project work or QA plans, including rationale
- Equipment problems
- Any other relevant field observations

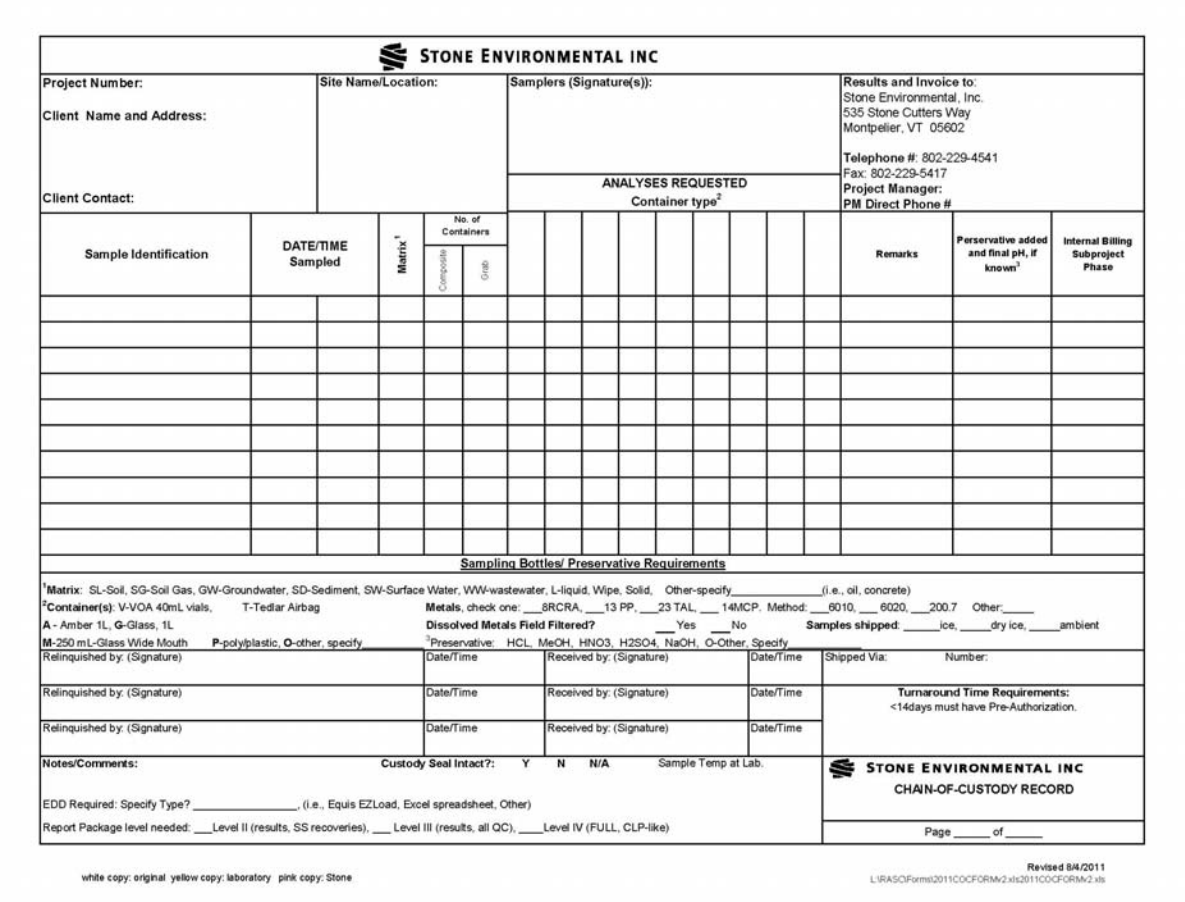
The author's signature and the date of the entry will accompany each page of Observations and Remarks forms. In addition, equipment calibration records and chain of custody records will be prepared and kept as part of the field records. If entries must be changed, the change should not obscure the original entry. Corrections shall be made by drawing a single line through the incorrect entry, inserting the correct information and the reason for the change, and initialing and dating the change.

As samples are collected and containerized in the field, the following information will be recorded on each label:

- Project identification
- Sample location
- Sample number (not to exceed 10 characters)
- Date and time of sample collection
- Parameters to be analyzed
- Initials of sample collector
- Preservatives (if applicable)

A chain of custody record (COC) identifying its contents will accompany each shipping container that goes to the laboratory. Upon sample receipt, the laboratory sample custodian will log the sample in, indicate the condition of each sample as received, and record (on the chain of custody record) whether the custody seal was intact. Additionally, the laboratory will record the temperature of the sample cooler upon receipt at the laboratory on the chain of custody or the laboratory login sheet. Any problems or discrepancies will be reported immediately to the Stone project manager and/or QA manager. The chain of custody forms will be used to identify all persons having contact with the samples. These forms will be kept in a permanent project file at the laboratory and will be available for inspection during normal business hours. Stone will provide the chain of custody form. An example of a chain of custody form is provided as Figure 6.

Sampling kits will be prepared by the designated laboratory custodian. The sampling kits will be packaged in coolers and will include the appropriate sample containers, preservatives, and trip blanks.



STONE ENVIRONMENTAL INC

Project Number: **Client Name and Address:** **Client Contact:**	**Site Name/Location:**	**Samplers (Signature(s)):** **ANALYSES REQUESTED** **Container type[2]**	**Results and Invoice to:** Stone Environmental, Inc. 535 Stone Cutters Way Montpelier, VT 05602 **Telephone #:** 802-229-4541 Fax: 802-229-5417 **Project Manager:** **PM Direct Phone #**

Sample Identification	DATE/TIME Sampled	Matrix[1]	No. of Containers: Composite	No. of Containers: Grab									Remarks	Perservative added and final pH, if known[3]	Internal Billing Subproject Phase

Sampling Bottles/ Preservative Requirements

[1]**Matrix**: SL-Soil, SG-Soil Gas, GW-Groundwater, SD-Sediment, SW-Surface Water, WW-wastewater, L-liquid, Wipe, Solid, Other-specify_____(i.e., oil, concrete)

[2]**Container(s)**: V-VOA 40mL vials, T-Tedlar Airbag, **A** - Amber 1L, **G**-Glass, 1L, **M**-250 mL-Glass Wide Mouth, **P**-poly/plastic, **O**-other, specify_____

Metals, check one: ___8RCRA, ___13 PP, ___23 TAL, ___ 14MCP. Method: ___6010, ___ 6020, ___200.7 Other:_____

Dissolved Metals Field Filtered? ___Yes ___No

[3]Preservative: HCL, MeOH, HNO3, H2SO4, NaOH, O-Other, Specify_____

Samples shipped: _____ice, _____dry ice, _____ambient

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	Shipped Via: Number:
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	**Turnaround Time Requirements:** <14days must have Pre-Authorization.
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	

Notes/Comments: **Custody Seal Intact?:** Y N N/A Sample Temp at Lab.

EDD Required: Specify Type? _____, (i.e., Equis EZLoad, Excel spreadsheet, Other)

Report Package level needed: ___Level II (results, SS recoveries), ___ Level III (results, all QC), ____Level IV (FULL, CLP-like)

STONE ENVIRONMENTAL INC
CHAIN-OF-CUSTODY RECORD

Page _____ of _____

white copy: original yellow copy: laboratory pink copy: Stone

Revised 8/4/2011
L:\RASC\Forms\2011COCFORMv2.xls2011COCFORMv2.xls

Figure 6: Sample Chain of Custody Form.

12.2. Site Sample Identification

Sample identifications will be presented as follows:

MM-IP-##-DD.DD-QA

where:

- MM is Matrix
- IP is Investigation Point
- DD.DD is Depth below ground surface in feet
- QA is Quality Assurance Code

Matrices used for this project will be as follows:

- SL: Soil

Investigation point codes for this project will be as follows:

- SB: Soil Boring;

Quality Assurance codes for this project will be as follows:

- FD: field duplicate;
- EQ: equipment blank;
- TB: trip blank.

13. FORM L: ANALYTICAL PRECISION AND ACCURACY

Table 13-1: Analytical precision and accuracy

Analyte	Analytical Method*	Detection Limit (Units)	Quantitation Limit (Units)	Precision RPD	Accuracy %
TPH-DRO	SW8015B	Soil: 14.172 mg/Kg	Soil: 50 mg/Kg	20	70-130
PAHs	SW8270C SIM PAH only	Soil: 0.003 ug/L	Soil: 0.010 mg/Kg	20	70-130
RCRA 8 Metals	SW6010B SW7471A SW7060	Soil: Metals: 0.08-4 mg/kg Mercury: 0.017 mg/kg	Metals: 0.25-12 mg/kg Mercury 0.05 mg/kg	20	75-125
PCBs as Aroclors	SW8082	Solid: 0.025 mg/kg	Solid: 0.05 mg/Kg	20	50-150
Herbicides	SW8151A	Soil: 2,4,5-T: 0.1 mg/Kg	Soil: 2,4,5-T: 0.1 mg/Kg	20	70-130
		2,4-D: 0.1 mg/Kg	2,4-D: 0.1 mg/Kg	25	40-135
		2,4-DB: 0.1 mg/Kg	2,4-DB: 0.1 mg/Kg	25	40-140
		Dalapon: 0.2 mg/Kg	Dalapon: 0.2 mg/Kg	25	50-140
		DCPA: 0.1 mg/Kg	DCPA: 0.1 mg/Kg	25	40-140
		Dicamba: 0.1 mg/Kg	Dicamba: 0.1 mg/Kg	25	55-110
		Dichlorprop: 0.1 mg/Kg	Dichlorprop: 0.1 mg/Kg	25	75-140
		Dinoseb: 0.1 mg/Kg	Dinoseb: 0.1 mg/Kg	25	40-140
		MCPA: 15 mg/Kg	MCPA: 15 mg/Kg	25	40-140
		MCPP: 15 mg/Kg	MCPP: 15 mg/Kg	25	40-140
		Pentachlorophenol: 0.1 mg/Kg	Pentachlorophenol: 0.1 mg/Kg	25	40-140
		Pichloram: 0.15 mg/Kg	Pichloram: 0.15 mg/Kg	25	40-140
		Silvex: 0.1 mg/Kg	Silvex: 0.1 mg/Kg	25	45-125
VOCs	SW8260B	Soil: VOCs: 12.5 – 625 ug/Kg (1,4 Dioxane)	Soil: VOCs: 25 – 1250 ug/Kg (1,4 Dioxane)	20	70-130

*Note: QA/QC limits in the laboratory may be more stringent and are reflected on the QA/QC summaries for that quality control sample.

14. FORM M: FIELD QUALITY CONTROL REQUIREMENTS

Table 14-1: Field Quality Control

QC Sample	Frequency	Acceptance Criteria	Corrective Action
Field duplicates	5 % per analyte group	Precision 50% soil	Assess sampling and lab precision, qualify data
Cooler temperature blanks	NA-temperature of the coolers should be taken upon receipt at the fixed laboratory.	4˚ C +/- 2ºC	Report to client, if >10ºC, use professional judgment to continue with analysis.

15. FORM M (CONT.): LABORATORY QUALITY CONTROL REQUIREMENTS

Standard fixed and onsite laboratory quality control requirements as described in the method or Laboratory Quality Manuals. Control limits will vary from laboratory to laboratory; Form L provides the base standards.

Table 15-1: Laboratory Quality Control

QC type	Frequency	Methodology	Acceptance Criteria	Corrective Action
method blank	1 per batch	Method blank subjected to same reagents and procedures as a field sample	Target analytes below RL, laboratory limits listed in method	System check, reanalysis of affected samples
LCS	1 per batch	Reagent water or a blank matrix is spiked with a known concentration of analyte(s); purpose is to determine if possible MS/MSD failure was result of matrix issues or other procedural issues	Limits listed in method	Review, reanalyze LCS and associated samples, based on technical judgment.
MS/MSD	1 per batch	Field sample is spiked with known concentration of analyte(s)	Limits listed in Method	Review LCS and recoveries. Report results in narrative regarding matrix affect.
ICV (independent)	After initial calibration	1 standard (ICP) or 3 standards (ICP-MS) or Internal standards 5 point average RF if <15% RSD or Linear reg. If >15% RSD (GC/MS)	%Recovery 70-130, unless otherwise listed in method	Review, reanalyze ICV, check standards, recalibrate if appropriate.
surrogate spike (organic analyses)	each sample, standard, blank		Limits listed in method	Review, reanalyze based on technical judgment

16. FORM N: DATA MANAGEMENT AND DOCUMENTATION

Types of information to request from the laboratory:

- Project Narrative which contains all observation and deviations
- Data Results Sheets (include any performance evaluation sample results)
- Method Blank Results
- Surrogate Recoveries and Acceptance Limits
- Matrix Spike/Matrix Spike Duplicate Results and Acceptance Limits, if applicable
- Spike/Duplicate Results and Acceptance Limits
- Laboratory Control Sample Results and Acceptance Limits

Documentation requirements are described in the BA QAPP.

17. FORM O: ASSESSMENT AND RESPONSE ACTIONS

Stone personnel and the quality assurance manager will review field sampling procedures, field sampling instrumentation, data quality, and project management systems at their discretion at any time during the study to assess the usability and validity of the data. Any reviews such as this will help ensure that the data collection and analysis methods presented in this QAPP will result in the collection data necessary to achieve the goals of this project.

17.1. Assessment and Response Actions

Assessment and response actions (internal and external) are generally implemented during project activities to ensure that elements of the QAPP are correctly implemented and that the quality of the data generated is adequate for project objectives. Section 3.3 describes corrective actions that will be followed if circumstances result in a deviation from approved work plans. Types of assessments may include:

- Management system reviews
- Surveillance
- Performance and Systems Audits
- Audits of data quality
- Data quality assessments

The following sections describe briefly each type of assessment activity listed above. The QA manager, who is organizationally independent of the project management team, usually performs internal audits. Technically qualified personnel of other organizations such as the US EPA or VT DEC, perform external assessments.

17.1.1. Management Systems Review (MSR)

This review consists of a qualitative assessment of organizational and data collection operations to ensure that sufficient management controls are in place and implemented to adequately plan, perform, and assess the results of the project. Stone Management conducts management system reviews annually.

17.1.2. Surveillance

Surveillance is continual or frequent monitoring of project status and records at each level of the organization to ensure that specified requirements are being fulfilled. Surveillance is conducted by the project manager formally with written documentation or informally.

17.1.3. Performance and System Audits

The project manager will monitor and audit the performance of the QA/QC procedures. All information produced or obtained in the course of the field activities is subject to audit at any time and therefore must be reliable, gathered with the appropriate attention to detail and maintained with full integrity in accordance with respective SOPs and laboratory quality assurance systems. If warranted (i.e., if problems are encountered) the

quality assurance manager may conduct a field audit and/or laboratory audit while field and laboratory activities are continuing.

Each laboratory addresses performance and system audits within their quality assurance systems.

17.1.4. Corrective Actions

The achievement and maintenance of the stated data quality objectives will be monitored by a continuous assessment of the work performed. At various times throughout the sampling and analysis program, situations may arise that will require some degree of corrective action, ranging from simple corrections on routine field documentation to systematic problems that necessitate shutting down sample analyses until the problem is identified and corrected. The following describe how situations requiring corrective action are to be handled and documented in both the field and the laboratory for the purposes of this project work.

17.1.5. Field Situations

In the field, numerous situations, such as equipment or instrument malfunction, may occur and require subsequent corrective action. Wherever possible, immediate corrective action should be taken, and must be clearly described in the field logbooks. No other formal documentation is required unless further corrective action is deemed necessary.

Any problem or situation which cannot be solved through immediate corrective action, will fall into the long-term corrective action category. The steps for long-term corrective action are as follows:

- Identification and definition of the problem.
- Investigation and determination of the cause of the problem.
- Determination and implementation of a corrective action to eliminate the problem.
- Verification that the corrective action has eliminated the problem.

The field personnel noting the problem will document it and bring it to the attention of the project manager. The project manager will investigate the situation and determine who will be responsible for implementing the corrective action. The project manager will verify that the corrective action has been taken, appears effective, and, at appropriate later dates, verify that the problem has been resolved. The successfully implemented corrective action must be fully documented by the project manager.

17.1.6. Laboratory Situations

As a result of system audit, a technical audit, or observation of or by laboratory personnel, discrepancies may be found which affect the validity or quality of analytical data. Corrective actions will be implemented to correct the deficiency or weakness and to identify any analytical data that may have been affected. Wherever possible, immediate corrective action procedures shall be employed. Immediate corrective actions taken must be noted in the laboratory logbooks, but no other formal documentation is required unless further corrective action is deemed necessary. The need for long-term corrective action should be addressed in the laboratory quality assurance system.

If any corrective actions taken affect the quality and integrity of the data generated during a specific site investigation then it is the responsibility of the laboratory to address the impact on the data generated and inform the project manager in writing.

18. FORM P: PROJECT REPORTS

Requirements for project reports are described in the BA QAPP. There are no changes for this Phase II ESA.

19. FORM Q-1: VERIFICATION OF SAMPLING PROCEDURES

Requirements for verification of sampling procedures are described in the BA QAPP. There are no changes for this Phase II ESA.

20. FORM Q-2: DATA VERIFICATION AND VALIDATION

Requirements for data verification and validation are described in the BA QAPP. There are no changes for this Phase II ESA.

21. FORM R: DATA USABILITY

21.1. Data Review, Verification, and Validation

Upon inspection by the data user and Stone of the field-collected and laboratory analytical data, the data are accepted for the study unless there is a noted occurrence of field instrumentation malfunction, or a laboratory note indicating that the required analysis was not performed in accordance with the SOP and/or one or more of the criteria associated with the particular method. These conditions will be clearly noted within field data collection notes and on laboratory analytical reports. Data will be reviewed and evaluated using the data quality objectives noted above and will be deemed usable for the overall study objectives. If a data point were deemed unusable the data would be flagged and noted as such.

21.2. Verification and Validation Requirements

The quality assurance manager and/or the designee will be responsible for verifying and validating all chain of custody forms and laboratory analysis data, while the sampling team under the direction of Stone is responsible for the verification and validation of measurements taken in the field. Chain-of-custody procedures are described in Section 12.1. Results will be conveyed to data users in the form of NELAC reporting format and spreadsheets.

Project participants are responsible for verifying that all deliverables submitted to Stone are consistent with the QAPP. Any deviations from the criteria specified in the QAPP must be disclosed to Stone at the time of submission.

Analytical chemistry data will be reviewed according to Region I, EPA-NE Data Validation Functional Guidelines for Evaluating Environmental Analysis (EPA, 1998) as described in Section 4.5. One hundred percent of the analytical data will be subjected to review modeled after the EPA Tier I guideline (EPA, 1998). The Tier I review will include a review of completeness. In addition, this review will compare selected QC parameters and data objectives with the acceptance criteria described in the QAPP. If necessary and appropriate as addressed by the laboratory analytical data that require changes to the analyte concentrations reported by the laboratory (e.g., analytes that are rejected or changed to “non-detect at the reporting limit” with an elevated reporting limit due to blank contamination) will be explained in the data validation narrative.

Qualifiers may be applied to data that fails to satisfy the acceptance criteria detailed in this QAPP. In addition to the laboratory data qualifiers detailed in the SOPs, Stone may use the following data qualifiers as outlined in the Region I validation guidelines:

> U – The analyte was analyzed for, but was not detected. The associated numerical value is the sample quantitation limit. The sample quantitation limit accounts for sample specific dilution factors and percent solids corrections or sample sizes that deviate from those required by the method.
>
> J – The associated numerical value is an estimated quantity.
>
> UJ – The analyte was analyzed for, but was not detected. The sample quantitation limit is an estimated quantity.

R – The data are unusable (analyte may or may not be present). Resampling and reanalysis is necessary for verification. The R replaces the numerical value or sample quantitation limit. In some instances (e.g., a dilution) a result may be indicated as "rejected" to avoid confusion when a more quantitatively accurate result is available.

JN – The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value is an estimated quantity.

EB, TB, BB – An analyte that was identified in an aqueous equipment (field) blank, trip blank, or bottle blank that was used to assess field contamination associated with soil/sediment samples. These qualifiers are to be applied to soil/sediment sample results only.

21.3. Reconciliation with User Requirements

At various times throughout the planned project, situations may arise that will require some degree of corrective action or reconciliation of data, ranging from simple corrections on routine field documentation to systematic problems that may necessitate shutting down a process until the problem is corrected. Described below are how situations requiring reconciliation are to be handled and documented in both the field and the laboratory for the purposes of this project.

Any or all deviations from stated work plans and this QAPP will be reconciled with VT DEC. Reconciliations will be documented as a memorandum to the project file with copies being sent (if appropriate) to all individuals noted on Form A. If there are limitations regarding the use of data these will be documented as such and reported to the project team.

21.3.1. Field Situations

In the field, numerous situations, such as equipment or instrument malfunction, may occur and require subsequent corrective action. Wherever possible, immediate corrective action should be taken, and clearly described on the Observations and Remarks form, but no formal documentation is required unless further corrective action is deemed necessary. Field personnel noting the problem will document it and bring it to the attention of Stone project manager. Reconciliation of the situation will be fully documented by the project manager.

21.3.2. Laboratory Situations

The samples are analyzed in conjunction with quality control samples. If all data are within quality control specifications, the data and the quality control information are reported to the client in the laboratory's standard format. If an out-of-control situation exists, the laboratory supervisor is notified and the problem is resolved. Stone is notified of all such occurrences in writing. If unusual or unexpected results are obtained on the project, Stone's project manager is notified, and a course of action is chosen.

Unless otherwise noted, all data validated using the methods noted above will be considered suitable for use in meeting the objectives of this study.

21.4. Data Recording and Transformation

An integral part of project activities will require the recording and transformation of data from logbooks. Personnel transcribing data will keep detailed records of their activities and document their method of data

collection. Care must be taken when recording or transcribing data from one media to another. Data review will be completed for each data set and an Observation and Remarks Sheet will be written to document the review process. Particular attention must be given to transcribing data units associated with particular data sets.

21.5. Data Usability

Data usability assessment activities are documented by the laboratory and by the project manager or his/her designee. As noted above the quality assurance manager or designee will evaluate data as described in this QAPP in order to determine the effect on the overall project objectives.

After data validation and verification, all data with the exception of rejected data will be used for project decisions. Rejected data will not be used for project activities.

APPENDICES

APPENDIX A: HISTORICAL FIGURES

Figure A.1: Historical Infrastructure

Figure A.2: Former Parcels

Figure A.3: Water Table Elevation Contour Map 8/26/1999

Figure A.4: Historic Uses / Features o Concern

Figure A.5: Previous Soil Testing Locations

Figure A.6: Surface Soils with PID Readings >20 ppm v/v

Figure A.7: VOC Sampling Results – Surface Soils

Figure A.8: PAH Screening Results – Surface Soils

Figure A.9: PAH Analytical Results – Surface Soils

Figure A.10: Arsenic Analytical Results – Surface Soils

Figure A.11: Subsurface Soils with PID Readings >20 ppm v/v

Figure A.12: VOC Exceedances of VGES in Shallow Groundwater

Figure A.13: Metals Exceedances of VGES in Shallow Groundwater

Figure A.14: Surface Soil Exposure Risk Areas



Legend

- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Historic Site Features
- Existing Site Features
- Bike Path
- Retaining Wall
- Historic Petroleum AST
- Historic building

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Enclosed areas surrounding ASTs represent earthen berms.

STONE ENVIRONMENTAL INC

TITLE: Historic Infrastructure

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A1

DATE: 10/31/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Bike Path
- Retaining Wall

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

STONE ENVIRONMENTAL INC

TITLE: Former Parcels

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A2

DATE: 10/31/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

100 Relative Water Table Elevation Contour (ft)

Urban Reserve Study Area Boundary Line

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Locations highlighted represent VOC concentrations in groundwater above VGES.

STONE ENVIRONMENTAL INC

TITLE: Water Table Elevation Contour Map: 8/26/1999

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A3

DATE: 5/23/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

- Urban Reserve Study Area Boundary Line
- Former Rail Line
- Current Rail Line
- Former Gasoline AST
- Former Fuel Oil AST
- Former Lumber Mill/Storage Area Boundary
- Former PAH-Contaminated Soil Pile
- Former Refrigerator Company Use Area
- Former UST Area Boundary

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

STONE ENVIRONMENTAL INC

TITLE: Historic Uses / Features of Concern

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A4

DATE: 11/5/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

- BH-1 ⊕ Boring Locations (1999)
- HA-1 ○ Hand Auger Locations (1999)
- ⊠ Approx. Location of Weston Test Pit (1986)
- ML#33 ⊗ H&N Hand Auger Locations (1997)

- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Area with Imported Fill Placed After Soil Testing
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

STONE ENVIRONMENTAL INC

TITLE: Previous Soil Testing Locations

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A5

DATE: 10/31/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

- BH-1 ⊕ Boring Locations (1999)
- HA-1 ○ Hand Auger Locations (1999)
- ⊠ Approx. Location of Weston Test Pit (1986)
- ML#33 ⊗ H&N Hand Auger Locations (1997)

- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Area with Imported Fill Placed After Soil Testing
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Samples collected in 1997 or 1999.

Locations highlighted in red had surface soils with PID readings >20 ppm v/v.

STONE ENVIRONMENTAL INC

TITLE: Surface Soils with PIDReadings >20 ppm v/v

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A6

DATE: 5/23/12 DRAWN BY: DJL CHECKED BY: EJS



Legend

- BH-1 ⊕ Boring Locations (1999)
- HA-1 ○ Hand Auger Locations (1999)
- ⊠ Approx. Location of Weston Test Pit (1986)
- ML#33 ⊗ H&N Hand Auger Locations (1997)
- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Area with Imported Fill Placed After Soil Testing
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Only sampling locations where VOCs were analyzed via EPA Method 8260 are displayed. No VOCs exceeded the Vermont Soil Screening Values.

STONE ENVIRONMENTAL INC

TITLE: VOC Sampling Results - Surface Soil

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A7

DATE: 10/31/12 DRAWN BY: DJL CHECKED BY: EJS



Legend

- BH-1 ⊕ Boring Locations (1999)
- HA-1 ○ Hand Auger Locations (1999)
- ⊠ Approx. Location of Weston Test Pit (1986)
- ML#33 ⊗ H&N Hand Auger Locations (1997)
- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Area with Imported Fill Placed After Soil Testing
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Only sampling locations where PAHs were analyzed via the Hanby Test Kit are displayed. Locations highlighted in red exceed the Vermont Soil Screening Value for TPH of 200 ppm (for a residential setting).

STONE ENVIRONMENTAL INC

TITLE: PAH Screening Results - Surface Soils

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A8

DATE: 10/31/12 DRAWN BY: DJL CHECKED BY: EJS



Legend

- BH-1 ⊕ Boring Locations (1999)
- HA-1 ○ Hand Auger Locations (1999)
- ⊠ Approx. Location of Weston Test Pit (1986)
- ML#33 ⊗ H&N Hand Auger Locations (1997)
- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Area with Imported Fill Placed After Soil Testing
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Only sampling locations where PAHs were analyzed via EPA Method 8270 are displayed. Locations highlighted in red exceed the Vermont Soil Screening Value for one or more parameter.

STONE ENVIRONMENTAL INC

TITLE: PAH Analytical Results - Surface Soils

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A9

DATE: 10/31/12 DRAWN BY: DJL CHECKED BY: EJS



Legend

- BH-1 ⊕ Boring Locations (1999)
- HA-1 ○ Hand Auger Locations (1999)
- ⊠ Approx. Location of Weston Test Pit (1986)
- ML#33 ⊗ H&N Hand Auger Locations (1997)

- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Area with Imported Fill Placed After Soil Testing
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Only sampling locations where arsenic was analyzed are displayed. Locations highlighted in red exceed the Vermont Soil Screening Value.

STONE ENVIRONMENTAL INC

TITLE: Arsenic Analytical Results - Surface Soil

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A10

DATE: 10/31/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

- BH-1 Boring Locations (1999)
- HA-1 Hand Auger Locations (1999)
- Approx. Location of Weston Test Pit (1986)
- ML#33 H&N Hand Auger Locations (1997)
- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Area with Imported Fill Placed After Soil Testing
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Samples collected in 1997 or 1999.

Locations highlighted in red had subsurface soils with PID readings >20 ppm v/v.

STONE ENVIRONMENTAL INC

TITLE: Subsurface Soils with PID Readings >20ppm v/v

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A11

DATE: 5/23/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

- MW-1 — Monitoring Wells
- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Former Site Features
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Groundwater samples from monitoring wells highlighted in red contained one or more VOC at concentrations above the VGES.

STONE ENVIRONMENTAL INC

TITLE: VOC Exceedances of VGES in Shallow Groundwater

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A12

DATE: 10/31/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

- MW-1 — Monitoring Wells
- Urban Reserve Study Area Boundary Line
- Approximate Boundary of Former Parcels
- Existing Site Features
- Former Site Features
- Bike Path

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

Groundwater samples from monitoring wells highlighted in red contained one or more heavy metal at concentrations above the VGES.

STONE ENVIRONMENTAL INC

TITLE: Metals Exceedances of VGES in Shallow Groundwater

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A13

DATE: 10/31/12

DRAWN BY: DJL

CHECKED BY: EJS



Legend

- Urban Reserve Study Area Boundary Line
- Area with Known Exposure Risk
- Area with Potential Exposure Risk / Lack of Data
- Former Rail Line / Bike Path (No Current Risk)
- Retaining Wall
- Area with No Current Exposure Risk

Note:

Building footprints and features digitized by Stone Environmental, Inc. based on Urban Reserve, Burlington VT site plan by Lamoureux & Dickinson (Phase II, May 2000).

All locations and dimensions are approximate. No claim is made to accuracy. This plan should not be used for construction purposes, property line determination, exact location of site features etc.

STONE ENVIRONMENTAL INC

TITLE: Surface Soil Exposure Risk Areas

PROJECT: Urban Reserve- Burlington, VT

FIGURE NO.: A14

DATE: 10/31/12

DRAWN BY: DJL

CHECKED BY: EJS

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APPENDIX B: CONTAMINANTS OF CONCERN TABLES

Table B.1: Site Metals Contaminants of Concern Quantitation, and Relevant Action Limits

Table B.2: Site PCBs Contaminants of Concern Quantitation, and Relevant Action Limits

Table B.3: Site PAHs Contaminants of Concern Quantitation, and Relevant Action Limits

Table B.4: Site Herbicides Contaminants of Concern Quantitation, and Relevant Action Limits

Table B.5: TPH Quantitation, and Relevant Action Limits

Table B.6: Site VOC Contaminants of Concern Quantitation, and Relevant Action Limits

Table B.1: Site Metals Contaminants of Concern Quantitation, and Relevant Action Limits

Contaminants of Concern, Matrix	Extraction Technique	Analytical Technique	Compound	Method Detection Limits (mg/kg)	Practical Quantitation Limits (mg/kg)	VT Action Limits[1] (mg/kg)
RCRA 8 Metals, Soil	**EPA SW3051**	**SW-7060**	Arsenic	0.47	5	0.39
		SW-846-6010B	Barium	0.20	10	1500
		SW-846-6010B	Cadmium	0.015	0.5	70
		SW-846-6010B	Chromium	0.047	1	0.29
		SW-846-6010B	Lead	0.49	4	400
		SW-7471A	Mercury	0.00241	0.05	10
		SW-846-6010B	Selenium	1.9	12	390
		SW-846-6010B	Silver	0.96	1.4	390

NS: No standard for this compound; [1]VT Soils Action Levels (EPA Regional Screening Levels [RSL]USEPA), June 2011

Table B.2: Site PCBs Contaminants of Concern Quantitation, and Relevant Action Limits

Contaminants of Concern, Matrix	Extraction Technique	Analytical Technique	Compounds	Method Detection Limits (mg/kg)	Practical Quantitation Limits (mg/kg)	VT Action Limits[1] (mg/kg)
PCBs, Soils	**EPA SW3540**	**GC/ECD EPA SW8082**	Aroclor 1016	0.025	0.05	3.9
			Aroclor 1221	0.025	0.05	0.14
			Aroclor 1232	0.025	0.05	0.14
			Aroclor 1242	0.025	0.05	0.22
			Aroclor 1248	0.025	0.05	0.22
			Aroclor 1254	0.025	0.05	0.22
			Aroclor 1260	0.025	0.05	0.22
			Aroclor 1262	0.025	0.05	NS
			Aroclor 1268	0.025	0.05	NS

NS: No standard for this compound; [1] PCBs Soils: VT Soils Action Levels (EPA Regional Screening Levels [RSL]USEPA)

Table B.3: Site PAHs Contaminants of Concern Quantitation, and Relevant Action Limits

Contaminants of Concern, Matrix	Extraction Technique	Analytical Technique	Compounds	Method Detection Limits (mg/kg)	Practical Quantitation Limits (mg/kg)	VT Action Limits[1] (mg/kg)
PAHs, Soil	SW3541	GC/MS, EPA 8270C by SIM	2-Methylnaphthalene:	0.025	0.01	310
			Acenaphthene:	0.025	0.01	3400
			Acenaphthylene:	0.025	0.01	NS
			Anthracene:	0.025	0.01	17000
			Benz(a)anthracene:	0.025	0.01	0.15
			Benzo(a)pyrene:	0.025	0.01	0.015
			Benzo(b)fluoranthene:	0.025	0.01	0.15
			Benzo(g,h,i)perylene:	0.025	0.01	NS
			Benzo(k)fluoranthene:	0.025	0.01	1.5
			Chrysene:	0.025	0.01	15
			Dibenz(a,h)anthracene:	0.025	0.01	0.015
			Fluoranthene:	0.025	0.01	2300
			Fluorene:	0.025	0.01	2300
			Indeno(1,2,3-cd)pyrene:	0.025	0.01	0.15
			Naphthalene:	0.025	0.01	3.6
			Phenanthrene:	0.025	0.01	NS
			Pyrene:	0.025	0.01	1700

NS: No standard for this compound; [1]VT Soils Action Levels (EPA Regional Screening Levels [RSL]USEPA), June 2011

Table B.4: Site Herbicides Contaminants of Concern Quantitation, and Relevant Action Limits

Contaminants of Concern, Matrix	Extraction Technique	Analytical Technique	Compound	Method Detection Limits (mg/kg)	Practical Quantitation Limits (mg/kg)	VT Action Limits[1] (mg/kg)
Chlorinated Herbicides, Soil	**SW 3550C**	**GC EPA 8151A**	Trichlorophenoxyacetic Acid, 2,4,5-	0.1	0.1	610
			Dichlorophenoxy Acetic Acid, 2,4-	0.1	0.1	690
			Dichlorophenoxy)butyric Acid, 4-(2,4-	0.1	0.1	490
			Dalapon	0.2	0.2	1800
			DCPA	0.1	0.1	NS
			Dicamba	0.1	0.1	1800
			Dichlorprop	0.1	0.1	NS
			Dinoseb	0.1	0.1	61
			MCPA	15	15	NS
			MCPP	15	15	NS
			Pentachlorophenol	0.1	0.1	3
			Picloram	0.15	0.15	4300
			Trichlorophenoxy) Propionic Acid, 2(2,4,5-	0.1	0.1	490

NS: No standard for this compound; [1]VT Soils Action Levels (EPA Regional Screening Levels [RSL]USEPA)

Table B.5: Site TPH Contaminants of Concern Quantitation, and Relevant Action Limits

Contaminants of Concern, Matrix	Extraction Technique	Analytical Technique	Compounds	Method Detection Limits (mg/kg)	Practical Quantitation Limits (mg/kg)	VT Action Limits (mg/kg)
TPH, Soil		**GC/MS EPA SW8015B DRO**	Diesel Range Organics	50	50	NA

Table B.6: Site VOC Contaminants of Concern Quantitation, and Relevant Action Limits

Contaminants of Concern, Matrix	Extraction Technique	Analytical Technique	Compounds	Method Detection Limits (mg/kg)	Practical Quantitation Limits (mg/kg)	VT Action Limits[1] (mg/kg)
VOCs in Soil	EPA SW Method 5035	EPA SW Method 8260	1,1,1,2-Tetrachloroethane	0.0125	0.025	1.9
			1,1,1-Trichloroethane	0.0125	0.025	8700
			1,1,2,2-Tetrachloroethane	0.0125	0.025	0.56
			1,1,2-Trichloroethane	0.0125	0.025	1.1
			1,1-Dichloroethane	0.0125	0.025	3.3
			1,1-Dichloroethene	0.0125	0.025	240
			1,1-Dichloropropene	0.0125	0.025	NS
			1,2,3-Trichlorobenzene	0.0125	0.025	49
			1,2,3-Trichloropropane	0.0125	0.025	5000
			1,2,4-Trichlorobenzene	0.0125	0.025	22
			1,2,4-Trimethylbenzene	0.0125	0.025	62
			1,2-Dibromo-3-chloropropane	0.05	0.125	0.0054
			1,2-Dibromoethane	0.0125	0.025	0.034
			1,2-Dichlorobenzene	0.0125	0.025	1900
			1,2-Dichloroethane	0.0125	0.025	0.43
			1,2-Dichloropropane	0.0125	0.025	0.94
			1,3,5-Trichlorobenzene	0.0125	0.05	NS
			1,3,5-Trimethylbenzene	0.0125	0.025	780
			1,3-Dichlorobenzene	0.0125	0.025	1900
			1,3-Dichloropropane	0.0125	0.025	1600
			1,4-Dichlorobenzene	0.0125	0.025	2.4
			1,4-Dioxane	0.625	1.25	4.9
			2,2-Dichloropropane	0.0125	0.025	NS
			2-Butanone	0.125	0.25	28000
			2-Chloroethyl vinyl ether	0.05	0.25	NS
			2-Chlorotoluene	0.0125	0.025	1600
			2-Hexanone	0.125	0.25	210
			4-Chlorotoluene	0.0125	0.025	1600
			4-Isopropyltoluene	0.0125	0.025	NS
			4-Methyl-2-pentanone	0.125	0.25	5300
			Acetone	0.125	0.25	61000
			Acrylonitrile	0.0125	0.025	870
			Benzene	0.0125	0.025	1.1
			Bromobenzene	0.0125	0.025	300
			Bromochloromethane	0.0125	0.025	160
			Bromodichloromethane	0.0125	0.025	0.27
			Bromoform	0.025	0.05	62
			Bromomethane	0.025	0.05	7.3
			Carbon disulfide	0.025	0.05	820
			Carbon tetrachloride	0.0125	0.025	0.61
			Chlorobenzene	0.0125	0.025	290
			Chloroethane	0.025	0.05	NS
			Chloroform	0.0125	0.025	0.29
			Chloromethane	0.025	0.05	120
			cis-1,2-Dichloroethene	0.0125	0.025	160
			cis-1,3-Dichloropropene	0.0125	0.025	1.7
			Cyclohexanone	0.0125	0.025	7000
			Dibromochloromethane	0.0125	0.025	0.68
			Dibromomethane	0.0125	0.025	5.4
			Dichlorodifluoromethane	0.0125	0.05	94

Contaminants of Concern, Matrix	Extraction Technique	Analytical Technique	Compounds	Method Detection Limits (mg/kg)	Practical Quantitation Limits (mg/kg)	VT Action Limits[1] (mg/kg)
			Dichlorofluoromethane	0.0125	0.025	NS
			Diethyl ether	0.125	0.25	16000
			Diisopropyl ether	0.025	0.05	2400
			Ethyl Tertiary Butyl Ether	0.0125	0.05	NS
			Ethylbenzene	0.0125	0.025	5.4
			Freon-113	0.0125	0.025	43000
			Hexachlorobutadiene	0.025	0.05	6.2
			Iodomethane	0.0125	0.025	NS
			Isopropylbenzene	0.0125	0.025	NS
			m,p-Xylene	0.0125	0.025	590
			Methyl methacrylate	0.0125	0.025	4800
			Methyl tert-butyl ether	0.0125	0.025	43
			Methylene chloride	0.025	0.05	11
			n-Butylbenzene	0.0125	0.025	3900
			n-Propylbenzene	0.0125	0.025	3400
			Naphthalene	0.025	0.05	3.6
			o-Xylene	0.0125	0.025	690
			sec-Butylbenzene	0.0125	0.025	NS
			Styrene	0.0125	0.025	6300
			tert-Butylbenzene	0.0125	0.025	NS
			Tertiary Amyl Methyl Ether	0.0125	0.05	43
			Tertiary Butanol	0.25	0.5	NS
			Tetrachloroethene	0.0125	0.025	0.55
			Tetrahydrofuran	0.125	0.25	NS
			Toluene	0.0125	0.025	5000
			trans-1,2-Dichloroethene	0.0125	0.025	150
			trans-1,3-Dichloropropene	0.0125	0.025	1.7
			trans-1,4-Dichloro-2-butene	0.0125	0.025	0.0069
			Trichloroethene	0.0125	0.025	2.8
			Trichlorofluoromethane	0.025	0.05	790
			Vinyl acetate	0.05	0.25	970
			Vinyl chloride	0.0125	0.025	0.06

NS: No standard for this compound; [1]VT Soils Action Levels (EPA Regional Screening Levels [RSL]USEPA)

APPENDIX C: STANDARD OPERATING PROCEDURES

NOTE: SOPs for this QAPP are included here. The approved hardcopy versions of the SOPs are filed on site at Stone Environmental's facility in Montpelier, Vermont. Approved versions of SOPs are available upon request as they are controlled documents.

As with all Stone SOPs, they may contain valuable, confidential, and proprietary information; therefore reproduction of these materials without written authorization by Stone is prohibited.

STANDARD OPERATING PROCEDURE

SEI-5.1.5

MAINTENANCE AND DECONTAMINATION OF FIELD EQUIPMENT

SOP Number: SEI-5.1.5
Revision Number: 5

Date Issued: 04/09/93
Date of Revision: 02/20/04

1.0 OBJECTIVE

Proper maintenance and decontamination of field equipment is an essential quality control measure and a safety precaution to ensure sampling equipment is in proper working condition and to remove any chemical or material contamination. The procedure prevents cross-contamination between sampling stations and helps maintain a clean working environment for the safety of all field personnel. Equipment decontamination helps prevent sampling bias. Maintenance records of equipment that "generates, measures, or assesses data" is required under the Good Laboratory Practices (GLPs) and is an important element of a compliant and scientifically sound operation. This standard operating procedure (SOP) defines the difference between maintenance of equipment and general decontamination procedures and sets forth the guideline SEI uses in determining if equipment records are required per Subpart D of 40 CFR Part 160. This SOP describes a general procedure for decontaminating a sampling device, more specific maintenance requirements and decontamination procedures may be described in SOPs specific to each type of equipment.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart D, Section 160.61, Equipment used in the generation, measurement, or assessment of data and equipment used for facility environmental control shall be of appropriate design and adequate capacity to function according to the protocol and shall be suitable located for operation, inspection, cleaning, and maintenance.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. All data generated during the conduct of a study, except those that are generated by automated data collection systems, shall be recorded directly, promptly, and legibly in indelible ink.
4. All data entries shall be dated on the day of entry and signed or initialed by the person entering the data. If multiple personnel enter data upon a single page, each entry should be uniquely attributed to its author.
5. Personnel will document the procedures performed in accordance with Good Laboratory Practices (GLP) Standards, 40 CFR Part 160.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. If necessary and appropriate, all chemicals are required to be received with a Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment types And Maintenance and Calibration Requirements

The majority of SEI's field equipment does not directly generate, measure or assess data and thus does not require the full documentation as described in Subpart D of the GLPs regarding written record keeping of all inspections, maintenance, testing, calibration or standardization operations.

Examples of exempt equipment include, but are not limited to:

1. Hand augers
2. Pump controllers/compressors
3. Generators
4. General hand tools (shovels, hammers, etc.)
5. Well pumps (bladder, electric submersible, etc)
6. Sampling Tools (bailers, dipping tools)

Maintenance documentation may be kept for this type of equipment to assist with inventory or the general condition of the equipment, but the records may not include all items required under 40 CFR Part 160, Subpart D.

Examples of equipment which fall under 40 CFR Part 160, Subpart D include, but are not limited to:

1. pH, conductivity meters.
2. Water level indicators.
3. Troll datalogger (for slug tests, etc.)
4. Guelph permeameter.
5. Scales (balances)

6. Flow meters.

7. Gas Chromatographs

For equipment, which falls under 40 CFR Part 160, Subpart D, at a minimum, the following records must be maintained:

1. An SOP must be written which details the methods, materials and schedules for the routine inspection, cleaning, maintenance, testing, and calibration for the equipment. Details will also include remedial action that will be taken in the event of failure or malfunction of the equipment. The SOP will include (by title if appropriate) the person responsible for maintenance of the equipment (both routine and non-routine).

2. Records will be maintained of all inspection, maintenance, testing, and calibrating operations. These records will contain, at a minimum:

    a. Dates of operation.

    b. Whether maintenance was routine or non-routine.

    c. Whether maintenance followed written SOPs.

3. If non-routine repairs are performed, at a minimum, the following will be documented in addition to the items in 2 above:

    a. Nature of the defect.

    b. How and when the defect was discovered.

    c. Any remedial or correction action taken in response to the defect.

Inspection, maintenance, testing and calibration efforts will be documented. In general, a checklist form or a maintenance logbook, specifically developed for each piece of equipment, should be used to document these procedures. This form or logbook should prompt the user for the maintenance, etc. to be performed. If a form or logbook is not available, actions must still be documented on an appropriate medium. Forms for equipment must be taken to the field for use. Once field efforts are complete, the forms should be returned to SEI's main office for filing. All original equipment maintenance forms will be retained in a filing cabinet. Copies may be made for inclusion in data packages at the request of a client. Records shall be maintained on each major item of equipment and all reference materials significant to the test performed. As indicated above these records shall include documentation on all routine and non-routinge maintenance activities and reference material verification. If available, the records shall include:

1. the name of the item of equipment;

2. The manufacturer's name, type identification, and serial number or other unique identification;

3. Date received and date place in service (if available);

4. Current location; where appropriate;

5. Condition when received (e.g., new, used, reconditioned);

6. Copy of the manufacturer's instructions,

7. Dates and results of calibrations and/or verifications and date of the next calibration and/or verification;

8. Details of maintenance carried out to date and planned for the future; and,

9. History of any damage, malfunction, modification or repair

10. Equipment which is malfunctioning and has been taken out of service, it shall be tagged as such.

If a piece of equipment is dedicated to a sampling site/study, the maintenance forms or logbook may be retained with that equipment until complete or the fieldwork is finished. Once either of these criteria has been fulfilled, the form should be filed in the project file or the general equipment file as noted above.

4.2 Support Equipment

The following are the calibration and maintenance requirements for laboratory analytical support equipment. This applies to all devices that may not be actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices (including thermometers and thermistors, thermal/pressure sample preparation devices and volumetric dispensing devices (such as Eppendorf®, or automatic dilutor/dispensing devices) if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume

1. All support equipment shall be maintained in proper working order. The records of all repair and maintenance activities including service calls shall be kept.

2. All support equipment shall be calibrated or verified at least annually, using NIST traceable references when available, over the entire range of use. The results of such calibration shall be within the specifications required of the application for which this equipment is used or:

    a. The equipment shall be removed from service until repaired; or

b. The laboratory shall maintain records of established correction factors to correct all measurements

3. Raw data records shall be retained to document equipment performance.

4. Prior to use on each working day, balances, ovens, refrigerators, freezers, and water baths shall be checked in the expected use range, with NIST traceable references where available. The acceptability for use or continued use shall be according to the needs of the analysis or application for which the equipment is being used.

5. Mechanical volumetric dispensing devices including burettes (except Class A glassware) shall be checked for accuracy on at least a quarterly use bases. Glass micro liter syringes are to be considered in the same manner as Class A glassware, but must come with a certificate attesting to established accuracy or the accuracy must be initially demonstrated and documented by the laboratory

6. For chemical tests, the temperature, cycle time, and pressure of each run auto claves must be documented by the use of appropriate chemical indicators or temperature recorders and pressure gauges.

4.3 Equipment Decontamination and Personnel

Decontamination of equipment does not constitute maintenance, but is a specific step required for the use of equipment between sampling points. Decontamination is a requirement for use of the equipment but does not effect whether the equipment is in working order. Improper decontamination may effect the quality or integrity of a sample, but does not effect the device collecting the sample. Thus, decontamination is not an action, which must be documented per 40 CFR Part 160, Subpart D.

Decontamination of sampling equipment refers to the physical and chemical steps taken to remove any chemical or material contamination. Equipment decontamination helps prevent sampling bias. All equipment that comes in contact with the sampled material should be free of components that could influence (contaminate) the true physical or chemical composition of the material. Besides the equipment used to collect the samples, any containers or equipment used for sample compositing or for field sub sampling should be free of contamination.

Equipment decontamination also prevents cross-contamination of samples when the equipment is used to collect more than one sample. Disposable equipment or the use of dedicated equipment provides the most effective means of avoiding cross-contamination; however, the use of such equipment is not always practical.

You should decontaminate equipment to a level that meets the minimum requirements for your data collection effort. Your decontamination steps (e.g., use of solvents versus use of only soap and water), therefore, should be selected based on the constituents present, their concentration levels in the waste

or material sampled, and their potential to introduce bias in the sample analysis results if not removed from the sampling equipment. You should describe the project specific decontamination procedures in your planning document for the sampling effort. In addition, items used to clean the equipment, such as bottlebrushes, should e free of contamination.

The following procedures is an example of one you could use to decontaminate sampling device to be used for collecting samples for trace organic or inorganic constituent analyses (from USEPA 1996):

1. Clean the device with tap water and soap, using a brush if necessary to remove particulate matter and surface films.

2. Rinse thoroughly with tap water.

3. Rinse thoroughly with analyte- or organic-free water.

4. If necessary and appropriate, rinse thoroughly with solvent. Do not solvent-rinse PVC or plastic items.

5. Rinse thoroughly with organic/analyte free water, or allow equipment to dry completely.

6. Remove the equipment from the decontamination area. Equipment stored overnight should be wrapped in aluminum foil and covered with clean, unused plastic.

The specifications for cleaning materials are as follows (you should justify and document the use of substitutes):

- "Soap" should be phosphate-free laboratory detergent such as Liquinox®. It must be kept in a labeled, clean, plastic, metal, or glass containers until used and poured directly from the container when in use.

- "Solvent" should be pesticide-grade isopropanol. It must be stored in the unopened original containers until used. It may be applied using the low-pressure nitrogen system fitted with Teflon nozzle, or using Teflon® squeeze bottles. For equipment highly contaminated with organics (such as oily wastes), a laboratory-grade hexane may be more suitable alternative to isopropanol or for volatile organic contaminates purge and trap grade methanol may be more appropriate.

- "Tap Water" may be used from any municipal water treatment system. Use of an untreated potable water supply is not an acceptable substitute unless the source has been determined to be free of contaminates of interest. Tap water may be kept in clean tanks, hand pressure sprayers, squeeze bottles, or applied directly form a hose of tap.

- "Analyte free water (deionized water) is tap water treated by passing through a standard deionizing resin column. At a minimum, it must contain no detectable heavy metals or other inorganic compounds as defined a standard ICP (or equivalent scan. It may be obtained by other methods as long as it meets the analytical criteria. Analyte free water must be stored in clean glass, stainless steel, or plastic containers that can be closed prior to use. IT can be applied from plastic squeeze bottles.

- "Organic/analyte free water" is tap water that has been treated with activated carbon and deionizing units. A potable system to produce such water under field conditions is available. At a minimum, the water must meet the criteria of analyte free water and not contain detectable pesticides, herbicides, or extractable organic compounds, and no volatile organic compounds above minimum detectable levels as determined for a given set of analyses. Organic/analyte free water obtained by other methods is acceptable, as long as it meets the analytical criteria. It may be applied using Teflon® squeeze bottles or with the portable system.

Clean the field equipment prior to field use. Designate a decontamination zone at the site and, if necessary, construct a decontamination pad at a location free of surface contamination. If necessary, you should collect wastewater from decontamination (e.g., via a sump or pit) and remove it frequently for appropriate treatment or disposal. The pad or area should not leak contaminated water into the surrounding environment. You also should collect solvent rinses for proper disposal.

You should always handle field-cleaned equipment in a manner that prevents recontamination. For example, after decontamination but prior to use store, the equipment in a location away from the cleaning area and in an area free of contaminants. If it is not immediately reused, you should cover it with plastic or aluminum foil to prevent recontamination.

At certain sites, decontamination will generate a quantity of wastes called investigation derived waste (IDW). You should address the handling and disposal of IDW in your sampling plan. You must handle this material in accordance with whether it is nonhazardous or suspect of, or know to be, hazardous. You should minimize the generation of hazardous IDW and keep it separated from the nonharzardous IDW. For example, you should control the volume of spent solvents during equipment decontamination by applying the minimum amount of liquid necessary and capturing it separately form the nonhazardous wash water. For additional guidance on handling IDW, see Management of Investigation-Derived Wastes (USEPA 1992).

Decontamination of personnel and their protective gear also is often necessary during hazardous waste sampling. This important type of decontamination protects personnel from chemical exposure and prevents cross-contamination when personnel change locations. The level or degree of such decontamination will depend on site-specific considerations, such as the decontamination procedures in your health and safety plan. For additional information regarding decontamination, See ASTM D5088, Standard Practice for Decontamination of Field Equipment Used at Nonradioactive Waste

Sites. Another source of additional information is "Sampling Equipment Decontamination (USEPA 1994) issued by EPA's Environmental Response Team.

If necessary and appropriate equipment specific decontamination procedures will be outlined in equipment specific SOPs. These SOPs will clearly state the type of documentation required for each type of equipment and state whether the equipment falls under 40 CFR part 160, subpart D. Decontamination may be documented by citing an SOP or directly on a sampling form or other task specific medium.

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

2. Prior to use and after use, all equipment will be appropriately cleaned and decontaminated in accordance to this SOP.

6.0 DEFINITIONS

1. *Archives* - Include an area for orderly storage and expedient retrieval of original raw data, documentation, protocols, specimens, and interim and final reports retained as a result of a study. Conditions of storage shall minimize deterioration of the documents or specimens in accordance with the requirements for the time period of their retention and the nature of the documents of specimens. An individual shall be identified as responsible for the archives. Only authorized personnel shall enter the archives. Material retained or referred to in the archives shall be indexed to permit expedient retrieval. A testing facility may contract with commercial archives to provide a repository for all material to be retained. Raw data and specimens may be retained elsewhere provided that the archives have specific references to those other locations.

2. *Calibration* – To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurement (NELAC).

3. *Decontamination* – Procedures followed to ensure cross contamination does not occur between sampling points or that potential contamination of equipment does not pose a hazard to sampling personnel.

4. *EPA* the U.S. Environmental Protection Agency.

5. *Equipment which Generates Data* – Equipment which directly collects a data point (or multiple data points) necessary for study goals and stores or displays the data for viewing.

6. *Equipment which Measures Data* – Equipment which directly collects a data point (or multiple data points) which includes a unit that can be used to compare the data point (or points which includes a unit that can be used to compare the data point (or points) with other data of the same unit.

7. *Equipment which Assesses Data* - Equipment which collects a data point (or multiple points) and manipulates the data in a manner in which inferences or calculations are made which create data other then what was directly collected. Or, equipment which measures the performance or accuracy of the data it collects.

8. *FIFRA* the Federal Insecticide, Fungicide, and Rodenticide Act as amended.

9. *Maintenance* – Actions performed on equipment to standardize and/or correct the accuracy and precision of a piece of equipment to ensure that the equipment is operating within the manufacturer's specifications and standard values.

10. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

11. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.

12. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

13. *Study* means any experiment at one or more test sites, in which a test substance is studied in a test system under laboratory conditions or in the environment to determine or help predict its effects, metabolism, product performance (pesticide efficacy studies only as required by 40 CFR 158.640) environmental and chemical fate, persistence, or residue, or other characteristics in humans, other living organisms, or media. The term "study" does not include basic exploratory studies carried out to determine whether a test substance or a test method has any potential utility.

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

USEPA. 1992. Management of Investigation-Derived Wastes. Directive 9345.3-03FS. NTIS PB92-963353. Office of Solid Waste and Emergency Response. Washington, DC.

USEPA . 1994. "Sampling Equipment Decontamination." Environmental Response Team SOP#2006, Revision#0.0. Edison, NJ. http://www.ert.org/

USEPA. 2000. National Environmental Laboratory Accreditation Conference (NELAC), EPA 600/R-00-0084Office of Research and Development, Las Vegas, NV.

USEPA. August 2002. RCRA Waste Sampling Draft Technical Guidance, EPA 530-D02-002, Office of Solid Waste and Emergency Response. Washington, DC. (5305W)

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: Kim B. Watson Date: 2-19-04

Kim B. Watson, Quality Assurance Manager

Approved by: Christopher T. Stone Date: 2-19-04

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision number 1

1. Added Section 3.0 number 10, for storage requirements.
2. Reformatted under new numbering scheme.

Revision number 2:

1. Changed wording of introduction to state that decontamination is a quality *control*, not a quality assurance measure.

Revision number 3:

1. Reformatted
2. Minor wording edits
3. Remove air dry requirement (section 3, # 8)

Revision number 4:

1. Change title.
2. Re-formatted.
3. Added definitions.
4. Added detail of the type of documentation required under subpart D of the GLPs.
5. Added section 3.4 and 3.5 regarding the use of checklists and filing of equipment documentation.
6. Added detail of what decontamination constitutes.

Revision number 5:

1. Minor wording edits in Section 1.0, Objective.
2. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
3. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, andSection10.0 Revision History.
4. Revised Section 4.2 on Decontamination of Equipment and Personnel to include general decontamination procedures as outlined in the RCRA Waste Sampling Draft Technical Guidance Document (USEPA 2002).
5. Added Section on Support Equipment for the on-site laboratory.
6. Included Section under Section 4.1 on records requirements for equipment.
7. Updated References

STANDARD OPERATING PROCEDURE

SEI-6.19.2

USE, MAINTENANCE AND CALIBRATION OF THE IONSCIENCE PHOCHECK 1000+ PHOTOIONIZATION DETECTOR (PID)

SOP Number: SEI-6.19.2
Revision Number: 2

Date Issued: 7/19/99
Date of Revision: 02/04/2011

1.0 OBJECTIVE

A photoionization detector PID is often used to determine the presence or absence of volatile organic compounds (VOCs) in soil and water with ionization potential. Although a PID cannot necessarily differentiate between individual VOCs, it is an indispensable tool for the initial screening of a potentially hazardous site. It is therefore important to understand its operation and to keep the PID in optimal operating conditions.

The goal of soil headspace screening is to acquire preliminary information regarding volatile organic or hydrocarbon contamination in the vadose zone. The process is not meant to identify specific VOCs or hydrocarbon compounds. Rather, it will detect the relative amount of volatile organic hydrocarbons accumulated in the headspace of the sample container within the ionization potential of the lamp. This information should be used with other site-specific information to formulate an assessment of the presence or absence of contamination at the subject site.

2.0 POLICIES

1. The PID should be calibrated each day before use. Maintenance, corrective actions, and calibration should be noted in the "Maintenance and Calibration Log".
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.
3. Field personnel will use a field logbook, observation and remark form (O&R) or other designated field form to record activities, measurements, and observations made in the field.
4. Any change in entries shall be made so as not to obscure the original entry. The recorder will initial corrections.
5. Field personnel will read this SOP prior to operating the PID.

3.0 SAFETY ISSUES

1. Whenever visiting a hazardous or potentially hazardous site, it is important to read, follow and sign the site health and safety protocol (HASP).

2. By itself, use of the PID poses no risk to human health; however, if elevated levels of VOCs are detected by the PID, it may be necessary to don personnel protective equipment as described in the site's HASP.

3. Field visits may involve accessing remote areas. Health and safety concerns regarding these field visits are minimal however; slip, trip and fall hazards, and poisonous plant and dangerous animal, as well as getting vehicles stuck in remote areas do present considerable health and safety issues. To help ensure field staff's health and safety with remote areas, all field staff is to have daily communication with the project manager or another appropriate Stone Environmental, Inc. (SEI) employee.

4.0 PROCEDURES

4.1 Introduction

The unit owned and operated by Stone Environmental, Inc. is the IonScience PhoCheck 1000+ Model PID. Although there are numerous models of PIDs available, all PIDs are based on the following principle:

> A sampling pump continuously draws air sample into the ionization chamber. The ionization chamber for the PhoCheck 1000+ PID unit is constructed as a small cavity in front of the UV lamp. The electrode is made of a wire mesh so that the high energy UV light can shine through the mesh and excite the gas molecules when they pass across the face of the UV window. The ions collected on the electrode are measured by an electrometer.
>
> A single chip microcomputer is used to control the operation of the lamp, alarm buzzer, LED, sampling pump, power supply and electrometer. It measures the electrometer readings and calculates the gas concentrations based on calibration to a known reference gas. An LCD display is used to show the readings. The user interacts with the unit though six keys on the front panel.
>
> A rechargeable lead acid battery powers the unit. A built-in charging unit allows the batteries to be charged directly from an AC power source.

4.2 General Considerations

The PhoCheck 1000+ is adversely affected by both damp and windy conditions, as well as temperature. Ensure that the PhoCheck 1000+ is used with its probe and a clean sample filter at all times. Replace the filter frequently. Refer to the operating manual to determine the lowest allowable operating temperature and humidity.

There is only one operating mode used by the PhoCheck 1000+ Model: Survey Mode. Survey mode is used for discrete VOC measurements where the peak concentration is desired. SEI will utilize the survey mode for its operations.

4.2.1 Equipment

- PhoCheck 1000+ PID and Operations Manual
- Calibration gas and regulator

- Carbon filter canister
- Spare sample filters
- Phillips head screwdriver, 7/16" wrench
- Maintenance and Calibration Log
- Clean, smooth-top jars (125 mL soil jars) and aluminum foil, or; freezer quality *zip-lock* baggies, one quart size
- Stainless steel spoon
- Distilled water
- Field notebook, soil boring/well log forms, or other appropriate medium for recording field data

4.2.2 Use

The PhoCheck 1000+ has only six controls: Power, Enter, Escape, Up Arrow, Down Arrow and Zero. The "Power" control turns the power on and off. The "Enter" control selects options and confirms settings. The "Escape" control is used to return the display to the previous screen and to abort an adjustment. Repeated pressing of this control key will return the display to the DISPLAY/FUNCTION/SELECTION screen. The "Up" and "Down" arrows allow the user to scroll up or down or to change selections. The "Zero" control zeroes out background readings.

The following provides basic instruction on the PhoCheck 1000+ menu and control functions. Once the PhoCheck 1000+ is powered on, press the "Escape" control repeatedly until the DISPLAY/FUNCTION/SELECTION screen is displayed (this screen has three icons: a graph, a number 1, and a wrench). Highlight the function menu (represented by the wrench icon) and press "Enter". Then use the "Up" and "Down" arrows to view the available functions, then press "Enter" to select the function of choice. Once a given function is selected, a cursor (highlighted symbol or number) can be moved from one selection to another by using the "Up" and "Down" keys. Pressing "Enter" will cause the selection to flash. When flashing, use the "Up" and "Down" keys to adjust its value. When you have adjusted the parameter to an acceptable level, press "Enter", which will store the adjusted parameter. In many cases, a checkmark will also appear to confirm the action. You can abort a selection by pressing the "Escape" control. Note: It is best to read the Operations Manual before attempting to operate the PID.

4.2.3 Calibration

Due to the linear output of the IonScience PID, the PhoCheck 1000+ only requires a two-point calibration. The PhoCheck 1000+ scales its linear output across a ZERO level (clean air reference) and the SPAN (a user defined gas concentration). There are two options for calibration: Factory Calibration and Custom Calibration. Factory calibration offers a safe set of calibration data. This should be used if the custom calibration fails and will keep the unit working until a good custom calibration is completed. Stone typically uses the Custom Calibration (see steps below).

The PhoCheck 1000+ is calibrated at zero using the provided carbon filter and at 100 ppm using the isobutylene calibration gas. The date and initials of the individual who performed the calibration will be documented in the maintenance and calibration log. Follow these steps for a custom calibration:

1. Select the Custom Calibration procedure from the Function Menu. A checkmark will appear to confirm the selection. Pressing "Enter" allows the user to enter the Custom Calibration menu.
2. Scroll down and select the flashing "ZERO".
3. Remove the caps from both ends of the carbon canister included in the Cal Kit Sample supplied with your PhoCheck 1000+, remove the probe and filter from the instrument and fit the carbon canister. Alternatively a known clean air supply can be used. Note – the carbon canister simply pushes onto end fitting – do not force; no turning is required.
4. Press "Enter" to start the ZERO process. A counter will count down, showing how long the carbon canister needs to remain connected.
5. At the end of the countdown, a checkmark will appear, indicating that the Zero has been accepted. Press "Escape".
6. Disconnect the carbon canister and replace the cap ends. The useful life of the carbon canister will be shortened if the canister is open to atmosphere for prolonged periods.
7. Scroll down and select the flashing "VOC".
8. The next screen shows the gas and gas concentration you have previously set up. If the information is correct, select "Enter".
9. A screen will show "VOC", a gas bottle icon and a flashing highlighted number.
10. Connect the calibration gas (Isobutylene) to the PhoCheck 1000+ and open the gas valve. Once gas is presented to the PhoCheck, press "Enter". The number will begin to count down, showing how long the instrument needs to be presented with the gas. A checkmark will appear indicating that the calibration has been accepted. "Escape" out of the calibration menu.
11. Now replace the instrument's filter and probe so PhoCheck is ready for use.
12. Carry out a Bump Test to ensure calibration was successful. In other words, expose the PID to a known concentration of gas (e.g., 100 ppm isobutylene calibration gas) and make sure it responds appropriately. Instrument is now ready for use.
13. Record value after calibrating in the Maintenance and Calibration Log.

If you are unsuccessful in calibrating the instrument, check the following points:

- The carbon canister may be contaminated
- The calibration gas may be low or empty
- If using a connector pipe, there may be a kink restricting the flow of gas

- PID lamp may require cleaning or replacement

4.2.4 Maintenance

Some tools are included with the field kit of the PhoCheck 1000+ that allow the user to perform minor maintenance. However, it is imperative that anyone attempting to clean or otherwise maintain the PID be in total comprehension of the inner controls of the PhoCheck 1000+ before opening the main body. **Please refer to the PhoCheck 1000+User Manual when performing maintenance.**

The following maintenance should be performed on a regular basis.

Maintenance	Schedule (Use dependent)
Change sample filter	Weekly
Blow out PID cell with can of computer dustoff	As needed
Replace secondary probe filter	Annually or as needed
Clean UV lamp with aluminum oxide	Annually or as needed

Detailed maintenance must be performed by a qualified technician and usually requires the shipping of the PID to the manufacturer. Stone typically sends the PID to the manufacturer annually for lamp cleaning and general maintenance – or more frequently as needed. Call the manufacturer for shipping instructions prior to sending the PID.

4.2.5 Documentation

A record of calibrations and any maintenance performed on the PID must be made in the Maintenance and Calibration log.

Typically in field use, PID values are recorded onto soil boring logs (see attached). If screening is being conducted of miscellaneous soil samples, such as those from surface samples, values should be recorded on "Observations and Remarks (O&R)" forms.

4.3 Collection of Soil Samples for Headspace Screening

Fill a clean jar or baggie approximately half full using the stainless steel spoon with a "consistent amount" of soil. Take special care to ensure that sample and container are not cross-contaminated from previous sample by rinsing spoon between samples with distilled water. It is also important to ensure that the sample is not frozen prior to analysis.

Seal top of jar with two layers of aluminum foil, or insure that bagged is properly sealed. Secure aluminum foil with either a rubber band or screw lid that fits jar. If using screw lid, do not include solid top with rubber gasket included with new mason jars.

Allow VOC concentrations to develop in the headspace of the sample container for five minutes. During this headspace development period, the sample should be shaken vigorously for a total of one minute.

Immediately pierce the aluminum foil seal (or un-zip an approximate one-inch section of baggie) with the probe of PID and note the highest (peak) reading, and sustained reading. Take care not to submerge PID tip into soil.

Record time of sample collection and peak value as ppm onto appropriate form (Soil Boring Log or O&R).

4.4 Documentation

A record of the soil screening must be made in the field at the time the peak sample headspace concentration is read from the PID. It may also be helpful to record the weather or any site-specific conditions such as excessive heat or cold that may affect the readings from the PID. Be sure that field notes are properly labeled to the project number and date.

5.0 RESPONSIBILITIES

1. Stone personnel must calibrate the PhoCheck 1000+ prior to use. Calibrations should be recorded in the Maintenance and Calibration Log.
2. It is the responsibility of Stone field staff to make sure that the PID is in proper working order prior to bringing it onto a site.
3. Stone personnel must read this SOP prior to using the PhoCheck 1000+. After reading the SOP, personnel should update their personnel training folder.

6.0 DEFINITIONS

1. *EPA* the U.S. Environmental Protection Agency.
2. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.
3. *PID*: Photoionization Detector
4. *PPM*: Parts per million.
5. *Quality Assurance Unit (QAU)* any person or organizational element except the study director, designated by testing facility management to perform the duties related to quality assurance of the studies.
6. *Raw data* any laboratory worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche

copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

7. *VOC*: Volatile Organic Compound

7.0 REFERENCES

1. Vermont State Agency of Natural Resources, Waste Management Division "Agency Guidelines for Petroleum Contaminated Soils and Debris" August 1996.
2. Ion Science Ltd., "PhoCheck+ Insturment User Manual v2.4", November 2008.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Revised by: _____ Date: 2/4/11

Morgan J. Greenwald, Staff Scientist

Approved by: _____ Date: 2-4-11

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Reformatted under new numbering scheme.
2. Combined with SOP draft 5.8.0. Title changed to "Use, Maintenance and Calibration of the Mini-Rae Photo Ionization Detector (PID)".
3. Added documentation and safety issues items from SOP 4.5.9.
4. Minor wording edits in Section 1.0, Objective.
5. Updated style to match SEI Style Guide – font and text. Reformatted using MS Word.
6. Added standardized section headers: 2.0 Policies, 3.0 Safety, 5.0 Responsibilities, 6.0 Definitions, 7.0 References, 8.0 Tables, Diagrams, Flowcharts and Validation data. Authorization moved to Section 9.0, and section10.0 Revision History.

Revision 2

1. Updated SOP to refer to our new PID, the IonScience PhoCheck 1000+ PID, rather than the old PID, the Mini-Rae. Changed title, any references to PID and updated all use and calibration instructions based on the PhoCheck 1000+ User Manual.

SOIL BORING LOG

Project Name: _____ Site Name: _____

SEI Study #: _____

BORING ID:	Date Drilled:	Location:
SOP followed:		
Deviations? (If YES, attach details):		
Drilling Method:		
Drilling contractor:	Inside diameter:	
Sampling method:		
Core Length: Inside diameter:		

Comments:

Depth Interval (feet bgs)	Blow Counts (per 0.5 ft)	PID (ppm)	Recovery (inches or feet)	Sample Description (color, texture, moisture, remarks)

Geoscientist:	Signature:	Date:

Geoscientist:	Signature:	Date:

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Page ____ of ____

SOIL BORING LOG (continued)			Boring ID:	SEI Study #
Depth Interval (feet bgs)	Blow Counts (per 0.5 ft)	PID (ppm)	Recovery (inches or feet)	Sample Description (color, texture, moisture, remarks)

Geoscientist:	Signature:	Date:
Geoscientist:	Signature:	Date:

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Page ____ of ____

STANDARD OPERATING PROCEDURE

SEI-6.42.2

GEOLOGIC DESCRIPTION OF UNCONSOLIDATED DEPOSITS USING THE WENTWORTH GRAIN SIZE SCALE

SOP Number: SEI-6.42.2
Revision Number: 2

Date Issued: 01/18/02
Date of Revision: 10/16/12

1.0 OBJECTIVE

The objective of this procedure is to provide a standardized geological method of describing the nature of unconsolidated deposits, commonly referred to as soils or sediments, using the Wentworth grain size scale. A description of unconsolidated geologic deposits is often part of site investigations as a critical element in understanding the properties that govern the movement of water and other fluids, including contaminants. Samples of unconsolidated deposits are typically collected with a split-spoon sampler or other soil coring tools. These samples are then described to aid in interpretations about the geologic and hydrologic characteristics of the site. The characteristics of major importance for this type of description are: color, texture (e.g., grain size, sphericity, and roundness), consistence, structure, moisture content and concretions.

The Wentworth scale was developed in 1922 and is based on the work of Udden. It is the generally accepted standard used by geologists and sedimentologists in North America (Pettijohn, 1975). It is a logarithmic scale in that each grade limit is twice as large as the next smaller grade limit (Folk, 1974). It is used to describe the texture of sedimentary rocks (e.g., sandstone) as well as unconsolidated sediments.

2.0 POLICIES

1. The Staff Scientist will follow the standardized geological method of describing the nature of the unconsolidated deposits (unless otherwise noted) and will document their fieldwork in a field notebook for all site investigations.
2. Staff Scientists will be properly trained in site investigations prior to conducting a site investigation as documented in their training record on file at Stone Environmental, Inc.

3.0 SAFETY ISSUES

Staff following the methodology outlined in this SOP will read the site Health and Safety plan for the individual project, as well as appropriate Material Safety Data Sheets (MSDS).

4.0 PROCEDURES

4.1 Equipment

1. A notebook computer, field notebook, logging forms, observations and remarks form or other acceptable recording medium.
2. Indelible pens
3. A measuring rule in feet and tenths and hundreds of feet (not inches)
4. Measuring devices, such as engineers scale, electronic balances, electronic water level indicator, interface probe, etc.
5. A rule in mm for measuring particle sizes
6. A grain size key
7. Knife or other appropriate tool for handling soil
8. Clean (new) poly/plastic sheeting or other material on which collected samples may be examined/preserved.
9. Appropriate personal protective equipment.
10. Sample containers, forms, labels, markers, preservatives and shipping materials.

4.2 Geologic Description Methodology

4.2.1 Separating Significant Layers

Each distinct sediment layer in the split spoon or other type of sample should be described separately. Layers may be differentiated based on any of the characteristics listed above. It may be most appropriate to describe layers less than 0.5 inches thick as laminae. In a situation where several laminae of similar properties are located in a sample, it may be best to describe them as a group. For example, 8 gray silty clay laminae between 5.5 and 6.5 feet, each about 1/4 inch thick. The following sections discuss each of the soil characteristics to be described.

4.2.1.1 Color

For most studies, a very general assessment of color based on simple observation is sufficient, but in some cases a study protocol may require that color be measured by comparing the soil to Munsell color charts. The Munsell system designates colors according to three variables: Hue, Value, and Chroma. The hue of a color indicates its relation to red, yellow, green, blue, or purple. The value indicates the lightness of the color, and the chroma indicates the strength of the color.

Stone Environmental uses eight pages of Munsell charts, displaying 251 separate color chips. Each page contains color chips of the same hue, ranging from reddish hues to yellow-green hues and including a one page "gley chart, which has colors in the green-blue-grey range.

Each hue is designated by a number followed by one or two letters. The eight pages of hues are as follows:

Hue	Color Range
10R	reds
2.5YR	reddish browns
5YR	reddish browns and reddish yellows
7.5YR	browns and reddish yellows
10YR	browns and yellowish browns
2.5Y	yellowish browns and olive browns
5Y	yellows and olives
gley	grays, blues, and greens

On each page, the chips vary according to value and chroma. Value increases toward the top of the page (colors become lighter), and chroma increases from left to right (colors become stronger and less grey). In a full color designation, hue is given first, followed by a space, then value, then chroma. Value and chroma are separated by a forward slash; for example:

Munsell Code	Hue	Value	Chroma	Color Name
5YR 6/3	5YR	6	3	light reddish brown

Note that each color chip has a color name as well as a color code, and the color chart book includes key charts to match the color names with the chips. It is not necessary to write down the color name in the field.

Matching soils to color chips is somewhat subjective, and results will vary not only with field personnel but also with the moisture content of the soil and the type of light available. The best strategy for matching colors is first to find the closest matching hue sheet, then choose a color chip on that sheet.

4.2.1.2 Texture

Texture (grain size distribution) of the sediment will be described according to the estimated percentages of gravel, sand, silt and clay in the sample. Textural classes and the particle sizes associated with these classes are shown in Attachment 1. Descriptions of soils will be formatted according to Attachment 2, with principle component listed first followed by secondary, tertiary and so forth. A reliable field guide to determining grain size and texture in the field can be found in the W.F. McCollough sand-gauge cards.

Also to be included in the description of the texture is a description of the rock fragments in the sediment sample. These will include particle sizes over approximately 5mm in diameter. The volumetric percentage of rock fragments should be recorded. The shape of the fragments will be described according to the following terminology: rounded, subrounded, angular or subangular, as illustrated in Attachment 3. The rock type or mineral type of the fragments should also be defined whenever possible.

4.2.1.3 Consistence

The consistence of the sediment will be described whenever it remains evident in the split spoon sample. The sample may be compacted excessively or otherwise disturbed during sampling and the natural consistence may not be evident during examination. If the consistence can be characterized, it will be described according to the terms and definitions listed in Attachment 4.

The key distinction for this parameter is the change from friable to firm. When handling a sample that is friable, it will take only slight pressure between the thumb and fingers to cause a clod or ped of the sediment to break apart. A sample that is firm will resist crushing to the point where it finally seems to explode when it is subjected to adequate pressure. A friable clod or ped will not explode or pop, it will simply fall apart when pressure is applied. The other descriptive classes, as show in Attachment 4, will be applied to add more information as to the degree of soil stability, or lack thereof.

4.2.1.4 Structure

Observations of structure will include a characterization of the orientation and continuity of contacts (e.g., sharp or gradational), angularity of grains (see Attachment 3), presence of fractures, cracks, partings or macropores and a qualitative description of the degree of sorting (poorly sorted means many different grain sizes, well sorted means uniform grain size).

Additional description of such as features as crossbeds, grading, ripples and varves will aid in identifying the depositional environment.

4.2.1.5 Moisture

The soil moisture will be described on the basis of visual and manual field observations. The moisture class will be based on the percent water in the soil as follows:

Moisture Class	Moisture Content (%)	Field Description
Dry	0	No dampness is evident in sample.
Humid	1-25	Slight damp mark seen on hand when sample is squeezed.
Moist	26-75	Grading from humid to wet.
Wet	76-99	Water is released when sample is squeezed.
Saturated	100	Water drips freely from sample.

4.2.1.6 Concretions

When iron or manganese concretions are present in a soil sample, they will be noted as to their abundance and size. The use of dilute hydrochloric acid is helpful in identifying the presence of carbonate minerals, though its use is not required.

4.2.1.7 Other Parameters

The protocols or work plan for a given study may require the determination of other parameters, e.g., screening for the presence of volatile organic compounds with a photoionization detector at a hazardous waste investigation. In such cases, the protocol, work plan, additional SOPs, or other authority or regulation will describe the additional or alternate method of sediment evaluation.

4.3 Data Recording

The information required by this SOP will be recorded in a field book during the sampling process. Required information includes:

Project name

SEI study number

Site name

Well/Profile ID number

Name of geoscientist

Driller's name

Date of exploration

Attachment 5 presents a format for recording field data as well as examples of the descriptors to be used. All of the data fields must be addressed for each discrete layer being described. All measurements should be in feet and tenths of feet (not inches). The lengths of the segments of the particular sample being described must add up to the linear footage of recovery noted. In the interest of standardization, if less than complete recovery is achieved it should be assumed that the missing portion of the sample interval is at the bottom of the sample.

5.0 RESPONSIBILITIES

5.1 Principals/Group Officers/Group Leaders/Personnel

Any staff conducting these procedures are responsible for signing, dating, and managing all field observation forms.

6.0 DEFINITIONS

1. *Concretions* A nodular or irregular concentration of certain authigenic constituents of sedimentary rocks and tuffs; developed by the localized deposition of material from solution, generally about a nucleus. Harder than enclosing rock.
2. *Lamina (ae)* Unit layer or sheet of sediment in which the stratification planes are one centimeter or less apart. Laminae need not be parallel to bedding. When they are at an angle to the bedding planes the term "cross-lamination" may be used.

7.0 REFERENCES

Davis, Richard A. Jr., 1992, Depositional Systems: An Introduction to Sedimentology and Stratigraphy, 2nd Edition, Prentice Hall, New Jersey, 604 p.

Folk, R.L., Petrology of Sedimentary Rocks, Hemphill Publishing Company, 1974.

Hunter/BCA. Standard Operating Procedure No. 6006, Identification of Soils

American Geological Institute, 1976, Dictionary of Geological Terms, Revised Edition, Anchor Press/Doubleday, Garden City, New York, 472 p.

Laboratory Manual for Hand Specimen Petrology, Department of Geology, University of Texas [Undated].

Pettijohn, F.J., Sedimentary Rocks, Harper & Row, 1975.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

1. Attachment 1: Wentworth Grain Size Scale
2. Attachment 2: Soil Description Format
3. Attachment 3: Angularity of Rock Fragment
4. Attachment 4: Consistence Classes
5. Attachment 5: Soil Boring Log

9.0 AUTHORIZATION

Revised by: Lee Rosberg Date: 10/16/12

Lee Rosberg, Project Scientist

Approved by: Christopher T. Stone Date: 10/16/12

Christopher T. Stone, President

10.0 REVISION HISTORY

Revision 1

1. Renumbered from Chapter SEI-10.11.1

Revision 2

1. Changed title from geologic description of unconsolidated deposits to geologic description of unconsolidated deposits using the Wentworth grain size scale
2. Added second paragraph of section 1.0
3. Expanded section 4.2.1.1 to describe color using the Munsell system
4. Updated Attachment 5 with current soil boring log
5. Several minor grammatical edits
6. Added Pettijohn (1975) and Folk (1974) references

ATTACHMENT 1

WENTWORTH GRAIN SIZE SCALE

Limiting particle diameter				Size class	
mm	phi units				
2048	-11	Very large		Boulders	GRAVEL
1024	-10	Large			
512	-9	Medium			
256	-8	Small			
128	-7	Large			
64	-6	Small		Cobbles	
32	-5	Very coarse			
16	-4	Coarse		Pebbles	
8	-3	Medium			
4	-2	Fine			
2	-1	Very fine		Granules	
1	0	Very coarse		SAND	
		µm			
1/2	1	500	Coarse		
1/4	2	250	Medium		
1/8	3	125	Fine		
1/16	4	62	Very fine		
1/32	5	31	Very coarse	Silt	MUD
1/64	6	16	Coarse		
1/128	7	8	Medium		
1/256	8	4	Fine		
1/512	9	2	Very Fine		
		<2 µm		Clay	

ATTACHMENT 2

SOIL DESCRIPTION FORMAT

Component	Identifying Term or Format	Defining Range of Percentage by Weight
Principle	Upper case letters	Generally >50%
Secondary Component	Use adjective (ex. silty, sandy)	35% to 50%
Third Component	some	20% to 35%
	little	10% to 20%
	trace	<10%

ATTACHMENT 3
ANGULARITY OF ROCK FRAGMENTS



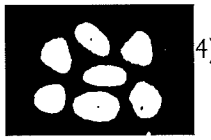
1) Angular: having sharp corners and edges, and therefore showing little or no effects of abrasion or wear.



2) Subangular: having edges and corners slightly rounded, so that wear is evident.



3) Subround: having most of the corners and edges worn down to smooth curves, and thus showing extensive abrasion.



4) Round: having all edges and corners smoothed off to gentle curves by prolonged wear.

ATTACHMENT 4
CONSISTENCE CLASSES

	Class	Description
Dry Soil:		
	dl —	loose- usually associated with sands
	ds —	soft- fragile
	dsh —	slightly hard- breaks easily
	dh —	hard- barely breakable with fingers
	deh —	extremely hard- can't break with fingers
Moist Soil:		
	ml —	noncoherent
	mvfr —	very friable- very easily broken apart
	mfr —	friable- easily broken apart
	mfi —	firm- resist crushing, "explode" when finally crushed
	mvfi —	very firm- barely breakable with fingers
Wet Soil:		
	wso —	nonsticky
	wss —	slightly sticky- soil sticks to one finger when squeezed and separated
	ws —	sticky- soil sticks to both thumb and finger, stretches slightly
	wvs —	very sticky- soil sticks strongly, resists separation of thumb and finger
	wsp —	slightly plastic- forms wire when rolled between hands, but breaks easily
	wp —	plastic- forms wire when rolled between hands, doesn't break easily
	wvp —	very plastic- forms a very durable wire when rolled between hands

Attachment 5

SOIL BORING LOG

Project Name: _____ Site Name: _____

Stone Project umber: Client:

BORING/WELL ID:	Date Drilled:		Location:
SOP followed:	Borehole Diam. (in)		
Deviations:	Well Diameter (in):		
Drilling Method:	Screen Length (ft):		
Driller's name:	Screen Slot Size:		
Sampling method:	Sand Pack (ft):		
Core Length:	Well Seal (ft):		
Comments:			

Depth Interval (ft bgs)	Recovery (feet)	PID		Soil Description (color, texture, moisture, remarks)	Sample Collected (Depth, ID & Time)	Well Const. Details
		Interval (ft)	Reading (ppm v/v)			

Geoscientist:	Signature:	Date:
Geoscientist:	Signature:	Date:

L:\RASC\FORMS\GENERAL HAZ-SITE SAMPLING FORMS\SOIL BORING LOG 020212.DOC

Page ____ of ____

Depth Interval (ft bgs)	Recovery (feet)	PID		Soil Description (color, texture, moisture, remarks)	Sample Collected (Depth, ID & Time)	Well Const. Details
		Interval (ft)	Reading (ppm v/v)			

Geoscientist:	Signature:	Date:
Geoscientist:	Signature:	Date:

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Page ____ of ____

STANDARD OPERATING PROCEDURE

SEI-6.45.0

Collection, Handling, and Preservation of Discrete Soil Samples

SOP Number: SEI-6.45.0
Revision Number: 0

Date Issued: 10/15/12
Date of Revision: NA

1.0 OBJECTIVE

The purpose of this procedure is to provide instruction for the collection, handling, and preservation of discrete soils for various analyses including, but not limited to: the potential presence of Volatile Organic Compounds (VOCs) along with representative samples for physical property analysis including porosity, bulk density, and fraction of organic carbon content (*foc*), Semi-Volatile Organic Compounds (SVOCs), Polycyclic Aromatic Hydrocarbons (PAHs), Metals, and Polychlorinated Biphenyls (PCBs).

VOC samples need to be obtained, logged, and preserved into methanol with a minimal loss of volatile analytes.

2.0 POLICIES

1. According to 40 CFR Part 160, Subpart E, Section 160.81, the NELAP standards and DOD 17025 ELAP standards, a testing facility shall have standard operating procedures in writing setting forth study methods that management is satisfied are adequate to insure the quality and integrity of the data generated in the course of a study.
2. Personnel will legibly record data and observations in the field to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

3.0 SAFETY ISSUES

1. If necessary and appropriate, a site-specific health and safety plan shall be created for each study site. A template for creating a proper health and safety plan is provided on the SEI network.
2. Care must always be taken when approaching a sampling location. Do not, under any circumstances, place yourself in danger to collect a sample.
3. If necessary and appropriate, all chemicals are required to be received with Material Safety Data Sheets (MSDS) or appropriate application label. These labels or MSDS shall be made available to all personnel involved in the sampling and testing.

4.0 PROCEDURES

4.1 Equipment and Materials

1. For VOCs: Pre-weighed VOA vials with screw cap and silicone septum containing 10 ml of purge-and-trap grade methanol (MSDS, Attachment 1). A pre-determined appropriate number of sample containers as needed for parameters of interest.

2. For VOCs: Sample collection device for minimally disturbed delivery of sample such as:
   - Stainless steel or disposable plastic syringe with barrel cut-off and smaller than vial opening
   - Stone's custom made stainless steel soil samplers with removable plungers
   - Encore piston sampler

3. Stainless steel spoons and bowls

4. Hand auger or other manual soil sampling tool

5. Portable balance, accurate to 0.01 grams, and calibration weights for vial preparation

6. Decontamination supplies if reusing stainless steel sampling tools: distilled water, alconox, scrub brushes, buckets, methanol if specified in project work plan

7. Field notebook, sample collection form, observation and remark form, or other acceptable medium for recording field notes. Ballpoint, indelible pens

8. Portable photoionization detector (PID) with calibration gas

9. Hacksaw

10. Soil core caps of appropriate diameter for the drill tooling being used to retrieve cores

11. Plastic wrap and zip top bags

12. Shipping materials: coolers with ice, bubble wrap or other packaging materials, packing tape

13. Measuring tape

14. As necessary or as described in the Site HASP appropriate personnel protection equipment (PPE) such as gloves, hardhat, hearing protection and safety glasses (minimal).

15. Chain of custody records and custody seals

4.2 Vial Preparation for VOC Sample Collection

VOC samples will be collected in 40 mL clear glass VOA vials with Teflon-lined septa and screw caps. This sampling requires purge and trap grade methanol for sample preservation. Methanol should be ordered before the start of field work and vials should be prepared in the laboratory prior to embarking into the field. If vials are prepared in the field it should be in a controlled environment (e.g. with an

appropriate balance on a flat surface and in a room free of wind). The following outlines the steps necessary to prepare vials for VOC sample collection of soils:

1. Affix sample label and label each sample container sequentially (e.g. VOC-001, VOC-002, etc.). Label the lid of each sample vial with the same ID as written on the label. Affixing the sample label prior to the sampling event assures the weight of the label will be included in the tare weight of the vial.

2. Check calibration of the balance being used to weigh vials. Record this calibration check in the appropriate calibration logbook.

3. Weigh each empty vial with affixed label and record weights on appropriate forms.

4. Add 10ml of purge and trap grade methanol using a calibrated pipetor or equivalent to each vial, screwing the cap back on each vial after filling it.

5. Re-weigh each vial once 10ml of purge and trap grade methanol has been added to accurately determine the weight and volume of methanol extract. Record these weights on appropriate forms.

6. Sample bottles will be stored at the laboratory in a freezer set between -10°C and -20°C until they are deployed into the field where the will be stored in a cooler with ice or fridge set to 4°C until samples are collected and are shipped to the laboratory. (A storage blank or trip blank will be stored with all sample bottles which will accompany all vials prepared)

Manufacturer, lot numbers, and other pertinent information of the methanol used to prepare vials will be recorded on appropriate forms. Recording this information enables Stone to track batches of methanol in the event contamination has partitioned into the methanol from various environmental sources.

4.3 Soil Core Retrieval, Handling, and VOC Sample Collection

Soil cores may be retrieved by several different methods, it is important to select a method that recovers soil cores representative of *in-situ* conditions and minimizes losses of VOCs. Examples of soil coring methods include, but are not limited to Geoprobe® Systems MC5, DT45, DT325/35, or DT22 coring tools, split spoon samplers, and AMS multi-stage soil core samplers. Refer to project specific protocols to determine which soil coring method will be utilized.

The rate of drilling and soil core retrieval shall not exceed the rate at which the sampler can collect samples and describe each core following SEI-6.42.*n Geologic Description of Unconsolidated Deposits.* This will prevent soil cores from being exposed to the atmosphere for extended periods of time prior to sampling, greatly reducing VOC losses.

Upon retrieval of soil cores the following steps shall be followed, working quickly to minimize the loss of VOCs:

1. Cores will be placed on a clean sampling surface, liners or spilt spoons shall be opened to reveal the core, and the core shall be photographed with labels indicating the site ID, borehole ID, depth interval, and recovery.

2. A portable PID will be utilized to screen the soil cores every 6 inches for the possible presence of VOCs. PIDs shall be calibrated daily prior to measurements according to manufactures instructions (SEI-6.19.*n*).

3. VOC sample frequency will follow site specific protocols. In general VOC samples will be collected from strata exhibiting the highest PID responses.

4. Using an appropriate sample device (see section 4.1 of this SOP), collect approximately 10-12 grams (g) of sample as soon as possible after the surface of the soil has been exposed to the atmosphere, generally within a few minutes at most. To the extent practical, samples should be obtained from undisturbed portions of the soil core.

5. Using the sample collection device, add the approximately 10-12g of sample to a pre-prepared sample vial containing purge-and-trap grade methanol.

6. Wipe any soil off the vial threads and immediately seal the vial with the septum and screw cap.

7. If project-specific work plan(s) specify, use a portable balance to weigh the sealed vial containing the sample (after either noting the laboratory-indicated weight or determining the tare weight of the vial in the field) to ensure 10-12g of sample was added. Samples will also be weighed upon receipt at the laboratory to determine sample mass.

8. Using the laboratory supplied label already fixed to the vials, label sample. Do not affix additional labels or custody seals to vials, as this will alter the tare weight.

9. Store samples on ice at 4°C until delivered to the analytical laboratory.

10. A trip blank comprised of a blank methanol vial should be supplied by the laboratory, and shall be submitted back to the laboratory with each sample delivery group.

11. A separate 10-12g aliquot of soil should be collected for moisture content determination using the same sampling device and methods described above, and placed in an un-preserved container. Label the moisture content sample appropriately.

12. Upon completion of VOC sample collection soil cores shall be described for color, texture, consistence, structure, and moisture, and other parameters as called for in site specific protocols following SEI-6.42.*n*. Collect discrete soil samples for other analytical parameters.

13. Decontaminate reusable sampling equipment prior to reuse according to site specific procedures and/or SEI-5.1.*n Maintenance and Decontamination of Field Equipment.*

4.4 Physical Properties Sample Collection

Soil cores will be retrieved following procedures described in section 4.3 of this SOP. Refer to site specific protocols for sample frequency. However, in general, an effort will be made to obtain samples from different stratigraphic units where variations in stratigraphy as assessed during core logging.

An effort shall be made to collect physical property samples from lesser contaminated sections of coil cores as determined by PID screening.

Each sample will be a cylindrical disc of the same diameter as the soil core retrieved, with a height of approximately 4 to 6 inches. These sections of core will be obtained such that the *in-situ* moisture conditions are retained. Soil samples will be collected for physical property analyses (porosity, bulk density, and *foc*) in the following manner:

1. Select a section of soil core to be sampled
2. Use a hacksaw to cut through plastic core liners on both ends of the desired sample. If split spoons were used carefully roll the soil core onto a clean piece of plastic wrap and remove unwanted soil with a gloved hand or knife.
3. Wrap sample tightly in clean plastic wrap. Tape plastic wrap to secure.
4. If available, place proper size end caps on both ends of sample to secure soil. If not available use additional plastic wrap to secure the core sample.
5. Clearly label each sample with the sample ID, location, depth, and date. Secure label with packing tape and wrap in bubble wrap or other padding.
6. Place samples individually in zip top polyethylene bags.
7. Store samples on ice at 4°C until delivered to the analytical laboratory.

4.5 Discrete Soil Sample Collection for Other Analytical Parameters

Discrete soil samples may be collected for other analytical parameters including, but not limited to, SVOCs, PAHs, Metals, and PCBs. These samples may be collected from soil cores retrieved from the subsurface using drilling techniques discussed in section 4.3 of this SOP or from surface or shallow soils collected using hand tools, such as a hand auger or AMS multi-stage soil core sampler. These discrete soil samples shall be collected following these steps:

1. Prior to fieldwork contact the laboratory conducting sample analysis and procure appropriate containers for the specific study. Container volume and amount of soil needed may vary by the analyses being conducted.
2. Using a stainless steel spoon or trowel collect the appropriate amount of soil needed for the analyses being performed into the appropriate sample containers provided by the laboratory.
3. Clearly label each sample with the sample ID, location, depth, and date. Secure label with packing tape and wrap in bubble wrap or other padding.
4. Store samples on ice at 4°C until delivered to the analytical laboratory.
5. Decontaminate reusable sampling equipment prior to reuse according to site specific procedures and/or SEI-5.1.*n Maintenance and Decontamination of Field Equipment.*

5.0 RESPONSIBILITIES

1. All personnel will legibly record data and observations (including phone conversations) in accordance with this SOP to enable others to reconstruct project events and provide sufficient evidence of activities conducted.

6.0 DEFINITIONS

1. *Observations & Remarks Form (O&R):* A pre-printed form, which contains mostly blank space for general note taking. The form typically prompts the user for the study or project designation, the SEI project number, the client or sponsor name, the total number of pages (page *n* of n) and requires a signature and date. The form is generally used to capture notes of one person when another, more specific forms is not available.

2. *Raw data* means any worksheets, records, memoranda, notes, or exact copies thereof, that are the result of original observations and activities of a study and are necessary for the reconstruction and evaluation of the report of that study. In the event that exact transcripts of raw data have been prepared (e.g., tapes which have been transcribed verbatim, dated, and verified accurate by signature), the exact copy or exact transcript may be substituted for the original source as raw data. Raw data may include photographs, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments.

3. *VOCs:* Volatile Organic Compounds

4. *SVOCs:* Semi-Volatile Organic Compounds

5. *PAHs*: Polycyclic Aromatic Hydrocarbons

6. *PCBs*: Polychlorinated Biphenyls

7. *PID*: Photoionization Detector

8. *MSDS*: Material Safety Data Sheet

9. *foc*: Fraction of Organic Carbon

7.0 REFERENCES

40 CFR Part 160 Good Laboratory Practice Standards, August, 1989.

8.0 TABLES, DIAGRAMS, FLOWCHARTS, AND VALIDATION DATA

None

9.0 AUTHORIZATION

Written by: Lee Rosberg Date: 10/16/12

Lee Rosberg, Staff Scientist

Approved by: Christopher T. Stone Date: 10/16/12

~~Seth Pitkin, Vice President~~ Christopher T. Stone

10.0 REVISION HISTORY

Not applicable.

Attachment I
Methanol MSDS

AMRO Environmental Laboratories Corporation

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POLYCHLORINATED BIPHENYLS (PCB's) BY GAS CHROMATOGRAPHY

SW-846 Method 8082

Procedure Number: OL-2006

Revision No.: 8

Revision Date: 02/03/10

Review Data: N/A

Prepared By: Kristin Albert Date: 2/3/10

Approved By: Sue Karan Date: 2/4/10
Organic Manager

Approved By: Mary Schmidt Date: 2/3/10
Laboratory Director

Approved By: Paula Barbour Date: 2/3/10
Quality Assurance Manager

Property of AMRO Environmental Laboratories Corporation

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1.0 SCOPE and APPLICATION

1.1 This SOP is for Method 8082 to determine the concentrations of various Polychlorinated Biphenyls (PCBs) in extracts from solid, aqueous, and other matrices.

1.2 Analytes and Reporting Limits:

ANALYTE	RL (Reporting Limit)*			
	Aqueous (µg/L)	Soil (µg/kg)	Oil (mg/kg)	Wipes (µg/wipe)
+PCB-1016	0.20	25	5.0	1.0
+PCB-1221	0.20	25	5.0	1.0
+PCB-1232	0.20	25	5.0	1.0
+PCB-1242	0.20	25	5.0	1.0
+PCB-1248	0.20	25	5.0	1.0
+PCB-1254	0.20	25	5.0	1.0
+PCB-1260	0.20	25	5.0	1.0

+= Designates multi-component analytes.
* = Aqueous based on 1000mL sample /4mL final volume
Soil based on 20.00g/10mL final volume
Oil based on 0.10g/10mL final volume
Wipe based on 10mL final volume per wipe

2.0 METHOD SUMMARY

2.1 This method describes chromatographic procedures for the detection of specific PCB's at ppb (µg/L and µg/kg) levels. Prior to the use of this method, appropriate extraction procedures must be used. Please refer to OL-2003 and OL-2004 for sample extraction. An aliquot of the sample extract is injected into a gas chromatograph (GC) equipped with dual columns and dual electron capture detectors (ECD). This allows for quantitation and confirmation to occur at the same time.

3.0 DEFINITIONS

3.1 **Multi-component Analyte and Single component Analyte:** Throughout this SOP, the terms "single component and multi-component analyte" are used. Multi-component analyte chromatograms contain a pattern of many peaks for one target analyte while a single component analyte chromatogram contains only a single peak for each target analyte.

3.2 **Aroclor:** Aroclor is the trade name for mixtures containing PCB's. Since each Aroclor is a mixture, they must be described as multi-component analytes. Aroclors may contain compounds in addition to PCB's, but the terms "PCB" and "Aroclor" are commonly used interchangeably. The names of the Aroclor mixtures such as 1260 refer to the 12-member biphenyl ring and the percent Chlorination by weight with the exception of 1016. Aroclor 1260 is a mixture of isomers composed of 12-member (carbon) biphenyl rings with 60% chlorination by weight. Chromatograms of Aroclors contain a pattern of multiple peaks due to different PCB compounds called congeners.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Extract must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

4.2 Sample extracts are custody transferred from the Extraction analyst to the GC analyst via signature in the extraction logbook, or the sample extracts are transferred to cold storage by the Extraction analyst. The extracts are either loaded directly on to GC instrument for analysis or they are stored at 4°C for future use. Extracts are kept for a minimum of two months after extraction and are then properly disposed of.

5.0 POTENTIAL PROBLEMS AND INTERFERENCES

5.1 The success of these methods frequently depends on the levels of interference rather than on instrumental limitations. If sample or matrix interference's prevent identification or detection of analytes, the analyst must evaluate the interference's and perform the proper corrective action.

5.2 Sources of interference's can be grouped into three categories:

5.2.1 Contaminated solvents, reagents, or glassware

5.2.1.1 Method Extraction Blanks and Instrument Blanks are used to determine possible contamination of solvents, reagents, glassware, and GC equipment.

5.2.1.2 If contamination is found from the extraction process, review of methodology, scrupulous cleaning of the glassware, and re-extraction of the samples may be required.

5.2.2 Contaminated carrier gas or GC equipment, i.e. columns, injection port liners, detectors and syringes.

5.2.2.1 If the GC equipment is found to be the source of contamination, proper cleaning and solvent rinsing of affected parts may be required or replacement with new ones.

5.2.3 Compounds extracted from the sample matrix to which the detectors respond.

5.2.3.1 If sample matrix is determined to be the cause of interference's in the extract, the analyst should evaluate whether extract cleanup procedures are needed. All PCB extracts are sulfuric acid cleaned prior to analysis. Additional cleanups may be needed.

5.2.3.2 Such interference's may include the presence of elemental sulfur, which results in broad peaks that interfere with the detection target compounds. (Analyst should refer to SOP OL-2020 for sulfur removal.).

5.2.3.3 Other sources of interference's could occur from phthalate esters which can be minimized by avoiding sample contact with any plastic materials. Phthalates are easily leached from plastic materials during laboratory operations or even sampling including equipment rinsing.

6.0 HEALTH AND SAFETY

6.1 AMRO has a laboratory safety program in place that applies to all employees. There is a safety officer as well as employees with 40 hours of OSHA training. The air quality of the laboratory and the extraction hood velocities are monitored. All employees receive fire and safety training upon employment.

6.2 Fire extinguishers are located in various areas of the lab. A lab evacuation plan is posted in every room, with a designated meeting place. Fire drills are held on an annual basis or "as needed" with building or personnel changes.

6.3 All new analysts receive training on the safe handling of samples, standards, the changing of gas cylinders, and the operation of instruments. In the lab, clean laboratory coats and gloves are worn when working with samples. Neat standards are made up in hoods. When gas tanks are replaced, the carrier with chain is used properly, and all gas tanks are chained in place.

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6.4 Care is taken with glassware and syringes to prevent injury. Broken glass and syringes are promptly removed to broken glass containers. First aid kits are available throughout the lab. There are also eyewash stations and an emergency shower.

6.5 Material Safety Data Sheets (MSDS's) are reviewed for each chemical used to ensure proper precautions are observed when handling chemicals.

6.6 Pollution prevention – The use of chemical exhaust hood are required when working with chemicals. They are specifically designed to minimize analyst exposure. The hood exhaust is monitored periodically to ensure environmental exposure is minimized.

6.7 Waste control – AMRO takes whatever steps possible to minimize waste. This includes sample and solvent volume minimization where appropriate. There is a Waste Coordinator officer designated with 40 hours of OSHA training. AMRO also properly segregates wastes based upon waste characterization (i.e. flammability, chlorination, acidity, etc.) and a commercial vendor is utilized for waste disposal.

7.0 INSTRUMENTS AND EQUIPMENT

7.1 Gas Chromatograph: Analytical system complete with gas chromatograph suitable for on column and split-splitless injections. The system includes all the required accessories such as syringes/autosampler systems, analytical columns, gases, electron capture detectors (ECDs) and a data system.

7.2 AMRO has two complete GC/ECD systems: GC-Trent and GC-Elvis. Both are Hewlett Packard Model 5890s capable of dual column analysis. Helium is the carrier gas and P5 (95% Argon/5% Methane) is the auxiliary make-up gas. Each GC is equipped with one of two equivalent capillary column pairs, either:

7.2.1 RXI-50 (Restek), 30m x 0.53mm I.D. 0.83µmdf
RTX50 (Restek), 30m x 0.53mm I.D. 1.5µmdf

or

7.2.2 RTX-CL PesticideII (Restek), 30m x 0.32mmID, 0.25µmdf.
RTX-CL PesticideI (Restek), 30m x 0.32mmID, 0.5µmdf

7.3 The data systems for all instruments are Hewlett-Packard Chemstation-Enviroquant software which is used to acquire and process all GC-ECD data. GC Elvis is the primary PCB analysis instrument. GC Trent is a back-up instrument for PCB analysis but not used routinely.

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7.4 Volumetric flasks, 10 and 25mL, and Gas-Tight syringes for preparation of standards.

8.0 INSTRUMENT PREVENTATIVE MAINTENANCE AND REPAIR

8.1 Daily Maintenance:

The following procedures are performed each day an instrument is used for analysis if in the analyst's judgement, system performance requires them:

8.1.1 The GC septum and glass liner are removed and replaced if judged necessary by the analyst.

8.1.2 Column performance is monitored by checking the response factor of key compounds which indicate column degradation, column bleed, or appropriate peak resolution. Clip two or more inches from the head of the column if necessary.

8.1.3 Check that carrier and make-up gases have adequate supply sufficient for the analytical sequence. Visual inspection of color indicating inline gas filters (hydrocarbon and moisture). Change tanks if necessary.

8.1.4 Check to ensure the hard drive memory capacity is sufficient for data acquisition using the HP Chemstation-EnviroQuant software .

8.2 Weekly Maintenance, or as needed:

8.2.1 Clipping column

8.2.2 Swab inlet weldment and injection port with hexane or other appropriate solvent(s).

8.2.3 Clean or replace single taper injection liner and glass wool, gold inlet seal, ferrules, and septa

9.0 REAGENTS AND STANDARDS

9.1 The following solvents are used for sample dilutions, preparation of standards or solvent rinsing of equipment:

9.1.1 Hexane, Reagent Grade and Pesticide Residue Grade chemicals or equivalent.

9.1.2 Acetone, Reagent Grade and Pesticide Residue Grade chemicals or equivalent.

9.1.3 Methanol, Reagent Grade and Pesticide Residue Grade chemicals or equivalent.

9.2 Commercially prepared stock standard solutions that are certified by the manufacturer are used in this analysis. All PCBs standards are purchased from Restek (e.g. cat. #32039 for 1000µg/mL Aroclor 1660) or an equivalent vendor. Second source standards are purchased from Ultra Scientific. Calibration standards for the special Aroclors 1262 and 1268 are also purchased from Ultra Scientific. The source of the surrogate, LCS, and MS/MSD spike solutions used in sample preparation is Restek.

9.3 Working standard solutions are made from stock standard solutions. All multi-component PCBs are purchased individually and surrogates are then added. Serial dilutions are made to create a seven point curve for "AR1660 (a mixture of PCB 1016 and PCB 1260). The remaining PCBs only require a single point analysis at the midpoint of the AR1660 curve, to be used to create a response factor appropriate for the individual Aroclor. Note for DoD projects all reportable quantitation of non-AR1660 PCBs must be preceded by at least a five point initial calibration so bracketing standards must be prepared as needed. All standards are brought to final volume with Hexane.

Calibration Stds	Vol. Of Stock	Final Vol.	Concentration (µg/mL)
AR1660*/TCX/DCB	0.05mL (2.0µg/mL)	2mL	0.05/0.001
AR1660*/TCX/DCB	0.2mL (2.0µg/mL)	2mL	0.20/0.004
AR1660*/TCX/DCB	0.4mL (2.0µg/mL)	2mL	0.40/0.008
AR1660*/TCX/DCB	0.6mL (2.0µg/mL)	2mL	0.60/0.012
AR1660*/TCX/DCB	0.8mL (2.0µg/mL)	2mL	0.80/0.016
AR1660*/TCX/DCB	1.0mL (2.0µg/mL)	2mL	1.00/0.02
AR1660*/TCX/DCB	0.05mL (1660 stock) 5µL TCX/DCB Stock	25mL	2.00/0.04

AR1221/TCX/DCB	25µL of Stock 2.5 µL of TXC/DCB Stock	25mL	1.0/0.02
AR1232/TCX/DCB	25 µL of Stock 2.5 µL of TCX/DCB Stock	25mL	1.0/0.02
AR1242/TCX/DCB	25 µL of Stock 2.5 µL of TCX/DCB Stock	25mL	1.0/0.02
AR1248/TCX/DCB	25 µL of Stock 2.5 µL of TCX/DCB Stock	25mL	1.0/0.02
AR1254/TCX/DCB	25 µL of Stock 2.5 µL of TCX/DCB Stock	25mL	1.0/0.02

*Please note AR1660 contains both AR1016 & AR1260.

9.4 TRACKING OF ANALYTICAL STANDARDS

9.4.1 Chemical Receipt:

The receipt of all standards, neat compounds and reagents from commercial sources is documented in the Chemical Receiving Logbook by sample receiving personnel or designate. Each solution or compound is assigned a unique standard receipt ID number, the letter "A" and a four digit sequential number. The date received, source, lot number, expiration date and disposal date are recorded. The expiration dates are provided from the manufacturer and the disposal dates are completed by AMRO employees when standards are used up or disposed. Refer to SOP " Sample Management" GL-1006.

9.4.2 Standards Preparation:

Standards are prepared as written in the respective SOPs. In order to assure the accuracy of standards the following general guidelines are used:

9.4.2.1 AMRO purchases pesticide residue grade solvents and purge and trap grade Methanol for organic standard preparation and organic sample extractions.

9.4.2.2 All stock standards that are purchased must include a certificate of analysis from the manufacturer.

9.5.3 Standard Tracking:

It is important to be able to track all analytical standards back to their sources. The following procedure is used by AMRO personnel to accomplish this:

9.5.3.1 Organic Standard Tracking: The Organic Standards Tracking System consists of three logbooks. One Standard ID Index logbook (Attachment 1) , one for Stock Standards, and one for Working Standards (Attachment 2) .

9.5.3.2 Stock Standard: Whenever a stock standard solution is opened or is prepared from a neat compound, this information is recorded in the Stock Standard Logbook section of the standard tracking log. The standard chemical receipt I.D. is used for the stock solution. This ID along with the compound name, concentration and initials of the person making the stock are included on the label. Stock standards are good one year from the date prepared or the date provided by manufacturer, whichever is shorter. Example - The fourth volatile standard prepared on April 15, 1989 would have the standard ID V041589D. This number will be recorded in the logbook and written on the container.

9.5.3.3 Working Section: Whenever a working standard solution opened or is prepared, whether from AMRO stocks or commercially obtained stocks, the information is recorded in the Working Standard section of the standard tracking log. The solution is assigned a new ID number based on the section "V" or "S", the date it is prepared plus a letter suffix. This number along with a descriptive name, concentration (or conc. varied) and the initials of the person preparing the solution are included on the label. The expiration of working standards vary depending on the use. Standards should be used in accordance with method specifications and manufacturer's instructions. Stock standards must be replaced no more than one year after opening or sooner if routine QC tests indicates a problem. Working standard expiration dates are derived from the assigned stock solution's expiration date or sooner if routine QC tests indicates a problem.

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9.5.3.4 Standard Storage: Standards and reagents are stored per method specifications and manufacturer's instructions. Semivolatile, Pesticide, PCB, Petroleum standards are stored in refrigerators at 4±2°C, freezers at minus 10 °C, and ambient temperature depending upon the method and manufacturer requirements.

9.5.3.5 Solvent Check Analysis

All extraction procedures are performed using pesticide residue grade solvents and the first method or procedural blank analysis is used when a new lot number is received to verify the absence of target analytes. Also, Before a new vendor's solvent is used for sample extraction a special check of the concentrated solvent (not a method blank) is required.

10.0 PROCEDURE

10.1 AMRO currently has two GC instruments: TRENT and ELVIS.

10.2 GC Operating Conditions:

PCB ANALYSIS (using Sec.7.2.1 columns on GC Elvis)
Injection volume: 1 µL
ECD Temperature: 310°C
Injection Port Temperature: 280°C
Initial Temperature: 150°C
Initial time: 0.5minutes
Rate: 1: 12°C/minute
Final Temperature 1: 270°C
Final Time hold: 15 minutes
Total Run Time: 25.5 min

* A signed hard copy of this acquisition method is stored in the Instrument run logbook.

10.3 Injection port maintenance:

10.3.1 Due to the low concentrations injected in this method, every attempt must be made to ensure that the analyte path in the GC is free from sites of absorption or activity by performing injection port maintenance. Such maintenance may include replacing septa, inlet liners, and the inlet seal. All maintenance must be recorded in the maintenance logbook.

10.4 Injection Runlog:

10.4.1 All injections are recorded with the EnviroQuant software and a signed hard copy of the sequence list (brief and full listing) is stored in the Instrument logbook.

10.5 Dual Column Analysis:

10.5.1 This method uses the dual column/dual detector approach. It involves the use of two 30m X 0.53mm ID fused- silica open tubular columns of different polarities, thus different selectivities towards the target compounds. The columns are connected to two separate injection ports with dual auto sampling towers. Each columns is then connected to its individual ECD detector.

10.6 Retention Time Windows (sections 10.6.1 through 10.6.5 refer to a setting up new instrument and determining analyte RT and elution orders):

10.6.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.

10.6.2 Make three injections of all single and all multi-component analytes over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

10.6.3 Record the retention times for all single analytes and three to five major peaks for multi-component analytes. Calculate the mean and standard deviation of the three absolute retention times of each single analyte and those peaks.

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10.6.4 If the standard deviation of the retention times for a target compound is 0.000, then the laboratory uses a default standard of 0.01 minutes. Recording retention times to three decimal places rather than two should minimize the instances in which the standard deviation is calculated as 0.000.

10.6.5 The width of the retention time window for the single analytes, each major constituent in multi-component analytes and surrogates is defined as ±3 times the standard deviation of the mean absolute retention time established during a 72-hour period. If the default standard deviation is employed, ½ the width of the window will be 0.07 minutes.

10.6.6 The absolute retention time windows must be calculated for each analyte and surrogate on each chromatographic column and instrument. New retention time windows and/or analyte elution order must be established when a new GC column is installed.

10.6.7 Establish the center of the window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

10.6.8 If the daily CCV falls outside the window, the analyst will evaluate the instrument and will recalibrate if necessary. For samples run during the same analytical shift as the Initial Calibration, use the RT of the midpoint standard. However, the experience of the GC analyst should weigh heavily in the interpretation of the chromatograms. The analyst may therefore qualify an analyte as present outside the RT window by using pattern recognition.

10.7 Initial Calibration:

10.7.1 Must contain a minimum 5-point curve of AR1660 (this is a mixture of Aroclors 1016 and 1260) to establish linearity for PCBs over the range of quanitifed Aroclors , along with the midpoint standards for the remaining 1221, 1232, 1242, 1248, 1254 Aroclors. These midpoints are to be used for pattern recognition and one-point calibration (the standards are used to determine the response factor appropriate for each individual Aroclor). The %RSD of the 1660 curve must be ≤20%. See Section 10.9.1 for action when the %RSD exceeds 20%. Note for DoD projects all reportable quantitation of Aroclors that are <u>not</u> 1016 or 1260 must be preceded by at least a five point initial calibration.

Bracketing standards must be prepared as needed and the sample re-run after the special initial calibration.

10.7.2 A minimum of 3 peaks must be chosen for quantitation but preferably 5 peaks should be used especially with 1016 and 1260. The chosen peaks should be within 25% of the height of the largest Aroclor peak and/or represent a unique peak height ratio. Peak selection should include at least one peak that is unique to each Aroclor.

10.7.3 When reporting PCBs two processes are used: qualitative and quantitative. The first is qualitative, which is done matching the unknown sample to the Aroclor standards evaluated during the initial calibration. In this case the pattern in the sample is compared to patterns of the standards. If a match is found, it is determined at what concentration the Aroclor is in the unknown, or its quantitative value. Three to five of the major peaks in the Aroclor pattern are chosen to measure the concentration of the positive hit. Of the seven commercial Aroclors on the 8082 target list, 1016, 1242, and 1232 have similar major peaks, the difference being the ratio of these peaks to one another. Although the same peaks may be used to quantitate a PCB hit, the same average response factors are not used. These are derived from that additional Aroclor's initial calibration.

10.8 Calibration Techniques:

There are two calibration techniques used by AMRO acceptable for quantitation: average response factor calibration and linear regression calibration curve. This is the order of preference:

10.8.1 The Average Response Factor Calibration:

10.8.1.1 Calculate the response factor of each compound using the equation:

$$\text{Response Factor (RF)} = \frac{\text{Peak Area of analyte}}{\text{Conc. of Analyte in standard}}$$

10.8.1.2 Calculate the Average of the Response Factors (Rfa) and the Standard Deviation (STD). Using the Rfa and STD, calculate the percent relative standard deviation (%RSD). When the RSD of Analytes are less than 20%, linearity through the origin can be assumed.

$$\%\ \text{RSD} = \frac{\text{Standard Deviation}}{\text{Average Response factor}} \times 100\%$$

10.8.1.3 Providing the % RSD for each analyte is less than 20%, the Average Response Factor (Rfa) can be using to calculate the sample results. If the %RSD is greater than 20% then a Linear Calibration Curve may be used providing the acceptance criteria is met.

10.8.2 Linear Calibration Curve:

10.8.2.1 In order to use this technique, the data system must plot and evaluate a calibration curve by calculating the correlation coefficient against the first order equation:

$$Y = mX + b$$

10.8.2.2 The software option Linear Regression must be selected and the computer will evaluate the calibration based on the above equation. Forcing the origin of the linear regression is not acceptable. The acceptance criteria for this equation is the coefficient of determination must be at least 0.990 and documented with a signed/dated graphics plot stored with the calibration.

10.8.2.3 Dropping of standard point should be performed ONLY on either the high or the low standard. Any point dropping should be documented for the reason of dropping on the initial calibration form. The analyst must sign and date the initial calibration form and will be reviewed by the appropriate data reviewer. The curve must be printed for visual verification and stored with the calibration records.

10.8.2.3.1 If the %RSD is greater than 20% and the coefficient of determination is less than 0.99 even after the adjustments in 10.8.2.3 are made , then the instrument must be re-calibrated.

10.9 Continuing Calibration Verification (CCV)

10.9.1 Verify calibration each 12 hour shift by injecting CCV's (Continuing Calibration Verification sample, a midpoint concentration standard) prior to any sample analysis. A CCV must also be injected at intervals not less than once every 20 client samples (or at the end of a 12 hour interval, whichever comes first). DoD samples will be analyzed at intervals of no greater than 10 field samples between CCV's.

10.9.2 A12-hour sequence begins with the injection of the first CCV. The shift ends after the completion of the analysis of the last sample or standard that can be injected within 12-hours of the beginning of the sequence. A closing standard **MUST** be injected at the end of the 12-hour window. However, in continuous analysis, the closing CCV of one shift may also serve as the CCV of the next 12-hour shift.

10.9.3 The CCV Standard response must be within ± 15% of the initial calibration curve. If the percent Difference (%D) of an analyte peak is > 15%, but the average %D for all the analyte peaks within any one Aroclor is within 15%, then the calibration has been verified.

$$\% \text{ Drift} = \frac{\text{Calculated Concentration - Theoretical Conc.}}{\text{Theoretical conc.}} \times 100\%$$

$$\text{Ave. \%Drift per Aroclor} = \frac{\text{\%Drift of Each Analyte Peak}}{\text{Total \# of Analyte Peaks}}$$

No sample results can be reported associated with a verified CCV > 15% for that analyte unless it is qualified as estimated. Reanalysis is recommended in these cases. If a CCV fails and correction action fails to produce a passing CCV, i.e. two CCVs fail in a row, then a new initial calibration is required *or* two consecutive CCVs must pass the method criteria described above in order for sample analysis to proceed.

10.9.4 CCV retention times must be compared to the RT windows established vs. starting CCV (RT updated and verified) of the analytical sequence.

10.9.4.1 If any analyte is outside the window, the chromatographic system must either be adjusted so re-analysis of the CCV falls within RT windows, or a new initial calibration must be performed and new retention time windows established.

10.9.4.2 Subsequent CCV retention times should be compared to the initial CCV of the 12-hour shift.

10.9.5 If the standard analyzed after a group of samples exhibits high response (>15%), and the analyte was not detected in the samples, it is acceptable to report ND results without qualification. The high bias must be demonstrated by one or both terminal CCVs. In contrast, if the response in the standard is low (by 15%), the sample must be reanalyzed.

10.10 Data Interpretation

10.10.1 The integration of compounds for standards and samples should be performed to be in the same manner as the initial calibration integration. Cases where the initial calibration is in question should be investigated to ensure it is not related to poor maintenance, damaged or defective column, or other issues which should be solved before further analysis.
The following must be checked for each compound present in standards and samples to ensure proper integration has been performed:

10.10.1.1 The peak shape of the calibration compound must be maintained for standards and samples. This should take into account any shouldering, peak tailing, and peak co-elution. See Sec.10.11.2.1 for specific information associated with PCBs.

10.10.1.2 The retention time windows for each compound of the calibration must be applied to standards and samples. If there is a retention time shift confirmed by comparison of the Continuing Calibration Standards and Surrogates then pattern recognition can be utilized for identification of multi-component analytes only. Otherwise re-calibration is required. Generally re-calibration is required whenever the column is significantly shortened during maintenance.

10.10.1.3 The baseline integration techniques for each compound of the calibration must be consistent for the standards and the samples. This should take into account elevated baselines, coelution, peak shape ands peak splitting, and negative peaks in samples.

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10.10.1.4 Make sure that positive identification and confirmation of compounds has been established. This is done using comparison between two columns.

10.10.1.5 Ensure any positive results are within the lower and upper levels of the calibration curve and above the reporting limits for each compound.

10.11.2 Data interpretation consists of two steps, qualitative interpretation and quantitative interpretation. Qualitative interpretation involves identifying the analytes that are present. Analytes are identified and confirmed according to their retention times on two different columns. Quantitative interpretation involves determining the concentrations of the analytes that are present. All analytes are calculated by using the initial calibration curve:

10.11.2.1 Qualitative Analysis:

10.11.2.1.1 Qualitative interpretation involves identifying the analytes that are present. Analytes are identified and confirmed according to their retention times on two different columns which is done matching the unknown sample to the Aroclor standards evaluated during the initial calibration. In this case the pattern in the sample is compared to patterns of the standards. If a match is found, it is determined at what concentration the Aroclor is in the unknown, or its quantitative value. Quantitative interpretation involves determining the concentrations of the analytes that are present. All analytes are calculated by using the initial calibration curve. Three to five of the major peaks in the Aroclor pattern are chosen to measure the concentration of the positive hit. Of the seven commercial Aroclors on the 8082 target list, 1016, 1242, and 1232 have similar major peaks, the difference being the ratio of these peaks to one another. Although the same peaks are used to quantitate a PCB hit, the same average response factors are not used.

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10.11.2.1.2 The first step in evaluating the data is qualitative analysis. Tentative identification of an analyte occurs when a peak from a sample falls within the established daily RT window for a specific target analyte. Confirmation is required on a second GC column of different characteristics. For multi-component analytes (Aroclors), identifications is based upon pattern recognition, but retention time windows are used to supplement this process.

10.11.2.1.3 Pattern recognition of multi-component analytes. Each multi-component analyte shows a distinctive chromatographic pattern which is used to identify that compound in a sample. To determine if a multi-component analyte is present, compare the sample chromatogram to the initial calibration standard. If the multi-component analyte is present in the sample, the relative response of the peaks in the standard should be similar in selected peak height ratios in standards. In addition, most major peaks observed in the standard should also appear in the sample. Be aware that degradation can alter the appearance and peak ratios of an Aroclor pattern. All Aroclors are mixes of congeners and may vary with the original manufactuer's raw materials.

10.11.2.1.4 When dual column analysis is being performed, the results for at least one column must meet the calibration acceptance criteria. The other column may be used for qualitative verification. Use of the designated qualitative verification column for quantitation is only acceptable if the initial and terminal CCVs on the verification column are in control and interference is suspected on the primary quantitation column. The maximum RPD between the final concentrations generated from the two columns is 40%. For DoD samples both columns must meet the acceptance criteria.

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10.11.2.1.5 If compound identification is precluded due to interference, cleanup of the extract may be warranted. Refer to method 3600 for cleanup procedures. Analyst judgement must consider options available vs. expected improvement in chromatography.

10.11.2.2 Quantitative Analysis:

The second step in evaluating data is quantitative analysis. This step can only take place after the qualitative techniques in Section 10.11.2.1 have been used to confirm a positive result. For all multi-component analytes, a minimum of three distinct peaks must be used for quantitative analysis.

10.11.2.2.1 Sample results are quantitated from the initial calibration based on the three techniques discussed in Section 10.9, following this order of preference.

10.11.2.2.2 Average Response Factor Calibration. Use the following equation to calculate the concentration of analytes:

$$\text{Concentration } (\mu g/L) = \frac{(Ax)\,(Vf)\,(Df)}{(Rfa)\,(Vi)}$$

Where:
Ax = Area counts for the analyte in the sample.
Vi = Initial volume of the sample extracted in mL.
Vf = Final volume of sample extract in mL.
Df = Dilution factor.
Rfa = Average response factor from the initial calibration curve.

10.11.2.2.3 Linear Fit Calibration. Use the following equation to calculate the concentration of analytes:

$$\text{Concentration } (\mu g/L) = \frac{([m]\,[Ax] + b)\,(Vf)\,(Df)}{(Vi)}$$

Where:

([m] [Ax] + b) = Concentration of analyte in sample extract, calculated by data system or integrator (ug/L).

Vi = Initial volume of sample extracted in mL.

Vf = Final volume of sample extract in mL

Df = Dilution factor.

The curve must be printed and stored with the calibration records to document visual evaluation.

10.11.3 To quantitate multi-component analytes, discern at least three to five distinctive, unobstructed, peaks in the sample chromatogram that correspond to characteristic peaks in the standard chromatogram used for initial calibration. The data system will calculate the average concentration of the selected characteristic peaks. The average response factors of the characteristic peaks or the calibration curve is used to calculate sample concentrations. Alternatively, the total area from each curve standard can be used to produce a linear or quadratic calibration curve.

10.11.4 Each sample analysis must be bracketed with an acceptable Initial Calibration, CCV's (each 12-hr. shift or interspersed every 20 samples, whichever comes first). DoD samples will be analyzed at intervals of no greater than 10 field samples between CCV's.

10.11.5 When a CCV fails to meet acceptance criteria, all the samples injected after the last acceptable CCV must be re-injected.

10.11.6 All sample responses must fall within the calibration range. Re-analysis at a dilution must occur if the sample response exceeds the calibration range.

10.11.7 Sample results are confirmed by using two dissimilar columns. The column used for reporting results depends upon acceptable daily quality control including CCVs, MS, MSD, and LCS criteria being met. In general, report data from the column with the best overall QC data. This is the designated quantitation column and should be indicated on the Data Review Checklist in the project folder and marked on the CCV Summary Form. For DoD samples both columns must be in control. Report the higher of two confirmed results unless overlapping peaks are

causing erroneously high results, then report the non-affected result and document in the case narrative.

10.11.8 For DoD samples any sample which is not confirmed must be reported with a "Q" flag.

10.11.9 When sample results are confirmed , the difference between the two columns is measured by calculating the Relative Percent Difference (RPD) between the two concentrations:

$$RPD = \frac{|R1-R2|}{((R1+R2)/2)} \times 100\%$$

*R1 & R2 are the results on 2 columns.

This data is calculated by the macro PCB_RT.mac when the information is required in a data deliverables package.

If the result from one column is significantly higher than the other column result, check the chromatogram for overlapping peaks causing erroneously high results. If no overlapping peaks are noted, examine the baseline parameters established during integration. If no abnormalities are noted, report the result from the designated primary quantitation column (except as noted in section 10.11.7 for DoD samples). If the results from the designated quantitation column appear to be in error due to interference's, and the CCV data is acceptable from the designated qualitative confirmation column, then use the confirmation column result for reporting. If the RPD exceeds 40% and the difference is not clearly attributable to matrix interference then report the RPD criteria exceedance in the case narrative and flag the result with a "J".

11.0 CALCULATIONS

11.1 All necessary calculations and formulas can be found throughout the text as applicable to each section.

12.0 QUALITY CONTROL

12.1 Initial Demonstration of Proficiency:

12.1.1 The ability of AMRO to produce accurate and precise data is demonstrated per instrument/per analyst for each method. It consists of a Method Detection Limit Study (MDL) and Precision and Accuracy Study (P&A) for each instrument and each matrix on an annual basis. The laboratory demonstrates initial proficiency with each combination of sample preparation and determinative methods that it utilizes by generating data of acceptable precision and accuracy for a reference sample containing the target analytes in a clean matrix. The reference samples are prepared from a spiking solution containing each analyte of interest at a concentration that is 10-50 times the MDL for each analyte in that matrix, or at the routine concentration of LCS's (Laboratory Control Samples). P & A Studies are required to be reviewed annually by evaluating a minimum of 4 LCS's and corrective action taken if necessary.

12.2 Method Detection Limit (MDL) Study:

12.2.1 The method detection limit is defined in 40 CFR 136 Appendix B. The purpose of an MDL is to establish the minimum concentration of each analyte greater than zero that can be reported with 99% confidence. An MDL study consists of at least 7 replicates of spiked deionized water (ideally standard set at ½ RL or lower), which are extracted and analyzed as normal samples. The standard deviation of the results of these analyses is multiplied by the appropriate student T-test value. The resulting value is deemed the method detection limit for that specific analyte.

12.2.2 MDL studies of each analyte must be reported annually for each instrument, column, matrix, and extraction method. To ensure that reasonable MDL values are determined, an MDL check sample is analyzed by spiking an interference free matrix with all target analytes at about two times the determined MDL. The MDL check sample is taken through the same process as the MDL. If any of the target analytes are not detected, or the characteristic Aroclor peak pattern cannot be identified, then the MDL study is modified and repeated until the MDL check sample is detectable. The MDL check is recommended on a quarterly basis and after major instrument maintenance or changes in the instrumentation or instrumental conditions to verify sensitivity of the method.

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12.3 Precision &Accuracy:

12.3.1 Accuracy is a measure of the degree to which the analytical result approaches the "true" value of the concentration of the analyte being determined. Precision in the laboratory is determined by the comparison of duplicates, where duplicate samples result from an original sample that has been split for identical analyses. Precision and Accuracy in the laboratory is assessed initially by the analyses of a minimum of 4 replicate samples at a mid-level point of the expected calibration range. The percent recoveries and Relative Standard Deviations (RSD) are evaluated and compared to method specifications or default limits where applicable.

12.3.2 Accuracy is also assessed by the regular analysis of known standards such as the Laboratory Control Samples (LCS), and matrix and analytical spikes. Accuracy within the laboratory is expressed in terms of percent recovery.

12.3.3 The precision of data is regularly evaluated by determining the relative percent difference (RPD) of duplicate (replicate) analyses.

12.4 Method Blanks are prepared at a frequency of one for every batch of up to 20 samples of the same matrix, prepared at the same time, by the same procedures. When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same procedures.

12.4.1 The results for the method blank should be < RL for all analytes for all sample batches and < ½ RL for batches containing DoD project samples (DoD samples can have common lab contaminants up to the RL). If the method blank results do not meet these acceptance criteria, then corrective action is taken to locate and reduce the source of the contamination and the associated sample extracts are reanalyzed or re-extracted if necessary. If all the samples in the batch are non-detects then re-extraction is not necessary. If insufficient sample volume remains for re-extraction then all the samples will be reported with the appropriate data qualifiers.

12.5 Matrix spike (MS) and Matrix Spike Duplicate (MSD) are prepared at a frequency of one for every batch of up to 20 samples of the same matrix, prepared at the same time, by the same procedures.

12.5.1 Samples must be selected for spiking at random and rotated among clients and sites from batch to batch. In some cases, the client may specify which sample is to used for MS/MSD; if not , the laboratory shall pick a representative sample based on the following:

A. MS/MSD shall not be performed on Trip Blank, Rinsate Blank or Field Blank.
B. Adequate sample weight/volume.
C. Client requested QC deliverables.

12.6 Laboratory Control Samples are prepared at a frequency of one for every batch of up to 20 samples of the same matrix, prepared at the same time, by the same procedures. The LCS consists of an aliquot of clean matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same compounds at the same concentrations as the MS and MSD. When the results of the MS and MSD analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the lab can perform the analysis in a clean matrix.

12.7 Analysis of the MS, MSD, and LCS require the same procedures employed for the original sample and the concentration of each analyte spiked is calculated.

12.8 Calculation of Recovery:

Accuracy is estimated from the recovery of spiked analytes from a sample matrix. The recovery of each spiked analyte is calculated using the following formula:

$$\text{Recovery} = \%R = \frac{Cs - Cu}{Cn} \times 100\%$$

Where:
Cs = Measured concentration of the spike sample aliquot
Cu = Measured concentration of the unspiked sample aliquot (use 0 for LCS)
Cn= Nominal (theoretical) concentration increase that results from spiking the sample, or the nominal concentration of the spike aliquot (for LCS).

12.9 Calculation of Precision:

Precision is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for the MS/MSD. To calculated the RPD, use the following formula:

$$RPD = \frac{|Conc.\ 1 - Conc.\ 2|}{((Conc.\ 1 + Conc.\ 2)/\ 2)} \times 100$$

Where:
Conc. 1 = Measured concentration of the first sample aliquot
Conc. 2 = Measured concentration of the second sample aliquot

12.10 All recoveries control limits for surrogates, matrix spikes and laboratory control sample control are based on the laboratory control charts. The control charts are updated annually.

12.11 The following additional Quality Control rules should be followed for all Massachusetts Contingency Plan (MCP) projects where MCP CAM methods are specifically requested:

12.11.1 MS/MSD: %REC's should be between 40-140%. RPD's should be <50% for all Aroclors.

12.11.2 LCS: %REC's must be between 40-140%.

12.11.3 Surrogates: %REC's must be between 30-150% for both surrogates on both columns.

12.11.4 Confirmation: All QC parameters must meet on both columns. Report the higher of the two columns results unless the highest result is attributable to interference.

12.11.5 Reporting: Report both sets if one is extracted inside the holding time and other outside the holding time. Provide chromatograms if the Quality Control problems are attributed to matrix effects. Report any exceedances of the above requirements in the narrative. Note if recoveries are outside the CAM acceptance criteria but inside AMRO's statistical control limits.

12.12 The following special default batch QC acceptance limit criteria apply to all DOD project samples unless superceded by a project specific QAPP, :

12.12.4 The LCS recovery acceptance limits are found in Attachment 3.

12.12.5 The MS/ MSD recovery acceptance limits are the same as for the LCS.

12.12.6 The maximum RPD allowed for MS/MSD pairs is 30%.

12.12.7 Surrogate recovery for Decachlorobiphenyl in water samples is 40-135%. Surrogate recovery for Decachlorobiphenyl in solid samples is 60-125%.

12.12.8 Corrective action must be taken if the MB or LCS surrogates or LCS target analyte spike recoveries are outside the acceptance criteria. If reanalysis does not fix the problem, or investigation does not prove the problem is confined to the MB and/or LCS, then re-extraction of the entire batch may be necessary. All data associated with failed MB or LCS QC must be flagged in the case narrative.

13.0 DATA REVIEW AND STORAGE

13.1 All cross-outs must be a single line and should be initialed and dated. Analyst review is documented on the GC and GC/MS Data Review Checklist (Attachment 4).

14.0 REFERENCES

14.1 40 CFR Part 136 Appendix B, 1984.

14.2 USEPA Office of Solid and Hazardous Waste - SW-846 Method Chapter 1, Rev. 1 7/92.

14.3 Method 8000B Revision 2, December 1996. SW846, Update III, USEPA.

14.4 Method 8082 Revision 1, December 1996. SW846, Update III, USEPA.

14.5 Shell for Analytical Chemistry Requirements, Version EM 200-1-3, 02/01/01.

14.6 OL-2003, Separatory Funnel Liquid-Liquid Extraction.

14.7 OL-2004, Automated Soxhlet Extraction.

14.8 OL-2019, Sulfur Cleanup.

14.9 Massachusetts Department of Environmental Protection, BWSC CAM V-A Method, Revision 3, May 1, 2003

Attachment 1

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STANDARD I.D. INDEX

Standard I.D.	Standard Logbook (Stock or Working)	Brief Description

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Attachment 2

STANDARD LOG - STOCK SOLUTIONS
ORGANIC SECTION

Date Opened/Prep'd	Open/ Prep'd by	Compound Name	Receipt/Stock ID	Source	Conc. or Purity	Amt (g) or (µl)	Final Vol. (ml)	Solvent ID/ number	Final Conc.	Units µg/µl or µg/ml	Solution ID	Storage Loc.	Exp. Date

Notes: 1) AMRO stock Std. ID is "V" or "S", date prepped, letter suffix(110498B is the second stock std prepared on 11/04/98)

2) Vials must be labeled with the compound name, conc., AMRO stock ID, and exp. Date

qc/qcmemos/forms/organicstock Rev. 4 05/00

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STANDARD LOG - WORKING STANDARDS
ORGANIC SECTION

Date Opened/Prep'd	Prep'd/Verified by	Compound Name	Receipt/Stock ID	Source	Conc. or Purity	Amt. (µl)	Final volume (ml)	Solvent ID/ number	Final Conc.	Units ng/µl or µg/ml	Sol'n ID	Storage Loc.	Exp. Date

Notes. 1) AMRO working Std. ID is "V" or "S", date prepped, letter suffix(110498C is the third working std prepared on 11/04/98)

2) Vials must be labeled with the compound name, conc., AMRO ID, and exp. Date

qc/qcmemos/forms/organicwork Rev.3 08/13/01

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Attachment 3
Attachment 3

TABLE D-16. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846 METHOD 8082 WATER MATRIX[27]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	85	20	25	145
Aroclor 1260	87	19	30	145

TABLE D-17. LCS CONTROL LIMITS FOR POLYCHLORINATED BIPHENYLS SW-846 METHOD 8082 SOLID MATRIX[27]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit
Aroclor 1016	90	16	40	140
Aroclor 1260	96	12	60	130

[27] LCS control limits are not available for Aroclors 1221, 1232, 1242, 1248, 1254, 1262, and 1268, although those compounds do appear on the target analyte list for method 8082 (Table C-9 in Appendix DoD-C). Sufficient data to perform statistically significant analyses were not received for those analytes during the LCS study. Additional limits for surrogate compounds can be found in section D.6.

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Attachment 4

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GC AND GC/MS DATA REVIEW CHECKLIST

LAB ORDER: _____ CLIENT: _____

Method: Sample(s) No.:

Analysis: Matrix: LIMS Batch ID:

Date of Extraction/Preparation: Date of Analysis

A.	GENERAL			
1.	Copy of Prep/Extraction Log?	Y	N	NA
2.	Copy of Instrument Run Log or Tune List?	Y	N	NA
3.	Copy of dry weight log present?	Y	N	NA
4.	Are all sample checked for preservation after VOA analysis?	Y	N	NA
5.	Are all VPH Soil sample received with mL Methanol/g soil 1:1 ±25% ?	Y	N	NA
6.	All samples met extraction and/or analysis holding time criteria?	Y	N	NA
7.	Did the Tune pass and OFTPP or BFB injected every 12 / 24 hours?	Y	N	NA
8.	Are the surrogate recoveries for each sample within the QC limits?	Y	N	NA
9.	Method Blank- contaminant free(<½RL)?	Y	N	NA
10.	Initial Calibration meets method criteria for all compounds reported?	Y	N	NA
11.	Continuing Calibration meets method criteria for all compounds reported?	Y	N	NA
12.	Are all reported results within calibration range?	Y	N	NA
13.	LCS % Recovery criteria met for all compounds?	Y	N	NA
14.	MS/MSD %R's and %RPD's are met for all compounds?	Y	N	NA
15.	%RPD criteria met between sample and duplicate?	Y	N	NA
16.	Samples checked for carry over from sample to sample due to target or nontarget overload?	Y	N	NA
17.	Were manual integrations performed properly?	Y	N	NA
18.	Are all data exported to LIMS, calculated and verified?	Y	N	NA

COMMENTS

Analyst (s) _____ DATE: _____

REVIEWER: _____ DATE OF REVIEW: _____

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Attachment 5

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QUALIFICATION CARD FOR SW-846 METHOD 8082

Trainee: _____ Signature: _____

Trainer: _____ Signature: _____

I. Knowledge Factors:

SOP Title: OL-2006 Revision Number/Date: Rev 7 01/24/08

1. Did the Trainee read and understand the above SOP?	Yes☐	No☐	NA☐
2. Was the Trainee instructed on how to properly fill out the documentation?	Yes☐	No☐	NA☐
3. Has the Trainee been instructed on the proper procedures for addressing potential problems?	Yes☐	No☐	NA☐

II. Practical Factors:

1. Has the Trainee completed a Precision and Accuracy Study?	Yes☐	No☐	NA☐
2. Has the Trainee completed an acceptable Performance Evaluation sample?	Yes☐	No☐	NA☐
3. Did the Trainee completed an acceptable Laboratory Control Sample (LCS)? or PT study*	Yes☐	No☐	NA☐
4. Has the Trainee performed manual integration as indicated in the SOP?	Yes☐	No☐	NA☐

* PT #240

Completion Date: _____ Trainer's Signature: _____

I _____ have read, understood and follow the above method/SOP, which is in use at AMRO Laboratories for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

Section Manager/ Supervisor: _____ Date: _____

QA Manager: _____ Date: _____

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SEMIVOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPH/MASS SPECTROMETRY

By

SW-846 METHOD 8270C

Procedure Number: OL-2008

Revision No.: 8

Revised Date: 02/02/2010

Review Date: N/A

Prepared By: _____ Date: 2/3/10

Approved By: _____ Date: 2/4/10
Organic Manager

Approved By: _____ Date: 2/3/10
Laboratory Director

Approved By: Paula Bentam Date: 2/3/10
Quality Assurance Manager

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1.0 SCOPE and APPLICATION

1.1 This Standard Operating Procedure (SOP) describes the analysis of semivolatile organic compounds in Methylene Chloride extracts prepared from many types of solid waste matrices, soils, and water samples. The compounds analyzed by AMRO and their typical reporting limits are listed in Table 1.

Table 1

Compound Name	CAS number	Reporting Limits	
		Water (µg/L)	Soil (mg/kg)
1,2,4-TRICHLOROBENZENE	120-82-1	10	0.25
1,2-Dichlorobenzene	95-50-1	10	0.25
1,2-Diphenylhydrazine (as Azobenzene)	122-66-7	10	0.25
1,3-Dichlorobenzene	541-73-1	10	0.25
1,4-Dichlorobenzene	106-46-7	10	0.25
2,4,5-Trichlorophenol	95-95-4	10	0.25
2,4,6-Trichlorophenol	88-06-2	10	0.25
2,4-Dichlorophenol	120-83-2	10	0.25
2,4-Dimethylphenol	105-67-9	10	0.25
2,4-Dinitrophenol	121-14-2	20	0.5
2,4-Dinitrotoluene	606-20-2	10	0.25
2,6-Dinitrotoluene	53-96-3	10	0.25
2-Chloronaphthalene	321-60-8	10	0.25
2-Chlorophenol	95-57-8	10	0.25
2-Methylnaphthalene	95-48-7	10	0.25
2-Methylphenol	91-59-8	10	0.25
2-Nitroaniline	88-75-5	20	0.5
2-Nitrophenol	119-93-7	10	0.25
3,3'-Dichlorobenzidine	56-49-5	10	0.25
3-Nitroaniline	534-52-1	20	0.5
4,6-Dinitro-2-Methylphenol	92-67-1	20	0.5
4-Bromophenylphenyl Ether	101-55-3	10	0.25
4-Chloro-3-Methylphenol	59-50-7	20	0.5
4-Chloroaniline	106-47-8	10	0.25
4-Chlorophenyl-Phenylether	7005-72-3	10	0.25
4-Methylphenol	106-44-5	10	0.25
4-Nitroaniline	57-97-6	20	0.5
4-Nitrophenol	3855-82-1	20	0.5
Acenaphthene	83-32-9	10	0.25
Acenaphthylene	208-96-8	10	0.25
Acetophenone	98-86-2	10	0.25

Table 1 (cont)

Compound Name	CAS number	Reporting Limits	
		Water (µg/L)	Soil (mg/kg)
ANILINE	120-12-7	10	0.25
Anthracene	140-57-8	10	0.25
Benz(a)Anthracene	56-55-3	10	0.25
Benzidine	92-87-5	20	0.5
Benzo(a)Pyrene	50-32-8	10	0.25
Benzo(b)Fluoranthene	205-99-2	10	0.25
Benzo(g,h,i)Perylene	191-24-2	10	0.25
Benzo(k)Fluoranthene	207-08-9	10	0.25
Benzoic Acid	65-85-0	20	0.5
Benzyl Alcohol	100-51-6	20	0.5
Bis(2-Chloroethoxy)Methane	111-91-1	10	0.25
Bis(2-Chloroethyl)Ether	111-44-4	10	0.25
Bis(2-Chloroisopropyl)Ether	108-60-1	10	0.25
Bis(2-Ethylhexyl)Phthalate	117-81-7	10	0.25
Butylbenzylphthalate	85-68-7	10	0.25
Carbazole	86-74-8	10	0.25
Chrysene	218-01-9	10	0.25
Dibenzo(a,h)Anthracene	53-70-3	10	0.25
Dibenzofuran	132-64-9	10	0.25
Diethylphthalate	84-66-2	10	0.25
Dimethyl Phthalate	131-11-3	10	0.25
Di-n-Butylphthalate	84-74-2	10	0.25
Di-n-Octyl Phthalate	117-84-0	10	0.25
Fluoranthene	62-50-0	10	0.25
Fluorene	206-44-0	10	0.25
Hexachlorobenzene	118-74-1	10	0.25
Hexachlorobutadiene	87-68-3	10	0.25
Hexachlorocyclopentadiene	77-47-4	10	0.25
Hexachloroethane	67-72-1	10	0.25
Indeno(1,2,3-c,d)Pyrene	193-39-5	10	0.25
Isophorone	78-59-1	10	0.25
Naphthalene	621-64-7	10	0.25
Nitrobenzene	62-75-9	10	0.25
N-Nitrosodimethylamine	86-30-6	20	0.5
N-Nitroso-di-n-propylamine	91-20-3	10	0.25

Table 1 (cont)

Compound Name	CAS number	Reporting Limits	
		Water (µg/L)	Soil (mg/kg)
N-Nitrosodiphenylamine	98-95-3	10	0.25
Pentachlorophenol	87-86-5	20	0.5
Phenanthrene	85-01-8	10	0.25
Phenol	108-95-2	10	0.25
Pyrene	129-00-0	10	0.25
Pyridine	110-86-1	20	0.5

2.0 SUMMARY OF METHOD

2.1 Soil and solid samples are prepared by SW-846 Method 3541, Automated Sohxlet Extraction. Water samples are prepared by SW-846 Method 3510, Separatory Funnel Extraction.

2.2 The semivolatile compounds are introduced into the GC/MS by injecting the sample extract into a gas chromatograph (GC) with a narrow-bore fused-silica capillary column. The GC column is temperature-programmed to separate the analytes, which are then detected with a mass spectrometer (MS) connected to the gas chromatograph.

2.3 Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a calibration curve with a minimum of five contiguous data points.

3.0 DEFINITIONS

3.1 **Method Blank:** An aliquot of reagent water, which is carried through the entire analytical procedure with the samples, its purpose is to determine whether contamination is present during the analysis. A method blank is analyzed 1 for every analytical batch of samples.

3.2 **Laboratory Control Sample:** An aliquot of reagent water which known quantities of the method analyte are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine the degree to which the analytical result approaches the "true value" of the concentration of the analyte being determined. The percentage recovery is calculated in order to assess the efficiency of the analysis. One (1) LCS is analyzed for every analytical batch of samples.

3.3 **Matrix Spike:** A specific aliquot of sample into which a known amount of analyte is added. The analytical spike is analyzed with the sample batch and the percent recovery is calculated in order to assess the matrix effect on the analytical system.

3.4 **Batch:** A group of 20 or fewer samples extracted (prepared) at the same time, in the same location, using the same method. Associated with each batch is a set of QC samples, including: Duplicate Samples, Matrix Spike(MS), Matrix Spike Duplicate (MSD), Laboratory Control Sample (LCS), and a Method Blank.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Store the sample extracts at 4°C ±2°C, protected from light, in sealed vials (e.g. screw-cap vials or crimp-capped vials) equipped with unpierced PTFE-lined septa. Analysis: 40 days after extraction.

5.0 POTENTIAL PROBLEMS AND INTERFERENCES

5.1 Contamination by carryover can occur whenever high-concentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, the analysis immediately following it is evaluated for cross-contamination.

5.2 The following compound specific problems are noteworthy.

5.2.1 Benzidine may be subject to oxidative losses during solvent concentration and its chromatographic behavior is poor.

5.2.2 Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition.

5.2.3 N-nitrosodimethylamine and Pyridine elute very early and become more difficult to separate from the solvent front as the column is shortened through maintenance clipping.

5.2.4 N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine.

5.2.3 Pentachlorophenol, 2,4-dinitrophenol, 4-nitrophenol, benzoic acid, 4,6-dinitro-2-methylphenol, 4-chloro-3-methylphenol, 2-nitroaniline, 3-nitroaniline, 4-chloroaniline, and benzyl alcohol are subject to erratic chromatographic behavior, especially if the GC system is contaminated with high boiling material.

6.0 HEALTH AND SAFETY

6.1 AMRO has a laboratory safety program in place that applies to all employees. There is a safety officer as well as employees with 40 hours of OSHA training. The air quality of the laboratory has been monitored and employees are sent for physicals. All employees receive fire and safety training upon employment.

6.2 Fire extinguishers are located in various areas of the lab. A lab evacuation plan is posted in every room, with a designated meeting place.

6.3 All new analysts receive training on the safe handling of samples, standards, the changing of gas cylinders, and the operation of instruments. In the lab, clean laboratory coats and gloves are worn when working with samples. Neat standards are made up in hoods. When gas tanks are replaced, the carrier with chain is used properly, and all gas tanks are chained in place. Soap is used to check for any leaks.

6.4 Care is taken with glassware and syringes to prevent injury. Broken glass and syringes are promptly removed to broken glass containers. First aid kits are available throughout the lab. There are also eyewash stations and an emergency shower.

6.5 MSDS's are reviewed for each chemical used to ensure proper precautions are observed when handling chemicals.

6.6 Pollution prevention: The use of chemical exhaust hood are required when working with chemicals. They are specifically designed to minimize analyst exposure. The hood exhaust is monitored periodically to ensure environmental exposure is minimized.

6.7 Waste control, AMRO takes whatever steps possible to minimize waste. This includes volume reduction where appropriate. AMRO also properly segregates wastes according to contaminants and a commercial vendor is utilized for waste disposal.

7.0 INSTRUMENTS AND EQUIPMENT

7.1 Gas Chromatograph/Mass Spectrometer Systems

7.1.1 Hewlett-Packard (HP) Model 5890 Series II (SV-2, SV-3) -temperature-programmable gas chromatographs equipped with Electronic Pressure Control (EPC), splitless injection, capillary-direct interface, and Model 7673 Autosampler/Injector. Hewlett-Packard (HP) Model 6890 (SV-4) - temperature-programmable gas chromatograph equipped with Electronic Pressure Control (EPC), split injection, capillary-direct Interface and Model 7683 Autosampler/injector.

7.1.2 Column - 30 m x 0.25 mm ID, 0.25 um film thickness 5% phenyl methyl silicone-coated fused-silica capillary column (J&W Scientific DB-5MS, Catalog No. 122-5532 or equivalent).

7.1.3 Hewlett-Packard (HP) Model 5972 Mass Selective Detector (MSD) , SV-2 and SV-3. Hewlett-Packard (HP) Model 5973 Mass Selective Detector (MSD) SV-4. Note SV-2 and SV-3 are backup instruments only for the SW-846 Method 8270 and do not routinely perform full BNA analyses.

7.1.4 Hewlett-Packard Chemstation PC Data system - Computer system interfaced to the GC/MSD.

The following system identifiers have the listed software versions:

SV-2	G1034C Version C.02.05
SV-3	G1701AA Version A.03.00
SV-4	G1701BA Version B.00.00

The system allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer has software that can search any GC/MS data file for ions of a specific mass and that can plot such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). The software allows integrating the abundances in any EICP between specified time or scan-number limits. The most recent version of the EPA/NIST Mass Spectral Library is also available.

7.2 Syringe - 10 µL, 25µl - 1.0 mL varying sizes

7.3 Volumetric flasks: Class A - Appropriate sizes with ground-glass stoppers.

7.4 Bottles - 1.5 to 2.0mL amber and clear glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops for standard storage.

7.5 5.0mL amber vials with PFTE-lined screw caps.

7.6 Compressed Helium - Ultra High Purity (UHP) grade.

8.0 REAGENTS AND STANDARDS

8.1 The stock standard solutions are purchased from commercial suppliers as certified solutions. Transfer the stock standard solutions into 2mL amber vials with PTFE-lined screw caps using a pasteur pipet. Label vial with content information, standard ID and expiration date. Store, protected from light, at 4°C±2°C. Stock standard solutions must be replaced after one year from the opening date of the vial, or on the manufacturer expiration date, whichever is shorter.

8.1.1 Stock Calibration Standards

Restek:

Semivolatiles Calibration Standards
Contain1mL of each of the following products at:
8270 Mega Mix (Catalog No.: 31850) 500-1000 µg/ml
Benzoic Acid Mix (Catalog No.: 31879) 2000 µg/ml
Benzidines Mix (Catalog No.: 31852) 2000 µg/ml
Custom Acetophenone (2,000 µg/ml) Catalog No. 558859

8.1.2 Calibration Standards: Second Source Verification Standards

Ultra Scientific, Inc.:

Pyridines Mixture	2000 µg/ml	Catalog No. US-120AN
Benzidines Mixture	2000 µg/ml	Catalog No. US-105N
SVOA Mix 1	1000 µg/ml	Catalog No. SVM-8270
SVOA Mix 2	1000 µg/ml	Catalog No. SVM-8271

8.1.3 Surrogate Solutions: The surrogates are Phenol-d_6, 2-Fluorophenol, 2,4,6-Tribromophenol, Nitrobenzene-d_5, 2-Fluorobiphenyl, and p-Terphenyl-d_{14}.

Restek, Inc.:

Base/Neutral Surrogates, 5,000 µg/mL (Catalog No. 31086)
Nitrobenzene-d5
Fluorobiphenyl
P-Terphenyl-d14

Acid Surrogates, 10,000µg/mL (Catalog No. 31063)
Phenol-d5
2-Fluorophenol
2,4,6-Tribromophenol

8.1.4 Internal Standard Solutions: The internal standards are 1,4-Dichlorobenzene-d_4, Naphthalene-d_8, Acenaphthene-d_{10}, Phenanthrene-d_{10}, Chrysene-d_{12}, and Perylene-d_{12}. Restek Catalog No.: 31206 is purchased and used as is directly from manufacturer.

8.1.4.1 The solution contains each compound at a concentration of 2,000 ng/µL. Each 1mL sample extract undergoing analysis should be spiked with 20µL of the internal standard solution, resulting in a concentration of 40ng/ µL of each internal standard. Store at 4°C±2°C when not in use.

8.1.5 GC/MS Tuning Standard: A mix of Decafluorotriphenylphosphine (DFTPP), Pentachlorophenol, Benzidine, and DDT is commercially available stock solution from Restek, Catalog No.: 31615 (1,000µg/mL).

8.1.6 Laboratory Control Sample spike stock solutions: Restek

Mega Mix	500-100 µg/ml	Catalog No.: 31850
Benzidines Mix	2000 µg/ml	Catalog No: 31852
Benzoic acid	2000 µg/ml	Catalog No.: 31879
CAL Mix 1	2000 µg/ml	Catalog No.: 31618
Custom Acetophenone	2000 µg/ml	Catalog No.: 558859

8.2 Intermediate Solutions

8.2.1 Preparation of Intermediate Calibration stock at 250µg/ml Using Primary Source Standards

Prepare each individual commercial calibration stock solutions according to the manufacturers instructions. Transfer each of the calibration stock solutions into 1.5mL amber vials with teflon lined screw caps using pasteur pipets. Label each vial with contents, standard ID and expiration date. Clean a 5mL volumetric flask and stopper with methylene chloride. Do not add any methylene chloride to the flask until all solutions have been added. Using a clean 500µl syringe, transfer 625µl of each 2,000 µg/mL calibration stock solution and 1250µl of the Mega Mix calibration stock solution (500-1000 µg/ml) into the volumetric flask. Rinse the syringe between each aliquot with clean methylene chloride and replace the stopper. Using a clean syringe, transfer 250µl of the Base Neutral Surrogate (5,000 µg/mL) to the volumetric flask and replace the stopper. Using a clean syringe, transfer 125µl of the acid surrogate (10,000 µg/mL) to the volumetric flask and replace the stopper. Using clean methylene chloride carefully bring the meniscus to the mark on the flask. Use extreme caution to ensure proper volume. Transfer contents of the volumetric to a clean 5mL amber vial with a PFTE-lined screw cap using a pasteur pipet. Label vial with contents, standard ID and expiration date. Using a Sharpee, mark the meniscus with a line to ensure no evaporation has taken place when not in use, store protected from light at 10°C-20°C.

8.2.2 Preparation of Intermediate Calibration Standard Stock at 250µg/mL - Secondary Source Verification

Prepare each individual commercial calibration stock solutions according to the manufactures instructions. Transfer each of the calibration stock solutions into 1.5mL amber vials with teflon lined screw caps using pasteur pipets. Label each vial with contents, standard ID and expiration date. Clean a 5mL volumetric flask and stopper with methylene chloride. Do not add any methylene chloride to the flask until all solutions have been added. Using a clean syringe, transfer 625µl of each 2,000 µg/mL calibration stock solution and 1250µl of each 1000 µg/mL calibration stock solution into the volumetric flask. Rinse the syringe between each aliquot with clean methylene chloride and replace the stopper. Using a clean syringe, transfer 250µl of the Base Neutral Surrogate (5,000 µg/mL) to the volumetric flask and replace the stopper. Using a clean syringe, transfer 125µl of the acid surrogate (10,000 µg/mL) to the volumetric flask and replace the stopper. Using clean methylene chloride carefully bring the meniscus to the mark on the flask. Use extreme caution to ensure proper volume. Transfer contents of the volumetric to a clean 5mL amber vial with a PFTE-lined screw cap using a pasteur pipet. Label vial with contents, standard ID and expiration date. Using a Sharpee mark the meniscus with a line to ensure no evaporation has taken place. When not using, store protected from light at -10 to -20C.

8.3 Working Standard Solution

8.3.1 Calibration standards - Eight calibration standards are prepared at eight different concentrations using the 250µg/mL intermediate standard, including 5.0, 10, 20, 50, 80, 120, 160 and 250µg/mL. Each point is prepared in a 1.5mL amber vial with a solid teflon-lined screw cap. Label each point with standard ID, concentration, expiration date and initials. Using a Sharpee mark the meniscus with a line to ensure no evaporation when not in use.

8.3.1.1 All working calibration standards are stored at -10 to -20°C and are freshly prepared if the calibration verification standard indicates a problem. The CCV working standard is stored at 4 ±2 °C.

8.3.1.2 A eight point curve is prepared by diluting the above stock with clean methylene chloride according to the chart below:

Calibration Point	Amt. of stock (µL)	Final Volume	Amt. of Internal Std
250ng/µl	500	0.5mL	10µl
160ng/µl	640	1.0mL	20µl
120ng/µl	480	1.0mL	20µl
80ng/µl also CCV	320	1.0mL	20µl
50ng/µl	200	1.0mL	20µl
20ng/µl	80	1.0mL	20µl
10ng/µl	40	1.0mL	20µl
5.0ng/µl	20	1.0mL	20µl

8.3.1.3 Low limit check standard.

A 1.0ng/µl standard is analyzed to check the lower limit of the system. This point is not used in the calibration curve.

8.3.2 Secondary Source Working Stock Calibration point using 225µg/mL Intermediate Calibration Stock

A single point 80µg/mL is prepared by diluting the secondary source stock with clean methylene chloride. Fill a clean 2mL volumetric frask with 500µl of clean methylene chloride. Using a clean 1mL syringe, transfer 640µl of the above stock into the 2mL volumetric and carefully bring to volume. Add 40µl of semivolatile internal standard to the volumetric and transfer to a clean 5mL amber screw cap vial, label with standard ID, concentration and expiration date. Using a Sharpee, mark the meniscus with a line to ensure proper volume.

8.3.3 Surrogate Standard Check: Dilute the working surrogate standard solution by a factor of two in methylene chloride. Inject this into the GC/MS to determine recovery of surrogate standards. Recovery of all surrogates must be between 95 - 110 % of true value before use in samples.

True values of the surrogates are as follows:

Base/Neutral Surrogates	50µg/mL on column
Nitrobenzene-d5	
Fluorobiphenyl	
p-Terphenyl-d14	
Acid Surrogates	75 µg/mL on column
Phenol-d5	
2-Fluorphenol	
2,4,6-Tribromophenol	

8.3.4 Laboratory Control Sample spike working solution:

Prepare each individual commercial LCS stock solutions according to the manufactures instructions. Transfer each of the calibration stock solutions into 1.5mL amber vials with teflon lined screw caps using pasteur pipets. Label each vial with contents, standard ID and expiration date. Clean a 10mL volumetric flask and stopper with methylene chloride. Do not add any methanol to the flask until all solutions have been added. Using a clean 500µL syringe, transfer 1.0mL of the CLP mix, 250µL of the phenols mix, 500µL of the benzidines mix, 200uL of the NNDMA stock, 500µL of the benzoic acid stock, 500µL of the benzyl alcohol stock, and 500uL of the pyridine stock into the volumetric flask. Rinse the syringe between each aliquot with clean

methylene chloride and replace the stopper. Using clean methanol carefully bring the meniscus to the mark on the flask. Use extreme caution to ensure proper volume. Transfer contents of the volumetric to a clean 5mL amber vial with a PFTE-lined screw cap using a pasteur pipet. Label vial with contents, standard ID and expiration date. Using a Sharpee mark the meniscus with a line to ensure no evaporation has taken place. When not using, store protected from light at -10 to -20°C.

8.3.5 GC/MS tuning standard:

Containing decafluorotriphenylphosphine (DFTPP), pentachlorophenol, benzidine, and DDT is prepared by diluting the stock solution with methylene chloride to form a solution containing 50 ng/µL. Clean a 2mL volumetric flask and stopper with methylene chloride. Add a small amount of clean methylene chloride to the flask and replace the stopper. Using a clean syringe transfer 100µL of DFTPP stock into the 2mL volumetric flask. Using clean methylene chloride slowly bring meniscus up to the mark on the volumetric flask. Transfer the contents of volumetric flask to a clean 5 mL amber PFTE-lined screw cap vial. Label vial with concentration, standard ID and expiration date. Using a Sharpee, mark the meniscus with a line to ensure no evaporation has occurred when not in use. Store at 4°C±2°C (per vendors recommendation) when not in use.

8.3.6 Matrix Spike Check:

Dilute the working matrix spike standard solution by a factor of two in Methylene Chloride. Inject this into the GC/MS to determine recovery of matrix spike compounds. Recovery of all matrix spike compounds must be between 95 -110 % of true value before use in samples. True values of the spiking solution are as follows:

BASE/NEUTRAL SPIKES	µg/mL on column
1,4-Dichlorobenzene	50
n-Nitroso-di-N-propylamine	50
1,2,4-Trichlorobenzene	50
Acenaphthene	50
2,4-Dinitrotoluene	50
Pyrene	50

ACID SPIKES	µg/mL on column
Phenol	75
2-Chlorophenol	75
4-Chloro-3-Methylphenol	75
4-Nitrophenol	75
Pentachlorophenol	75

8.3.6.1 Some projects may require the spiking of the specific compounds of interest. When this occurs, the matrix and LCS spiking standards should be prepared in methanol, with each compound present at a concentration appropriate for the project.

8.4 Acetone, pesticide residue quality or equivalent.

8.5 Hexane, pesticide residue quality or equivalent.

8.6 Methanol, pesticide residue quality or equivalent.

8.7 Methylene chloride, pesticide residue quality or equivalent.

8.8 TRACKING OF ANALYTICAL STANDARDS

8.8.1 Chemical Receipt

The receipt of all standards, neat compounds and reagents from commercial sources is documented in the Chemical Receiving Logbook by sample receiving personnel or designate. Each solution or compound is assigned a unique standard receipt ID number, the letter "A" and a four digit sequential number. The date received, source, lot number, expiration date and disposal date are recorded.

8.9 Standards Preparation:

Standards are prepared as written in the respective SOPs. In order to assure the accuracy of standards the following general guidelines are used:

8.9.1 AMRO purchases pesticide residue grade solvents and purge and trap grade Methanol for organic standard preparation and organic sample extractions.

8.9.2 ACS reagent grade chemicals are used when the method warrants.

8.9.3 Any stock standards that are purchased are accompanied by a certificate of analysis from the manufacturer.

8.9.4 Class A volumetric glassware is used to prepare standards.

8.10 Standard Tracking:

It is important to be able to track all analytical standards back to their sources. The following procedure is used by AMRO personnel to accomplish this:

8.10.1 Standards, neat compounds and reagents from commercial sources is documented in the Chemical Receiving Logbook by sample receiving personnel or designate. Each solution or compound is assigned a unique standard receipt ID number, which consists of the letter "A" and a four digit sequential number. The date received, source, lot number, expiration date and disposal date are recorded. The expiration dates are provided from the manufacturer and the disposal dates are completed by AMRO employees when standards are used up or disposed. Refer to the Certificate of Analysis Logbook for standard concentration information. The Certificates are kept on file in the GC/MS department.

8.10.2 Standards Preparation

Standards are prepared as written in the respective SOPs. In order to assure the accuracy of standards the following general guidelines are used:

AMRO purchases pesticide residue grade solvents and purge and trap grade Methanol for organic standard preparation and organic sample extractions.

Any stock standards that are purchased are accompanied by a certificate of analysis from the manufacturer.

Class A volumetric glassware is used to prepare standards.

8.10.3 Organic Standards:The Organic Standards Tracking Log consists of three sections, an Index, a Stock section and a Working section. Stock Standards are defined as any solution that requires a dilution or addition to multiple solutions and are used to make Working Standards. Working Standards are defined as anything that is added directly to a sample or analyzed directly on an instrument.

8.10.3.1 The ID index assures that no two standards are assigned the same ID number. The Index requires the Standard ID, the section of the Standard Log (Stock or Working) the Standard information is located, and a brief description of the standard.

8.10.3.2 Stock Section: Whenever a stock standard solution is opened or prepared, this information is recorded in the Stock Standard Logbook section of the standard tracking log.

8.10.3.2.1 Date prepared and Prepared by are the first two items to be completed. The next is the compound name, which should be the name of the solution being used. Do not use vendor catalog numbers alone.

8.10.3.2.2 The Receipt/Stock ID is the ID of the solution being used. This can be the "A" number or a Stock ID number.
The Source of the solution is the manufacturer.
The concentration or purity of the solution is the next item.

8.10.3.2.3 The amount in grams (g) or microliters (μL) of the solution used and the final volume of the solution made is documented. The final concentration of the new solution and the units is recorded.

The stock solution is assigned a new ID number consisting of the letter "S" for semivolatile, the date of preparation plus a letter suffix starting with the letter "A". This ID along with the compound name, concentration and initials of the person making the stock are included on the label. Example - The fourth semivolatile standard prepared on April 15, 1989 would have the standard ID S041589D. This number will be recorded in the logbook and written on the container along with expiration date and analyst initials.
The storage location is recorded in the logbook.
The expiration date is the last item recorded. Stock standards are good one year from the date opened or the date provided by manufacturer, whichever is shorter. The analyst must verify that 1 year from the date opened is within the manufacturers expiration date window. If it is not, then the manufacturer's expiration date must be used for the AMRO expiration date.

8.10.4 Working Section - Whenever a working standard solution opened or is prepared, whether from AMRO stocks or commercially obtained stocks, the information is recorded in the Working Standard section of the standard tracking log.

- Date opened/prepared and Opened/Prepared by are the first two items to be completed.

The next is the compound name, which should be the name of the solution being used. Do not use vendor catalog numbers alone.
The Receipt/Stock ID is the ID of the solution being used. This can be the "A" number or a Stock ID number. If a stock ID number was previously assigned, use the stock ID number rather than the "A" number for traceability.
The Source of the solution is the manufacturer.
The concentration or purity of the solution is the next item.
The amount in microliters (uL) of the solution used and the final volume of the solution made is documented.
The final concentration of the new solution and the units is recorded.
The stock solution is assigned a new ID number consisting of the letter "S" for semivolatile, the date of preparation plus a letter suffix starting with the letter "A". This ID along with the compound name, concentration and initials of the person making the stock are included on the label. Example - The fourth semivolatile standard prepared on April 15, 1989 would have the standard ID S041589D. This number will be recorded in the logbook and written on the container along with expiration date and analyst initials.
The storage location is recorded in the logbook.
The expiration date is the last item recorded. Working standards are good one year from the date opened or the oldest date prepared of any compound stock standard or the date provided by manufacturer, whichever is shorter. The daily CCV standards are good for one week only. The analyst must verify that 1 year from the date opened is within the manufacturers expiration date window. If it is not than the manufacturer date must be used for the expiration date.

8.11 Standard Storage - Standards and reagents are stored per method specifications or manufacturer's instructions. Semivolatile standards are stored in refrigerators at 4 +/- 2°C, or in freezers at below minus 10 °C, and/or ambient temperature depending upon the method and manufacturer requirements.

8.12 Solvent Check Analysis

All extraction procedures are performed using pesticide residue grade solvents and a method or procedural blank analysis is performed when a new lot number is received to verify the absence of target analytes.

9.0 PROCEDURE

9.1 Sample Preparation

9.1.1 All Samples are normally prepared by one of the following Methods prior to GC/MS analysis

Matrix	SW-846 Methods	AMRO SOP
Water	3510	OL-2003
Soil/Solids	3541	OL-2004
Waste	See supervisor	

9.1.2 Sample extracts are prepared by adding 2µl of internal standard solution per 100µl of extract (or diluted extract) that is dispensed into a 2mL crimp vial.

9.1.3 AMRO does not routinely perform any cleanups of extracts for semivolatile analyses, however if significant non-target analyte interference exists for aniline and aniline derivatives, or phthalate esters, or nitrosamines, or nitroaromatics and cyclic ketones, or haloethers, or chlorinated hydrocarbons, then the Florisil cleanup (OL-2018, SW-846 Method 3620B) may be used to remove interferences.

9.2 GC/MS Conditions

Attachment 7 contains the specific instrument operating conditions. These parameters are contained in method file *BNAmmdd.m;* where mm = the month, and dd=the day of calibration, or any similar format incorporating the date of calibration. Method calibration files should be archived with the data files at the time of back-up on the file server

9.3 Hardware Tuning - The GC/MS system must be hardware-tuned using a 50 ng injection of DFTPP. Analyses must not begin until the tuning criteria are met.

9.3.1 Under the HP ChemStation drop down menu item, select Tune. In the drop down menu select Autofind DFTPP to screen or printer. This macro selects the peak apex plus or minus one scan average mass spectrum and does an automatic background correction. Alternately, the whole peak average spectrum and a single scan background correction may be evaluated. Another alternative is the use of a single passing scan near

the TIC peak apex after the background scan correction. All background correction scans must be before the DFTPP peak but not more than 20 scans before the DFTPP apex. In either case, a hard copy showing the passing spectrum and passing criteria must be printed, dated and initialed by the analyst.

9.3.2 Use the DFTPP mass intensity criteria as tuning acceptance criteria.

MASS	ION ABUNDANCE CRITERIA 8270
51	30-60 % of mass 198
69	present
68	< 2% of mass 69
70	< 2% of mass 69
127	40-60 % of mass 198
197	< 1% of mass 198
198	Base peak, 100% relative abundance
199	5-9 % of mass 198
275	10-30 % of mass 198
365	> 1% of mass 198
441	Present but less than mass 443
442	> 40% of mass 198,
443	17-23 % of mass 442

NOTE: All subsequent standards, sample, MS/MSDs, and blanks associated with a DFTPP analysis must use the identical mass spectrometer instrument conditions.

9.4 Initial Calibration

9.4.1 Using the HP 7673 autosampler, inject 1µl of each calibration standard (containing internal standards). Seven calibration levels are injected: 5.0 ng/µl, 10 ng/µl, 20 ng/µl, 50 ng/µl, 80 ng/µl, 120 ng/µl, and 160 ng/µl. An injection volume of 1µl is used for all standards and sample extracts. A set of at least 5 calibration standards is required. The default quantitation ions and association of target analytes and internal standards is listed in Attachments 1A, 1B, and 1C.

The data system software calculates relative response factors (RRFs) for each target analyte relative to one of the internal standards as follows:

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

where:

A_s = Peak area of the analyte's quantitation ion
A_{is} = Peak area of the internal standard's quantitation ion
C_s = Mass of analyte injected in nanograms (ng)
C_{is} = Mass of internal standard injected in nanograms (ng)

9.4.2 System performance check compounds (SPCCs)

9.4.2.1 System Performance Check Compounds (SPCCs) are: N-nitroso-d-n-propylamine; hexachlorocyclopentadiene; 2,4-dinitrophenol; and 4-nitrophenol. The minimum acceptable average RRF for these compounds is 0.050 in the initial calibration curve. If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

9.4.3 Calibration check compounds (CCCs) are as follows:

Base/Neutral Fraction	Acid Fraction
Acenaphthene	4-Chloro-3-Methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitrosodiphenylamine	Phenol
Di-n-octyl phthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

9.4.3.1 The data system calculates the mean response factor and the relative standard deviation (RSD) of the response factors for each target analyte. The RSD should be less than or equal to 15% for each target analyte. However, the RSD for each individual CCC must be less than or equal to 30%.

$$RSD = \frac{SD}{RF} * 100$$

Where: SD = Standard Deviation
RF = Relative Response Factor

9.4.3.2 If the RSD of any CCC is greater than 30%, then the chromatographic system is too reactive for analysis to begin. Evaluate possible causes of the problems, perform additional maintenance which may include replacing the injector liner, clipping 2 to 3 inches off the front of the capillary, or replacing the capillary column, then repeat the calibration procedure.

9.4.4 Evaluation of retention times - The relative retention time (RRT) of each target analyte in each calibration standard must agree within 0.06 RRT units.

9.4.5 Linearity of target analytes - If the RSD of any target analyte is 15% or less, then the relative response factor is assumed to be constant over the calibration range, and the average relative response factor can be used for quantitation.

9.4.5.1 If the RSD of any target analyte is greater than 15%, linear regression or quadratic equation may be used but the Coefficient of Determination (COD) (r^2) must be greater than 0.99 for the calibration to be valid for the analyte.

9.4.5.2 Several compounds may not exhibit sufficient response at the lower calibration levels due to their chemical properties. These compounds are indicated in Table 1 by a Reporting Limit of 20 ug/L in water. If the %RSD criteria cannot be met for any of these compounds with all seven data points then up to the two lowest points (i.e. the 5.0 and 10 ug/ml calibration points) may be excluded from the calibration curve to meet the criteria. If the criteria cannot be met in this manner then further investigation into the problem must be initiated. If there is a subset of compounds to be reported, then the curve may be used for those which meet the criteria; e.g PAHs only.

9.4.6 A second source initial calibration verification standard (ICV) is analyzed after the initial calibration. All analytes must be within 25% of the expected value.

9.5 GC/MS calibration verification - Calibration verification consists of five evaluation steps that are performed prior to sample extract analysis at the beginning of each 12 hour shift.

9.5.1 Prior to the analysis of samples or calibration standards, inject 50 ng of the tune standard into the GC/MS system. The resultant mass spectrum for DFTPP must meet the criteria given in section 9.3.2 before sample analysis begins. Using the Enviroquant "Tailing" macro in QEDIT, evaluate and

hardcopy the tailing factor for pentachlorophenol and benzidine. See Section 9.5.2.3 for the formula used. The tailing factors must be less than 5 and 3, respectively. Evaluate the DDT degradation using the following formula: ((DDD+DDE) / (DDT+DDD+DDE))*100 = %degradation. DDT degradation must be less than 20%. These criteria must be demonstrated before each 12-hour shift during which samples are analyzed.

9.5.2 The initial calibration for each compound of interest is verified once every 12 hours prior to sample analysis, using the introduction technique and conditions used for samples. This is accomplished by analyzing the 80 ng/µl calibration standard and verifying that the following criteria are met. All analytical queues containing USACE project samples must be followed by a closing CCV. CCV criteria do not apply to the closing standard and the results do not need to be reported, but the data must be available to USACE inspection if requested.

9.5.2.1 System performance check compounds (SPCCs) - A system performance check must be made during every 12-hour analytical shift. Each SPCC in the calibration verification standard must meet a minimum response factor of 0.050. This is the same check that is applied during the initial calibration.

If the minimum response factors are not met, the system must be evaluated, and corrective action must be taken before sample analysis begins. Possible problems include standard mixture degradation, injection port inlet contamination, contamination at the front end of the analytical column, and active sites in the column or chromatographic system. This check must be met before sample analysis begins.

9.5.2.2 Calibration check compounds (CCCs) - After the system performance check is met, the CCCs listed in Section 7.4.3 are used to check the validity of the initial calibration. Use percent difference when performing the average response factor model calibration. Use percent drift when calibrating using a regression or quadratic fit model. If the percent difference or drift for each CCC is less than or equal to 20%, then the initial calibration is assumed to be valid. If the criterion is not met (i.e., greater than 20% difference or drift) for any one CCC, then corrective action must be taken prior to the analysis of samples. If the CCCs are not included in the list of analytes for a project, then all analytes which will be reported must meet the 20% difference or drift criterion. Problems similar to those listed under SPCCs could affect the CCCs. If the problem cannot be corrected by other measures, a new initial

calibration must be generated. The CCC criteria must be met before sample analysis begins.

9.5.2.3 Peak tailing will be evaluated by using the Enviroquant "Tailing" macro in QEDIT for each tune mix standard run and printed. Tailing factor is calcluated by the software and must be <5 for Pentachlorophenol and <3 for Benzidine. The formula is as follows:

$$\text{Peak Tailing} = \frac{B*C}{A*B}$$

9.5.3 A method or solvent blank must be analyzed after the calibration standard, or at any other time during the anlytical shift, to ensure that the total system (introduction device, transfer lines and GC/MS system) is free of contaminants. If the sample extracts in the 12 hour analysis shift are being analyzed solely for dilution of target analytes exceeding calibration range, and the method blank analyzed with the sample extract was previously demonstrated to be free of contaminants, then a method or solvent blank is not required to be analyzed with the reanalysis for dilution. See the following for method blank performance criteria.

9.5.3.1 Less than half of the Reporting Limit for the matrix for the specific analyte.

9.5.3.2 Less than a level specified in a quality assurance project plan specific to the associated samples.

9.5.3.3 Less than the MDL check sample concentration for all batches containing USACE project samples. See Section 9.1.1.

If the method blank exceeds these criteria then an investigation will be initiated.

9.5.4 Internal standard retention time - The retention times of the internal standards in the calibration verification standard must be evaluated immediately after data acquisition. If the retention time for any internal standard changes by more than 30 seconds from that in the mid-point standard level of the most recent initial calibration sequence, then the chromatographic system must be inspected for malfunctions and corrections must made, as required. When corrections are made, reanalysis of samples analyzed while the system was malfunctioning is required. AMRO also evaluates all samples and batch QC against the daily CCV using the same

criteria. The decision to re-analyze samples due to internal standards falling outside of the windows is based on analysts' judgement of data impact.

- If examination of the total ion chromatogram shows no evidence of matrix interference or elevated baseline, the extract should be reanalyzed to determine if there is some other matrix effect behavior or that the added internal standard solution is no longer acceptable for use. If reanalysis exhibits the same behavior then the analyst will report the data and indicate the problem on the Data Review Checklist with some assessment of data impact.

- If examination of the total ion chromatogram shows evidence of matrix interference or elevated baseline, the analyst should evaluate whether a dilution is warranted due to peak distortion. If there are no chromatographic anomalies, the data is reported as is since the internal standard is designed to allow for some matrix effects or inlet discrimination.

9.5.5 Internal standard response - if the EICP area for any of the internal standards in the calibration verification standard changes by a factor of two (-50% to +100%) from that in the mid-point standard level of the most recent initial calibration sequence, the mass spectrometer must be inspected for malfunctions and corrections must made, as required. When corrections are made, reanalysis of sample analyzed while the system was malfunctioning is required. AMRO also evaluates all samples and batch QC against the daily CCV using the same criteria. The decision to re-analyze samples due to internal standards falling outside of the windows are based on analysts' judgement of data impact.

- If examination of the total ion chromatogram shows no evidence of matrix interference or elevated baseline, the extract should be reanalyzed to determine if there is some other matrix effect behavior or that the added internal standard solution is no longer acceptable for use. If reanalysis exhibits the same behavior then the analyst will report the data and indicate the problem on the Data Review Checklist report with some assessment of data impact.

- If examination of the total ion chromatogram shows evidence of matrix interference or elevated baseline, the analyst should evaluate whether a dilution is warranted due to peak distortion. If there are no chromatographic anomalies, the data is reported as is since the internal standard is designed to allow for some matrix effects or inlet discrimination.

9.6 GC/MS analysis of samples

9.6.1 Allow the sample extract to warm to room temperature. Add 2 µL of the internal standard per 100 µL of concentrated sample extract or diluted extract obtained from sample preparation.

9.6.2 Inject a 1µL aliquot of the sample extract into the GC/MS system, using the same operating conditions that were used for the calibration.

9.6.3 If the response for any quantitation ion exceeds the initial calibration range of the GC/MS system, the sample extract must be diluted and reanalyzed.

9.6.4 Monitor internal standard retention times and responses (area counts) in all samples, spikes, blanks, and standards to effectively check drifting method performance, poor injection execution, and anticipate the need for system inspection and/or maintenance.

9.6.5 The use of Selected Ion Monitoring (SIM) is acceptable for applications requiring detection limits below the normal range of electron impact mass spectrometry. However, SIM provides a lesser degree of confidence in compound identification.

9.7 Qualitative analysis

9.7.1 The qualitative identification of compounds determined by this method is based on retention time and on comparison of the sample mass spectrum, after background correction, with characteristic ions in a reference mass spectrum. The reference mass spectrum must be generated by using the conditions of this method. The characteristic ions from the reference mass spectrum are defined as the three ions of greatest relative intensity, or any ions over 30% relative intensity, if less than three such ions occur in the reference spectrum. Compounds are identified when the following criteria are met.

9.7.1.1 The intensities of the characteristic ions of a compound must maximize in the same scan or within one scan of each other. Selection of a peak by the HP data system target compound search routine where the search is based on the presence of a target chromatographic peak containing ions specific for the target compound at a compound-specific retention time will be accepted as meeting this criterion.

9.7.1.2 The RRT of the sample component is within +/- 0.06 RRT units of the RRT of the standard component.

9.7.1.3 The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

Isometric pairs are resolved >90% to baseline except in the case of Benzo(b)fluoranthene and Benzo(k)fluoranthene. In this case, manual integration is performed by drawing parallel to the baseline and then stopping at the baseline point where the valley between the two isomers is deepest. This integration is performed on all calibration standards. Samples are integrated in the same way as the standards.

9.7.1.4 Identification is hampered when sample components are not resolved chromatographically and produce mass spectra containing ions contributed by more than one analyte. When gas chromatographic peaks obviously represent more than one sample component, i.e. a broadened peak with shoulder(s) or a valley between two or more maxima, appropriate selection of analyte spectra and background spectra is important.

9.7.1.5 Examination of extracted ion current profiles of appropriate ions can aid in the selection of spectra and in qualitative identification of compounds. When analytes co-elute (i.e. only one chromatographic peak is apparent), the identification criteria may be met, but each analyte spectrum will contain extraneous ions contributed by the co-eluting compound.

9.8 Quantitative analysis

9.8.1 Once a compound has been identified, the quantitation of that compound will be based on the integrated abundance of the primary characteristic ion from the EICP.

9.8.2 Isomeric pairs will be reported as individual compounds by performing integration in the same manner that the standards were integrated.

9.8.3 The integration of compounds for standards and samples should be performed to be in the same manner as the initial calibration integration. Cases where the initial calibration is in question should be investigated to

ensure it is not related to poor maintenance, damaged or defective column, or other issues which should be solved before further analysis.

9.8.4 The following must be checked for each compound present in standards and samples to ensure proper integration has been performed.

9.8.4.1 The peak shape of the calibration compound must be maintained for standards and samples. This should take into account any shouldering, peak tailing, and peak co-elution.

9.8.4.2 The retention time windows for each compound of the calibration must be applied to standards and samples. If there is a retention time shift confirmed by comparison of the Internal Standards and Surrogates then pattern recognition can be utilized for identification of multi-component analytes or known isomer relative retention time patterns, e.g., benzo(b) and benzo(k) fluoranthene, dibenz(a,h)anthracene and indeno(1,2,3,cd)pyrene. If the retention time shifts are serious enough to cause potential false negatives or quantitation errors for many compounds, then re-calbration is required.

9.8.4.3 The baseline integration techniques for each compound of the calibration must be maintained for the standards and the samples. This should take into account elevated baselines, column compensation corrections and negative peaks in samples.

9.8.4.4 Make sure that positive identification of compounds has been established thru EICP's. Pay special attention to the isomer pairs commonly misidentified by the data system, i.e. phenanthrene + anthracene, benzo(a)anthracene +chrysene, benzo(b)fluroanthene + benzo(k)fluoranthene, indeno(1,2,3,cd)pyrene + dibenz(a,h)anthracene, the trichlorophenol isomers, the dichlorobenzene isomers, the dueterated dichlorobenzene isomers, the methylphenol isomers, and benzyl alcohol.

9.8.4.5 Ensure any positive results are within the lower and upper levels of the calibration curve and above the reporting limits for each compound.

9.8.4.6 If there are any cases where the integration of a compound is questionable after ensuring the above steps have been followed then consult another qualified analyst. Chromatograms should be printed out in these cases, dated and initialed by the analyst and the qualified consultant.

10.0 INSTRUMENT PREVENTATIVE MAINTENANCE AND REPAIR

Instrument maintenance and repair records are maintained to document satisfactory instrument performance. The operator is generally responsible to see that records are well kept. Records of major maintenance and repair are kept in the instrument maintenance log per instrument. Routine maintenance procedures such as changing the GC septum need only be recorded in the instrument analysis log.

10.1 Daily maintenance procedures:

10.1.1 The following procedures are performed each day an instrument is used for analysis.

10.1.1.1 The GC septum and glass liner are removed and replaced if judged necessary by the analyst.

10.1.1.2 Column performance is monitored by checking the response factor of key compounds which indicate column degradation and/or column bleed. Clip two or more inches from the head of the column if necessary.

10.1.1.3 Check that gases have adequate supply to last until the following day. Change tanks if necessary.

10.1.1.4 Check for sufficient data file space on the data system hardrive.

10.2 Weekly maintenance procedures:

10.2.1 Clip column at the inlet end.

10.2.2 Swab shell weldment with methylene chloride or other appropriate solvent.

10.2.3 Clean or replace gold seal.

10.3 Six month maintenance procedure:

10.3.1 Six month maintenance procedure. Every six months the vacuum pump oil and the adsorbent trap packing should be changed.

10.4 Maintenance "As needed":
The following service should be performed when system performance Indicates:

10.4.1 Clean the analyzer source. This will become necessary if a high background becomes apparent which is not attributable to column degradation or the system cannot meet DFTPP criteria.

10.4.2 Replace the electron multiplier when the maximum gain no longer provides the required sensitivity.

10.4.3 Replace the ion source filament as needed.

11.0 CALCULATIONS

11.1 Sample results are calculated automatically using a Microsoft EXCEL spreadsheet macro called *calcmul2*. The macro requires that specific information be included in the Sample and Miscellaneous fields in the HP Chemstation quantitation report. Provided that the information has been entered correctly, the results are provided as direct read-out in units of ug/liter for water samples and mg/kg for soil samples. The information and format of the sample information is as follows:

Sample : [AMRO ID#] DF= {8270-SX}
Misc : Matrix FV= Samp= S= X

Where: [AMRO ID#] = AMRO # in square brackets
DF = dilution factor
{8270-SX} = LIMS test code in curly brackets
FV = Final volume of sample extract in mLs
Samp = sample weight in grams for soils, sample volume in mLs for water
S = Dry weight % (i.e. % Solids)
X = Extraction date of sample

Example follows for a soil sample:

Sample : [20152-03] DF=1 {8270_SX}

Misc : Soil FV=1ML SAMP=20.10G S=94.5 X083198

12.0 QUALITY CONTROL

12.1 Initial Demonstration of Lab Performance

The ability of AMRO to produce accurate and precise data is demonstrated per instrument/per analyst for each method. This information was entered on an Excel® spreadsheet and is kept on file with the QC department along with the associated chromatograms and quantitation output pages. It consists of a Method Detection Limit Study (MDL) and Precision and Accuracy Study (P&A) for each instrument and each matrix on an annual basis.

12.1.1 The method detection limit is defined in 40 CFR 136 Appendix B. The MDL is determined annually by the analysis of a minimum of seven replicate samples, all containing an analyte(s) of the same low-level concentration. These replicate samples are put through all the preparation steps that a sample is put through. The standard deviation of the results of these analyses is multiplied by the appropriate student T-test value and the resulting value is deemed the method detection limit for that specific analyte.

This is performed annually for each method, each instrument and each matrix analyzed by AMRO. Quarterly preparation and analysis of MDL check samples are performed and the results compared to the USACE and Navy requirements. MDL check sample concentrations should be about twice the target analyte MDL values. MDL values must be less than about 1/3 of the RL value for each analyte.

12.1.2 Precision &Accuracy

Accuracy is a measure of the degree to which the analytical result approaches the "true" value of the concentration of the analyte being determined. Precision in the laboratory is determined by the comparison of duplicates, where duplicate samples result from an original sample that has been split for identical analyses. Precision and Accuracy in the laboratory is assessed initially by the analyses of a minimum of 4 replicate samples at a mid-level point of the expected range. The percent recoveries and Relative Standard Deviations (RSD) are evaluated and compared to method specifications or default limits where applicable.

Accuracy is also assessed by the regular analysis of known standards such as the Laboratory Control Samples (LCS), and matrix and analytical spikes. Accuracy within the laboratory is expressed in terms of percent recovery.

The precision of data is regularly evaluated by determining the relative percent difference (RPD) of duplicate (replicate) analyses.

12.2 The GC/MS system must be tuned to meet the DFTPP criteria listed in Section 9.3.2.

12.3 There must be an initial calibration of the GC/MS system as described in Section 9.4.

12.4 The GC/MS system must meet the calibration verification acceptance criteria in Section 9.5, each 12 hours.

12.5 Sample Quality Control for Preparation and Analysis - The laboratory analyzes QC samples for every analytical batch of up to 20 samples or each day, whichever is more frequent. QC samples include a method blank, matrix spike, matrix spike duplicate, and a laboratory control sample (LCS) in each analytical batch and the addition of surrogates to each field sample and QC sample.

12.5.1 Method blanks are evaluated for every extraction batch. See Section 9.5.3 for method blank performance criteria.

12.5.2 Full list Matrix spike (MS)/matrix spike duplicate (MSD) pairs are prepared with every extraction batch if sufficient sample was provided. The MS/MSD are evaluated for % Recovery and Relative Percent Difference (RPD). Quality Control limits are updated annually for the water and soil matrices (non-soil solid samples follow the rules for soil samples). Note the special USACE acceptance criteria limits for the LCS apply to all USACE or Navy project sample matrix spikes by default.

12.5.2.1 If all analytes are within QC limits then accuracy and precision objectives have been met for the matrix.

12.5.2.2 If any analyte exceeds QC limits then an investigation of possible causes begins. This may include, but is not limited to, the following:

12.5.2.2.1 Review of the preparation worksheet to see if any problems were encountered by the technicians during sample preparation such as emulsions, unusually high or low initial pH, observations or descriptions of a

strange physical appearance of the sample, extract losses during handling, etc;

12.5.2.2.2 review of the chain-of-custody to see if any descriptions or notations were provided by client;

12.5.2.2.3

12.5.2.2.4 previous knowledge of the history of the particular matrix;

12.5.2.2.5 evaluation of the total ion chromatogram (TIC) for interferences in the region of interest.

12.5.3. A Full List Laboratory Control Sample (LCS) is included with each extraction batch. If sufficient sample was not provided for an MS/MSD, then a LCS Duplicate is also extracted. The LCS consists of an aliquot of a clean (control) matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with a full analyte list at the same relative concentrations (acid and base) as the matrix spike. When the results of the matrix spike analyses indicate a potential problem due to the sample matrix itself, the LCS results are evaluated to verify that the laboratory extraction systems are in control. Note the special default USACE acceptance criteria limits for LCS samples are described in the Attachments 3A, 3B, 3C and 4A, 4B, and 4C and these apply to all USACE and Navy project sample LCS reports. Note that 3 Sporadic Marginal Exceedances of the criteria below are allowed for each full list LCS.

12.5.4 Surrogate recoveries - The laboratory must evaluate surrogate recovery data from individual samples versus the surrogate control limits developed by the laboratory. The same evaluation criteria for sections 12.5.2.1 through 12.5.2.3 are applied to the surrogates.

12.5.3 Special surrogate recovery limits apply to all USACE and Navy project samples and associated QC samples. These limits are listed in Attachment 2 and the USACE test codes: Note that Sporadic Marginal Exceedances of surrogate recoveries is not allowed. All surrogate failures must be discussed in the narrative and results flagged. Samples must be re-extracted and re-analyzed if sufficient volume remains.

12.5.4 Samples must be selected for spiking at random and rotated among clients and sites from batch to batch. In some cases, the client may specify which sample is to used for MS/MSD; if not , the laboratory shall pick a representative sample based on the following:

A. MS/MSD shall not be performed on Trip Blank, Rinsate Blank or Field Blank.
B. Adequate sample weight/volume.
C. Client requested QC deliverables.

12.6 The following additional Quality Control criteria should be met for all Massachusetts Contingency Plan (MCP) projects where MCP methods are requested:

12.6.1 MS/MSD: Must contain all target compounds. Recoveries should be between 40-140% for base-neutral targets and between 30-130% for acids. RPDs should be≤20% for waters and ≤30% for soils. Document exceedance in narrative with laboratory statistical control limits if recoveries are *within* AMRO statistical limits.

12.6.2 LCS- Must contain all target compounds.Recoveries must be between 40-140% for base-neutral targets and between 30-130% for acids. Re-extract if >20% of all analytes fall outside the criteria or if >15% or either fraction fall outside the criteria. Document exceedance in narrative with laboratory statistical control limits if recoveries are *within* AMRO statistical limits.

12.6.3 Surrogates- Recoveries must be between 30-130% for all surrogates in soils. Recoveries must be between 30-130% for all base-neutral surrogates in water and between 15-110% for acids in water. Document exceedance in narrative with laboratory statistical control limits if recoveries are *within* AMRO statistical limits.

12.7 USACOE and Navy Projects:

12.7.1 For all project submitted from the USACOE and Navy follow the specific requirements as indicated in Attachment 2, 3A, 3B, 3C, 4A, 4B, 4C. LCS failure occurs if: (1) a project specific analyte exceeds the control limits; or (2) if more than the allowable number of Sporadic Marginal Exceedances occur; or (3) if any single analyte exceeds the SME limits. Corrective action must be applied to all analytes that exceed the primary control limits used as acceptance criteria. If the problem appears to be isolated to the LCS then a new LCS may be prepared and run with the original batch reruns. If the problem appears to affect all the samples in the batch, indicated by similar problems in the surrogates and matrix spikes, then the entire batch must be reprepared and reanalyzed. If the new LCS has similar problems then all the associated data must be flagged in the results and discussed in the

narrative. If the new LCS passes, report both LCS results and discuss the problem analytes in the narrative.

13.0 DATA REVIEW AND STORAGE

AMRO's goal for completeness is 100%. The following data calculating, reviewing and reporting procedures are followed to obtain that goal.

13.1 Data Review - performed by the analyst:

When an analysis of a sample is complete, the analysis for that sample should be checked and the data calculated as soon as possible to identify any problems while still within the holding time for the analysis. The following steps should be taken to ensure that a good analysis has been obtained.

13.1.1 Check that all calibration analyses associated with the sample meet criteria to ensure acceptance criteria is met.

13.1.2 Check the internal standard areas. They should be -50 to +200% in comparison to the I.S. areas in the Initial Calibration standard.

13.1.3 Compare the surrogate recoveries to the AMRO limits. The default SVO surrogate recovery limits are as follows and are used until in house limits are generated.

13.1.4 Check the LCS to be sure the results are inside the limits, as well as the matrix spike and matrix spike duplicate results.

13.1.5 Check the analyzed times to be sure the tuning requirements are met.

13.1.6 Check that no compounds exceed the calibration range. If there are, these samples must be reanalyzed at a dilution.

13.1.7 Check the chromatography (good peak shape, baseline not noisy, good sensitivity).

13.1.8 Check the analysis for carryover or contamination. If any carryover is evident, these samples must be reanalyzed.

13.1.9 Check that the amount of sample analyzed is correctly recorded in the chromatogram header.

13.1.10 Check spectrum, area, and retention time of any positive hit on the quant output page.

13.1.11 Perform the final calculations on any positively identified compound in µg/L, taking into account dilution factors, etc.

13.1.12 Check the chromatogram for very large peaks not picked up by quant. If any are present, check the spectrum to be sure it is not a target compound that is over the calibration range or outside the normal retention time window. If not a target compound, make a note on the chromatogram that the peak is an unknown and use the library to identify if possible.

13.1.13 Check to see if there are any other analyses of the sample. If so, check to see if the data agrees.

13.1.14 Any problems discovered during this preliminary data check will necessitate re-analysis unless the lab manager authorizes use of the data in writing with an explanation of why the data is being used.

13.1.15 All calculations should be written in black ballpoint pen ink. All cross-outs should be a single line and should be initialed. The person performing these data calculations and checks should sign and date the bottom of the quant page.

13.2 Data Review - performed by the department supervisor or designate.

When all samples for a given project have been analyzed and the results have been calculated, the project goes through the following data review procedure:

13.2.1 The reviewer reads over all associated paperwork to become familiar with the requirements of the project.

13.2.2 The project is checked for completeness.

13.2.3 Data sheets are checked for accuracy.

13.2.4 The data calculations are spot-checked. If any problems are encountered then the whole package should be checked.

13.2.5 The QC data sheets are checked. Any problems encountered by the reviewer are brought to the attention of the lab manager.

13.2.6 All cross-outs must be a single line and should be initialed and dated. Analyst review is documented on the GC and GC/MS Data Review Checklist (Attachment 8).

13.3 Data Reporting

Many report options are available to clients of AMRO. It is AMRO policy to discuss with all clients their reporting requirements soon after receipt of the samples. The cover letter for the data reports will address any quality control problems, either with the condition of the samples upon receipt or with the analyses, or contain a statement that no quality control problems were encountered. The letter will also contain a statement that it is an integral part of the data report.

13.4 Data Archival

13.4.1 GC/MS Data Files:
All raw GC/MS data files are archived through the Network and are backed up on a tape drive through the server.

13.4.2 Data Reports:

Final data and QC reports are input into Excel and spreadsheet files. Template files exist on the Network for final data reports, daily QC check reports, sample spike reports, initial precision and accuracy data, wastewater method accuracy data, surrogate recovery data, and MDL data. When data is input template files are renamed as follows:

Data reports: XXX#####

Where X 's are the first three characters of the AMRO client code and #'s are the AMRO project and sample number.

14.0 REFERENCES

14.1 USEPA Office of Solid and Hazardous Waste - SW-846 Method Chapter 1, Rev. 1 7/92.

14.2 USEPA Method 3500. Physical and Chemical Methods for the Determination of Soild/Hazardous Waste, SW 846, Update III, Revision 2, 12/96

14.3 USEPA Method 8000B. Physical and Chemical Methods for the Determination of Soild/Hazardous Waste, SW 846, Update III, Revision 2, 12/96.

14.4 USEPA Method 8270C. Physical and Chemical Methods for the Determination of Soild/Hazardous Waste, SW 846, Update III, Revision 3, 12/96.

14.5 40 CFR Part 136 Appendix B, 1984

14.6 USACE Shell for Analytical Chemistry Requirements, EM 200-1-3, Feb 01, 2001.

14.7 Massachusetts Department of Environmental Protection, Massachusetts Contingency Plan (MCP) Method BWSC – CAM IIB , Revision 3, May 22, 2003.

14.8 USEPA Superfund Contract Laboratory Program Statement of Work for Organics Analysis OLM04.2 , May 1999.

14.9 Department of Defense Quality Systems Manual For Environmental Laboratories, Final Version 3, January 2006.

Attachment 1A

Semi-Volatile Internal Standards With Corresponding Target Compounds and Surrogates Assigned For Quantitation

	Compound	Quant ion	Qual #1	Qual #2	Qual#3
Internal Standard	1,4-DICHLOROBENZENE-d4	152	115		
	N-NITROSODIMETHYLAMINE	74	42		
	PYRIDINE	79	52	50	73
Surrogate	2-FLUOROPHENOL	112	64		
Surrogate	PHENOL-d5	99	42	71	
	PHENOL	94	65	66	
	ANILINE	93	65	66	
	bis (2-CHLOROETHYL) ETHER	93	63	95	
	2-CHLOROPHENOL	128	64	130	
	1,3-DICHLOROBENZENE	146	148	113	
	1,4-DICHLOROBENZENE	146	148	113	
	BENZYL ALCOHOL	108	79	77	
	2-METHYLPHENOL	108	107		
	1,2-DICHLOROBENZENE	146	148	113	
	bis(2-CHLOROISOPROPYL) ETHER	45	77		
	ACETOPHENONE	105	77	120	51
	4-METHYLPHENOL	108	107		
	n-NITROSO-DI-N-PROPYLAMINE	70	42	130	
	HEXACHLOROETHANE	117	201	199	
Internal Standard	NAPHTHALENE-d8	136	68		
Surrogate	NITROBENZENE-d5	82	128	54	
	NITROBENZENE	77	123	65	
	ISOPH0RONE	82	138		
	2,4-DIMETHYLPHENOL	107	121	122	
	BENZOIC ACID	122	105	77	
	2-NITROPHENOL	139	65	109	
	bis(2-CHLOROETHOXY) METHANE	93	95	123	
	2,4-DICHLOROPHENOL	162	164	98	
	1,2,4-TRICHLOROBENZENE	180	182	145	
	NAPHTHALENE	128	129	127	
	4-CHLOROANILINE	127	129		
	HEXACHLOROBUTADIENE	225	223	227	
	4-CHLORO-3-METHYLPHENOL	107	144	142	
	2-METHYLNAPHTHALENE	142	141		

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Attachment 1B

Semi-Volatile Internal Standards With Corresponding Target Compounds and Surrogates Assigned For Quantitation

	Compound	Quant ion	Qual #1	Qual #2	Qual#3
Internal Standard	ACENAPHTHENE-d10	164	162	160	
	HEXACHLOROCYCLOPENTADIENE	237	235	272	
	2,4,6-TRICHLOROPHENOL	196	198	200	
	2,4,5-TRICHLOROPHENOL	196	198	200	
Surrogate	2-FLUOROBIPHENYL	172	171		
	2-CHLORONAPHTHALENE	162	164	127	
	2-NITROANILINE	65	92	138	
	DIMETHYLPHTHALATE	163	194	164	
	2,6-DINITROTOLUENE	165	89	121	
	ACENAPHTHYLENE	152	151	153	
	3-NITROANILINE	138	108	92	
	4-NITROPHENOL	109	139	65	
	2,4-DINITROPHENOL	184	63	154	
	ACENAPHTHENE	153	152	154	
	2,4-DINITROTOLUENE	165	63	182	
	DIBENZOFURAN	168	139		
	DIETHYLPHTHALATE	149	177	150	
	4-CHLOROPHENYL-PHENYLETHER	204	206	141	
	FLUORENE	166	165	167	
	4-NITROANILINE	138	92	108	
Internal Standard	PHENANTHRENE-d10	188	94		
	4,6-DINITRO-2-METHYLPHENOL	198	51	121	
	n-NITROSODIPHENYLAMINE	169	168	167	
	AZOBENZENE	77	51	182	
Surrogate	2,4,6-TRIBROMOPHENOL	330	332	141	
	4-BROMOPHENYL-PHENYL-ETHER	248	250	141	
	HEXACHLOROBENZENE	284	142	249	
	PENTACHLOROPHENOL	266	264	268	
	PHENANTHRENE	178	179	176	
	ANTHRACENE	178	179	176	
	CARBAZOLE	167	166	139	
	DI-N-BUTYL-PHTHALATE	149	150	104	
	FLUORANTHENE	202	101		

Attachment 1C

Semi-Volatile Internal Standards With Corresponding Target Compounds and Surrogates Assigned For Quantitation

	Compound	Quant ion	Qual #1	Qual #2	Qual#3
Internal Standard	CHRYSENE-d12	240	120	236	
	BENZIDINE	184	185	92	
	PYRENE	202	101	100	
Surrogate	TERPHENYL-d14	244	122	212	
	BUTYLBENYLPHTHALATE	149	91	206	
	BIS(2-ETHYLHEXYL)PHTHALATE	149	167	279	
	3,3'-DICHLOROBENZIDINE	252	254	126	
	BENZO(A)ANTHRACENE	228	229	226	
	CHRYSENE	228	226	229	
	DI-N-OCTYL PHTHALATE	149	150		
Internal Standard	PERYLENE-d12	264	260	265	
	BENZO(B)FLUORANTHENE	252	253	125	
	BENZO(K)FLUORANTHENE	252	253	125	
	BENZO(A)PYRENE	252	126	253	
	DIBENZO(A,H,)ANTHRACENE	278	139	279	
	INDENO[1,2,3-CD]PYRENE	276	138		
	BENZO[G,H,I]PERYLENE	276	138	277	

Attachment 2

SURROGATE RECOVERY CONTROL LIMITS FOR USACOE AND NAVY PROJECTS

	8270C Water		8270C Solid	
Analyte	**Lower**	**Upper**	**Lower**	**Upper**
	Control Limit	**Control Limit**	**Control Limit**	**Control Limit**
2-Fluorobiphenyl	50	110	45	105
Terphenyl-d14	50	135	30	125
2,4,6-Tribromophenol	40	125	35	125
2-Fluorophenol	20	110	35	105
Phenol-d5/d6	10	115	40	100
Nitrobenzene-d5	40	110	35	100

Attachment 3A

LCS and MS Control Limits For Semi-volatile Organic Compounds SW-846 Method 8270C

Water Matrix

Analyte	Lower Control Limit	Upper Control Limit	Lower ME	Upper ME
1,2,4-Trichlorobenzene	35	105	25	120
1,2-Dichlorobenzene	35	100	20	115
1,2-Diphenylhydrazine (as Azobenzene)	55	115	45	120
1,3-Dichlorobenzene	30	100	20	110
1,4-Dichlorobenzene	30	100	20	110
2,4,5-Trichlorophenol	50	110	40	120
2,4,6-Trichlorophenol	50	115	40	125
2,4-Dichlorophenol	50	105	40	115
2,4-Dimethylphenol	30	110	15	125
2,4-Dinitrophenol	15	140	10	160
2,4-Dinitrotoluene	50	120	40	130
2,6-Dinitrotoluene	50	115	35	130
2-Chloronaphthalene	50	105	40	115
2-Chlorophenol	35	105	25	115
2-Methylnaphthalene	45	105	35	115
2-Methylphenol	40	110	25	120
2-Nitroaniline	50	115	35	125
2-Nitrophenol	40	115	25	125
3,3´-Dichlorobenzidine	20	110	10	125
3,3´-Dimethylbenzidine	20	150		
3-Nitroaniline	20	125	10	145
4,6-Dinitro-2-methylphenol	40	130	25	145
4-Bromophenyl phenyl ether	50	115	40	125
4-Chloro-3-methylphenol	45	110	35	120
4-Chloroaniline	15	110	10	125
4-Chlorophenyl phenyl ether	50	110	40	120
4-Methylphenol	30	110	20	125
4-Nitroaniline	35	120	20	130
4-Nitrophenol[3]	*0*	*125*	*0*	*145*
Acenaphthene	45	110	35	120
Acenaphthylene	50	105	40	115
Acetophenone	45	135		
Aniline*	10	150		

Notes:

1. All **bold** analytes do not have DoD QSM acceptance criteria and "Shell" criteria are used.
2. **Bold*** analytes are designated Poor Performing Analytes (PPA) in the "Shell"
3. PPA DoD Table D-2 analytes are designated poor performers in the DoD QSM.

Attachment 3B

LCS and MS Control Limits For Semi-volatile Organic Compounds SW-846 Method 8270C
Water Matrix

Analyte	Lower Control Limit	Upper Control Limit	Lower ME	Upper ME
Anthracene	55	110	45	120
Benz(a)anthracene	55	110	45	120
Benzidine*	10	150		
Benzo(a)pyrene	55	110	45	120
Benzo(b)fluoranthene	45	120	35	130
Benzo(g,h,i)perylene	40	125	25	135
Benzo(k)fluoranthene	45	125	30	135
Benzoic acid[3]	*0*	*125*	*0*	*150*
Benzyl alcohol	30	110	15	125
Bis(2-chloroethoxy)methane	45	105	35	115
Bis(2-chloroethyl)ether	35	110	25	120
Bis(2-chloroisopropyl)ether	25	130	10	150
Bis(2-ethylhexyl)phthalate	40	125	30	140
Butyl benzyl phthalate	45	115	35	130
Carbazole	50	115	35	130
Chrysene	55	110	45	120
Dibenz(a,h)anthracene	40	125	30	140
Dibenzofuran	55	105	45	115
Diethyl phthalate	40	120	30	130
Dimethyl phthalate	25	125	10	145
Di-n-butyl phthalate	55	115	45	125
Di-n-octyl phthalate	35	135	20	155
Fluoranthene	55	115	45	125
Fluorene	50	110	40	120
Hexachlorobenzene	50	110	40	120
Hexachlorobutadiene	25	105	15	115

Notes:

1. All **bold** analytes do not have DoD QSM acceptance criteria and "Shell" criteria are used.
2. **Bold*** analytes are designated Poor Performing Analytes (PPA) in the "Shell"
3. PPA DoD Table D-2 analytes are designated poor performers in the DoD QSM. Batch acceptance should not be evaluated based on these control limits.

Attachment 3C

LCS and MS Control Limits For Semi-volatile Organic Compounds SW-846 Method 8270C
Water Matrix

Analyte	Lower Control Limit	Upper Control Limit	Lower ME	Upper ME
Hexachlorocyclopentadiene	20	150		
Hexachloroethane	30	95	15	105
Indeno(1,2,3-cd)pyrene	45	125	30	140
Isophorone	50	110	40	125
Naphthalene	40	100	30	115
Nitrobenzene	45	110	35	120
N-Nitrosodimethylamine	25	110	10	125
N-Nitrosodi-n-propylamine	35	130	20	145
N-Nitrosodiphenylamine	50	110	35	120
Pentachlorophenol	40	115	25	130
Phenanthrene	50	115	40	130
Phenol[3]	0	115	0	135
Pyrene	50	130	35	140
Pyridine	NA	NA		

Notes:

1. All **bold** analytes do not have DoD QSM acceptance criteria and "Shell" criteria are used.
2. **Bold*** analytes are designated Poor Performing Analytes (PPA) in the "Shell"
3. PPA DoD Table D-2 analytes are designated poor performers in the DoD QSM.

Attachment 4A

LCS and MS Control Limits For Semi-volatile Organic Compounds SW-846 Method 8270C Soil Matrix

Analyte	Lower Control Limit	Upper Control Limit	Lower ME	Upper ME
1,2,4-Trichlorobenzene	45	110	30	120
1,2-Dichlorobenzene	45	95	35	105
1,2-Diphenylhydrazine (as Azobenzene)	45	135		
1,3-Dichlorobenzene	40	100	30	110
1,4-Dichlorobenzene	35	105	25	115
2,4,5-Trichlorophenol	50	110	40	120
2,4,6-Trichlorophenol	45	110	30	120
2,4-Dichlorophenol	45	110	35	120
2,4-Dimethylphenol	30	105	20	115
2,4-Dinitrophenol	15	130	10	150
2,4-Dinitrotoluene	50	115	35	130
2,6-Dinitrotoluene	50	110	35	125
2-Chloronaphthalene	45	105	35	115
2-Chlorophenol	145	105	35	115
2-Methylnaphthalene	45	105	35	115
2-Methylphenol	40	105	30	115
2-Nitroaniline	45	120	30	130
2-Nitrophenol	40	110	30	120
3,3'-Dichlorobenzidine[3]	10	130	0	145
3-Nitroaniline	25	110	15	125
4,6-Dinitro-2-methylphenol	30	135	10	155
4-Bromophenyl phenyl ether	45	115	35	130
4-Chloro-3-methylphenol	45	115	35	125
4-Chloroaniline[3]	10	95	0	110
4-Chlorophenyl phenyl ether	45	110	35	120
4-Methylphenol	40	105	30	120
4-Nitroaniline	35	115	20	125
4-Nitrophenol	15	140	10	160
Acenaphthene	45	110	35	120
Acenaphthylene	45	105	35	115
Acetophenone	45	135		
Aniline	10	150		

Notes:

1. All **bold** analytes do not have DoD QSM acceptance criteria and "Shell" criteria are used.
2. **Bold*** analytes are designated Poor Performing Analytes (PPA) in the "Shell"
3. PPA DoD Table D-2 analytes are designated poor performers in the DoD QSM.

Attachment 4B

LCS and MS Control Limits For Semi-volatile Organic Compounds SW-846 Method 8270C
Soil Matrix

Analyte	Lower Control Limit	Upper Control Limit	Lower ME	Upper ME
Anthracene	55	105	45	115
Benz(a)anthracene	50	110	40	120
Benzidine	10	150		
Benzo(a)pyrene	50	110	40	120
Benzo(b)fluoranthene	45	115	35	125
Benzo(g,h,i)perylene	40	125	25	140
Benzo(k)fluoranthene	45	125	30	135
Benzoic acid[3]	0	110	0	130
Benzyl alcohol	20	125	10	140
Bis(2-chloroethoxy)methane	45	110	30	120
Bis(2-chloroethyl)ether	40	105	25	115
Bis(2-chloroisopropyl)ether	20	115	10	130
Bis(2-ethylhexyl)phthalate	45	125	35	140
Butyl benzyl phthalate	50	125	35	135
Carbazole	45	115	30	130
Chrysene	55	110	45	120
Dibenz(a,h)anthracene	40	125	25	140
Dibenzofuran	50	105	40	110
Diethyl phthalate	50	115	40	125
Dimethyl phthalate	50	110	40	120
Di-n-butyl phthalate	55	110	45	120
Di-n-octyl phthalate	40	130	25	145
Fluoranthene	55	115	45	125
Fluorene	50	110	40	115
Hexachlorobenzene	45	120	35	130
Hexachlorobutadiene	40	115	25	130

Notes:

1. All **bold** analytes do not have DoD QSM acceptance criteria and "Shell" criteria are used.
2. **Bold*** analytes are designated Poor Performing Analytes (PPA) in the "Shell"
3. PPA DoD Table D-2 analytes are designated poor performers in the DoD QSM.

Attachment 4C

LCS and MS Control Limits For Semi-volatile Organic Compounds SW-846 Method 8270C
Soil Matrix

Analyte	Lower Control Limit	Upper Control Limit	Lower ME	Upper ME
Hexachlorocyclopentadiene	NA	NA		
Hexachloroethane	35	110	20	120
Indeno(1,2,3-cd)pyrene	40	120	35	135
Isophorone	45	110	30	125
Naphthalene	40	105	30	120
Nitrobenzene	40	115	30	125
N-Nitrosodimethylamine	20	115	10	130
N-Nitrosodi-n-propylamine	40	115	30	125
N-Nitrosodiphenylamine	50	115	40	125
Pentachlorophenol	25	120	10	135
Phenanthrene	50	110	40	120
Phenol	40	100	30	110
Pyrene	45	125	35	135
Pyridine	NA	NA		

Notes:

1. All **bold** analytes do not have DoD QSM acceptance criteria and "Shell" criteria are used.
2. **Bold*** analytes are designated Poor Performing Analytes (PPA) in the "Shell".
3. PPA DoD Table D-2 analytes are designated poor performers in the DoD QSM.

Attachment 5

INSTRUMENT CONTROL PARAMETERS

Sample Inlet: GC
Injection Source: GC ALS
Mass Spectrometer: Enabled

HP GC Injector

Front Injector:
No parameters specified

Back Injector:

Sample Washes	1
Sample Pumps	1
Injection Volume	1.0 microliters
Syringe Size	10.0 microliters
On Column	Off
Nanolitre Adapter	Off
Post-Inj Solvent A Washes	1
Post-Inj Solvent B Washes	1
Viscosity Delay	0 seconds
Plunger Speed	Fast

HP5890 Temperature Parameters

Zone Temperatures:	State	Setpoint
Inlet A:	Off	50 C
Inlet B:	On	280 C
Detector A:	Off	50 C
Detector B:	On	280 C
Auxiliary:	Off	50 C

Oven Parameters:

Oven Equilb Time:	0.30 minutes
Oven Max:	325 C
Oven State:	On
Cryo State:	Off
Cryo Blast:	Off
Ambient:	25 C

Oven Program:

Initial Temperature:	50 C
Initial Time:	1.50 minutes

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	Valve 1: Off	Valve 2: Off	Valve 3: Off	Valve 4: Off
19405 Valves:				
	Valve 5: Off	Valve 6: Off	Valve 7: Off	Valve 8: Off
19405 Relays:				
	Relay 1: Off	Relay 2: Off	Relay 3: Off	Relay 4: Off

HP5890 Detector Information

Detector	Type	State
A	---	Off
B	---	Off

HP5890 Signal Information

Not saving signal data.

Signal	Source	Peak Width	Data Rate	Start Data	Stop Data
1	Tecplot	0.053	5.000	0.00	1.00
2	Tecplot	0.053	5.000	0.00	1.00

MS ACQUISITION PARAMETERS

General Information

Tune File : df8pp.u
Acquisition Mode : Scan

MS Information

Solvent Delay : 1.00 min

EM Absolute : False
EM Offset : 0
Resulting EM Voltage : 1317.6

(Scan Parameters)

Low Mass : 35
High Mass : 500
Threshold : 500
Sample # : 2 A/D Samples 4

END OF MS ACQUISITION PARAMETERS

END OF INSTRUMENT CONTROL PARAMETERS

Level	Rate (C/minute)	Final Temperature (C)	Final Time (minutes)
1	10.0	100	0.00
2(A)	15.0	240	0.00
3(B)	3.0	320	1.50

Next Run Time: 24.80 minutes

HP5890 Inlet Pressure Programs

GC Pressure Units: psi

Inlet A:

Constant Flow:	Off
Constant Flow Pressure:	0.0 psi
Constant Flow Temperature:	50 C
Initial Pressure:	0.0 psi
Initial Time:	650.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	0.00	0.0	0.00
2(A)	0.00	0.0	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 650.00 minutes

Column Length:	30.00 m
Column Diameter:	0.530 mm
Gas:	He
Vacuum Compensation:	Off

Inlet B:

Constant Flow:	Off
Constant Flow Pressure:	0.0 psi
Constant Flow Temperature:	50 C
Initial Pressure:	7.7 psi
Initial Time:	0.00 minutes

Level	Rate (psi/minute)	Final Pressure (psi)	Final Time (minutes)
1	99.00	50.0	0.15
2(A)	99.00	7.7	0.00
3(B)	0.00	0.0	0.00

Total Program Time: 1.00 minutes

Column Length:	30.00 m
Column Diameter:	0.250 mm
Gas:	He
Vacuum Compensation:	On

HP5890 Packed Column Flow Control

Inlet A not used to control packed column flow.

Inlet B not used to control packed column flow.

HP5890 Purge Valve Settings

Inlet Purge	Init Value	On Time	Off Time	Splitless Injection
A	Off	0.00	0.00	No
B	Off	0.52	0.00	Yes

HP5890 Valve and Relay Information

Initial Sequence:
5890 Valves:

Method: BNASV1.M	Mon Jul 21 12:54:19 2001	Page: 1

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Attachment 6

AMRO Environmental
Laboratories Corporation

111 Herrick Street
Merrimack, NH 03054
(603) 424-2022

GC AND GC/MS DATA REVIEW CHECKLIST

LAB ORDER: _____ CLIENT: _____

Method: | Sample(s) No.:

Analysis: | Matrix:

A.	GENERAL			
1.	Copy of Prep/Extraction Log?	Y	N	NA
2.	Copy of Instrument Run Log or Tune List?	Y	N	NA
3.	Copy of dry weight log present?	Y	N	NA
4.	Are all sample checked for preservation after VOA analysis?	Y	N	NA
5.	All samples met extraction and/or analysis holding time criteria?	Y	N	NA
6.	Did the Tune pass and DFTPP or BFB injected every 12 hours?	Y	N	NA
7.	Are the surrogate recoveries for each sample within the QC limits?	Y	N	NA
8.	Method Blank- contaminant free(< ½ RL)?	Y	N	NA
9.	Initial Calibration meets method criteria for all compounds reported?	Y	N	NA
10.	Continuing Calibration meets method criteria for all compounds reported?	Y	N	NA
11.	Are all reported results within calibration range?	Y	N	NA
12.	LCS % Recovery criteria met for all compounds?	Y	N	NA
13.	MS/MSD %R's and %RPD's are met for all compounds?	Y	N	NA
14.	%RPD criteria met between sample and duplicate?	Y	N	NA
15.	Samples checked for carry over from sample to sample due to target or nontarget overload?	Y	N	NA
16.	Are all data exported to LIMS: calculated and verified?	Y	N	NA

COMMENTS:

Analyst (s) _____ DATE: _____

REVIEWER: _____ DATE OF REVIEW: _____

qc/qcmemos/Forms/gcmschecklist Rev. 2 08/13/01

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Attachment 7

AMRO Environmental
Laboratories Corporation

111 Herrick Street
Merrimack, NH 03054
(603) 424-2022

QUALIFICATION CARD FOR SW-846 METHOD 8270C

Trainee: _____ Signature: _____

Trainer: _____ Signature: _____

I. Knowledge Factors:

SOP Title: _____ Revision Number/Date: _____

1. Did the Trainee read and understand the above SOP?	Yes ☐	No ☐	NA ☐
2. Was the Trainee instructed on how to properly fill out the documentation?	Yes ☐	No ☐	NA ☐
3. Has the Trainee been instructed on the proper procedures for addressing potential problems?	Yes ☐	No ☐	NA ☐

II. Practical Factors:

1. Has the Trainee completed a Precision and Accuracy Study?	Yes ☐	No ☐	NA ☐
2. Has the Trainee completed an acceptable Performance Evaluation sample?	Yes ☐	No ☐	NA ☐
3. Did the Trainee completed an acceptable Laboratory Control Sample (LCS)? or PT study	Yes ☐	No ☐	NA ☐
4. Has the Trainee performed manual integration as indicated in the SOP?	Yes ☐	No ☐	NA ☐

Completion Date: _____ Trainer's Signature: _____

I _____ have read, understood and follow the above method/SOP, which is in use at AMRO Laboratories for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

Section Manager/ Supervisor: _____ Date: _____

QA Manager: _____ Date: _____

qc/qcmemos/forms/8270Cqualcard Rev. 0 08/24/01

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TOTAL PETROLEUM HYDROCARBONS BY GAS CHROMATOGRAPHY

AND

DIESEL RANGE ORGANICS (DROs)

SW-846 Method 8015B Modified

Procedure Number: OL-2009

Revision No.: 5

Revision Date: 02/04/2010

Review Date: N/A

Prepared By: Kristin Albert Date: 2/4/10

Approved By: Sue Kerr Date: 2/4/10
Organic Manager

Approved By: [signature] Date: 2/4/10
Laboratory Director

Approved By: Paula Benham Date: 2/4/10
Quality Assurance Manager

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1.0 SCOPE and APPLICATION

This method is used to analyze Total Petroleum Hydrocarbons with qualitative "fingerprint" identification, diesel range organics (DRO), the Connecticut DEP Extractable Total Petoleum Hydrocarbons (CT ETPH), and the Maine Health and Environmental Testing Method 4.1.25 (Maine DRO), and the Oklahoma Department of Environmental Quality's method for Diesel Range Organics Revision 4.1(OK DRO) from extracts of waters, solids, soils and sludges, for purposes of identification and quantification. In general diesel range organics cover the range of nC10-nC28 alkanes and covers a boiling point range of approximately 170°C to 430°C. AMRO extends this method to include lubricating oil range organics (LRO) up to C36+. The CT ETPH range extends from nC9 to nC36. The identification of specific fuel types may be accomplished but may be complicated by environmental processes such as evaporation, biodegradation, or when more than one fuel type is present.

1.1 This SOP is for Method 8015B Modified, used to determine the concentrations of various hydrocarbons present in extracts from solid, aqueous, and other matrices.

1.2 Analytes and Reporting Limits:

ANALYTE	REPORTING LIMIT Aqueous (µg/L) (1)	REPORTING LIMIT Soil (mg/kg) (2)
Diesel Range Organics	50	50
Diesel Fuel	50	50
Gasoline	50	50
Motor Oil	100	100
Kerosene	50	50
Mineral Spirits	50	50
Oklahoma DRO	100	10
Maine DRO	50	5
CT ETPH	100	50
Unidentified Hydrocarbons	200	100

Notes: 1. Aqueous based on 1000mL sample/1mL final volume.
2. Soil based on 20.0g/10mL final volume (Diesel Range Organics).
3. Soil based on 20.0g/20mL final volume (Fingerprint Analysis).
4. Soil Reporting Limits based on 100% dry weight.

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2.0 METHOD SUMMARY

2.1 This method describes chromatographic procedures for the detection of various hydrocarbons using a Flame Ionization detector (FID). Prior to the use of this method, appropriate extraction procedures must be used. Refer to the following SOP's: OL-2003 (Separatory Funnel Liquid-Liquid Extraction, OL-2016 (Microscale Solvent Extraction (MSE) and OL-2004 (automated Soxhlet Extraction for Soil) for sample extraction. The identification of petroleum products is accomplished by comparison with reference oil standards, and quantitation of identified products using the appropriate calibration curve(s).

3.0 DEFINITIONS

3.1 **Diesel range organics:** All chromatographic material eluting from C-10 (decane) and C-28 (octacosane) inclusive.

3.2 **Fingerprint Analysis:** Analysis of samples for specific hydrocarbon products such as fuels and lubricating oils.

3.3 **Unknown Hydrocarbons** are defined as the collective concentration of all compounds eluting from n-nonane to n-hexatriacontane inclusive.

3.4 **Analytical Batch:** is defined as a group of field samples with similar matrices which are processed as a unit. For Quality Control purposes, if the number of samples in such a group is greater than 20, then each group of 20 samples or less are defined as separate analytical batches.

3.5 **Calibration Check Standard** or **Calibration Verification Standard (CCV)** is defined as a calibration standard used to periodically check the calibration state of an instrument. The calibration check standard is prepared from the same stock standard solution as calibration standards, and is generally one of the mid-level range calibration standard dilutions.

3.6 **Calibration Standards** are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte, surrogate or petroleum product of interest.

3.7 **Method Blank** is defined as an aliquot of reagent water or clean sand spiked with a surrogate standard. The laboratory method blank is treated exactly as a sample, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is analyzed with every batch of samples, to

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determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

3.8 **Surrogate Standards** are compounds spiked into all samples, blanks, and matrix spikes to monitor the efficiency of sample extraction, chromatographic, and calibration systems.

3.9 **Matrix Spike Samples** are separate aliquots of field samples spiked with #2 fuel oil to monitor the efficacy of sample extraction, chromatographic, and calibration systems.

3.10 **Laboratory Control Samples** are aliquots of analyte free water or soil spiked with #2 fuel oil to monitor the efficacy of sample extraction, chromatographic, and calibration systems. Maine and Oklahoma DRO methods also require an LCS/LCSD spike pair using the Diesel Component Standard spike of even carbon n-alkanes from nC10 to nC28.

3.11 **Unresolved Complex Mixture (UCM)** is the "hump" in the chromatogram caused by a complex mixture of unresolved hydrocarbon peaks.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Water samples are collected in 1L glass bottles. Samples to be analyzed by Maine DRO and OK DRO are preserved with 5 ml of 1:1 HCl (check sample pH when received, preserve any samples with pH >2 and note this fact on the sample receiving checklist). Soil, sludge and oil samples are collected in 8 oz. wide mouth jars or other suitable containers.

4.2 Extract must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

4.3 Sample extracts are custody transferred from the Extraction analyst to the GC analyst via signature in the extraction logbook, or they are transferred to cold storage by the Extraction analyst. The extracts are either loaded directly on to GC instrument for analysis or they are stored at 4°C for future use. Extracts are kept for a minimum of two months after extraction and are then properly disposed of.

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5.0 POTENTIAL PROBLEMS AND INTERFERENCES

5.1 The success of these methods frequently depends on the levels of interference rather than on instrumental limitations. If interferences prevent identification or detection of analytes, the analyst must evaluate the interferences and perform the proper corrective action.

5.2 Sources of interferences can be grouped into three categories:

5.2.1 Contaminated solvents, reagents, or glassware

5.2.1.1 Method Extraction Blanks and Instrument Blanks are used to determine possible contamination of solvents, reagents, glassware, and GC equipment.

5.2.1.2 If samples are found to be contaminated from the extraction process, scrupulous cleaning of the glassware and re-extraction of the samples may be required.

5.2.2 Contaminated carrier gas or GC equipment, i.e. columns, injection port liners, & syringes.

5.2.2.1 If the GC equipment is found to be the source of contamination, proper cleaning and solvent rinsing of affected parts may be required or replacement with new ones.

5.2.3 Most organic compounds will show some response on the FID. Naturally occurring long chain alcohols and fatty acids are frequently found in waste sludges that are analyzed for the presence of TPH's. These will respond as large tailing peaks that are not part of the TPH response.

5.2.4 Contamination by carryover can occur whenever high concentration and low concentration samples are analyzed in sequence. Whenever an unusually high sample is encountered, it should be followed by injection of solvent blanks until the system is demonstrated to be free of carryover.

5.2.5 Other sources of interferences could occur from phthalate esters which can be minimized by avoiding sample contact with any plastic materials Phthalates are easily leached from plastic materials during laboratory operations or even sampling including equipment rinsing.

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6.0 HEALTH AND SAFETY

6.1 AMRO has a laboratory safety program in place that applies to all employees. There is a safety officer as well as employees with 40 hours of OSHA training. The air quality of the laboratory has been monitored and employees are sent for physicals. All employees receive fire and safety training upon employment.

6.2 Fire extinguishers are located in various areas of the lab. A lab evacuation plan is posted in every room, with a designated meeting place.

6.3 All new analysts receive training on the safe handling of samples, standards, the changing of gas cylinders, and the operation of instruments. In the lab, clean laboratory coats and gloves are worn when working with samples. Neat standards are made up in hoods. When gas tanks are replaced, the carrier with chain is used properly, and all gas tanks are chained in place.

6.4 Care is taken with glassware and syringes to prevent injury. Broken glass and syringes are promptly removed to broken glass containers. First aid kits are available throughout the lab. There are also eyewash stations and an emergency shower.

6.5 MSDS's are reviewed for each chemical used to ensure proper precautions are observed when handling chemicals.

6.6 Pollution prevention – The use of chemical exhaust hood are required when working with chemicals. They are specifically designed to minimize analyst exposure. The hood exhaust is monitored periodically to ensure environmental exposure is minimized.

6.7 Waste control – AMRO takes whatever steps possible to minimize waste. This includes volume reduction where appropriate. AMRO also properly segregates wastes according to contaminants and a commercial vendor is utilized for waste disposal.

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7.0 INSTRUMENTS AND EQUIPMENT

7.1 Gas Chromatograph: Analytical system complete with gas chromatograph suitable for on column and split-splitless injections. The system includes all the required accessories such as syringes, analytical columns, gases, flame ionization detectors (FIDs) and a data system.

7.2 AMRO has one complete GC/FID system: GC-FING 1. It is a Hewlett Packard Model 5890 Series II gas chromatographs equipped with electronic pressure control (EPC) and a Model 7673 autosamplers. Helium is the carrier and auxiliary make up gas. UHP hydrogen and ultra zero air are the detector gases. The GC is equipped with the following analytical column.

7.2.1 HP-5ms capillary column, 30m x 0.32mm from Agilent or comparable

7.3 The data systems for all instruments are run by Enviroquant software which is used to acquire and process all data.

7.4 Volumetric flasks, 10-mL to 100-mL, for preparation of standards.

8.0 INSTRUMENT PREVENTATIVE MAINTENANCE AND REPAIR

8.1 Daily Maintenance:

The following procedures are performed each day an instrument is used for analysis if, in the analyst's judgement, system performance requires them:

8.1.1 The GC septum and glass liner are removed and replaced if judged necessary by the analyst.

8.1.2 Column performance is monitored by checking the response factor of key compounds which indicate column degradation and/or column bleed. Clip two or more inches from the head of the column if necessary.

8.1.3 Check that gases have adequate supply to last until the following day. Change tanks if necessary.

8.1.4 Check data file space and quant space on the MS Chem Station system.

8.2 Weekly Maintenance, or as needed:

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8.2.1 Clip column

8.2.2 Swab shell weldment with methylene chloride or other appropriate solvent.

8.2.3 Clean or replace inlet seal

9.0 REAGENTS AND STANDARDS

9.1 The following solvents are used for sample dilutions, preparation of standards or solvent rinsing of equipment:

9.1.1 Methylene chloride, Reagent Grade and Pesticide Residue Grade chemicals or equivalent.

9.1.2 Acetone, Reagent Grade and Pesticide Residue Grade chemicals or equivalent.

9.1.3 Methanol, Reagent Grade and Pesticide Residue Grade chemicals or equivalent.

9.2 Commercially prepared stock standard solutions and solutions prepared from pure reference materials are used in this analysis.

9.3 Stock calibration standards

	Concentration µg/ml	Vendor	Catalog Number
Diesel fuel	50,000	Restek	31259
o-Terphenyl	10,000	Restek	31097
Gasoline	50,000	Restek	30206
Motor Oil	50,000	Restek	31464
Kerosene	50,000	Restek	31257
Mineral Spirits	50,000	Restek	31261
DRO mix	2,000	Restek	31064
CT ETPH Mix	1,000	Restek	31614

9.4 Preparation of calibration standards.

Diesel Fuel, Kerosene, Mineral Spirits, Gasoline and Motor Oil are all prepared in methylene chloride as described below (all stock at 50,000 µg/ml)

Calibration Point (ng/µL)	Amount of Stock (µL)	Final Volume (mL)
50*	10	10
250	50	10
1000	200	10
2500	500	10
5000	200	2
10000	400	2
20000	800	2

* note this fuel oil #2 standard is also used as the resolution check mix for Maine DRO

The o-Terphenyl (Surrogate) calibration curve is prepared in methylene chloride as described below (Stock solution at 10,000 ug/ml).

Calibration Point (ng/(µL)	Amount Of Stock (µL)	Final Volume (mL)
2.0	2.0	10
10	10	10
25	25	10
50	50	10
100	100	10
150	150	10
250	250	10

9.5 The CT ETPH standard contains 15 n-alkanes ranging from C-9 to C-36 at 1000µg/mL each. This is used as a retention time marker for the range analytes. It is also used to prepare a calibration curve for methods using the response factors of a specified set of alkanes for quantitation. The Maine and OK DRO required Diesel Component Standard contains all the even carbon number normal alkanes (10 of them) from nC10 to nC28

9.5.1 Preparation of retention time marker standard. 100µL of the n-alkane stock is brought to 10 ml final volume with methylene chloride. Final concentration is 10 ng/µL for each alkane.

9.5.2 Preparation of n-alkane calibration curve.

Calibration point (ng/µL)	Conc. of stock (µg/mL)	Amount of stock (µL)	Final volume (mL)
1	1000	5	5.0
5*	1000	5	1.0
20	1000	20	1.0
50*	1000	50	1.0#
100	1000	100	1.0
200	1000	200	1.0
500*	1000	500	1.0
# 50 ng/µL standard also used as CCV	* minimum 3 points for ME and OK DRO		

9.6 Surrogate, matrix spike and second source standards.

	Concentration	Vendor	Catalog Number
Fuel Oil #2	neat	Rochette Oil	N/A
Diesel component mix	2,000 ug/mL	Restek	31064
n-Nonane	neat	Chem Service	F1099
o-Terphenyl	neat	Chem service	F1054

9.6.1 Preparation of standard mixes from neat material.

	Amount (g)	Final volume (mL)	Concentration (µg/mL)	Solvent
Fuel Oil #2	2.0	100	20,000	Acetone
n-Nonane	1.0	5.0	20,000	MeCl2
o-Terphenyl	0.25	5.0	50,000	Acetone

9.6.2 Preparation of second source, initial calibration verification working standards.

	Amount (µL)	Final volume (mL) in methylene chloride	Concentration (µg/mlL
Fuel Oil #2 (20,000 µg/ml)	250	10.0	500
n-Nonane (20,000 ug/mL)	12.5	5.0	50
o-Terphenyl (50,000 µg/ml)	10	10.0	50

9.6.3 Preparation of surrogate and matrix spike solutions.

The #2 fuel oil standard (see sec 9.6.1) prepared in acetone from neat material is used as is to spike laboratory control samples and matrix spike samples.

The o-terphenyl standard (see sec 9.6.1) prepared in acetone from neat material is diluted 5X with acetone to prepare the working solution with a final concentration of 1,000 µg/ml and 20ug/mL (for Maine DRO)

Diesel component spike mix (for Maine and OK DRO): Prepared from a certified solution containing the 10 alkanes listed in Table 1 of the Maine DRO Method 4.1.25 and Table 1 of the OK DRO method. The stock solution at 2,000ug/mL is dilulted in acetone to make a working solution at 50 ug/mL per component.

9.7 TRACKING OF ANALYTICAL STANDARDS

9.7.1 Chemical Receipt:

9.7.1.1 The receipt of all standards, neat compounds and reagents from commercial sources is documented in the Chemical Receiving Logbook by sample receiving personnel or designate. Each solution or compound is assigned a unique standard receipt ID number, the letter "A" and a four digit sequential number. The date received, source, lot number, expiration date and disposal date are recorded. The expiration dates are provided from the manufacturer and the disposal dates are completed by AMRO employees when standards are used up or disposed. Refer to SOP " Sample Management" GL-1006.

9.7.2 Standards Preparation: Standards are prepared as written in the respective SOPs. In order to assure the accuracy of standards the following general guidelines are used:

9.7.2.1 AMRO purchases pesticide residue grade solvents for organic standard preparation and organic sample extractions.

9.7.2.2 All stock standards that are purchased must include a certificate of analysis from the manufacturer.

9.7.3 Standard Tracking: It is important to be able to track all analytical standards back to their sources. The following procedure is used by AMRO personnel to accomplish this:

9.7.3.1 Organic Standard Tracking: The Organic Standards Tracking System consists of three logbooks. One Standard ID Index logbook (Attachment 1) , one for Stock Standards, and one for Working Standards (Attachment 2).

9.7.3.2 Stock Standard: Whenever a stock standard solution is opened or is prepared from a neat compound, this information is recorded in the Stock Standard Logbook section of the standard tracking log. The stock solution is assigned a new ID number consisting of the letter "V" or "S", the date of preparation plus a letter suffix starting with the letter "A". This ID along with the compound name, concentration and initials of the person making the stock are included on the label. Stock standards are good one year from the date prepared or the date provided by manufacturer, whichever is shorter. Example - The fourth volatile standard prepared on April 15, 1989 would have the

standard ID V041589D. This number will be recorded in the log book and written on the container.

9.7.3.3 Working Section: Whenever a working standard solution opened or is prepared, whether from AMRO stocks or commercially obtained stocks, the information is recorded in the Working Standard section of the standard tracking log. The solution is assigned a new ID number based on the section "V" or "S", the date it is prepared plus a letter suffix. This number along with a descriptive name, concentration (or conc. varied) and the initials of the person preparing the solution are included on the label. The expiration of working standards vary depending on the use. Standards should be used in accordance with method specifications and manufacturer's instructions.

9.7.3.4 Standard Storage: Standards and reagents are stored per method specifications and manufacturer's instructions. Semivolatile, Pesticide, PCB, Petroleum standards are stored in refrigerators at 4±2°C, freezers at minus 10 °C, and ambient temperature depending upon the method and manufacturer requirements.

9.7.3.5 Solvent Check Analysis

All extraction procedures are performed using pesticide residue grade solvents and the first method or procedural blank analysis is used when a new lot number is received to verify the absence of target analytes. Also, Before a new vendor's solvent is used for sample extraction a special check of the concentrated solvent (not a method blank) is required.

10.0 PROCEDURE

10.1 AMRO currently has one GC-FID instrument: FING 1.

10.2 GC Operating Conditions: all analysis:

10.2.1 Injection volume: 2 µL

10.2.2 FID Temperature: 320°C

10.2.3 Injection Port Temperature: 300°C

10.2.4 Initial Temperature: 50°C

10.2.5 Initial time: 2.0minutes

10.2.6 Rate 1: 35°C/minute

10.2.7 Final Temperature 1: 100°C

10.2.8 Final Time 1: hold 0.0 minutes

10.2.9 Rate 2: 25°C/minute

10.2.10 Final Temperature 2: 320°C

10.2.11 Final Time 2: hold 15.77 minutes

10.2.12 Total Run Time 28 min

10.3 Injection Run Log:

10.3.1 All injections must be recorded in the injection runlog created by the macro inject.mac and stored in the Excel/InjLogs directory on each instrument. The runlog must indicate the appropriate sample, standard, and the instrument. Each page in the hardcopy logbook must be signed by the analyst who is responsible for its accuracy.

10.4 Retention Time Windows:

10.4.1 Before establishing retention time windows, make sure that the chromatographic system is operating reliably and that the system conditions have been optimized for the target analytes and surrogates in the sample matrix to be analyzed.

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10.4.2 Make three injections of all single and all multi-component analytes over the course of a 72-hour period. Serial injections or injections over a period of less than 72 hours may result in retention time windows that are too tight.

10.4.3 Record the retention times for all single analytes and three to five major peaks for multi-component analytes. Calculate the mean and standard deviation of the three absolute retention times of each single analyte and those peaks.

10.4.4 If the standard deviation of the retention times for a target compound is 0.000, then the laboratory uses a default standard of 0.01 minutes. Recording retention times to three decimal places rather than two should minimize the instances in which the standard deviation is calculated as 0.000.

10.4.5 The width of the retention time window for the single analytes, each major constituent in multi-component analytes and surrogates is defined as ±3 times the standard deviation of the mean absolute retention time established during a 72-hour period. Determination of the retention time windows for the DRO range marker analytes and surrogates are required . If the default standard deviation is employed, ½ the width of the window will be 0.07 minutes.

10.4.6 The absolute retention time windows must be calculated for each analyte and surrogate on each chromatographic column and instrument. New retention time windows must be established when a new GC column is installed.

10.4.7 Establish the center of the window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

10.4.8 If the daily CCV falls outside the window, the analyst will evaluate the instrument and will recalibrate if necessary. For samples run during the same analytical shift as the Initial Calibration, use the RT of the midpoint standard. However, the experience of the GC analyst should weigh heavily in the interpretation of the chromatograms. The analyst may therefore qualify an analyte as present outside the RT window by using pattern recognition.

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10.5 Initial Calibration

10.5.1 A minimum of a five point curve is used for the initial calibration curve (*three points minimum for Maine DRO Method 4.1.25 and the Oklahoma DRO method Rev.4.1*). The following components and their corresponding concentrations are listed in the reagent section of this document. The response factor (RF) for each component is calculated.

$$\frac{\text{Area of component}}{\text{ng injected}} = \text{RF}$$

10.5.2 The average RF is calculated by averaging the RF's from each calibration Level. The %RSD must be less than or equal to 20%. Any curve that is over 20% must be evaluated using linear regression. The criteria for linear regression is the coefficient of determination must be 0.99 or greater. If this criteria cannot be met a quadratic equation must be used. The criteria is a coefficient of determination of 0.99 or greater. If none of these criteria are met the instrument must be recalibrated. A 6^{th} point must be added to use a quadratic regression. Do not force the origin, and do not use (0,0) as a calibration point.

10.5.3 The response factors for the Connecticut EPTH method are calculated by summing the areas of the 15 components listed in the CT ETPH Mix and dividing by the total amount injected for each calibration level.

10.5.4 The response factors for the Maine DRO method 4.1.25 and OK DRO Rev.4.1 are calculated by summing the integrated area, baseline to baseline, of the 10 alkane components in the Diesel Component Mix and dividing this area by the total amount injected for each calibration level.

10.6 Continuing Calibration Verification (CCV)

10.6.1 Baseline subtraction is performed at the instrument level by electronic subtraction of a stored "blank run". This "column compensation data" needs to be updated periodically as column bleed and length change. Do not update the column compensation to compensate for an elevated baseline due to contamination of the GC system with high boiling material. This will result in a negative baseline once the system has cleared itself of this material.

10.6.2 Updating the column compensation data.

10.6.2.1 Close the data acquisition software (Chemstation) for the instrument.

10.6.2.2 On the GC keypad press col comp 1 (or 2) and enter.

10.6.2.3 The GC will go through the normal EPC and oven program.

10.6.2.4 Check the result by running a solvent blank.

10.6.3 Verify calibration at the start of each analytical sequence by injecting a #2 fuel oil standard at 2500 ng/ul and an o-terphenyl standard at 50 ng/ul. The #2 fuel oil standard response must be ± 15% of the true value, the o-terphenyl standard response must be ± 15% of the true value. A closing or continuing standard is required every 12 hours and is evaluated by the same criteria. The closing CCV concentration should not be the same as the initial CCV. If two or more initial CCVs fail in a row, then after corrective action to fix the problem, two passing CCVs in succession must be analyzed to demonstrate calibration verification before continuing with sample analyses.

10.6.4 Verify calibration for the CT ETPH method at the start of each analytical sequence containing CT ETPH samples by injecting the n-alkane CCV mix and o-terphenyl standard (50ug/mL). If the response is within 30% of the true value then the calibration is verified and CT ETPH samples can be analyzed. A passing continuing or closing standard is required every 12 hours. A discrimination check must be performed on the CCV at the beginning of each sequence and pass the requirement that the RFs for each alkane must be within 20% of the average response factor. This discrimination check requirement does not apply to the closing CCV.

10.6.5 Verify calibration for the Maine DRO method and OK DRO methods at the start of each sequences containing Maine or OK DRO samples by injecting the n-alkane CCV mix and o-terphenyl standard (50ug/mL). If the response is within 20% of the true value then the calibration is verified and Maine DRO / OK DRO samples can be analyzed. A passing continuing standard is required every 20 samples and at the end of each sequence. A resolution check should be performed at the beginning and end of each analytical batch if Maine DRO samples are present in the queue. Pristane should resolve from n-C17 and phytane should resolve from n-C18 in the resolution check mix (50ug/mL fuel oil #2 standard). The peaks are considered resolved

when the smallest peak height to valley difference is greater than 60% of the smaller peak height. If this criteria is not met then all the associated data must be rerun or flagged. The recovery of the resolution check standard should be between 60% and 140%.

10.6.6 The verification of calibration curves for other products such as gasoline, kerosene, and mineral spirits is only done at the start of an analytical sequence. The standard response must be ± 15% of the true value. A closing CCV is required only for fuel oil#2 and o-terphenyl because fuel oil #2 is representative of all analyte fuel types. Samples can be analyzed for all these products for 24 hours as long as the #2 fuel and o-terphenyl CCV's continue to pass.

10.6.7 Retention time marker standard (sec 9.5.1). This standard is run before and after each analytical sequence but need not be injected within the 12 hour window. If a #2 fuel oil standard and an o-terphenyl standard are serving as continuing calibration standards (marking the end of one analytical sequence and the start of another) the retention time marker standard may be run either before or after the continuing calibration standards. The n-alkane CCV can also serve as the retention time marker.

10.6.8 An instrument blank is run after the opening or continuing standards. This may be an extraction blank or a methylene chloride blank. The o-terphenyl standard can also serve as the instrument blank.

10.6.9 If the standard analyzed after a group of samples exhibits high response (over the applicable limit), and the analyte was not detected in the samples, it is acceptable to report ND results without qualification. If Department of Defense (DOD) samples are present in the queue then two terminal CCVs must be analyzed at the end of the sequence to demonstrate a consistent high bias. In contrast, if the response in the standard is low (below the applicable limit), the sample must be reanalyzed.

10.6.10 Note the OK DRO method requires an instrument or method blank analysis after every 10 samples to check for target analyte carryover.

10.7 Data Interpretation

10.7.1 Begin to examine the sample chromatograms. Check the baseline integration and compare it to the baseline for the sample blank. The baseline should be about the same as the sample blank. In the case of poor chromatography or matrix interferences, this baseline may be difficult to determine. The technical judgement and experience of the analyst must be used in making accurate baseline determinations. Baseline determinations must be verified by a supervisor or data reviewer to confirm analyst interpretation of the baseline. In some cases adjustment to the GC may be required.

10.7.2 Locate the Surrogate Standard peak (OTP) and quantify it. The computer will automatically calculate the recovery. If the % recovery of OTP in the sample is off by more than the current limits, re-extraction may be necessary. The analyst may re-analyze the samples to determine if a bad injection was made.

10.7.3 Check to determine if the surrogate is identified correctly by matching retention times with the daily standard. If any interferences are observed associated with the surrogate the data should be flagged and noted in the narrative. The analyst should consult a supervisor if retention time shifts occur.

10.8 Petroleum Product Identification in 'Fingerprint Analysis'

10.8.1 Use a current hard copy of the daily standard to compare to the samples for identification purposes. Look for the presence of a gaussian shaped window of un-resolved peaks in the chromatogram, the presence of such an envelope is indicative of a possible petroleum product and should be examined for a positive match with reference materials.

10.8.2 Light-weight products such as gasoline, kerosene, jet fuel or mineral spirits may not show any un-resolved hydrocarbon window at low concentrations. If there is a group(s) of early eluting peaks in the sample chromatogram check them against the reference standards for the aforementioned products for retention time matches. Please note that these low boiling point products are particularly susceptible to weathering effects, so differences in the relative amounts of the peaks should be expected. Samples showing a reasonable degree of match with the reference material patterns should be quantitated as the matching petroleum product with the notation that it is weathered. If

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there is no identifiable match to a calibrated petroleum product then report the results as an unidentified hydrocarbon (nC9-nC36 range).

10.8.3 Gasoline is more appropriately analyzed by purge and trap (8015B GRO Mod) and should be reported as an estimated value by this method.

10.8.4 To identify which petroleum product is present in an extract, a detailed evaluation must be made. Listed below is guidance information to use in identifying components.

10.8.4.1 Shape, width and elution time of the UCM 'hump'.

10.8.4.2 Range and relative ratios of n-alkane components.

10.8.5 Characteristics of typical petroleum products:

10.8.5.1 Unleaded Gasoline - Asymmetrical, C8-C12 no unresolved area

10.8.5.2 Mineral Spirits - symmetrical, C8 - C14

10.8.5.3 Kerosene - symmetrical, C8-C18

10.8.5.4 Fuel Oil #2 - symmetrical, C9 - C24

10.8.5.5 Motor Oil - symmetrical, C18 - C36+

10.8.6 Sample chromatograms that do match any of the calibration standards are quantitated as 'unknown hydrocarbons' using the #2 fuel oil response factor. Integration is done by projecting a horizontal baseline from nC-9 to nC-36.

10.8.7 Sample chromatograms that are to be reported as Diesel Range Organics (DRO) are Integrated by projecting a horizontal baseline from C-10 to C-28. These samples are quantitated using a response factor generated by integrating the #2 fuel oil curve using the same criteria, or in the case of Maine and OK DRO analyses using the Diesel Component Standard response factor.

10.8.8 Sample chromatograms that are to be reported as CT ETPH are integrated by projecting a horizontal baseline from nC9 to nC36 and quantified using the initial calibration specified in Sec.10.5.3.

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10.8.9 Sample chromatograms that are to be reported as Maine DRO or OK DRO are integrated by projecting a horizontal baseline from nC10 to nC28 and quantified using the initial calibration specified in Sec. 10.5.4. Note the individual calibrated n-alkanes in the DRO window should also be reported for OK DRO.

10.8.10 Note the presence of any extraneous peaks in the sample that are not present in the reference standards used for qualitative identification. Report the possible presence of gasoline or significant UCM area outside the DRO window for Maine DRO reports.

10.8.11 Note additional evidence of weathering, including degradation of low boiling point components and degradation of the low boiling point end of the unresolved complex mixture.

10.8.12 All sample responses must fall within the calibration range. Re-analysis at a dilution must occur if the sample response exceeds the maximum calibration range.

10.8.13 Identification based on the guidelines in ASTM Method 3328 is recommended. Four possible levels of identification are given.

10.8.13.1 MATCH - This level of identification indicates an exact or near exact match between the sample and reference standard Indicate the name of the component.(i.e. "Fuel Oil #2", conc. = 500 mg/kg, reporting limit (50 mg/kg.).

10.8.13.2 PROBABLE MATCH - This indicates a nearly exact match, with the exception of small differences that can be specifically attributed to the presence of extraneous components or moderate weathering effects. Indicate the name of the component same as above but flag that the sample is weathered.

10.8.13.3 INDETERMINATE - This level indicates that the sample is too different from any reference standard to make a definitive identification. Include a carbon range and quantify as an unknown hydrocarbon against a known standard in the same range. Indicate unidentified hydrocarbon, concentration, and reporting limit with a flag on the results to indicate what the result was quantitated as.

10.8.13.4 MISMATCH - This level of identification should only be used when a client has submitted a possible source standard for comparison with the sample and the source does not match the sample. Use this level in conjunction with one of the other 3 levels of identification for both the sample and the suspected source.

10.8.13.5 If there is no indication of the presence of a petroleum product report "No Petroleum Product Detected" (N.D.) and the reporting limit. All samples and blanks that are reported as ND are integrated either as diesel range organics or as unidentified hydrocarbons (C-9 to C-36). This is done to confirm the response is below the reporting limit.

10.8.13.6 The integration of compounds for standards and samples should be performed to be in the same manner as the initial calibration integration. Cases where the initial calibration is in question should be investigated to ensure it is not related to poor maintenance, damaged or defective column, or other issues which should be solved before further analysis.

10.8.13.7 The initial calibration curve must be printed and stored with the calibration records to document passing the required QC criteria.

10.8.14 Each sample analysis must be bracketed with an acceptable continuing calibration check CCV's (each 12-hr. shift or interspersed every 20 samples, whichever comes first). DOD samples will be analyzed at intervals of no greater than 10 injections between CCV's.

11.0 CALCULATIONS

11.1 Necessary calculations and formulas can be found throughout the text as applicable to each section.

11.2 Average Response Factor Calibration. Use the following equation to calculate the concentration of analytes in aqueous samples:

$$\text{Concentration (mg/L)} = \frac{(As)\,(Vf)\,(Df)}{(Rfa)\,(Vs)\,(Vi)}$$

Where:
As = Area counts for the analyte in the sample.
Vs = Initial volume of the sample extracted in mL.
Vf = Final volume of sample extract in mL.
Df = Dilution factor.
Vi = Volume of extract injected in ul. The injection volume for samples and standards must be the same
Rfa = Average response factor from the initial calibration curve (area per ng injected).

11.3 Average Response Factor Calibration. Use the following equation to Calculate the concentration of analytes in non aqueous samples:

$$\text{Concentration (mg/KG)} = \frac{(As)\,(Vf)\,(Df)}{(Rfa)\,[(W)(\%S)]\,(Vi)}$$

Where:
As = Area counts for the analyte in the sample.
W = Initial weight of the sample in grams.
%S = Percent solids expressed in decibel form (91% = 0.91). If not applicable set at 1.0.
Vf = Final volume of sample extract in mL.
Df = Dilution factor.
Vi = Volume of extract injected in ul. The injection volume for samples and standards must be the same
Rfa = Average response factor from the initial calibration curve (area per ng injected).

12.0 QUALITY CONTROL

12.1 Initial Demonstration of Proficiency:

12.1.1 The ability of AMRO to produce accurate and precise data is demonstrated per instrument/per analyst for each method. It consists of a Method Detection Limit Study (MDL) and Precision and Accuracy Study (P&A) for each instrument and each matrix on an annual basis. The laboratory demonstrates initial proficiency with each combination of sample preparation and determinative methods that it utilizes by generating data of acceptable precision and accuracy for a reference sample containing the target analytes in a clean matrix. The reference samples are prepared from a spiking solution containing each analyte of interest at a concentration that is 10-50 times the MDL for each analyte in that matrix, or at the routine concentration of LCS's (Laboratory Control Samples). P & A Studies are required to be updated annually by evaluating a minimum of 4 LCS's.

12.2 Method Detection Limit (MDL) Study:

12.2.1 The method detection limit is defined in 40 CFR 136 Appendix B. The purpose of an MDL is to establish the minimum concentration of each analyte greater than zero that can be reported with 99% confidence. An MDL study consists of at least 7 replicates of spiked deionized water (ideally standard set at ½ RL or lower), which are extracted and analyzed as normal samples. The standard deviation of the results of these analyses is multiplied by the appropriate student T-test value and the resulting value is deemed the method detection limit for that specific analyte.

12.2.2 MDL studies of each analyte must be reported annually for each instrument, column, matrix, and extraction method. To ensure that reasonable MDL values are determined, an MDL check sample is analyzed by spiking an interference free matrix with all target analytes at about two times the determined MDL. The MDL check sample is taken through the same process as the MDL. If any of the target analytes are not detected, the MDL study is modified and repeated until the MDL check sample is detectable. The MDL check will be analyzed quarterly and after major instrument maintenance or changes in the instrumentation or instrumental conditions to verify sensitivity of the method.

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12.3 Precision &Accuracy:

12.3.1 Accuracy is a measure of the degree to which the analytical result approaches the "true" value of the concentration of the analyte being determined. Precision in the laboratory is determined by the comparison of duplicates, where duplicate samples result from an original sample that has been split for identical analyses. Precision and Accuracy in the laboratory is assessed initially by the analyses of a minimum of 4 replicate samples at a mid-level point of the expected range. The percent recoveries and Relative Standard Deviations (RSD) are evaluated and compared to method specifications or default limits where applicable.

12.3.2 Accuracy is also assessed by the regular analysis of known standards such as the Laboratory Control Samples (LCS), and matrix and analytical spikes. Accuracy within the laboratory is expressed in terms of percent recovery.

12.3.3 The precision of data is regularly evaluated by determining the relative percent difference (RPD) of duplicate (replicate) analyses.

12.4 Method Blanks are prepared at a frequency of one for every batch of up to 20 samples of the same matrix, prepared at the same time, by the same procedures. When sample extracts are subjected to cleanup procedures, the associated method blank must also be subjected to the same procedures.

12.4.1 The results for the method blank should be < the RL for all analytes and ½ the RL if DoD project samples are in the batch. If the method blank results do not meet the acceptance criteria, then corrective action is taken to locate and reduce the source of the contamination and the associated sample extracts are reanalyzed or re-extracted if necessary.

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12.5 Matrix spike (MS) and Matrix Spike Duplicate (MSD) are prepared at a frequency of one for every batch of up to 20 samples of the same matrix, prepared at the same time, by the same procedures.

12.5.1 Samples must be selected for spiking at random and rotated among clients and sites from batch to batch. In some cases, the client may specify which sample is to used for MS/MSD; if not , the laboratory shall pick a representative sample based on the following:

12.5.1.1 MS/MSD shall not be performed on Trip Blank, Rinsate Blank or Field Blank.

12.5.1.2 Adequate sample weight/volume.

12.5.1.3 Client requested QC deliverables.

12.6 Laboratory Control Samples are prepared at a frequency of one for everybatch of up to 20 samples of the same matrix, prepared at the same time, by the same procedures. The LCS consists of an aliquot of clean matrix similar to the sample matrix and of the same weight or volume. The LCS is spiked with the same compounds at the same concentrations as the MS and MSD. When the results of the MS and MSD analysis indicates a potential problem due to the sample matrix itself, the LCS results are used to verify that the lab can perform the analysis in a clean matrix. Note the OK DRO method requires an LCSD per batch and a maximum batch size of 10 samples.

12.7 Analysis of the MS, MSD, and LCS require the same procedures employed for the original sample and the concentration of each analyte spiked is calculated.

12.8 Calculation of Recovery: Accuracy is estimated from the recovery of spiked analytes from the matrix of interest. The recovery of each spiked analyte is calculated using the following formula:

$$\text{Recovery} = \%R = \frac{Cs - Cu}{Cn} \times 100\%$$

Where:
Cs = Measured concentration of the spike sample aliquot
Cu = Measured concentration of the unspike sample aliquot (use 0 for LCS)
Cn= Nominal (theoretical) concentration increase that results from spiking the sample, or the nominal concentration of the spike aliquot (for LCS).

12.9 Calculation of Precision: Precision is estimated from the relative percent difference (RPD) of the concentrations (not the recoveries) measured for the MS/MSD. To calculated the RPD, use the following formula:

$$RPD = \frac{|Conc.\ 1 - Conc.\ 2|}{((Conc.\ 1 + Conc.\ 2)/\ 2)} \times 100$$

Where:
Conc. 1 = Measured concentration of the first sample aliquot
Conc. 2 = Measured concentration of the second sample aliquot

12.10 Unless specifically excepted below, all recoveries control limits for surrogates, matrix spikes and laboratory control sample control are based on the laboratory control charts. The control charts are updated annually.

12.11 For all batches containing DOD project samples the default acceptance criteria for LCS recoveries is 60% to 140%.

12.12 Maine DRO LCS and MS spike recovery contorl limits are 60% to 140% and the RPD for LCS/LCSD or MS/MSD results should be less than 20%.

12.13 OK DRO LCS/LCSD pairs must demonstrate accuracy and precision within 20% for waters and 40% for soils.

12.14 If the surrogate recovery for a Maine DRO sample is less than 50% then repeat the analysis and flag the results if it fails this criteria in the rerun.

13.0 DATA REVIEW AND STORAGE

13.1 All cross-outs must be a single line and should be initialed and dated. Analyst review is documented on the GC and GC/MS Data Review Checklist (Attachment 3).

14.0 REFERENCES

14.1 40 CFR Part 136 Appendix B, 1984.

14.2 USEPA Office of Solid and Hazardous Waste - SW-846 Method Chapter 1, Rev. 1 7/92.

14.3 Method 8000B Revision 2, December 1996. SW846, Update III, USEPA.

14.4 Method 8015B Revision 2, December 1996. SW846, Update III, USEPA.

14.5 Shell for Analytical Chemistry Requirements, Version EM 200-1-3, 02/01/01.

14.6 Separatory Funnel, Liquid-Liquid Extraction, OL-2003.

14.7 Microscale Solvent Extraction (MSE), OL-2016.

14.8 Automated Soxhlet Extraction for Soil, OL-2004.

14.9 Department of Defense Quality Systems Manual For Environmental Laboratories, Version 3, January 2006.

14.10 Method For Determining Diesel Range Organics, Maine, Method 4.1.25, 09/06/95.

14.11 Analysis of Extractable Total Petroleum Hydrocarbons (ETPH) using Methylene Chloride Gas Chromatograph/Flame Ionization Detection , CT DEP , March 1999

14.12 Oklahoma Department of Environmental Quality, Methods 8000/8100 (modified) Diesel Range Organics, Revision 4.1, 10/22/97

Attachment 1

AMRO Environmental Laboratories Corporation

111 Herrick Street
Merrimack, NH 03054
(603) 424-2022

STANDARD I.D. INDEX

Standard I.D.	Standard Logbook (Stock or Working)	Brief Description

Attachment 2

AMRO Environmental Laboratories Corporation
111 Merrick Street
Merrimack, NH 03054

STANDARD LOG - STOCK SOLUTIONS
ORGANIC SECTION

Date Opened/Prepared	Opened/Prepared by	Compound Name	Reagent/Stock ID	Source	Conc. or Purity	Amt (g) or (µl)	Final Vol. (ml)	Solvent ID# Number	Final Conc.	Units (g/µl or µg/ml)	Solution ID	Storage Loc.	Exp. Date

Notes: 1) AMRO stock Std. ID is "V" or "S", date prepared, letter suffix(11/04/98 is the second stock std prepared on 11/04/98)

2) Vials must be labeled with the compound name, conc., AMRO stock ID, and exp. Date

qc/[illegible]/standardslog.wpd Rev. 4 05/00

AMRO Environmental Laboratories Corporation

111 Merrick Street
Merrimack, NH 03054
(603) 424-03054

STANDARD LOG - WORKING STANDARDS
ORGANIC SECTION

Date Opened/Prepared	Prepared/Received by	Compound Name	Reagent/Stock ID	Source	Conc. or Purity	Amt. (ml)	Final volume (ml)	Solvent ID# number	Final Conc.	Units (g/µl or µg/ml)	Soln ID	Storage Loc.	Exp. Date

Notes: 1) AMRO working Std. ID is "V" or "S", date prepared, letter suffix(11/04/98C is the third working std prepared on 11/04/98)

2) Vials must be labeled with the compound name, conc., AMRO ID, and exp. Date

qc/qc-reviews/[illegible]standardslog.wpd Rev. 3 08/11/01

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Attachment 3

AMRO Environmental
Laboratories Corporation

111 Herrick Street
Merrimack, NH 03054
(603) 424-2022

GC AND GC/MS DATA REVIEW CHECKLIST

LAB ORDER: _____ CLIENT: _____

Method:	Sample(s) No.:
Analysis:	Matrix:

A.	GENERAL			
1.	Copy of Prep/Extraction Log?	Y	N	NA
2.	Copy of Instrument Run Log or Tune List?	Y	N	NA
3.	Copy of dry weight log present?	Y	N	NA
4.	Are all sample checked for preservation after VOA analysis?	Y	N	NA
5.	All samples met extraction and/or analysis holding time criteria?	Y	N	NA
6.	Did the Tune pass and DFTPP or BFB injected every 12 hours?	Y	N	NA
7.	Are the surrogate recoveries for each sample within the QC limits?	Y	N	NA
8.	Method Blank - contaminant free(< ½ RL)?	Y	N	NA
9.	Initial Calibration meets method criteria for all compounds reported?	Y	N	NA
10.	Continuing Calibration meets method criteria for all compounds reported?	Y	N	NA
11.	Are all reported results within calibration range?	Y	N	NA
12.	LCS % Recovery criteria met for all compounds?	Y	N	NA
13.	MS/MSD %R's and %RPD's are met for all compounds?	Y	N	NA
14.	%RPD criteria met between sample and duplicate?	Y	N	NA
15.	Samples checked for carry over from sample to sample due to target or nontarget overload?	Y	N	NA
16.	Are all data exported to LIMS, calculated and verified?	Y	N	NA

COMMENTS:

Analyst (s) _____ DATE: _____

REVIEWER _____ DATE OF REVIEW: _____

qc/qcmemos/Forms/gcmschecklist Rev. 2 08/13/01

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Attachment 4

AMRO Environmental
Laboratories Corporation

111 Herrick Street
Merrimack, NH 03054
(603) 424-2022

QUALIFICATION CARD FOR TPH/DRO SW-846 METHOD 8015Mod.

Trainee: _____ Signature: _____

Trainer: _____ Signature: _____

I. Knowledge Factors:

SOP Title: _____ Revision Number/Date: _____

1. Did the Trainee read and understand the above SOP?	Yes ☐	No ☐	NA ☐
2. Was the Trainee instructed on how to properly fill out the documentation?	Yes ☐	No ☐	NA ☐
3. Has the Trainee been instructed on the proper procedures for addressing potential problems?	Yes ☐	No ☐	NA ☐

II. Practical Factors:

1. Has the Trainee completed a Precision and Accuracy Study?	Yes ☐	No ☐	NA ☐
2. Has the Trainee completed an acceptable Performance Evaluation sample?	Yes ☐	No ☐	NA ☐
3. Did the Trainee completed an acceptable Laboratory Control Sample (LCS)? or PT study*	Yes ☐	No ☐	NA ☐
4. Has the Trainee performed manual integration as indicated in the SOP?	Yes ☐	No ☐	NA ☐

Completion Date: _____ Trainer's Signature: _____

I _____ have read, understood and follow the above method/SOP, which is in use at AMRO Laboratories for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

Section Manager/ Supervisor: _____ Date: _____

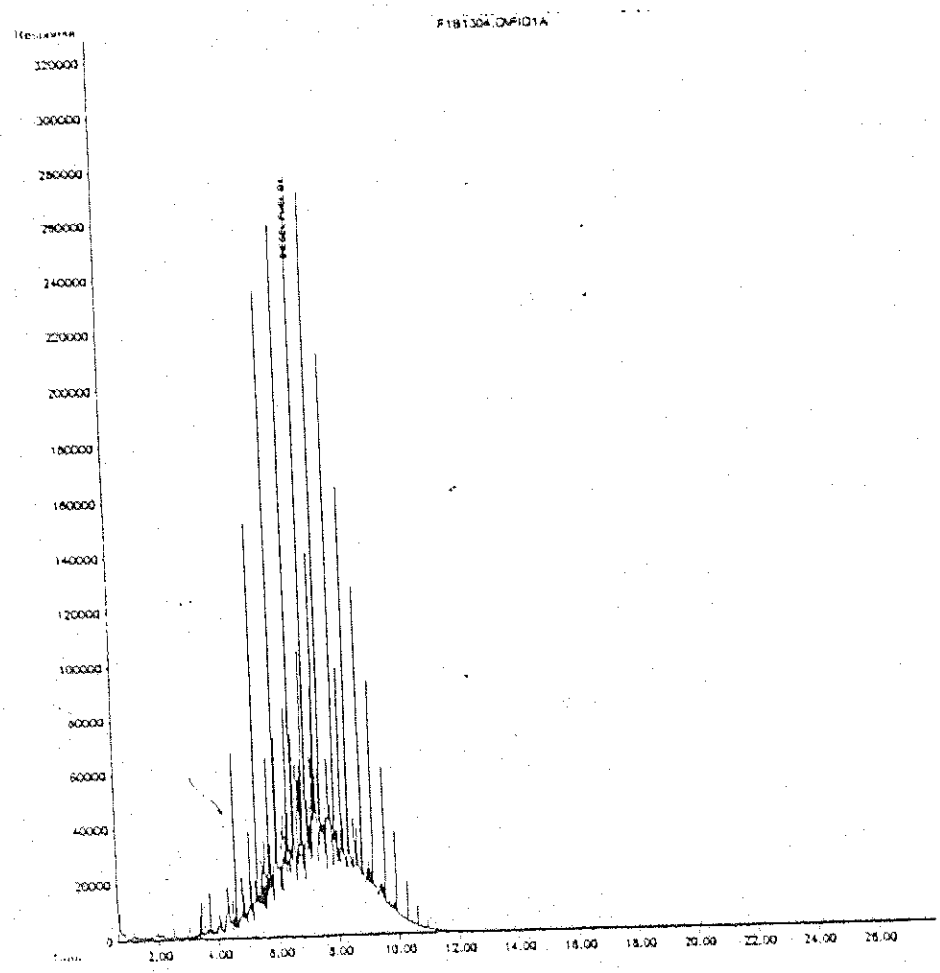
QA Manager: _____ Date: _____

qc/qcmemos/forms/TPH/DRO8015Mod.qualcard Rev. 0 08/13/01

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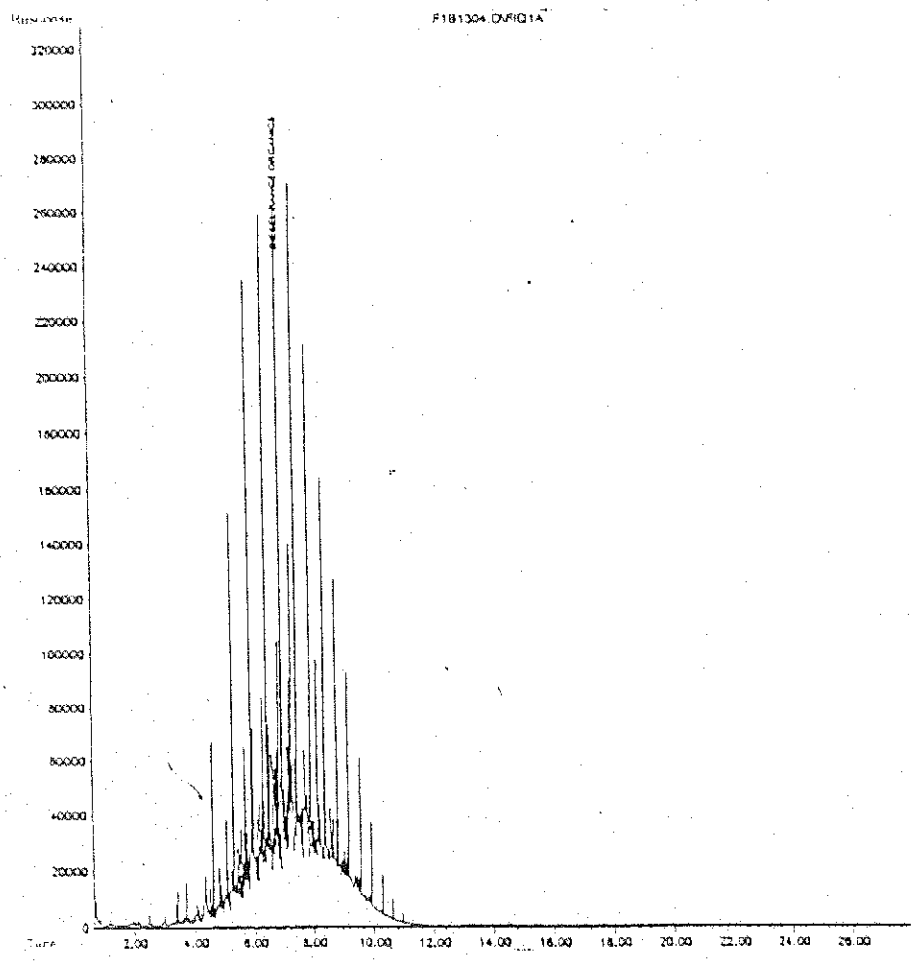
Attachment 5A

```
File       : C:\HPCHEM\1\DATA\082703\F1B1304.D
Operator   : KD
Acquired   : 27 Aug 2003   2:18 pm using AcqMethod FING1_32.M
Instrument :   Fing1
Sample Name: (#2 FUEL OIL 2500 NG/UL) DF-1 (FING_S)
Misc Info  : S0701031
Vial Number: 6
```



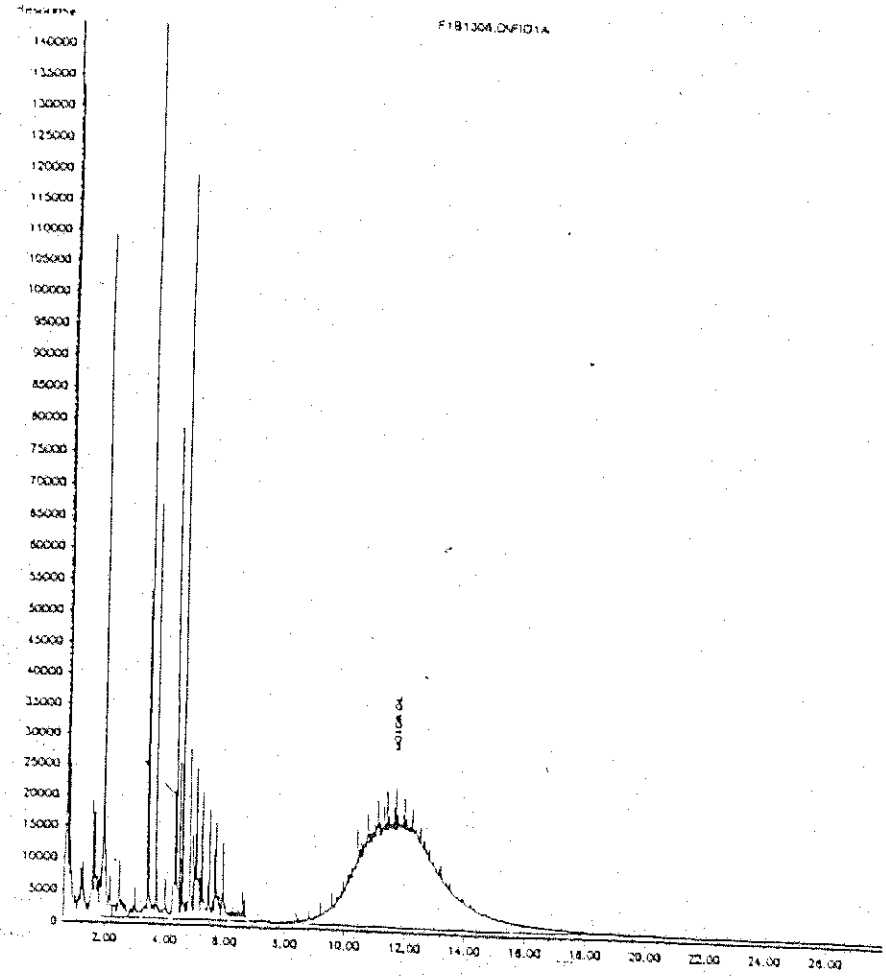
Attachment 5B

```
File       : C:\HPCHEM\1\DATA\082703\F1B1304.D
Operator   : KD
Acquired   : 27 Aug 2003   2:18 pm using AcqMethod FING1_32.M
Instrument :   fing1
Sample Name: (#2 FUEL OIL 2500 NG/UL) DF=1 (FING_S)
Misc Info  : S0701031
Vial Number: 6
```



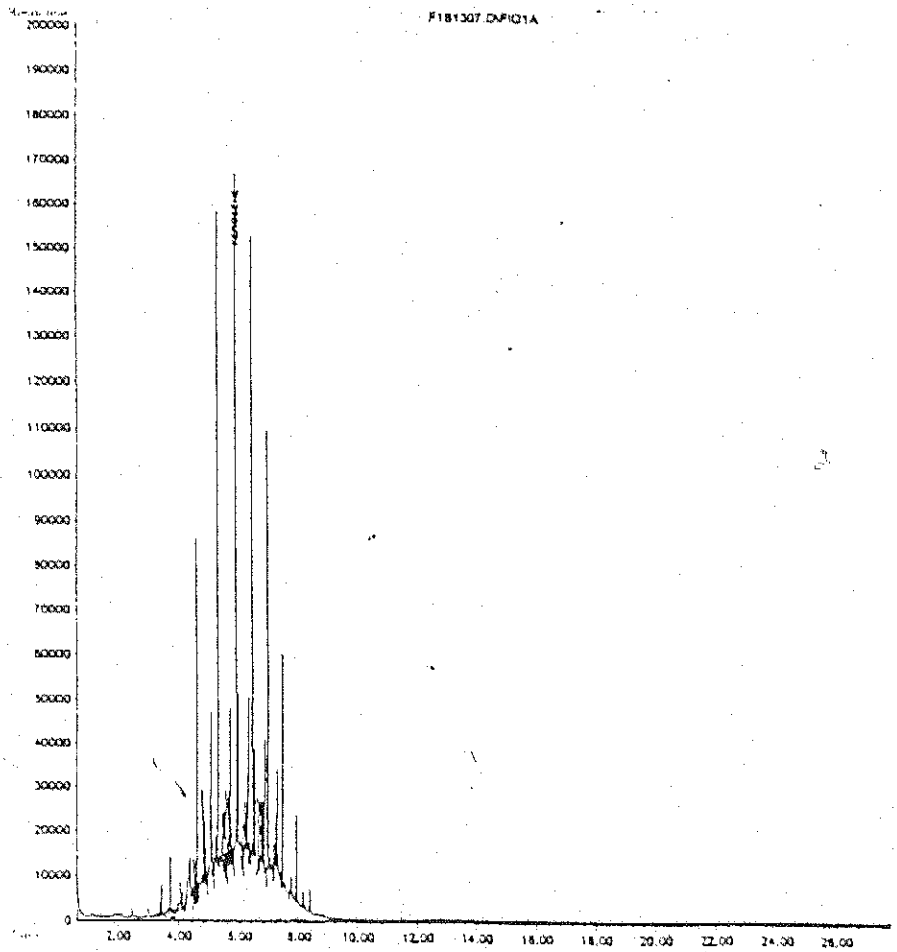
Attachment 5C

```
File        : C:\HPCHEM\1\DATA\082703\F1B1306.D
Operator    : KD
Acquired    : 27 Aug 2003   3:31 pm using AcqMethod FING1_32.M
Instrument :   Fing1
Sample Name: (GASOLINE/MOTOR OIL 1000 NG/UL) DF=1 (FING_S)
Misc Info   : S070103K
Vial Number: 8
```



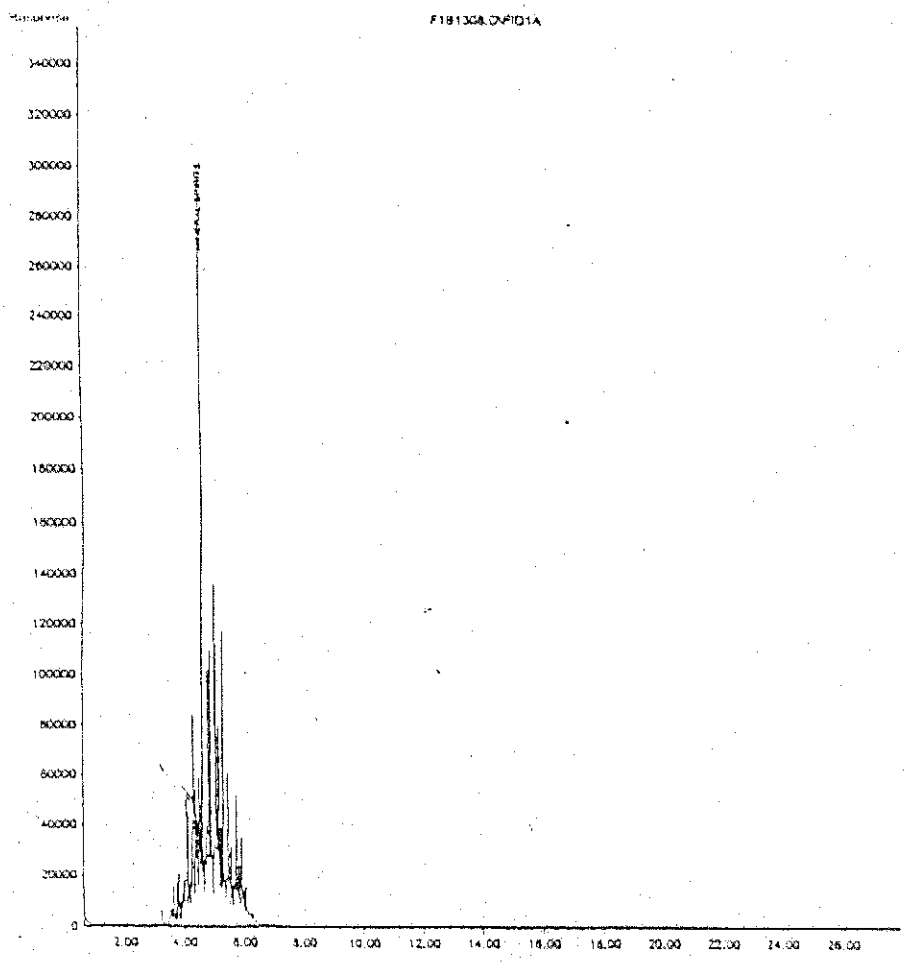
Attachment 5D

```
File       : C:\HPCHEM\1\DATA\082703\F1B1307.D
Operator   : KD
Acquired   : 27 Aug 2003   4:08 pm using AcqMethod FING1_32.M
Instrument :   Fing1
Sample Name: (KEROSENE 1000 NG/UL) DF=1 (FING_S)
Misc Info  : S070103L
Vial Number: 9
```



Attachment 5E

```
File        : C:\HPCHEM\1\DATA\082703\F1B1308.D
Operator    : KD
Acquired    : 27 Aug 2003    4:44 pm using AcqMethod FING1_32.M
Instrument  :   Fing1
Sample Name: (MINERAL SPIRITS 1000 NG/UL] DF=1 (FING_S)
Misc Info   : S070103M
Vial Number: 10
```



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800-929-9906
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Laboratory Method for the Determination of Herbicides in All Matrices

Approved By:

QA Officer: _____ Date: 8·10·2009

Laboratory Director: _____ Date: 8·10·2009

Revision History

Revision	Description of Change	Approval	Date
9	Updated to DOD 4.1	MAG	08/2009

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1. Title

Laboratory Method for the Determination of Herbicides in All Matrices.

EPA References:

Test Methods For Evaluating Solid Waste Physical/Chemical Methods- Method 8000B, Revision 2, December 1996

Test Methods For Evaluating Solid Waste Physical/Chemical Methods- Method 8151A, Revision 1, December 1996

EPA Method 615-The Determination of Chlorinated Herbicides in Municipal and Industrial Wastewater.

Quality Systems Manual For Administrative, Organic and Limited Inorganic Departments of ANALYTICS Environmental Laboratory, LLC. Current Revision

Analytics Environmental Laboratory (AEL) SOP QA-L001- Sample Receipt, Preservation and Storage

AEL SOP QA-H002: Separatory Funnel Liquid/Liquid extraction for the Determination of Chlorinated Acid Herbicides in Aqueous samples

AEL SOP QA-H003: Sonication Extraction for the Determination of Chlorinated Acid Herbicides in Solid samples

AEL SOP QA-H004: Extraction for the Determination of Chlorinated Acid Herbicides in Non Aqueous Liquid samples

AEL SOP QA-M009-Laboratory Method for Balance Calibration

AEL SOP QA-D001- Standard Operating Procedure for Data Review and Validation

AEL SOP-W001- Method for Containing, Cataloging, and Disposing of Solid/Liquid Waste

2. Scope and Application

This method is used for the determination of chlorinated phenoxy acid herbicides that are partitioned into an organic solvent (Diethyl Ether or Methylene Chloride:Acetone) and derivatized to methyl esters. The resulting methyl esters are amenable to gas

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chromatography. The parameters listed (Table 1) can be detected by this method. The quantitation range for aqueous sample is 1.0 μg/L-10 μg/L and 16.7 μg/kg-666.7μg/kg for solids. This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of GC data. Organic acids, especially chlorinated acids, may result in interferences with the determination.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware and other sample processing hardware that introduce artifacts or cause elevations in baselines. Method blanks must routinely be analyzed to show an absence of these interferences.

3.2 Matrix interferences may include contaminants that are co-extracted from the sample. These may include but are not limited to, phthalate esters, petroleum hydrocarbons, and other organic acids. Alkaline hydrolysis may be used to remove some of these interferences. However, hydrolysis may result in the loss of some of the target analytes.

3.3 Sample extracts should be dry prior to methylation or else poor recoveries may be obtained.

4. Safety

4.1 The toxicity or carcinogenicity of all the reagents included in this method have not been precisely defined. Therefore, each chemical compound should be treated as a potential health hazard.

4.2 Exposure will be minimized by wearing solvent resistant gloves, safety goggles, a lab coat and by working in a fume hood when samples or standards are handled.

4.3 Material Safety Data sheets for all of the compounds and reagents used in this method are on file in the laboratory.

5. Method Summary

A 1µL volume of the sample extract is injected into the gas chromatograph (GC) using a Hewlett Packard auto-ampler. Analytes are detected with an electron capture detector (ECD). Quantification of target compounds is performed by external standard technique, using Hewlett Packard Enviroquant software. Target Compounds are confirmed by additional analysis on a dissimilar capillary column. Refer to Figure 1 for the method summary.

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6. Sample Collection, Handling and Preservation

6.1 Solid samples should be collected in glass jar with a screw top Teflon lid.

6.2 Aqueous samples should be collected in 1 Liter Amber glass containers with teflon-lined caps.

6.3 Samples should be iced immediately after collection. All samples should be received cold 0°-6°C: the temperature should be recorded upon receipt. Refer to AEL SOP QA-L001 for proper cooler handling instructions.

6.4 Samples should be refrigerated at (1.1-4.4°C) as soon as possible after they are received at the lab. Refer to AEL SOP QA-L001 for laboratory receipt and log-in procedures.

6.5 Aqueous samples must be extracted within 7-days of collection.

6.6 Solid samples must be extracted within 14 days of collection.

6.7 Sample extracts must be analyzed within 40 days of extraction.

6.8 Extracts must be stored in a freezer at –10°C to –20°C protected from light.

7 Definitions

7.1 μL= micro-liter

7.2 mL = milliliter

7.3 L= liter

7.4 μg= microgram

7.5 g= gram

7.6 kg= kilogram

7.7 GC= Gas Chromatograph

7.8 ECD= Electron Capture Detector

7.9 DCAA= 2,4-dichlorophenylacetic acid

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7.10 MW= Molecular Weight

7.11 °C= Degrees Celsius

7.12 wt.= weight

7.13 vol.= volume

7.14 Conc.= concentration

7.15 psi= pounds per square inch

7.16 CF= Calibration Factor

7.17 inj.= injections

7.18 CAS= Chemical Abstract Service

7.19 ALS= Automatic Liquid Sampler

7.20 RSD= Relative Standard Deviation

7.21 min= minute

7.22 ID= Internal Diameter

7.23 CV= Calibration Verification

8. Apparatus and Materials

8.1 GC System

8.1.1 Gas Chromatograph: Agilent Technologies 6890 N or equivalent

8.1.2 Auto-sampler Sequence:

Hewlett Packard 7683 auto-sampler or equivalent
ALS Conditions from HP Chemstation:

Method:	HERB.M
Sample washes	2
Sample Pumps	3
Injector volume	1.0 μL
Syringe size	10.0 μL

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On column	off
Nano-liter adapter	off
Post-Inj. solvent A washes	2
Post-Inj. solvent B washes	2
Viscosity delay	1 second
Plunger speed	fast

8.2 GC Columns

8.2.1 Primary column:
Stx- CLPesticides 2, 30 m, 0.25 mm ID, 0.2 µm film thickness or equivalent

8.2.2 Confirmatory column:
Rtx – CLPesticides, 30 m, 0.25 mm ID, 0.25µm film thickness or equivalent

8.2.3 Guard column fused silica 10 meter, 0.25 mmID, Phenomenex Part #7CG-G000-00-GZ0 or equivalent

8.2.4 Oven Temperature Program:

Inlet A	275°C
Inlet B	off
Detector A	300°C
Detector B	300°C
Oven equib time	0 minutes
Initial temperature	80°C
Initial time	1.50 minutes

Level	Rate (°C/min)	Final Temp (°C)	Final Time (min)
1	100	120	0.00
2(A)	40.0	325	3.00

Next run time 10.03 minutes

8.3 Gases

Helium, Grade 5, for carrier gas
Argon Methane (95:5), Grade 5, for make-up gas for ECD
Carrier Gas Flow rate: 6.2 mL/min
Purge Vent Flow rate: 8 mL/min

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Split Vent Flow rate: 75 mL/min
Make-up Gas Flow rate: 45-50 mL/min

Inlet Purge	Init Value	On Time	Off Time	Split-less Injection
A	off	0.50	0.00	yes
B	off	0.50	0.00	yes

8.4 Electronic Pressure Program

Constant Flow: ON
Constant Flow Pressure 47.1 psi
Constant Flow Temp 80 °C

Total program time 10.03 minutes

8.5 Data System: Hewlett Packard Environmental Chemstation

8.6 Instrument run logbook

8.7 Sample preparation logbook

8.8 Instrument maintenance logbook

9. Standards and Reagents

9.1 Stock Standard Solutions: use the following standards or equivalent

9.1.1 Herbicide Curve Stock: 100 ug/mL, Accustandard M-8151

9.1.2 Herbicide Surrogate Stock: 100 ug/mL, Accustandard M-515-SS

9.1.3 Herbicide Second Source Stock: 100 ug/mL, Ultra HBM-8151m-1

9.1.4 Herbicide Surrogate Second Source Stock: 100 ug/mL, Absolute Standard #30022

9.2 Preparation of Working Solutions and Standards

9.2.2 Standards Preparations Logbook. All information pertinent to standards preparation must be recorded in black ink in a permanently bound logbook

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with numbered pages entitled "Herbicide Standard Prep". When a standard is prepared, it is assigned a unique, sequential number as follows: "HXXX, where H indicates that the standard is for herbicide analysis and XXX is the next sequential number (e.g. H001). Record this number on the left side of the page. The preparation date and analyst's initials are recorded in the lower right had corner of the preparation area. To the right of the standard number, describe the standard, including the concentration. Assign an expiration date and record it. Record the volume of the standard used, the standard description, manufacture, manufacturer's expiration date, catalog number and the lot number and the lot number. Record the amount, manufacture and lot number of the dilution solvent (Figure 3).

9.2.3 Store standards with a minimum of headspace in the freezer designated for herbicide standard storage. Label standard containers with the standard number, a brief description of the standard, the preparation date, analyst's initials, and the expiration date.

9.2.4 Expiration Dates

Stock standards may be stored in sealed ampules in the freezer until the manufacturer's expiration date. Stock standards should be replaced sooner if comparison with previous calibration standards indicates poor performance. Once opened, stock standards are good for six months. Expiration dates are for prepared intermediates standards are based on the oldest stock standard used, but will be no longer than 6 months from the preparation date. Replace standards if poor performance is indicated.

Note: Equivalent Dilutions may be used.

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9.2.5 Herbicide Calibration Curve

Std.Concentration ug/mL	Stock Standard	Stock standard Conc.	Vol. Of Stock Std. Sol.	Final Volume of Ether
Herb Std 0.025*	Herb. Stock Std. (9.1.1)	100 ug/mL	2.5 uL each	10 mL
Herb Std 0.05*			5.0 uL each	10 mL
Herb Std 0.1	Herb. Sur. Stock (9.1.2)	100 ug/mL	10 uL each	10 mL
Herb Std 0.2			20 uL each	10 mL
Herb Std 0.3			30 uL each	10 mL
Herb Std 0.5			50 uL each	10 mL
Herb Std 0.75			75 uL each	10 mL
Herb Std 1.0			100 uL each	10 mL

*These standards are used for low level pentachlorophenol analysis and are not always used for other analytes.

9.2.6 Herbicide Second Source Check Standard

Std.Concentration ug/mL	Stock Standard	Stock standard Conc.	Vol. Of Stock Std. Sol.	Final Volume of Ether
0.5 ug/mL	Herb. Second Source Stock (9.1.3) Herb Sur. Second Source Stock (9.1.4)	100 ug/mL 100 ug/mL	50 uL of each	10 mL

9.3 Hexane - Pesticide quality or equivalent for auto-sampler rinseate #2 and column washing.

9.4 Acetone - Pesticide quality or equivalent for column washing.

9.5 Methylene Chloride - Pesticide quality or equivalent for column washing.

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9.6 Methanol – Purge &Trap grade or equivalent auto-sampler rinseate if needed.

9.7 Ether- Pesticide Quality or equivalent for dilutions and rinseate #1.

10. Procedure

10.1 Refer to the following Standard Operation Procedures for sample preparation.

AEL SOP QA-H002: Separatory Funnel Liquid/Liquid extraction for the Determination of Chlorinated Acid Herbicides in Aqueous samples

AEL SOP QA-H003: Sonication Extraction for the Determination of Chlorinated Acid Herbicides in Solid samples

AEL SOP QA-H004: Extraction for the Determination of Chlorinated Acid Herbicides in Non Aqueous Liquid samples

10.2 Instrument Run Logbook

A bound paginated laboratory notebook is assigned to each instrument for use as a sample run log. The date is noted above each day's analyses along with the analyst's initials. Each day starts a new page. Each run is recorded with the following information: the run number, the auto-sampler vial number, the AEL sample # or standard #, the dilution, the method file name, relevant comments (i. e. rerun for low surrogate, standard passes, dilution necessary, etc.). Each instrument is identified with a unique letter. Data files (run numbers) are assigned using this letter followed by the next consecutive number. The letter designation for this instrument is "H". Also on the run log is a "loading check" and "unloading check". Prior to loading the vials, check to ensure that the tape label agrees with the label on the vial. When unloading the samples, verify that the order in the auto-sampler tray agrees with that in the run log. Record initials in the loading check boxes. See Figure 2 for an example of the run log page.

10.3 Initial Calibrations

Before sample analysis can begin, the instrument must be calibrated for each parameter. Calibration standards are prepared as described in section 9.2 above. The lowest level standard must be at or below the quantitation limit for each target compound. The remaining levels are chosen based on the linear range of each parameter with the highest level not exceeding the concentration limits of the column or detector. For herbicides, analysis of a minimum of five calibration levels is required, **unless a quadratic curve fit is used, which requires a minimum of six calibration levels.** Refer to Table 2 for the specific calibration levels.

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10.3.1 The method is named by incorporating the date the calibration was run as follows: HMMDDY where;

H is the designation for analysis of Herbicides
MM is a two number designation for the month
DD is a two number designation for the day
Y is a one number designation for the last digit of the year

Saving the previous 5-point using the new name creates the new name. Calibration standards will be quantitated automatically upon completion of the analytical run. Load the data file of the first standard in the 5-point.

10.3.2 Evaluate the low standard for analyte sensitivity. Peak area should be approximately three times the baseline noise level. Analytes may be selectively dropped from the low standard as long as the curve still contains five levels.

10.3.3 Verify that the computer is properly integrating all analytes and selecting the correct peaks. All manual integrations must be conducted in accordance with AEL SOP QA-M022.

10.3.4 Update each of the calibration levels by loading each datafile and using the "InitCal" menu pull-down "Update Levels". It is important to save the method after updating.

10.3.5 Determination of Linearity

Quantitation of herbicides is based on the area or height of the analyte peak at the known retention time. Linearity is determined from the calibration factor (CF) of the standards or the generation of a calibration curve using either linear regression or quadratic fit.

10.3.5.1 One of the following options should be used.

- Relative standard deviation (RSD) for each analyte must be$\leq$ 20%. Calculate a calibration factor for each compound. The calibration factor is the peak area (or height) of the compound divided by the mass of the compound injected. When the relative standard deviation (RSD) of the calibration factors is $\leq$20% the standard curve is considered to be linear and the average calibration factor can be used for quantitation.
- Linear regression - If a linear calibration curve is used the correlation coefficient (r) must be 0.995 (coefficient of determination r^2 0.990) or greater to meet acceptance criteria.

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- Quadratic Fit (non-linear regression)-For a quadratic fit, the coefficient of determination must be (r^2) 0.995 or greater to meet acceptance criteria. **Quadratic fits requires a minimum of 6 calibration standards.**
- **It is never acceptable to force a calibration curve (linear or quadratic) through the origin. Nor is it acceptable to include the origin (0,0) as a sixth calibration point.**

10.3.5.2 If the linearity criteria are acceptable, file the initial calibration printout, the analyte plots for compounds set to linear or quadratic or the custom report evaluating curve fits, and the standard quantitation reports in the Herb QC book.

10.3.6 Determination of Retention Time (RT) Windows: Herbicides are identified using absolute retention times. Retention time windows are generated as follows:

1. Make three injections of a standard over the course of 72 hours. Serial injections or injections over a period of less than 72 hours may result in retention time windows which are to tight.

2. Calculate the mean and standard deviation (SD) of the absolute retention time (RT) for each analyte in all three standards.

3. The width of the RT window is defined as the mean RT +/- (3*SD). Note: When the SD=0 a default SD of 0.01 shall be used.

4. The center of the RT window is determined for each analyte by using the absolute RT for each analyte once per initial calibration and at the beginning of each analytical window. For initial calibration the mid-point standard is used to determine the RT window and for analytical windows the calibration verification standard to open the window is used for RT window establishment.

RT windows shall be determined annually or when a new column is installed.

10.3.7 Immediately following an initial calibration a Second Source Calibration Verification (herbicide standard prepared from an independent source) is analyzed (Table 2) to verify the calibration. The measured value must agree within 20% of the true value for the calibration to be considered valid.

10.3.8 Calibration verification (CV) or Continuing calibration standard Calibration verifications are conducted on a daily basis, prior to sample analysis, every 20th analysis (10 recommended to minimize reanalysis), and at the end of each sequence to validate the calibration of the system for each parameter. NOTE: DOD projects require calibration verification be analyzed after every 10 field samples and at the end of the analysis sequence.

10.3.8.1 A continuing calibration standard at the mid point concentration shall be used for this purpose. If the measured value is within ±15% of the initial calibration, then the initial calibration is considered valid. DOD projects allow a difference of 20%.

10.3.9 Corrective Action

If the calibration verification or initial calibration is unacceptable, the analyst must consider that the analytical system is out of control and corrective action is needed. Once the problem is determined, correct it and re-analyze the initial calibration if necessary. Instrument corrective action must be documented in the instrument's maintenance logbook. Each log entry should contain the date of the action, the initials of the analyst, a description of the problem and the corrective action taken and the result of the corrective action. Hard copies of the unacceptable initial calibration and/or calibration verification standard should be stored in the QC notebook with an explanation of the problem and how it was corrected.

10.4 Analyzing Samples

10.4.1 Inject 1µL of sample or blank extract onto the GC column using the Hewlett Packard 7683 auto-sampler. Analyze the sample using the GC conditions in the method HERB. Record the sample analyses in the instrument's run logbook (Section 10.2).

10.5 Quantitation of Sample Analytes

10.5.1 Target compounds are identified qualitatively based on the retention time comparison with the calibration standard. All analytes must fall within the RT windows defined above for positive qualification. The final interpretation of peak identification is based on the discretion of an experienced GC analyst. Target compounds are quantitated using a

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method file stored on the HP Enviroquant. The method file stores calibration factors and retention times. The calibration files are named as follows: HMMDDY where;

H= is the designation for Herbicide analysis
MM = is a two number designation for the month
DD = is a two number designation for the day
Y = is a one number designation for the year

Example: H06071, for the calibration analyzed on June 7, 2001.

10.5.2 Enviroquant quantitates the sample extract result by calculating the corresponding analyte concentration for the area found based on the calibration curve (or average calibration factor).

10.5.3 Dilutions

If the concentration of any target compounds exceeds the upper calibration, the extract must be diluted in Ether to achieve an on column concentration that will be in the upper half of the calibration range. Analyze the diluted extract and recalculate the sample result. Dilutions are documented in the Herbicide Sequence Logbook, noting the volume of extract used, the volume of Ether used and the Ether lot number.

10.5.4 The sample result is calculated, correcting to sample weight and percent solids or volume using the following equation:

$$\text{Sample result } (\mu g/L \text{ or } \mu g/kg) = \frac{\text{Sample extract conc. } (\mu g/ml) * \text{actual vol of extract (ml)}}{\text{sample wt or vol (kg or L)} * \% \text{ solids}/100} \times DF$$

where:

1) Sample extract conc (µg/ml) is found on the raw data report from HP Enviroquant.
2) vol of extract (ml) and sample wt or vol is found on the extraction transfer sheet (Figure 3).
3) % solids is found on the extraction transfer sheet (Figure 3).

10.5.5 If target compounds are detected on the primary analysis, a confirmatory analysis is performed simultaneously to verify the presence of the target compound. This confirmation analysis is performed on a different column type, i. e. Stx -CLPesticides. The result from the confirmatory analysis should be within 40% of the result from the primary analysis. Report the higher of the two results unless there are overlapping peaks or other anomalies present. In these cases clearly document on the data file the

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reason for reporting the lower result and initiate a QC memo so results can be reported with a comment to this affect. When the % difference is >40% the data user should be advised of the disparity. This is done by flagging the result with a "P" and entering a comment on the results page.

11 Quality Control

11.1 Each time the five point calibration is analyzed an herbicide standard prepared from an independent source is analyzed (Table 2) to verify the calibration. The measured value must agree within 20% of the true value for the calibration to be considered valid.

11.2 The initial calibration is verified at the beginning of each day, every 20th sample analysis (10 recommended to minimize reanalysis required for DOD projects), and at the end of each sequence with a calibration verification (CV). If the calibration verification is unacceptable, the problem must be corrected and documented. It may be necessary to analyze a new initial calibration. Each sample analysis must be bracketed with an acceptable initial or calibration verification standard. However, if the samples are "U" and the ending calibration verification fails high, the proceeding runs are considered acceptable. Initiate a QC Documentation Memo for approval by the Quality Control Officer and the Laboratory Director. See Figure 4.

11.3 Per sample

11.3.1 A surrogate (2,4-Dichlorophenylacetic acid) is added to every sample, sample matrix spike, sample matrix spike duplicate, laboratory control sample and blank before extraction to test the efficiency of the extraction procedure. Percent recoveries are calculated by dividing the amount found by the amount added. The recovery acceptance ranges are listed in the instrument method per matrix. Whenever a surrogate is outside of the acceptable range, an investigation of the extraction and analytical procedures is conducted. If the surrogate recovery is outside the laboratory acceptance criteria the following steps should be taken:

1.) Re-inject the extract to verify the surrogate result.
2.) Re-extract the affected sample if sample volume and holding time permits.
3.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. Unusual circumstances during the extraction are noted in the sample preparation log

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notebook. Once the deviation report is completed it is given to the QA officer.

11.3.2 Surrogate recoveries are charted by matrix in batches of 20. The analyst uses this information to observe trends, which might indicate a problem with the instrument or extraction procedure. The following conditions shall be considered out-of-control events for all control charts:

- One point outside control limits
- Three consecutive points outside warning limits
- Six consecutive points such that each point is larger (or smaller) than its immediate predecessor
- Eight consecutive points on the same side of the centerline

11.4 Per extraction batch- These are AEL's general acceptance criteria. Projects that have required QAPPs take precedence to AEL's general limits. These requirements are indicated using controlled project sheets to be filled out by the laboratory project manager.

11.4.1 A method blank must be performed with every sample batch (20 samples or daily). The blank is analyzed prior to the associated samples to monitor interferences or contaminants that may have occurred during the extraction process. If a blank is found to contain more than the reporting limit (1/2 RL for DOD projects) of any compound the following steps should be taken:

1.) Re-inject the extract to verify the results.
2.) Re-extract the samples in the batch if sample volume and holding time permits. It may be possible to accept samples that are "U" with the approval of the QA Officer.
3.) If insufficient sample exists to re-extract, the contaminant will be qualified with a "B" flag.
4.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Extraction personnel may be asked to determine if a matrix effect has contributed to the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

11.4.2 A laboratory control sample (LCS) must be extracted with every sample batch 20 samples or daily, whichever is more frequent. The LCS is spiked with 0.5 mL of the matrix spiking solution. The laboratory control samples are subjected to the same extraction procedure as the client samples, thus indicating extraction accuracy. The GC/ECD analyst will calculate the percent recoveries using the following formula:

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$$\text{Recovery}=\%\text{Rec}=\frac{Cs-Cu}{Cn}X100$$

Where:
Cs = Measured concentration of the spiked sample aliquot
Cu = Measured concentration of the unspiked sample aliquot (use 0 for non-detects)
Cn = Nominal (true) concentration

If the recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. An LCS with recoveries above the laboratory acceptance criteria is considered acceptable for samples with no analytes detected. The following steps should be taken for recoveries below the acceptance limits or samples that have analytes detected:

1.) Re-inject the extract to verify the result.
2.) Re-extract the affected sample if sample volume and holding time permits.
3.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

11.5 Per QC Batch-These are AEL's general acceptance criteria. Projects that have required QAPPs take precedence to AEL's general limits. These requirements are indicated using controlled project sheets to be filled out by the laboratory project manager.

11.5.1 A matrix spike/matrix spike duplicate sample (MS/MSD) must be extracted with every QC batch (20 samples or monthly). The MS/MSD's are spiked with 0.5 mL of the matrix spiking solution. MS/MSD samples are subjected to the same extraction procedure as the client samples, thus indicating extraction accuracy and precision. Spike recoveries are calculated and charted in accordance with AEL SOP QA-M023 in batches of 20 by matrix. Acceptable water or soil spikes shall be within the limits listed in table 3. Matrix spikes (MS) and Matrix spike duplicate (MSD) recoveries are calculated by the following formula:

$$\text{Recovery}=\%\text{R}=\frac{Cs-Cu}{Cn}X100$$

Where:
Cs = Measured concentration of the spiked sample aliquot

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Cu = Measured concentration of the un-spiked sample aliquot (use 0 for non-detects)
Cn = Nominal (true) concentration

If the recoveries are not within the laboratory's acceptance limits, the cause must be determined and appropriate corrective action taken. A MS/MSD with recoveries above the laboratory acceptance criteria is considered acceptable for samples with no analytes detected. The following steps should be taken for recoveries below the acceptance limits or samples that have analytes detected:

1.) Re-inject the extract to verify the result.
2.) Re-extract the affected sample if sample volume and holding time permits.
3.) Initiate a QA Deviation Report (Figure 4) that explains the corrective actions taken and possible causes for the deviation. Unusual circumstances during the extraction are noted in the sample preparation log notebook. Once the deviation report is completed it is given to the QA officer.

For each set of spikes extracted the Relative Percent Difference (RPD) must be calculated. Precision is estimated from the RPD of the concentrations (not recoveries) measured in the MS/MSD pairs. To be acceptable the RPD should be
≤ 25%for aqueous samples and ≤ 30%for solid samples. Calculate the RPD according to the formula below.

$$RPD = \frac{|C1-C2|}{\frac{C1+C2}{2}} X100$$

11.6 Minimum Detection Limits

Minimum detection limit studies or method detection limit check samples are performed according to AEL SOP QA-M021.

11.7 Run log sequences are confirmed when loading and unloading sample extracts.

11.8 Data Validation- All data is reviewed completely before reporting to the client. Data review and Validation is conducted following AEL SOP QA-D001.

12. Routine Instrument Maintenance

12.1 Inlet Maintenance

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A diagram of the GC inlet can be found in the Agilent Technologies 6890N manual Volume 2-Inlets. All inlet maintenance is recorded in the maintenance logbook for the instrument.

12.1.1 The inlet septum is replaced after 20-40 injections. An 11 mm septum is used. (Supelco thermogreen™ LB-2 Septa catalog #2-0654 or equivalent).

12.1.2 A clean deactivated/silanized split-less single taper liner should be installed each day, or as needed. Install the liner in the GC with a new O-ring (Hewlett Packard catalog #5180-4182 or equivalent), being careful not to over-tighten the nut. Refer to AEL SOP QA-M011 for cleaning and silanization of inlet liners.

12.1.2 The injection port inlet seal and washer must be replaced periodically. This should be checked bi-monthly. If the gold seal is black or pitted a new seal and washer is required. Refer to Agilent Technologies 6890N manual Volume 2-Inlets for proper seal/washer placement.

12.2 Column Maintenance

All column maintenance is recorded in the maintenance logbook for the instrument.

12.2.1 The front end of the columns and the guard column are inspected when instrument performance is poor. Compound and particle deposits will degrade the chromatography by the broadening of peaks and the reduction of sensitivity. One loop (about 12 inches) of the front end can be clipped from each of the columns and/or the guard column to remove these contaminants.

12.2.2 Periodic baking of the column at its upper temperature limit will clean the column of contaminants that have remained on the column during the day's normal operating temperatures. This should be performed for a maximum of 24 hours.

12.2.2 When the previous two steps do not lead to improvement of the chromatography it may be necessary to replace the guard column. If chromatography still doesn't improve a new column may also be required. Generally, a column will last 6 - 10 months under normal operating conditions. Document the installation of a new column in the instrument logbook. Each entry includes the date the column was installed a description of the column which includes manufacturer, column length,

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internal diameter, film thickness, catalog and lot numbers and maximum temperature. Conditions under which the column was conditioned and any changes in installation procedures shall be documented as well.

12.3 The molecular sieve in-line trap for the helium gas is changed annually.

12.4 Charcoal trap for ECD vent is changed annually.

12.5 ECD Leak tests are performed every six months. Testing materials are purchased from National Leak Test or a similar company. Procedures vary so it is necessary to read the instructions received with each kit.

12.6 Gas tanks are replaced when tank pressure falls below 500 psi.

12.6 Periodic tightening of the detector's make-up gas fitting is necessary. The oven temperature programming loosens the fitting. Check weekly.

13. Pollution Prevention and Waste Management

13.1 See AEL SOP QA-W001 for handling of waste and unused portions of sample.

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TABLE 1

Target Compounds

Compound	CAS#
Dalapon	75-99-0
Dicamba	1918-009
2,4-D	94-75-7
2,4,5-TP (Silvex)	93-72-1
2,4,5-T	93-76-5
Dichloroprop	120-36-5
2,4-DB	94-82-6
Dinoseb	88-85-7
MCPA	94-74-6
MCPP	93-65-2
Acifluorfen*	50594-66-6
Bentazon	25057-89-0
Chloramben *	133-90-4
DCPA,Diacid	2136-79-0
3,5-Dichlororbenzoic acid	51-36-5
4-Nitrophenol	100-02-7
Picloram	1918-02-1
Pentachlorophenol	87-86-5
DCAA (surrogate)	19719-28-9

***These analytes are not routinely reported but may be requested.**

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TABLE 2

Calibration Levels

Herbicide (HERB):

Analytes Level:	1*	2*	3	4	5
Dalapon	0.025µg/mL	0.05µg/mL	0.1µg/mL	0.2µg/mL	0.3µg/mL
Dicamba	0.025	0.05	0.1	0.2	0.3
2,4-D	0.025	0.05	0.1	0.2	0.3
2,4,5-TP (Silvex)	0.025	0.05	0.1	0.2	0.3
2,4,5-T	0.025	0.05	0.1	0.2	0.3
Dichloroprop	0.025	0.05	0.1	0.2	0.3
2,4-DB	0.025	0.05	0.1	0.2	0.3
Dinoseb	0.025	0.05	0.1	0.2	0.3
MCPP	2.5	5.0	10	20	30
MCPA	2.5	5.0	10	20	30
Acifluorfen	0.025	0.05	0.1	0.2	0.3
Bentazon	0.025	0.05	0.1	0.2	0.3
Chloramben	0.025	0.05	0.1	0.2	0.3
DCPA, diacid	0.025	0.05	0.1	0.2	0.3
3,5-Dichlorobenzoic acid	0.025	0.05	0.1	0.2	0.3
4-Nitrophenol	0.025	0.05	0.1	0.2	0.3
Picloram	0.025	0.05	0.1	0.2	0.3
Pentachlorophenol	0.025	0.05	0.1	0.2	0.3
DCAA (Surrogate)	0.025	0.05	0.1	0.2	0.3

Analytes Level:	6	7	8
Dalapon	0.5µg/mL	0.75µg/mL	1.0µg/mL
Dicamba	0.5	0.75	1.0
2,4-D	0.5	0.75	1.0
2,4,5-TP (Silvex)	0.5	0.75	1.0
2,4,5-T	0.5	0.75	1.0
Dichloroprop	0.5	0.75	1.0
2,4-DB	0.5	0.75	1.0
Dinoseb	0.5	0.75	1.0
MCPP	50	75	100
MCPA	50	75	100
Acifluorfen	0.5	0.75	1.0
Bentazon	0.5	0.75	1.0
Chloramben	0.5	0.75	1.0
DCPA, diacid	0.5	0.75	1.0
3,5-Dichlorobenzoic acid	0.5	0.75	1.0
4-Nitrophenol	0.5	0.75	1.0
Picloram	0.5	0.75	1.0

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Pentachlorophenol	0.5	0.75	1.0
DCAA (Surrogate)	0.5	0.75	1.0

*These levels are below the reporting limits for most compounds and are generally used for low level analysis of Pentachlorophenol.

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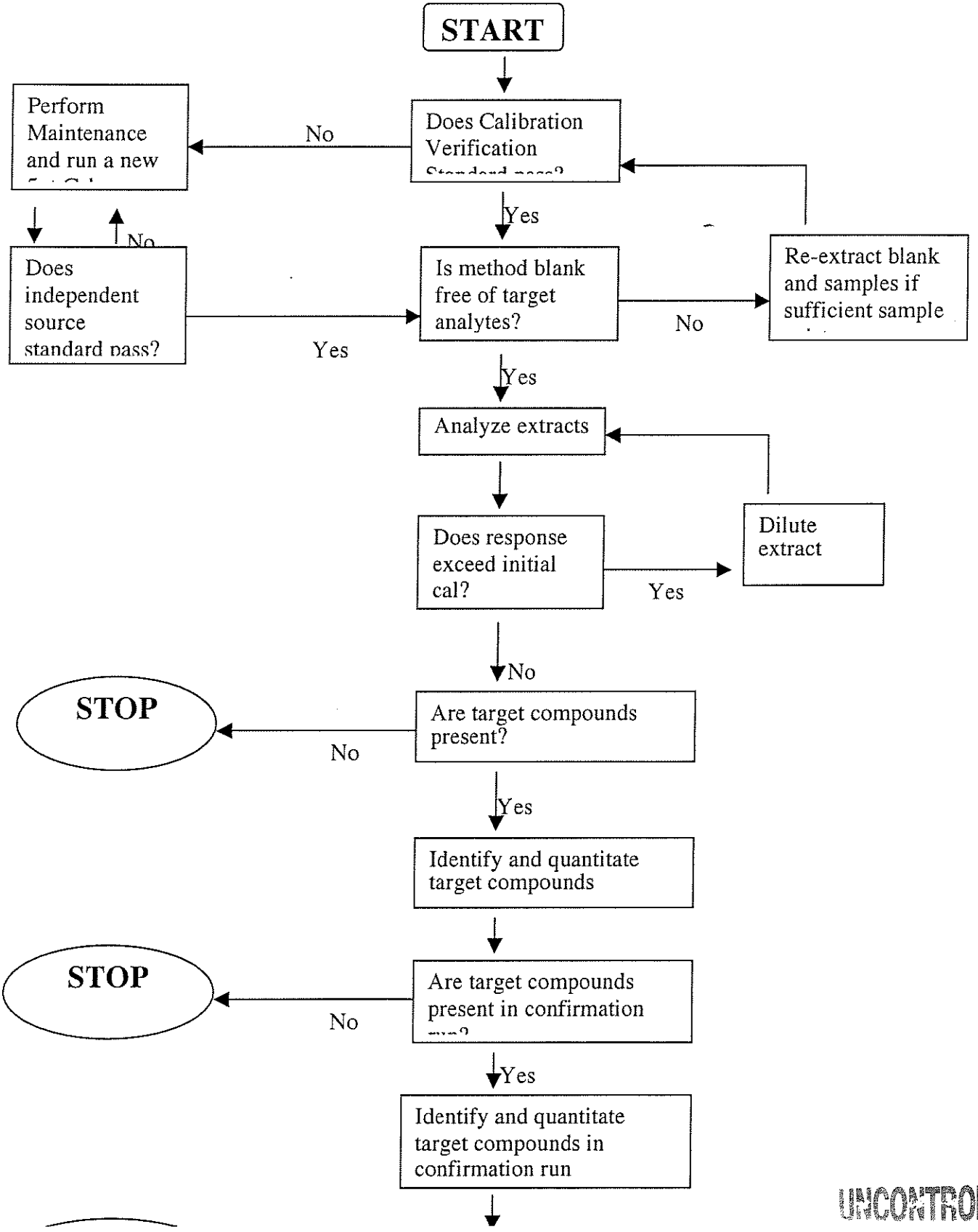
TABLE 2 (Cont.)

Second Source:

Analytes	Concentration (μg/mL)
Dalapon	0.5
Dicamba	0.5
2,4-D	0.5
2,4,5-TP (Silvex)	0.5
2,4,5-T	0.5
Dichloroprop	0.5
2,4-DB	0.5
Dinoseb	0.5
DCAA	0.5
MCPP	50
MCPA	50
Acifluorfen	0.5
Bentazon	0.5
Chloramben	0.5
DCPA, diacid	0.5
3,5-Dichlorobenzoic acid	0.5
4-Nitrophenol	0.5
Picloram	0.5
Pentachlorophenol	0.5
DCAA (Surrogate)	0.5

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FIGURE 1



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FIGURE 2

PEST/PCB/HERB
Instrument F
Sequence Log Book

Load: _____
Unload: _____

Data File	Vial #	Sample	Method	Comments

Standard Dilutions: 1:2- 500 uL sample +500 uL hexane
1:5- 200 uL sample +800 uL hexane
Hexane Lot : _____ 1:10- 100 uL sample + 900 uL hexane
1:20- 50 uL sample + 950 uL hexane
1:100- 10 uLsample +990 uL hexane
1:1000- 1 uLsample +1000 uL hexane

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FIGURE 3

Transfer Date: 11/21/97

Initials: MR

EXTRACT TRANSFER SHEET

Organochlorine HRB

Sample	Analysis	Extraction Date	Matrix	Initial Volume (mL)	Initial Weight (g)	Final Volume (mL)	% Solids	Extraction Comments
39059-1	615	11/20/97	Aqueous	1060		5.0		
38209-1	T8151W	11/20/97	Sludge	200		5.0		

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FIGURE 4

Quality Assurance Deviation

Analysis Code:
QC Batch ID:
(attach copy or list samples affected by this memo)

Out of Control Event with Brief Explanation

☐ Method Blank

☐ Continuing Calibration or Initial Calibration Issue

☐ Laboratory Control Spike

☐ Matrix Spike/MSD

☐ Surrogate Recovery

☐ Other

SOP Corrective Action Followed:

☐ Re-extract in Hold time

☐ Re-inject to confirm

☐ Other

☐ Document SOP corrective action or "See QA Memo" on raw data file

QA Officer Review: _____ _____

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METHOD 8151 QC ANALYSES

QC Analysis	Minimum Frequency	Acceptance Criteria	Corrective Action
Minimum five-point Initial Calibration for all analytes	At start-up and when continuing calibration fails	One of the options below: % RSD ≤20% for each analyte linear regression :r>0.995 (r^2>0.990) Non-linear regression: r^2>0.995(requires 6 points)	use linear curve if %RSD is out or re-calibrate
Initial Calibration Verification (ICV)-Second Source Standard Check	Immediately following initial calibration	All target analytes within ±20% of the expected value	Correct problem, re-run ICV If that fails recalibrate
Method Blank (MB)	1X per extraction batch of 20 samples or less	All analytes < RL DOD <1/2 RL	Clean system, Re-analyze Re-extract and Rerun Report with flag
Continuing Calibration Standards	A mid point standardanalyzed: prior to sample analysis after every 20 field samples (10 recommended required for DOD) at the end of the analysis sequence	%D ≤15% for all analytes DOD %D ≤20% for all analytes	Correct problem re-run Cont. Cal. If still out recalibrate Reanalyze all samples since the last successful continuing calibration standard.
MS/MSD	Per Batch of 20 samples or less as designated by client	Within acceptance criteria established by the laboratory	Check analysis and calculations, check LCS/LCSD %R If LCS/LCSD in control write QA

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			Memo
Surrogate spike- (DCAA)	All field and QC samples	Within acceptance criteria established by the laboratory	Check analysis - then Re-run sample Re-extract Write QA Memo
Laboratory Control Sample (LCS)/LCS Duplicate (LCSD)	1X per extraction batch for LCS LCSD per batch of 20 samples or less	Within acceptance criteria established by the laboratory	Check for errors, Re-run LCS/LCSD Re-extract and reanalyze entire batch of samples
Second Column Confirmation	For each detection above the RL on the primary column	Primary and secondary result must compare within 40% RPD	Report result with "P" and notify client
Results reported between MDL and PQL	none	"J" flag as estimated data	none

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Mercury in Solid or Semisolid Waste

SW-846 Method 7471A by Perkin Elmer FIMS 100 Automated Mercury Analyzer

Procedure Number: IL-3005

Revision No.: 4

Revision Date: 01/23/2004

Review Date: 01/18/2010

Prepared By: Paula Benham Date: 1/18/10

Approved By: Robert G. Sargent Date: 01/19/10
Inorganic Manager

Approved By: [signature] Date: 1/19/10
Laboratory Director

Approved By: Paula Benham Date: 1/19/10
Quality Assurance Manager

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1.0 SCOPE and APPLICATION

1.1 This procedure is used for the preparation/determination of mercury in soils, sediments, bottom deposits and sludge-type materials.

1.2 AMRO routinely digests MS and MSD and Duplicate sample per batch, to meet all varying project QC requirements.

1.3 AMRO uses a Perkin-Elmer FIMS (Flow Injection Mercury System) 100 automated mercury analyzer. This instrument is an automated flameless AA mercury analyzer.

1.4 The Aqua Regia is generated in the sample tube by adding concentrated Hydrochloric Acid and concentrated Nitric Acid to ensure a fresh reagent for each sample and standard.

2.0 METHOD SUMMARY

2.1 Aqua Regia (generated in the sample tube) is added to a representative 0.6g aliquot of sample and heated for two minutes in a hot block at 95°C (± 2°C). Potassium Permanganate is added and the mixture is heated for an additional 30 minutes in a hot block at 95°C (± 2°C). Mercury in the digested sample is reduced with stannous chloride to elemental mercury, which is volatile and swept from the solution with argon and measured by a flameless AA procedure at wavelength of 253.7nm.

3.0 DEFINITIONS

3.1 **Analytical Batch -** A group of samples of a similar matrix analyzed together with the same procedure, using the same reagents and within the same time period. Samples in each batch should be of similar composition (i.e. soil, water, oil, etc.). A batch shall not exceed 20 samples.

3.2 **Matrix Spike (MS)/ Duplicate (MSD) -** Predetermined quantities of stock solutions of certain analytes, which are added to a sample matrix prior to sample digestion and analysis. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at the regulatory standard level.

3.3	**Sample Duplicate - (DUP)**	A duplicate sample aliquots of sample which is carried through the same digestion and analysis. The relative percent difference between the sample and sample duplicate is calculated and used to assess analytical precision.
3.4	**Laboratory Control-Sample(LCS)/ Duplicate (LCSD)**	A control sample of known composition. Aqueous and solid laboratory control samples are analyzed using the same sample preparation, reagents and analytical methods employed for samples received. Percent recoveries are calculated for each of the analytes detected. The relative percent difference between the samples is calculated and used to assess analytical precision. The concentration of the spike should be at the regulatory standard level.
3.5	**Method Blank**	An analytical control consisting of all reagents which is carried through the entire analytical procedure. It is used to define the level of laboratory background contamination.
3.6	**Instrument Detection Limit (IDL)**	Determined by multiplying by 3 the standard deviation obtained for the analysis of a standard solution (each Analyte in reagent water) at a concentration of 3 to 5 times instrument detection limit within 7 consecutive measurements per day.
3.7	**Method Detection Limit (MDL)**	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater then zero. It is determined by analyzing seven replicates of a standard solution that is 2–3 times the IDL. The standard deviation is then multiplied by 3.143 (the student t test for seven replicates) to obtain the MDL or 2.998 for eight replicates. For soil MDL studies play sand must be used (7.11).
3.8	**Reporting Limit (RL)**	The lowest level that can be reliably achieved within specified limits of precision and accuracy during routine laboratory operating conditions.
3.9	**Linear Range**	The highest concentration of mercury that can be analyzed which is true value of the highest calibration standard.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 Soil samples shall be refrigerated at 4°± 2°C until analyzed. The holding time for mercury is 28 days from collection date.

4.2 All sample containers must be cleaned according to the SOP on glassware cleaning.

5.0 POTENTIAL PROBLEMS AND INTERFERENCES

5.1 Possible interference from sulfide is eliminated by the addition of Potassium Permanganate.

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

5.3 Sea waters, brines and industrial effluents high in chlorides require additional permanganates. During the oxidation step, chlorides are converted to free chlorine which will also absorb radiation at 253nm. Care must be taken to assure free chlorine is absent before the mercury is reduced and swept into the cell. This may be accomplished by using an excess of hydroxylamine hydrochloride reagent. Both inorganic and organic mercury spikes have been quantitatively recovered from sea water using this technique.

5.4 Low level mercury sample preparation, digestion and analysis may be subject to environmental contamination. This analysis should not be performed in areas with high ambient backgrounds where mercury was previously employed as an analytical reagent in analyses such as total Kjeldahl nitrogen (TKN) or chemical oxygen demand (COD).

6.0 HEALTH AND SAFETY

6.1 AMRO has a laboratory safety program in place that applies to all employees. There is a safety officer as well as employees with 40 hours of OSHA training. The air quality of the laboratory has been monitored and employees are sent for physicals. All employees receive fire and safety training upon employment.

6.2 Fire extinguishers are located in various areas of the lab. A lab evacuation plan is posted in every room, with a designated meeting place.

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6.3 All new analysts receive training on the safe handling of samples, standards, the changing of gas cylinders, and the operation of instruments. In the lab, clean laboratory coats and gloves are worn when working with samples. Neat standards are made up in hoods. When gas tanks are replaced, the carrier with chain is used properly, and all gas tanks are chained in place. Soap is used to check for any leaks.

6.4 Care is taken with glassware and syringes to prevent injury. Broken glass and syringes are promptly removed to broken glass containers. First aid kits are available throughout the lab. There are also eyewash stations and an emergency shower.

6.5 MSDS's are reviewed for each chemical used to ensure proper precautions are observed when handling chemicals.

6.6 Pollution prevention – The use of chemical exhaust hood are required when working with chemicals. They are specifically designed to minimize analyst exposure. The hood exhaust is monitored periodically to ensure environmental exposure is minimized.

6.7 Waste control – AMRO takes whatever steps possible to minimize waste. This includes volume reduction where appropriate. AMRO also properly segregates wastes according to contaminants and a commercial vendor is utilized for waste disposal.

7.0 INSTRUMENTS AND EQUIPMENT

7.1 Perkin-Elmer FIMS 100 automated mercury analyzer. This instrument is an automated flameless AA mercury analyzer. It is controlled by a Dell Octiplex computer with WinLab/FIMS software Ver 2.5. Samples and Standards are introduced by an AS-90 autosampler.

7.2 Environmental Express Hot block capable of reaching 95°C (±2°C).

7.3 Argon Cylinder with regulator

7.4 100µL, 200µL,500µL and 1000uL Eppendorf pipets for spiking (Fisher # 03402-69)
or equivalent).

7.5 Repipet Universal Dispensers, 5 mL volume range in 0.05 mL graduations

7.6 100mL containers for sample storage

7.7 100mL, 50mL, 500mL volumetric flasks. Only Class A volumetric glassware is used to prepare standards.

7.8 Plastic autosampler tubes (Fisher #14-961-29)

7.8 Autosampler racks B for samples and standards (Perkin-Elmer # B300-0132)

7.9 Analytical balance, capable of weighing to 0.0001g.

7.10 Thermometer

7.11 Play Sand, heated to 475 C, is added to the Method Blank and LCS/LCSD.

8.0 INSTRUMENT PREVENTATIVE MAINTENANCE AND REPAIR

8.1 All maintenance must be documented in the maintenance logbook.

8.2 Daily:

8.2.1 Check calibration curve correlation (>0.995).
8.2.2 Rinse before and after running samples for 10 minutes with 10% HCL.

8.3 Weekly:

8.3.1 Change pump and drain tubing.
8.3.2 Wipe off instrument with a damp cloth.
8.3.3 Archive files.
8.3.4 Clean racks and standard cups with DI water.

8.4 Monthly:

8.3.1 Change waste drain tubing.
8.3.2 Clean external optics.

8.5 Annually:

8.5.1 Replace liquid/gas separator.

8.6 As Needed

8.6.1 Replace the mercury lamp.

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9.0 REAGENTS AND STANDARDS

Note: ***All reagents should be of analytical grade, suitable for mercury determination***

9.1 Nitric acid for Trace Metals Analysis, Omni Trace, EM (VWR# NX0407/2)

9.2 Sulfuric acid for Trace Metals Analysis, Fisher (Fisher # AS10SK-212)

9.3 Hydrochloric acid for Trace Metals Analysis, Omni Trace, EM (VWR# HX0607/2)

9.4 Deionized (DI) Water, ASTM Type I.

9.5 Potassium Permanganate: 5% solution. (w/v)

9.5.1 Dissolve 50g of potassium permanganate (Fisher P279-212) in 1000mL of DI water

9.6 Hydroxylamine Hydrochloride

7.7.1 Dissolve 120g of Hydroxylamine Hydrochloride in 1000 mL of DI water (JT Baker 2196-01).

9.7 Stannous Chloride Solution: Add 13.2g of stannous chloride (Fisher TI42-500) to 1000mL of 3%hydrochloric acid. Prepare fresh daily.

9.8 0.5N Hydrochloric Acid: Add 2.8mL Hydrochloric acid for Trace Metals Analysis, Omni Trace, EM (VWR# HX0607/2) to 200mL of DI water.

9.9 3% Hydrochloric Acid Rinse Solution: Add 30mL of Hydrochloric acid for Trace Metals Analysis, Omni Trace, EM (VWR# HX0607/2) to 1000mL DI water.

9.10 Refer to Mercury Standard Log (Attachment 1) for all standard preparation information. It is readily available near the FIMS. This log contains copies of the Certificates of Analysis for each stock purchased. It also gives an in depth explanation of all the standards required for analysis. This log is used to document the preparation of all Hg calibration standards and instrument QC standards.

9.11 Stock Mercury Solution: Hg 5 mg/L Standard. Use to prepare Working Standards for Calibration Standards, LCS and Spikes.

9.12 Calibration Standards - The Calibration Standard is a purchased standard. The Calibration Standard is replaced annually. The analytical solutions are prepared as needed.

9.12.1 Mercury Spike and LCS solution (500 ug/L): To a 50mL volumetric flask, add 5mL of DI and 2.5mL of HNO3 **THEN** 5.0mL of the Mercury Calibration Solution (5.0 mg/L) and dilute to the mark with DI. This solution is prepared Daily.

9.12.2 Working Mercury Calibration Solution (0.20mg/L): Use to prepare Calibration Standards. To prepare, add 2.5mL Nitric Acid and 2.0mL Of 5.0mg/L Hg Calibration Standard up to 50mL with DI water. Document in Mercury Standard Tracking Log (Attachment 1). This solution is prepared Daily.

9.13 Stock Mercury Solution: Hg 20 mg/L Standard. Use to prepare Initial Calibration Verification (ICV). Note: ICV standard must be from a different source from the Hg standard. Initial Calibration Verification Standard (ICV) and be replaced annually. The ICV solution is run at the beginning of each run with a control limit of 90-110%. Analytical solutions are diluted as needed:

9.13.1 Working Mercury ICV Solution (0.60mg/L): Use to prepare ICV. To prepare, bring 1.25mL Nitric Acid and 3.0mL of 20mg/L Hg Standard. Bring to 100mL with DI water. Document in Mercury Standard Tracking Log. This standard is prepared Daily.

9.14 Hg Soil Laboratory Control Standard (LCS): ERA Priority Pollutant/CLP #540, purchased from ERA. The concentration varies each time it is purchased.

9.15 Continuing Calibration Verification Standard (CCV)

9.15.1 The CCV is prepared daily from the calibration standard. (See Hg Standard Log.) CCVs are run every 10 samples with a control limit of 80-120%.

9.16 Standards

9.16.1 Refer to Mercury Standard Log for all standard preparation information. It is readily available near the PS200II. This log contains copies of the Certificates of Analysis for each stock purchased. It also gives an in depth explanation of all the standards required for analysis. This log is used to document the preparation of all Hg calibration standards and instrument QC standards.

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9.16.2 Calibration Standards

9.16.2.1 The Calibration Standard is a purchased standard.

9.16.2.2 The Stock Calibration Standard is replaced annually.

9.16.2.3 The analytical solutions are prepared fresh.

9.16.3 Initial Calibration Verification Standard (ICV):

9.16.3.1 The analyst must document that the source of this standard is different than the calibration standard. The ICV Standard is a purchased standard. It is run at the beginning of each run. The acceptance criteria: 90-110%.

9.16.3.2 The Stock ICV Standard is replaced annually.

9.16.3.3 Analytical solutions are diluted as needed.

9.16.4 Continuing Calibration Verification Standard (CCV):

9.16.4.1 The CCV is prepared daily from the calibration standard. (Refer to Hg Standard Log.) CCV's are run every 10 samples and at the end of the analytical sequence. The acceptance criteria: 80-120%.

9.16.4.2 The Stock Calibration Standard is replaced annually.

9.16.5 Reporting Limit (RL) Standard for Hg (concentration = 0.20µg/L).

9.16.5.1 The RL Standard is prepared from the Calibration Standard. Refer to Mercury Standard Log (Attachment 1).

9.16.5.2 The Stock Calibration Standard is replaced annually.

9.16.5.3 Analytical solutions are prepared fresh.

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9.17 TRACKING OF ANALYTICAL STANDARDS

9.17.1 Chemical Receipt:

The receipt of all standards, neat compounds and reagents from commercial sources is documented in the Chemical Receiving Logbook by sample receiving personnel or designate. Each solution or compound is assigned a unique standard receipt ID number, the letter "A" and a four digit sequential number. The date received, source, lot number, expiration date and disposal date are recorded. The expiration dates are provided from the manufacturer and the disposal dates are completed by AMRO employees when standards are used up or disposed as indicated in the SOP "Sample Management GL-1006".

9.17.2 Standards Preparation:

Standards are prepared as written in the respective SOPs. In order to assure the accuracy of standards the following general guidelines are used:

9.17.2.1 ACS reagent grade chemicals are used when the method warrants.

9.17.2.2 Any stock standards that are purchased are accompanied by a certificate of analysis from the manufacturer.

9.17.3 Standard Tracking:

It is important to be able to track all analytical standards back to their sources. The following procedure is used by AMRO personnel to accomplish this:

9.17.3.1 Metals Standards

Hg Standards Log (PS200II) - Preparation and Standard ID's are listed in the Standard Log. Standards include: S1 (Calibration Blank, ICB, CCB), S2 (0.20 µg/L Calibration Standard and RL Standard), S3 (2.0 µg/L Calibration Standard), S4 (4.0 µg/L

Calibration Standard), S5 (8.0 µg/L Calibration Standard) and S6 (10.0µg/L Calibration Standard), CCV 6.0 µg/L Standard, ICV (3.0 µg/L Independent Source QC Standard), and Stock Solutions for Matrix/LCS spike additions (Matrix/LCS spike addition = 5.0 µg/L). The Solution ID is written as the ID followed by the date prepared (example: S1042798 is standard S1 prepared on 042798).

9.17.3.2.1 Wet Chemistry and Metals Reagent Log - Working reagents are made from reagents listed in the Chemical Receiving Log and are documented in the Reagent Log. The corresponding analysis, preparation date, reagent name, AMRO ID, initial concentration, amount used, final volume, final concentration, Solution ID, expiration date, storage location, and analyst initials are recorded. The initial concentration is obtained from the certificate received from the manufacturer. The Solution ID is written as an "R" followed by a 4 digit sequential number. The expiration of prepared reagents varies. Reagents should be used in accordance with method specifications and manufacturer's instructions as indicated in the SOP "Sample Management GL-1006".

10.0 PROCEDURE

10.1 Determination of Instrument Detection Limit (IDL):

10.1.1 Pour a calibration blank solution into seven autosampler tubes, and analyze as individual samples.

10.1.2 Determine the standard deviation for seven replicates.

10.1.3 The IDL = 3 x (standard deviations).

10.1.4 Provide all required information on the current Instrument Detection Limit study worksheet provided by the QA Department.

10.2 Determination of Method Detection Limit (MDL):

10.2.1 Digest at least seven individual aliquots of an MDL Solution using an appropriate digestion procedure which is 3-5 times the IDL or 20% of the AMRO reporting limit. For soil MDL studies play sand must be used (7.11).

10.2.2 Determine the standard deviation for the seven digestions.

10.2.3 The MDL = 3.143 x (average of the standard deviations). If eight samples re digested, use 2.998.

10.2.4 Provide all required information on the current Method Detection Limit study on the worksheet provided by the QA department.

10.2.5 The MDL must be performed annually.

10.3 Precision and Accuracy:

10.3.1 Run four LCS's. Acceptance limits are 80-120%. Precision and accuracy is done annually by each analyst for each matrix and analysis type.

10.4 Linear Range:

10.4.1 The highest concentration of mercury that can be analyzed which is the true value of the highest calibration standard. This must be determined initially and verified annually.

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10.5 Calibration Curve:

10.5.1 Take clean, labeled 100mL tubes and make the 7 standards listed below by adding the amount of Deionized Water shown plus 4.0mL of concentrated Hydrochloric Acid and 1.0 mL of concentrated Nitric Acid followed by the amount of Mercury standard listed below:

Standard (µg/L)	DI Water	Hg Calibration Working Std.
0 (CCB)	40.0 mL	0
0.2	39.6mL	100µL (Reporting Limit)
2.00	36.0mL	1.0mL
4.00	32.0mL	2.0mL
6.00 (CCV)	28.0mL	3.0mL
8.00	24.0mL	4.0mL
10.00	20.0mL	5.0mL
3.0 (ICV)	39.6mL	500µL Hg ICV Working Std

10.5.2 Place tubes in water bath at 95°C (±2°C) for two minutes. After two minutes remove the tubes.

10.5.3 After cooling, add 8 mL of DI and 15mL of Potassium Permanganate.

10.5.4 Mix thoroughly.

10.5.5 Place tubes back in hot block at 95°C (±2°C) and heat for 30 minutes. Document the temperature of the hot block in the mercury logbook. The temperature of the hot block MUST be at 95 ±2°C. Add the samples and wait until the temperature returns to 95 before starting the 30 minutes count.

10.5.6 Remove after 30 minutes, cool and bring to 100mL volume with DI water.

10.5.7 Add 6mL hydroxylamine hydrochloride to all samples to reduce the excess permanganate.

10.5.8 Pour samples into appropriately labeled standard tubes. Document preparation of the curve in the Mercury Standard Tracking Log.

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10.6 Obtaining a Homogeneous Sample

10.6.1 Refer to SOP GL-1013 "Sample Homogenization for Soil, Solid and Semisolid Waste" for sample homogenization guidance and how to obtain a representative subsample.

10.6.2 If multiphase samples are submitted, the client is called to determine which phase is to be digested.

10.7 Sample Digestion

10.7.1 Transfer three well-mixed portions of sample, totaling 0.6g into an appropriately labeled tube. Weigh sample using an analytical balance to the 0.0001g place. Document weight in Hg logbook.

10.7.2 Prepare blank by adding 5.0mL DI, 4.0mL of HCl, and 1.0mL of HNO_3 to an appropriately labeled tube. Each batch of twenty samples requires a blank. Add 0.6 g of Blank Sand to the method blank.

10.7.3 Prepare LCS by adding 5.0mL DI, 4.0mL of HCl, and 1.0mL of HNO_3 to an appropriately labeled tube then 1.0 mL of spiking solution (Sec 9.10.2). Each batch of twenty samples requires an LCS (true value = 5.0µg/L). Add 0.6 g of Blank Sand to the LCS & LCSD.

10.7.4 To each soil sample add 5.0mL DI, 4.0mL of HCl, and 1.0mL of HNO_3.

10.7.5 Prepare the Matrix Spike (MS) and Matrix Spike Duplicate (MSD) by chosing a sample and adding three well-mixed portions of sample, totaling a 0.6g aliquot of sample into tubes labeled with the sample ID plus MS or MSD. Add 1.0mL Spiking Solution (Sec 9.10.2) to the appropriate commercial MS and MSD samples. Add 5.0mL DI, 4.0mL of HCl, and 1.0mL of HNO_3. Each batch of twenty samples requires an MS and an MSD. (true value = 5.0µg/L)

10.7.6 If needed for training or internal reference, an ERA soil sample can be prepped as an LCS.

10.7.7 Place tubes in the hot block at 95°C (±2°C) for two minutes then remove and cool. Add DI water to a volume of 40mL by using the 40mL line on the centrifuge tube.

10.7.8 Add 15mL Potassium Permanganate to each sample.

10.7.9 Mix thoroughly.

10.7.10 Loosely cap samples.

10.7.11 Place samples in the hot block at 95°C (± 2°C) for 30 minutes. Document the temperature of the hot block in the mercury logbook. The temperature of the water bath MUST be at 95 ± 2°C. Add the samples and wait until the temperature returns to 95 before starting the 30 minute count.

10.7.12 After 30 minutes, remove the samples and record the hot block temperature. Remove samples from water bath, allow cooling and adding DI to a final volume of 100mL.

10.7.13 Add 6mL hydroxylamine hydrochloride to all samples to reduce the excess permanganate.

10.7.14 Pour samples into appropriately labeled glass centrifuge tubes. Centrifuge as needed. They can also be filtered w/ a syringe filter and a 45µL syringe filter.

10.6.2 Operational Parameters: See Attachment A for operational parameters

10.6.3 Instrument Start-up:

10.6.3 Switch on the spectrometer with the switch at the left-hand side of the spectrometer's front panel.

10.6.4 Switch on the computer and monitor with power switches on the front of each one.

10.6.5 Windows 95 will come up.

10.6.6 Double click AAWinLab folder on desktop. Instrument software will come up.

10.6.7 One the initial screen click Menus and Toolbar.

10.6.8 Put pump tubing in place, place reductant line in the Stannous Chloride and carrier line in 3% HCl.

10.6.9 Display FIAS Control window by choosing FIAS on the tool bar.

10.6.10 Press the FIAS on/off button. Instrument will go into an internal test mode.

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10.6.11 Go to Autorun or press pump 1 on the FIAS control screen to rinse.

10.7 Creating and Modifying Sample Information Files

10.7.1 The system uses this file to label the data from samples both on the printed log of the results and in the results file. Information in this file is also used by the system to calculate final concentrations. A sample information file is especially useful for other data handling programs to manipulate the data.

10.7.2 To create a new sample information file, click on New Sample Info File in the File menu. The Sample Information Editor will appear.

10.7.3 Enter the sample information including all IDs in the cup where the sample will be placed. The amount of information that is entered depends on how the analysis results are to be reported, in weight/weight units or weight/volume units. The first eight positions are dedicated to Standards and Instrument QC as follows:

Position No.	Sample ID
1	Calibration Blank
2	Std. 0.20µg/L
3	Std. 2.0µg/L
4	Std. 4.0µg/L
5	Std. 8.0µg/L
6	Std. 10.0µg/L
7	ICV 3.0µg/L
8	CCV 6.0µg/L

The next nine positions are reserved for future QA/QC samples. Start analytical samples at Auto Sampler Position 17.

10.7.4 Save the sample information file by clicking on Save As Sample Info File in the File menu. The Save As dialog will appear. Type a name for the file (Mmmddyy for example, M031099A) and click on OK.

10.7.5 To modify a stored sample information file click on Open Sample Info File in the File menu.

10.7.6 In the Open dialog select the sample information file to be opened and click OK.

10.7.7 In the Tools menu, click on Sample Information Editor and enter the new sample information.

10.7.8 Save the sample information file by clicking on Save As Sample Info File in the File menu. The Save As dialog will appear.

10.7.9 In this dialog, type a name for the file and click OK.

10.8 Setting Up the Automated Analysis Window:

10.8.1 Select the correct methods by double clicking on an entry field in the Method column in the Automated Analysis window. The Open Method dialog appears. Use this dialog to select the methods that are required.

10.8.2 Set a delay time between methods to allow the lamps to warm up before the system makes measurements by typing a value in the Delay column. This is not needed for mercury analysis.

10.8.3 Select the samples to be analyzed by using a sample information file or by selecting individual sample tray locations. To use a sample information file click on Open next to the Sample Information File entry field. Then Open Dialog appears. This dialog is used to select the name of the sample information file. In the Automated Analysis window, in the table, select the box in the Defined in Sample Info. File column. To select individual sample tray locations clear the box in the Defined in Sample Info. File column in the table in the Automated Analysis window. Type the locations of the samples in the Use Autosampler Locations Listed Below column. Type individual locations or a range of locations. Use commas to separate the locations and the ranges. Example: 10-15, 18,20,25-30 for the analyte addition technique, type only the locations of the pure sample solutions. If the locations of blanks, QC, checks or calibration solutions are typed in, they will be analyzed as if they were normal samples.

10.8.4 Select the data saving and print options by clicking Open next to the Results Data Set entry field. The Choose Results Data Set dialog will appear. Use this dialog to select the results data set. If the data set exists, new data will be added to it. Select the Save Data box to save the results in the selected data set. To print the results during the analysis, select the Print Log box.

10.8.5 Select items to switch off after the analysis by selecting the Off After Analyses options that are required.

10.9 Starting a Calibration Curve Analysis

There are two different options for performing a calibration curve analysis.

10.9.1 In the Automated Analysis Window, click on Analyze All. The system analyzes the calibration solutions first, immediately followed by the samples and any other solutions, for example, the QC and check samples.

10.9.2 In the Automated Analysis Window, click on Calibrate. The system will analyze the calibration solutions. If necessary edit the calibration curve. When the calibration curve is acceptable, click on Analyze Samples in the Automated Analysis window.

10.9.3 Following calibration, the instrument will print out a "Calibration data for Hg." The Correlation Coefficient MUST be better than 0.995, and it's usually better than 0.999. The Calculated Concentrations should be in good agreement with the True Values for the standards. The %RSD is usually less than 1.0%, with the possible exception of the 0.20 ug/L standard.

10.10 Loading the Autosampler Tray

10.10.1 Flow Injection autosampler – Make sure that the correct sample tray (Tray B) has been selected in the Method Editor on Calib. – Autosampler page. In the Automated Analysis window, click on the Analyze tab. Click on Load Tray. Place the loaded sample tray in the autosampler, or load the samples and other solutions into the sample tray in the autosampler. Make sure that the solutions are in the locations specified in the Method Editor, in the sample information file and in the Automated Analysis – Setup window. Click on Load Tray again.

10.11 Rinsing the Fluid System

10.11.1 Place the inlets of the pump tubes for the carrier, reductant and any other reagents in a container of the first rinsing solution.

10.11.2 Place the inlet of the sampling tube in a container of the first rinsing solution or if the autosampler is being used, place a container of the first rinsing solution in the wash location of the autosampler. In the Automated Analysis window, click on Select Location. In the Autosampler dialog, select Go to wash. Click on OK.

10.11.3 In the FIAS Control window set a speed in 100 for the pump. Click on Pump # for each pump to start the pumps.

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10.11.4 In the FIAS Control window, switch the FIAS-valve between the Fill and Inject positions a few times to rinse the inside of the valve.

10.11.5 Rinse the system for as long as necessary to remove all traces of the solution used previously.

10.11.6 If more than one rinsing solution is used, place the inlets of the pump tubes for the carrier, reductant and any other reagents in a container of the next rinsing solution.

10.11.7 Place the inlet of the sampling tube in a container of the next rinsing solution or in the Automated Analysis window, click on Probe Up/Down, then place a container of the next rinsing solution in the wash location of the autosampler.

10.11.8 Repeat rinsing and setup for each rinsing solution.

10.11.9 After rinsing with the final rinsing solution, remove the inlets for the pump tubes from the rinsing solution. If the autosampler is used, in the Automated Analysis window, click on Probe Up/Down to raise the probe. Allow the pumps to run until the fluid system is empty. In the FIAS Control window, click on Pump # for each pump to stop the pumps.

10.12 Analysis Start up

10.12.1 When analysis is started, the system first checks for errors in the method and in the sample information file. If the system finds a fault that must be corrected before analysis can start, a message will appear and the fault must be corrected before the system will start the analysis.

10.12.2 When the button is clicked to start an analysis procedure, a green indicator will appear on the button and the other buttons will become inactive. The system uses the analytical conditions in the active method for each replicate. When all the solutions have been analyzed, the green indicator button disappears and the other buttons will become active.

10.12.3 The progress of the analysis is shown in the Automated- or Manual Analysis window.

10.13 Shutting Down the Flow Injection System

10.13.1 Prepare the correct rinsing solutions.

10.13.2 Rinse the fluid system with the correct rinsing solutions.

10.13.3 Switch the FIAS pumps off by clicking on On/Off for each pump in the FIAS Control window.

10.13.4 Switch off the spectrometer with the switch at the left-hand side of the front panel.

10.13.5 Turn off the carrier gas supply

10.13.6 Release the pressure on the pump tubes. Swing the pressure levers away from the pump tube magazines.

10.13.7 Empty the waste bottle. Dispose of waste solutions correctly, according to the safety regulations.

10.13.8 Switch off the computer and printer.

11.0 CALCULATIONS

11.1 Instrument Calibration Concentrations are in µg/L and Sample Concentration are in mg/kg. To verify soil results, perform the following calculation:

$$\text{Reported Conc. (mg/kg)} = \frac{\text{Determined conc. (µg/L) x Final Vol(0.025L) xDilution}}{\text{sample weight (g) x total solids (in decimal form)}}$$

11.2 % Recovery Laboratory Control Sample (water) = Determined concentration (µg/L)/true value x 100.

11.3 % Recovery MS/MSD = Determined concentration - sample concentration/true value x 100

11.4 %RPD MS/MSD or DUP = (sample - dup)/((sample + dup)/2) x 100. Use concentrations of each in calculations.

12.0 QUALITY CONTROL

12.1 Method Blank:

12.1.1 One Method Blank must be digested with every 20 samples or for every batch. The acceptable criteria: < RL and <½ RL for Department of Defense (DoD) projects.

12.1.2 The Method Blank results above the reporting limit indicate contamination. The source of contamination will be investigated and corrected. If a sample is greater than the reporting limit and less than ten times the Method Blank results, the sample will be re-digested and re-analyzed.

12.2 Matrix Spike and Matrix Spike Duplicate:

12.2.1 One matrix spike and one matrix spike duplicate are required for every 20 samples or for every preparation batch.

12.2.2 Samples must be selected for spiking at random and rotated among clients and sites from batch to batch. In some cases, the client may specify which sample is to used for MS/MSD; if not , the laboratory shall pick a representative sample based on the following:

A. MS/MSD shall not be performed on Trip Blank, Rinsate Blank or Field Blank.
B. Adequate sample volume.
C. Client requested QC deliverables

12.2.3 The concentration of the analyte in the unspiked sample aliquot should be no more than four times the amount added. If the sample result is greater than four times the amount added, the LCS is the only valid assessment of accuracy for the batch and the spike recovery is not evaluated for control. If the control limits are not met for Relative Percent Difference, the control limits for Percent Recovery in the Matrix Spike are not applicable, since sample heterogeneity is indicated and an inaccurate sample result used to calculate percent recovery is suspected. The acceptable criteria: % R=75-125%

12.3 Sample Duplicate:

12.3.1 One sample duplicate is required for every 20 samples or for every preparation batch .

12.3.2 Duplicate samples are two aliquots of a sample which are carried through the same sample preparation and analysis procedure to verify the precision of the method for that sample matrix. The RPD (Relative Percent Difference) is calculated from the original and the duplicate sample. If one sample result is above the reporting limit and one is less than five times the reporting limit the data is acceptable. An RPD can also be calculated between a matrix spike and matrix spike duplicate. The RPD is calculated using the concentration of the matrix spike and matrix spike duplicate. If the

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control limits are not met for Relative Percent Difference, the control limits for Percent Recovery in the Matrix Spike are not applicable, since sample heterogeneity is indicated and an inaccurate sample result used to calculate percent recovery is suspected. The control limits are ≤ 20%.

12.4 Laboratory Control Sample

12.4.1 One LCS is required for every 20 samples or for every preparation batch .

12.4.2 The %R control limit for the LCS is 80-120%.

12.4.3 If the Control limits which are not met for percent recovery in the LCS indicates inaccuracy during the procedure and requires re-digestion and re-analysis.

12.5 The following chart contains the frequency and control limits for QC required during the analytical run.

QC Sample	Frequency	Hg Action Level	Hg Action	Analyst Response
ICV	Once, after calibration	±10% of True Value for SW-846 Method 7471A	Stop	Correct problem and recalibrate
ICB*	Once, after ICV	±RL	Stop	Correct problem and recalibrate
RL	Start & Finish	±20% of True Value	Flag Data	Use caution regarding low level samples
CCV	10% of analytical samples	±20% of True Value for SW-846 Method 7470A	Flag Data	Correct problem and reanalyze affected samples
CCB*	Following each CCV	± RL	Flag Data	Correct problem and reanalyze affected samples

* ± ½ RL for Department of Defense (DoD) projects.

12.6 Corrective Action for Batch QC:

12.6.1 The initial investigation of the problem involves:

12.6.1.1 Checking sample concentrations for being high compared to the spike or too low for the RPD limits.

12.6.1.2 Checking calculations and raw data transcriptions (both QC and sample).

12.6.1.3 Checking sample concentrations and analytical sequence for influences such as carryover and mislabeling.

12.6.1.4 Reviewing the notes in the Mercury Digestion worksheet (Attachment 2) and analytical notebook for signs of problems or heterogeneity. A conclusion of heterogeneity is not made as an afterthought.

12.6.2 When the control limits are not met for any batch QC, the batch QC is rerun to verify that the out-of-control result is not a function of analytical variability.

13.0 DATA REVIEW AND STORAGE

13.1 All related preparation information and observations will be noted in the digestion logbook.

13.2 All cross-outs must be a single line and should be initialed and dated. Analyst review is documented on the GFAA and CVAA Data Review Checklist (Attachment 4).

13.3 All data from each run will be properly labeled and placed in the central file in an appropriate storage area. Each central file will include a run log, sample digestion sheet, and all original data.

14.0 REFERENCES

14.1 SW846 Method 7471A. Office of Solid and Hazardous Waste, rev. 1, 9/94.

14.2 FIMS, Software Guide, Publication B3118.30, Release 1.0/July 1994.

14.3 Flow Injection Mercury Analysis, Setting Up and Performing Analyses, Publication B3118.20, Release 1.1/March 1994.

14.4 Flow Injection Mercury/Hydride Analyses, Recommended Analytical Conditions and General Information, Publication B3505, Release 4.0.

14.5 FIMS, Installation Maintenance System Description, Publication B3118.10, Release 1.1/June 1994.

14.6 40 CFR Part 136 Appendix B, 1984

14.7 Department of Defense Quality Systems Manual for Environmental Laboratories, Revision 3, January 2006.

Mercury Standard Log

Attachment 1

Storage: Hg Hood

Standard ID	Manufacturer	Lot No.	Expiration Date	Stock Conc.	Analyst
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	
Hg Calib. Stock ID				µg/L	
Hg ICV Stock ID				10,000 µg/L	

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Mercury Standard Log
Working Standards and Solutions

Storage: Hg Hood

Matrix: Water

Prep. Date/ Exp. Date	Standard ID	Stock Conc.	Amount Used (ml)	Final Vol. (ml)	Final Conc.	Hg Working Std. ID	Hg ICV Std. ID	LCS Std. ID	HNO3 Lot No.	Prepared By
	Hg Calib Std. Working	1,000 µg/L (Hg Calib. Solution)	1.00	100	10 µg/L					
	Hg ICV Std. Working	10,000 µg/L (Hg ICV Solution)	1.00	100	600 µg/L					
	LCS									
	0.0 Std	NA	0.00	25	0 µg/L					
	0.2 Std	50 µg/L	0.10	25	0.2 µg/L					
	2.0 Std	10 µg/L	1.00	25	2.0 µg/L					
	4.0 Std	10 µg/L	1.00	25	4.0 µg/L					
	6.0 Std. (CCV)	10 µg/L	1.00	25	6.0 µg/L					
	8.0 Std	10 µg/L	1.00	25	8.0 µg/L					
	10.0 Std.	10 µg/L	1.00	25	10.0 µg/L					
	2.4 ICV	600 µg/L	0.1	25	2.4 µg/L					
	Hg Calibration Working	1,000 µg/L (Hg Calib. Solution)	1.00	100	10 µg/L					
	Hg ICV Working	10,000 µg/L (Hg ICV Solution)	1.00	100	600 µg/L					
	LCS									
	0.0 Std	NA	0.00	25	0 µg/L					
	0.2 Std	50µg/l	0.10	25	0.2 µg/L					
	2.0 Std	10µg/l	1.00	25	2.0µg/L					
	4.0 Std	10µg/l	1.00	25	4.0 µg/L					
	6.0 Std (CCV)	10µg/l	1.00	25	6.0 µg/L					
	8.0 Std	10µg/l	1.00	25	8.0 µg/L					
	10.0 Std	10µg/l	1.00	25	10µg/L					
	2.4 ICV	600 µg/l	0.1	25	2.4 µg/L					

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Attachment 2

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Mercury Digestion Logbook

Methods: EPA 245.1(IL-3028); SW846- 7470A (IL-3002); SW846- 7471A (IL-3005)

Weighed Date: _____
Digestion Date: _____
Digested By: _____
Curve Prep. Date: _____
Wk. Hg Cal. Sol. ID: _____
Wk. Hg ICV. Sol. ID: _____
WK. Hg Spike/LCS ID: _____
Bath Temp. In: _____
Time In: _____
Bath Temp. out: _____
Time out: _____

H2SO4 Lot No.: _____
SnCl2 Lot No.: _____
KMnO4 Lot No.: _____
K2SO4 Lot No.: _____
HNO3 Lot No.: _____
HCl Lot No.: _____
Hydroxylamine
HCl Lot No.: _____
LIMS Batch ID No.: _____

Water Bath Temp.= 93°C - 97°C

Lab. Sample ID	Sample Wt./ Vol. (g) or (ml)	Final Volume (ml)	Spike Amount (ml)	Spike Conc.	Comments

qc/qcmemos/forms/Hgdigestionlog Rev. 5 09/03/02

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Attachment 3

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GFAA and CVAA DATA REVIEW CHECKLIST

LAB ORDER: _____ CLIENT: _____

Method:

Analysis:

	A. GENERAL			
1.	Copy of Digestion Log?	Y	N	NA
2.	Copy of Instrument Run Log?	Y	N	NA
3.	All samples met digestion and/or analysis holding time criteria?	Y	N	NA
4.	Initial Calibration meets method criteria for GFAA, min. 3 std plus a blank? R >0.995	Y	N	NA
5.	Initial Calibration meets method criteria for CVAA, min. 5 std plus a blank? R >0.995	Y	N	NA
6.	Initial Calibration Verification (ICV) meets method criteria: ±10%?	Y	N	NA
7.	Is the RL1 check QC at ±20%?	Y	N	NA
8.	Initial Calibration Blank (ICB) free of contamination?(<½RL)	Y	N	NA
9.	Initial Performance Calibration(IPC) meets criteria: ±5% ?	Y	N	NA
10.	Continuing Calibration Verification(CCV) meets method criteria: ±10% ?	Y	N	NA
11.	Continuing Calibration Blank(CCB) free of contamination?(<½RL)	Y	N	NA
12.	Are CCV and CCB analyzed after every 10 samples ?	Y	N	NA
13.	Was post digestion performed? If yes was %R=85-115?	Y	N	NA
14.	Was the serial dilution performed? If yes was %R= 90-110?	Y	N	NA
15.	Method Blank- contaminant free(<½RL)?	Y	N	NA
16.	Are all reported results within calibration range?	Y	N	NA
17.	LCS % Recovery criteria met for all compounds?	Y	N	NA
18.	MS/MSD %R's and %RPD's are met for all compounds?	Y	N	NA
19.	%RPD criteria met between sample and duplicate?	Y	N	NA

COMMENTS

Analyst (s) _____ DATE: _____

REVIEWER: _____ DATE OF REVIEW: _____

qc/qcmemos/Forms/GFAACVAAchecklist Rev. 1 07/05/01

Attachment 4

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QUALIFICATION CARD FOR SW-846 METHOD 7471A

Trainee: _____ Signature: _____

Trainer: _____ Signature: _____

I. Knowledge Factors:

SOP Title: _____ Revision Number/Date: _____

1. Did the Trainee read and understand the above SOP?	Yes☐	No☐	NA☐
2. Was the Trainee instructed on how to properly fill out the documentation?	Yes☐	No☐	NA☐
3. Has the Trainee been instructed on the proper procedures for addressing potential problems?	Yes☐	No☐	NA☐

II. Practical Factors:

1. Has the Trainee completed a Precision and Accuracy Study?	Yes☐	No☐	NA☐
2. Has the Trainee completed an acceptable Performance Evaluation sample?*	Yes☐	No☐	NA☐
3. Did the Trainee completed an acceptable Laboratory Control Sample (LCS)?	Yes☐	No☐	NA☐
4. Has the Trainee performed manual integration as indicated in the SOP?	Yes☐	No☐	NA☐

Completion Date: _____ Trainer's Signature: _____

I _____ have read, understood and follow the above method/SOP, which is in use at AMRO Laboratories for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

Section Manager/ Supervisor: _____ Date: _____

QA Manager: _____ Date: _____

qc/qcmemos/forms/7471Aqualcard Rev. 0 09/25/01

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Analysis of Metals by Inductively Coupled Plasma Optical Emission Spectrometry according to SW-846 Method 6010B

Perkin-Elmer Optima 3300 Dual View
SN – 069N9061701 07/14/99

Procedure Number: IL-3021

Revision No.: 6

Revision Date: 4/17/2006

Review Date: 01/18/2010

Prepared By: Paula Benham Date: 1/18/10

Approved By: Robert E. Benham Date: 01/19/10
Inorganic Manager

Approved By: [signature] Date: 1/19/10
Laboratory Director

Approved By: Paula Benham Date: 1/19/10
Quality Assurance Manager

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1.0 SCOPE and APPLICATION

1.1 Application

1.1.1 This SOP describes the hardware and software operations of the Optima-DV ICP Spectrometer for the analysis of: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Lithium, Magnesium, Manganese, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Vanadium, Zinc, Boron, Tin, Titanium, Strontium, and Molybdenum in acidic, aqueous solutions. The instrument is capable of analyzing other elements as required following appropriate start-up procedures.

1.1.2 The Optima has the capability to determine elements simultaneously at different wavelengths. Whenever possible, this feature is employed to:

1.1.2.1 Confirm primary analytical result at secondary wavelength,
1.1.2.2 Extend Linear Range,
1.1.2.3 Permit reporting of data from secondary wavelength in instances where primary wavelength is out-of-control, useful for fast turn-around-samples.

1.1.3 This SOP is an equivalent to EPA Method 6010B with exceptions noted for TCLP analyses according to 40 CFR Appendix II.

1.1.4 This SOP is applicable to waters, wastewaters, soils, oils, and sludges prepared by acid digestions such as 3005, 3010,3050B, and 3051.

2.0 METHOD SUMMARY

2.1 An aliquot of the sample is mixed with an Internal Standard Solution (Yttrium/Scandium) then pumped to the nebulizer where it is met by a precisely controlled flow of argon creating a very fine aerosol. This aerosol is transported by the argon to the center of the plasma. Within this axial channel, the sample is atomized and the elements are excited to emission at temperatures in the range of 4000-80000K. The emitted energy is directed through the entrance slit, diffracted by the grating, refocused on the exit slits, and then projected on to two appropriate segmented-Array charge-coupled devices (SCD). The SCD's convert the light energy into electrical signals that are digitized and processed by the computer.

2.2 AMRO routinely digests MS, MSD and sample duplicate to meet all varying project QC requirements.

3.0 DEFINITIONS

3.1 **Analyte:** The element the analyst seeks to determine.

3.2 **Analytical Run:** A continuous analytical sequence consisting of digested samples plus all associated quality assurance measurements as described in the AMRO QA/QC Manual.

3.3 **Analytical Sample:** - Any solution introduced into the instrument on which an analysis is performed, excluding instrument calibration, calibration verification, and calibration blanks. The following are all analytical samples: digested samples, Duplicate Samples, Serial Dilutions, Post-Digestion Spikes, Matrix Spikes, Interference Check Standard A (ICSA), Interference Check Standard AB (ICSAB), Reporting Limit Standard (RL), Laboratory Control Sample (LCS & LCSD), and Method Blanks.

3.4 **Post-Digestion Spike (PDS):** The addition of a known amount of standard after digestion.

3.5 **Background Correction:** A technique to compensate for variable background contribution to the instrument signal.

3.6 **Batch:** A group of 20 or fewer samples digested (prepared) at the same time, in the same location, using the same method. Associated with each batch is a set of QC samples, including: Duplicate Samples, Matrix Spike (MS), Matrix Spike Duplicate (MSD), Serial Dilution (SD), Laboratory Control Sample (LCS & LCSD), and a Method Blank. Teflon boiling chips are used for solid/soil samples for Method Blanks, LCS and LCSD.

3.7 **Interelement Correction Factor (IEC):** Interelement interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. This intensity contribution will then cause a high and erroneous concentration to be reported for the analyte. IEC's must be applied to the analyte to remove the effects of these unwanted emissions.

3.8 **Interference Check Standard A (ICSA):** A sample with Aluminum, Calcium, and Magnesium at 500 mg/L, Iron at 320 mg/L.

3.9 **Interference Check Sample (ICSAB):** A sample with aluminum, Calcium, and Magnesium at 500 mg/L Iron at 320 mg/L and all other Analyte elements at 0.5-1.0 mg/L except Na & K at 2.5mg/L..

3.10 **Instrument Detection Limit (IDL):** A value determined by analyzing seven calibration blanks. The standard deviation is multiplied by 3 to obtain the IDL. The IDL limits must be ≤ MDL limits.

3.11 **Linear Range:** The highest concentration of an analyte that can be analyzed with a recovery of the True Value between 90-110 %.

3.12 **Method Detection Limit (MDL):** The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater then zero. It is determined by analyzing seven replicates of a low level standard that is 2-3 times the IDL. The standard deviation is then multiplied by 3.143 (the student t test value for seven replicates) to obtain the MDL. For soild/soils an MDL study is performed using teflon boiling chips.

3.13 **Reporting Limit Standard (RLP):** A low level standard used to verify linearity near the detection limit. The concentration is generally 3-5 times the MDL. See Table 1 for a list of metals RLs.

3.14 **Internal Standard (IS):** An internal standard solution containing Yttrium and Scandium at 10 mg/L is mixed with all calibration standards, all QC standards, and all samples. The intensity of Yttrium is used by the instrument as an internal standard to ratio the analytical intensity signals for both calibration and quantitation. This technique is useful in overcoming matrix interferences especially those caused by high dissolved solids. A second element, Scandium, is added to the IS solution in the rare event that Yttrium is present in the sample.

3.15 **Calibration:** Multi-element standards are used for calibration. For routine applications, a Blank and Four or Five Standards are used for the majority of the elements while Four Standards are employed for Aluminum, Calcium, Iron, and Magnesium.

4.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING AND STORAGE

4.1 All sample digestates (calibration, QC, and unknowns) must be in an aqueous matrix of Hydrochloric Acid (HCl) and Nitric Acid (HNO_3).

4.2 All standards and samples are corrosive. Care must be taken when handling, including the use of vinyl gloves, safety glasses and a lab coat. Hands must be washed before eating or smoking.

4.3 AMRO routinely stores all digestates for three months after digestion in case further analyses are required.

5.0 POTENTIAL PROBLEMS AND INTERFERENCES

5.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

5.1.1 Spectral interferences:

5.1.1.1 Overlap of a spectral line from another element-Interelement correction factors (IEC's) are required to compensate for any spectral interferences. These are mandatory for Aluminum, Calcium, Iron, and Magnesium. They should be determined and utilized for the remaining analytical channels. As an example of the impact of IEC's, if Arsenic were being determined in the presence of 500,000µg/L (500 mg/L) of Aluminum, an apparent (but false) Arsenic concentration of 4500 ug/L would be reported in an Arsenic-free sample if only background correction were being applied. With a properly selected IEC, this false signal would be reduced to a value less than the MDL. One additional point should be made. Note the effect of Cobalt on Lead. As with Arsenic, a false signal would be reported but this time the spurious signal is a negative value, -1,800µg/L. A negative concentration value indicates that the interfering line from Cobalt is on, or near, the background correction point selected for the lead wavelength. An IEC would be determined, however, instead of entering a positive value, a negative value would be entered into the ICP software. Once entered and verified, nebulization of the interferent (Cobalt) should produce an apparent concentration with an absolute value of less than the MDL for Lead.

5.1.1.2 Unresolved overlap of molecular band spectra - may require selection of an alternate wavelength.

5.1.1.3 Background contribution from continuous or recombination phenomena and Background contribution from stray light from the line emission of high concentration elements - 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 analyte wavelength measured. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array.

5.1.2 Physical Interferences:

5.1.2.1 Generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by employing the Internal Standard technique, dilution of the sample and/or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow rate causing instrumental drift. Wetting the argon prior to nebulization or sample dilution has been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

5.1.3 Chemical Interferences:

5.1.3.1 Characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique. However, if observed, they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

5.2 The instrument is programmed to analyze *two exposures* and report an average between the two results. The standard deviation and percent relative standard deviation (%RSD) along with the average result are printed for each metal. The %RSD must be <5%. This data is reviewed and the professional judgement of the analyst is used to determine if the precision between the replicates is acceptable. Typically, this type of review is not necessary with ICP unless there is a problem with the sample introduction system.

6.0 HEALTH AND SAFETY

6.1 AMRO places a high value on laboratory safety. The air quality of the laboratory has been monitored. Fire extinguishers are located in various areas of the lab. The laboratory evacuation plan is posted in every room, with a designated meeting place.

6.2 All new analysts receive training on the safe handling of samples, standards, the changing of gas cylinders, and the operation of instruments. In the lab, clean lab coats and gloves are worn when working with samples. Neat standards are made up in hoods. When gas tanks are replaced, the carrier with chain is used properly, and all gas tanks are chained in place. Soap is used to check for any leaks.

6.3 Care is taken with glassware and syringes to prevent injury. Broken glass and syringes are promptly removed to broken glass containers. First aid kits are available throughout the lab. There are also eye-wash stations and an emergency shower.

6.4 MSDS's are reviewed for each chemical used to ensure proper precautions are observed when handling chemicals.

6.5 Pollution prevention – The use of chemical exhaust hood are required when working with chemicals. They are specifically designed to minimize analyst exposure. The hood exhaust is monitored periodically to ensure environmental exposure is minimized.

7.0 INSTRUMENTS AND EQUIPMENT

7.1 Equipment:

7.1.1 Dual View ICP with the following specifications:

7.1.1.1 Optics – Two segmented-Array charged-coupled Device Detectors each cooled to 40° C.

7.1.1.2 Resolution - 0.006 in UV region.

7.1.1.3 Wavelength Range – 165-782 nm.

7.1.2 Source 1.5 KW R.F. generator operating at 40 Mhz with automatic power control and automatic tuning.

7.1.3 Cooling Water Supply – Neslab chiller CTF # 33 (Use Tap Water ONLY !!).

7.1.4 Mass Flow Controller for argon flow to the nebulizer.

7.1.5 Three Channel Peristaltic Pump.

7.1.6 AS-90 Autosampler with automated restandardization and automated analysis of quality control check samples.

7.1.7 Dell Computer: 233 MHz Pentium II running Microsoft Windows NT with a 17" monitor, a 2 GB hard drives, a CD ROM, and an 8 GB tape backup.

7.1.8 WinLab32 Software version 2.2

7.1.9 Torch, Perkin-Elmer Dual View, PE Cat. # N069-1662

7.1.10 Radial Purge Quartz Window, PE Cat # N069-1689

7.1.11 Axial Purge Quartz Window, PE Cat # B006-6549

7.1.12 Gem Cone, Low Flow Nebulizer, PE Cat # N069-0671

7.1.13 Cyclonic Spray Chamber, PE Cat. # N812-2188.

7.1.14 Alumina Injector Tip, 2.0 mm I.D., PE Cat # N069-5362

7.1.15 Quartz Torch Bonnet, PE Cat # N069-1664

7.1.16 RF Coil, PE Cat # N069-0772 (Requires PE Service to install).

7.1.17 Sipper for Auto Sampler, PE Cat. # B300-0159

7.1.18 Standard & QC Tubes, PE Cat. # B019-3234

7.1.19 17-mL Polypropylene Sample Tubes, 100mm round bottom, non-sterile, culture tubes, Elkay, Cat # 000-2018-001 (H&L)

7.1.20 Eppendorf Metal Free Clear Pipet Tips, (1000 µL)

7.1.21 Peristaltic Pump Tubing

7.1.21.1 Black/Black, VHG Cat # D180203

7.1.21.2 Green/Orange, VHG Cat # DCPT-007, Fisher Cat # 14-190-103

7.1.21.3 Red/Red, VHG Cat # D180201

7.1.22 Eppendorf Clear (Metal Free) Pipet Tips, 100 µL size

7.1.23 Nalgene Syringe Filters (0.45 u size),

7.1.24 10cc Syringe, Luer Lok,

7.1.25 Graduated, Conical Tubes with Caps, 15 mL polypropylene (used for IEC studies), Becton Dickinson, Cat. # Falcon 2097 Blue Max.

7.1.26 Serological Pipets, disposable and non-sterile, 10x1/10 mL size, Baxter, Cat. # P4650-110.

7.2 Materials:

7.2.1 Liquid Argon – 99.999 % at 70-120psig.

7.2.2 High Purity Nitrogen to purge the optical system at 30-120psig.

7.2.3 Compressed Air or Liquid Nitrogen for Shear Gas Supply at 75-100psig with pressure at the air dryer filter at 60psig.

7.2.4 Labware

7.2.4.1 For the determination of trace levels of elements, contamination and loss are of prime consideration. Potential contamination sources include improperly cleaned laboratory apparatus and general contamination within the laboratory environment from dust, rust, etc. A clean laboratory work area, designed for trace element sample handling must be used. Sample containers can introduce positive and negative errors in the determination of trace elements by (1) contributing contaminants through surface desorption or leaching, (2) depleting element concentrations through adsorption processes. All re-useable labware (glass, quartz, polyethylene, Teflon, etc.) including the sample container should be cleaned prior to use. Labware may be soaked overnight and thoroughly washed with laboratory-grade detergent and water, rinsed with water, and soaked in a mixture of dilute nitric and hydrochloric acid. (See AMRO dishwashing SOP.)

7.2.5 Glassware - 500mL and 1000mL volumetric flasks.

7.2.6 Storage - Teflon and Polyethylene bottles of 500 mL and 1000 mL size.

7.2.7 Disposable, metal free pipet tips.

7.2.8 Air Displacement Pipettes (Eppendorf) - 100µL 500µL, and 1000µL volumes.

8.0 REAGENTS AND STANDARDS

8.1 Nitric acid, VWR Omni Trace for Trace Metals Analysis or equivalent.

8.2 Hydrochloric acid, VWR Omni Trace for trace Metals Analysis or equivalent.

8.3 Water, Deionized Water, ASTM Type II (DI).

8.4 Standards

8.4.1 See ICP Standard Log OPTIMA for all standard preparation information. It is readily available near the ICP. This log contains copies of the Certificates of Analysis for each stock purchased for OPTIMA. It also gives an in depth explanation of all the standards required for OPTIMA ICP analysis. This log is used to document the preparation of all ICP calibration standards and instrument QC standards.

8.4.2 Calibration Standards (for concentrations, see Table 5)

8.4.2.1 The calibration standards are prepared as needed as described in the ICP Standard Log.

8.4.2.2 The superstock solutions are replaced annually.

8.4.3 Initial Calibration Verification Standard (ICV) (for concentrations, see Table 1)

8.4.3.1 The independent standards MUST be purchased from a different company than the calibration standards or, alternatively, the manufacturer must supply documentation that they trace back to different original stocks than the calibration standards do.

8.4.3.2 The ICV standard is prepared by adding 5mL of each ICV Superstock solution to a 500mL volumetric flask containing 5mL nitric acid and 25mL hydrochloric acid and diluting to the mark with DI water.

8.4.3.3 The ICV superstock solutions are replaced annually.

8.4.3.4 Analytical solutions are diluted from superstock as needed as described in the standards log.

8.4.4 Continuing Calibration Verification Standard (CCV) (for concentrations see Table 1).

8.4.4.1 The CCV is the same as ICP Standard S2. (See ICP Standard Log.)

8.4.4.2 The Custom Multielement Stock Standards are replaced annually.

8.4.4.3 Analytical solutions are diluted from the stock as needed.

8.4.5 Reporting Limit Standards for ICP (RL) (for concentrations see Tables 1-4).

8.4.5.1 See ICP Standard Log for prep instructions.

8.4.5.2 The stock solutions are replaced annually.

8.4.5.3 Analytical solutions are prepared as needed.

8.4.6 Interference Check Standard AB (ICSA) and Calibration Standard S4.

8.4.6.1 See ICP Standard Log for prep instructions.

8.4.6.2 The stock solutions are replaced annually.

8.4.6.3 Analytical solution is prepared as needed

8.4.7 Interference Check Standard AB (ICSAB).

8.4.7.1 See ICP Standard Log for prep instructions.

8.4.7.2 The stock solutions are replaced annually.

8.4.7.3 Analytical solution is prepared as needed.

8.4.8 Individual Element Solutions at 1000mg/L.

8.5 Chemical Receipt

8.5.1 The receipt of all standards, neat compounds and reagents from commercial sources is documented in the Chemical Receiving Logbook by sample receiving personnel or designate. Each solution or compound is assigned a unique standard receipt ID number, the letter "A" and a four digit sequential number. The date received, source, lot number, expiration date and disposal date are recorded. The expiration dates are provided from the manufacturer and the disposal dates are completed by AMRO employees when standards are used up or disposed.

8.6 Standards Preparation

Standards are prepared as written in the respective SOPs. In order to assure the accuracy of standards the following general guidelines are used:

8.6.1 ACS reagent grade chemicals are used when the method warrants.

8.6.2 Any stock standards that are purchased are accompanied by a certificate of analysis from the manufacturer.

8.6.3 Class A volumetric glassware is used to prepare standards.

8.7 Standard Tracking

It is important to be able to track all analytical standards back to their sources. The following procedure is used by AMRO personnel to accomplish this:

8.7.1 Metals Standards

8.7.1.1 Optima Standards Log (OPTIMA) - Preparation and Standard ID's are listed in the Standard Log. Standards include: Low S1 = RL1 (Reporting Limit), S1 (Calibration #1 & RL2), S2 (Calibration #2 & CCV), S3 (Calibration #3), S4 (Calibration #4 and ICSA), RL1 (Reporting Limit), RL2 (two times the Reporting Limit), ICSA &ICSAB (Interference Check Standards), ICV (Independent Source QC Standards), and Stock Solutions for Matrix/LCS spike additions. The Solution ID is written as the ID followed by the date prepared (example: S1042798 is standard S1 prepared on 042798). After a conformational analysis to verify the new standard and just prior to it's use, the new ID is entered into the OPTIMA software. Until updated, this will be the ID printed on every run log and standard page for hardcopy tracking.

8.7.2 Wet Chemistry and Metals Reagent Log - Working reagents are made from reagents listed in the Chemical Receiving Log are documented in the Reagent Log, The corresponding analysis, preparation date, reagent name, AMRO ID, initial concentration, amount used, final volume, final concentration, Solution ID, expiration date, storage location, and analyst initials are recorded. The initial concentration is obtained from the certificate received from the manufacturer. The Solution ID is written as an "R" followed by a 4 digit sequential number. The expiration of prepared reagents varies. Reagents should be used in accordance with method specifications and manufacturer's instructions.

9.0 PROCEDURE

9.1 Determination of IEC's

9.1.1 After the ICP has been calibrated, nebulize the single element solutions listed in Table 1 and note the "apparent concentration" values produced at each channel.

9.1.1.1 The elements with an "*" are analyzed every two to four weeks (as needed or after maintenance) and the IEC's are updated. These elements comprise the majority of the interfering elements routinely encountered in environmental samples.

9.1.1.2 IEC's for the remaining elements are determined annually or as needed. No corrections have been found to be necessary for: Antimony, Boron, Cadmium, Sodium, Potassium, Strontium, Selenium, Silver, and Thallium.

9.1.2 The correction factor, K1, is then calculated from the equation, K1 = (Apparent Analyte Concentration, µg/L)/(Interferent Concentration, mg/L).

9.1.3 The IEC is then entered into the ICP software.

9.1.4 To verify the IEC's, reprocess the data file and observe the "apparent concentrations."

9.1.4.1 A value of less than 2 x MDL for each analyte should be obtained if the IEC is correct.

9.1.4.2 This data can also be used to verify the linear range for the elements

9.1.5 To update an existing IEC, analyze pure solutions as previously described and calculate the correction factor from the equation:

9.1.5.1 K1 = [(Current IEC x Conc. of interfering element) + (Apparent Conc.)]/Conc. of interfering element.

9.2 Determination of Linear Ranges

9.2.1 After calibration, analyze solutions of high concentration for all elements for which the instrument is calibrated.

9.2.1.1 The same solution of pure single elements may be used to determine Linear Range and verify IEC's in the same analytical run (See IEC Section)

9.2.2 The Linear Range for an element is established if the recovery of the high standard is within 90-110%.

9.2.2.1 For any element, which does not meet this requirement, the linear range will default to the highest calibration standard

9.3 Determination of IDL's

9.3.1 Pour a calibration blank solution into seven autosampler tubes, and analyze as individual samples.

9.3.2 Determine the standard deviation for seven replicates for each analyte.

9.3.3 The IDL = 3 x (standard deviation).

9.3.4 Provide all required information on the current Instrument Detection Limit study worksheet provided by the QA Department.

9.3.5 An IDL study is required at start-up and after any significant modification to the instrument.

9.4 Determination of MDL's

9.4.1 Digest seven individual aliquots of an MDL Solution using an appropriate digestion procedure which is 3-5 times the IDL for 6010B or 20% of the AMRO reporting limit. For soils the MDL study is performed using teflon boiling chips.

9.4.2 Determine the standard deviation for the seven digestions for each analyte.

9.4.3 The MDL = 3.143 (the student T test value for seven replicates) x (average of the standard deviations).

9.4.4 Provide all required information on the current Method Detection Limit study worksheet provided by the QA department.

9.4.5 The MDL is performed annually.

9.4.6 To ensure that reasonable MDL values are determined, an MDL check sample is analyzed by spiking an interference free matrix with all target analytes at about two times the determined MDL. The MDL check sample is taken through the same process as the MDL. If any of the target analytes are not detected, the MDL study is modified and repeated until the MDL check sample is detectable. The MDL check will be analyzed quarterly and after major instrument maintenance or changes in the instrumentation or instrumental conditions to verify sensitivity of the method.

9.5 Precision and Accuracy

9.5.1 Precision and accuracy is done annually by each analyst for each matrix and analysis type. Digest and Analyze four Laboratory Control Samples with an acceptance limit of 80-120%.

9.6 Operational Parameters for the Optima Dual View

9.6.1 Plasma Information

9.6.1.1 Nebulizer argon flow – Approximately 0.55 LPM (Adjust for each nebulizer)

9.6.1.2 Plasma argon flow - 15 LPM

9.6.1.3 Auxiliary argon flow – 0.30 LPM

9.6.1.4 Forward RF power – 1450 watts

9.6.2 Peristaltic pump flow rate (sample) - 1.8mL/min.

9.6.3 Torch observation height - 18mm

9.6.4 Method Information (Optima_DV)

9.6.4.1 Number of elements – 27 (Variable – multiple wavelengths used)

9.6.4.2 Replicates – 2

The method states "multiple exposures" without giving a number. AMRO analyzes a comprehensive list of metals to meet all client needs while maintaining optimal use of the instruments. In order to accomplish this, three replicates of each sample are analyzed. For the Optima ICP three replicates would seriously degrade the analytical performance. There is a concern with buildup on the Nebulizer tip and other Sample Introduction problems from the length of sample period. Each replicate is read in the axial and radial mode which result in a "sample nebulization time" of nearly eight minutes. An %RSD of 2.5% between replicates is required to ensure the accuracy of the data. If the %RSD acceptance limit is exceeded, the sample is re-analyzed.

9.6.4.3 Read Delay – 45 seconds.

9.6.4.4 Flush time – 30 seconds.

9.6.4.5 Analysis data file name – MMDDYY A, B, C where: MM is the month, DD is the day and YY is the year of the analysis. A, B, C are analytical runs during the day.

9.6.4.6 Autosampler (SIF) table name – same as Analysis file.

9.6.4.7 Approximate analysis time – 4 to 7 minutes.

9.6.4.8 Integration Time – 5 to 25 seconds depending on elemental concentrations.

9.6.5 Rinse time between QC samples - 90 seconds

9.6.6 Additional 70 second rinse will be performed if previous sample contained high concentrations (> CCV) of prior selected analytes.

9.7 Start-Up ICP

9.7.1 Turn on Air Compressor or Liquid Nitrogen Tanks.

NOTE: Argon and chiller are always on!

9.7.2 Turn on the Nitrogen regulator to a pressure of 60 psig. Set the Argon pressure to 85 psig. Chiller temp should be <25 C.

9.7.3 Tighten the pump tubing, check flow of pump tubing and check the levels in the internal standard bottle, autosampler wash bottle, and the drain bottle.

9.7.4 Check DIAGNOSTIC WINDOW for any instrument "Warning Flags" and correct.

9.7.5 Open PLASMA CONTROL window.

9.7.6 Click the "PLASMA ON" button.

9.7.7 After ignition, wait about 60min. before starting to ensure temperature equilibrium. (This time can be used to prepare the autosampler table in the software and to pour the samples into the autosampler tubes).

9.8 Autosampler Table Setup (Sample Information Editor)

9.8.1 Open appropriate SIF format file (Water-200.7, Water-SW, Soil-3050B, Soil-3051, TCLP). These have the correct "Samp Types" and "Test Codes" necessary to transfer the data to LIMS.

9.8.2 Enter sample ID's and weight/ volume for soil samples and dilution factors as appropriate.

9.8.3 Save SIF as MMDDYY A, B, C. (Same as data files.)

9.8.3.1 Enter a CCV and CCB between every 10 samples at a minimum.

9.8.3.2 When all the samples have been entered, review the table and make sure the correct sample units (ug/L or mg/kg) have been selected and the appropriate initial, final, and continuing QC samples have been entered. Save the file.

9.8.3.3 Print the completed table and save in the Run Log File.

9.9 Profile - Profiling is used to establish optimum alignment of the spectrometer entrance slit image with respect to the exit slits. This is only necessary after cleaning sample introduction system and prior to Automatic Alignment of viewing position for Axial and Radial modes. To profile, select View Mode (Axial or Radial) then select Spectrometer Control.

9.9.1 Nebulize a 1.0 ppm manganese solution for Axial Viewing and a 10 ppm Mn for Radial Viewing and Click on "Optimize X & Y."

9.9.2 Optimize both Axial and Radial modes.

9.10 **Calibration** – Calibration is accomplished in the Automated Analysis Mode which has two options: (1) Calibrate and stop or (2) Run All (Calibrate and run Qc and samples. After calibration, the instrument will print a "Calibration Summary". This includes: Analyte, Number of Standards, Equation Used, Intercept, Slope, Curvature, and Correlation Coefficient. The Correlation Coefficient MUST be ≥0.995 to report the data. The "Mean Corrected Intensities" for Standard # 2 (CCV – mid-standard) should be constant (within ±30%) over time and they provide an excellent indicator of correct instrument performance in the following areas:

9.10.1 Optical Alignment,

9.10.2 Transfer optics,

9.10.3 Nebulizer,

9.10.4 Torch and spray chamber,

9.10.5 Torch alignment,

9.10.6 Argon flow rates to the nebulizer and the torch,

9.10.7 RF power,

9.10.8 Peristaltic pump rate, and

9.10.9 Calibration standard solutions.

9.11 Sample Analysis

9.11.1 Select Automated Analysis Control

9.11.1.1 Select method "Dual View," for example.

9.11.1.2 Enter the SIF and the Result Set (Data File).

9.11.1.3 For auto shutdown, select SET then check "Shutdown Enabled for: End of Run, Wash 30 minutes after analysis, Turn off Plasma & Pump.

9.11.1.4 Select Analyze.

9.11.1.5 Click on Analyze All.

9.12 Shut-down ICP

9.12.1 Open PLASMA CONTROL and click on "PLASMA OFF".

9.12.2 DO NOT turn off Argon or Chiller.

9.12.3 Turn off Nitrogen, Air Compressor (if used), and Vent..

9.12.4 Cap the Autosampler QC tubes.

9.12.5 Fill the Autosampler rinse bottles for the next day's run, (Deionized Water).

9.12.6 Empty autosampler drain bottle as needed.

9.12.7 Preventive Maintenance.

10.0 INSTRUMENT PREVENTAIVE MAINTENANCE AND REPAIR

Preventative Maintenance is designed to keep the instrument operating at a consistent performance level. A maintenance program must be followed with some "common sense" regarding the quality of samples that are introduced. For example:

10.1 General Guidelines

10.1.1 Samples containing particulate must be filtered before analysis.

10.1.2 The TDS (Total Dissolved Solids) in a sample should not exceed 3,000mg/L (this is about the level in TCLP samples).

10.1.3 The acid matrix of HCl and HNO3 should be similar between standards and samples.

10.1.4 Organic samples **must** not be analyzed unless the entire sample introduction system is cleaned afterwards and an analytical method is developed, tested, and verified.

10.2 DAILY Schedule (D):

10.2.1 Check autosampler Rinse and Waste solutions and Argon & Nitrogen pressures.

10.2.2 Verify that intensity counts for first standard are in a range that indicates efficient sample introduction. Verify the precision values between replicates for the high standard are less than 1% RSD.

10.2.3 Change pump tubing every other day.

10.2.4 Make any standards that are low and verify concentration prior to use.

10.3 BI-WEEKLY (W):

10.3.1 Clean torch and Injector Tip. Replace O-rings as needed. Acid wash torch and soak tip in dilute Aqua Regia.

10.3.2 Check coolant level and top off water. Watch for green growth in water lines.

10.3.3 Check Nebulizer (if sputtering clean in Ultrasonic Bath) and Spray Chamber and clean if necessary.

10.3.4 Do Alignment (Axial and Radial) then run IEC single element standards.

10.3.5 Tweak IEC's as necessary. Update IEC Table and Method.

10.4 MONTHLY (M):

10.4.1 Clean filters.

10.4.2 Wipe off Rail on Auto Sampler and oil lightly.

10.4.3 Dust off instrument.

10.4.4 Archive database Results Data to LIMS. Then Delete data from database, Check database and Pack database.

10.5 SEMIANNUAL (S):

10.5.1 Drain chiller and refill with tap water.

10.6 REPAIRS/SERVICE (RS)

10.6.1 Record problem and resolution.

11.0 CALCULATIONS

11.1 All calculations pertaining to the conversion of intensity units into concentration units are done by the PE software. After calibration, the instrument will print a "Calibration Summary". This includes: Analyte, Number of Standards, Equation Used, Intercept, Slope, Curvature, and Correlation Coefficient. The Correlation Coefficient MUST be ≥ 0.995 to report the data.

11.2 Data from the ICP are rounded to three significant figures by the PE software. (This can be changed easily in the method.)

11.3 MSA-corrected concentrations (mg/L) = the absolute value of (-b/m)

11.3.1 b = y-intercept (Constant) of the regression analysis performed in the MSA.WK3 worksheet.

11.3.2 m - slope (X Coefficient) of the regression analysis performed in the MSA.WK3 worksheet.

11.4 Interelement Correction factor (K1) = (Apparent Analyte Concentration, µg/L)/(Interferent Concentration, mg/L).

11.5 Hardness, mg equivalent CaCO3/L = ({Ca} in mg/L x 2.497) + ({Mg} in mg/L x 4.118

11.6 Instrument concentrations are in µg/L.

11.7 For water samples: The results are reported in µg/L. The sample concentration reported will be in µg/L units times the dilution factor.

11.8 For soil samples: Multiply the results in µg/L times the final volume of digestate in liters divided by the dry wt of sample in grams times the dilution factor to give results in mg/kg. In the sample info file if the sample weight has been entered in wet weight, and units entered in as mg/Kg the PE software will calculate the sample result in mg/Kg as wet weight. For sample results being reported in LIMS. The raw data being imported into LIMS is in calibration units of ug/L. The LIMS has the prep info entered separately such as weight and volume. The percent solids info is entered separately. The final sample result will be mg/Kg accounting also for dilution factor. To check this result for correctness take the sample result on the raw data in mg/Kg and divide by the percent solids and multiply times 100. This will account for the dry weight. Always multiply times the dilution factor.

11.9 % Recovery MS/MSD = Determined concentration – sample concentration/true value x 100

11.10 %RPD MS/MSD or DUP = (sample - dup)/((sample + dup)/2) x 100. Use concentrations of each in calculations.

12.0 QUALITY CONTROL

12.1 Periodic Demonstrations of Performance

12.1.1 Interelement Correction Factors: IEC's are updated verified on an annual basis or as required.

12.1.2 Linear Ranges: Linear Ranges are updated on an annual basis or as required

QC Sample	Frequency	ICP QC Table	ICP Action Level	ICP Action	Analyst Response
ICV	Once, after calibration	ICV	± 10% of True Value	Stop	Correct problem and recalibrate.
ICB	Once, after ICV	ICB	>±2 x MDL	Flag Data	Correct problem and reanalyze affected samples.
RLP	Start	RLP	±30% of True Value	Flag Data	Correct problem; then recalibrate/reanalyze samples.
ICSA	Start	ICSA	<2 x MDL	Stop	Correct problem; then recalibrate/reanalyze samples.
ICSAB	Start	ICSAB	±20% of True Value	Stop	Correct problem; then recalibrate/reanalyze samples.
CCV	10% of analytical samples	CCV	±10% of True Value	Flag Data	Correct problem and reanalyze affected samples
CCB	Following each CCV	CCB	>±2 x MDL	Flag Data	Correct problem and reanalyze affected samples.

12.2 Calibration Curves are verified using a ICV solution from an independent source. The Correlation Coefficient for each element reported must be ≥ 0.995.

12.3 The ICB and CCB's are a solution of 1% HNO3 and 5% HCl in DI.

12.4 The frequency of Batch QC samples is 5% for Method 6010B, Method Blank, Laboratory Control Sample (LCS), Matrix Spikes and Serial Duplication sample (SD).

Analyte	Matrix Spike (MS) %R Limit	Matrix Spike Duplicate (MSD) %R Limit	Post-Digestion Spike (PDS) %R Limit	Serial Dilution (SD) RPD Limit	Matrix Duplicate (DUP) RPD Limit	MS/MSD Duplicate (DUP) RPD Limit	LCS Laboratory Control Sample
All Elements	75-125 (DoD limit 80-120%)	75-125 (DoDlimit 80-120%)	75-125	10	20	20	80-120

12.4.1 Method Blanks:

A sample of deionized (DI) water is put through the procedure and analyzed as a Method Blank. Method Blank results above the Laboratory Reporting Limits indicate contamination. The source of contamination will be investigated and corrected. A Method Blank is required 1 for every 20 samples. For DOD projects the method blank must be less than one half the reporting limit.

12.4.2 Laboratory Control Sample:

A sample using DI water plus a spike of the appropriate analytes is put through the entire procedure and analyzed as a laboratory control sample (LCS). Control limits which are not met for percent recovery in the LCS indicate errors during the procedure and require reprep unless there is a definite assignable cause. The LCS control limits are 80%-120% for 6010B. A LCS is required 1 for every 20 samples.

12.4.3 Matrix Spike/ Matrix Spike Duplicate:

Duplicate aliquots of a commercial sample are spiked as a Matrix Spike and a Matrix Spike Duplicate and are put through the entire procedure. The concentration of the analyte in the unspiked sample aliquot should be no more than four times the amount added. If the sample result is greater than four times the amount added, the LCS is the only valid assessment of accuracy for the batch and the spike recovery is not evaluated for control. If the control limits are not met for Relative Percent Difference, the control limits for Percent Recovery in the Matrix Spike are not applicable, since sample heterogeneity is indicated and an inaccurate sample result used to calculate percent recovery is suspected. The control limits are 75% - 125%. For DOD projects the limit is 80-120%. A matrix spike and matrix spike duplicate is required for every 20 samples for 6010B. In some cases, the client may specify which sample is to used for MS/MSD; if not the laboratory shall pick a representative sample based on the following:

A. MS/MSD shall not be performed on Rinsate Blank or Field Blank.
B. Adequate sample weight/volume.
C. Client requested QC deliverables.

12.4.4 Duplicate:

12.4.4.1 Duplicate samples are two aliquots of a sample, which are carried through the same sample preparation and analysis procedure to verify the precision of the method for that sample matrix. The %RPD (Relative Percent Difference) is calculated from the original and the duplicate sample. The %RPD can also be calculated between a matrix spike and matrix spike duplicate. The %RPD is calculated using the concentration of the matrix spike and matrix spike duplicate. If the control limits are not met for Relative Percent Difference, the control limits for Percent Recovery in the Matrix Spike are not applicable, since sample heterogeneity is indicated and an inaccurate sample result used to calculate percent recovery is suspected. The control limits are 0%-20% for 6010B. The duplicate for 6010B often refers to the %RPD between the matrix spike and the matrix spike duplicate. In some cases, the client may specify which sample is to be used for MS/Dup; if not, the laboratory shall pick a representative sample based on the following:

A. Method of extraction or digestion.
B. Duplicate shall not be performed on Trip Blank, Rinsate Blank or Field Blank.
C. Adequate sample weight/volume.
D. Client requested QC deliverables

12.4.5 Serial Dilution sample:

An aliquot of the analytical solution of a representative sample is diluted five-fold and analyzed as the Serial Dilution Sample to determine the presence of interferences during analysis. The control limit is 0%-10% for 6010B. A serial dilution is required 1 every 20 samples for 6010B.

12.4.5.1 The ICP Serial Dilution Analysis must be performed on a sample from each group of samples of a similar matrix type (i.e., water, soil).

12.4.5.2 Samples identified as field, site, or rinse blanks cannot be used for Serial Dilution Analysis.

12.4.6 Post-Digestion Sample:

An aliquot of analytical solution from a representative solution is spiked and analyzed as the Post-Digestion Sample to determine the presence of interferences during analysis. A post digestion spike is performed whenever a new or unusual sample matrix is encountered. It is also used when a matrix spike fails to verify matrix effects.

12.4.6.1 An analyte spike added to a portion of a prepared sample, or its dilution, should be recovered to within 75-125% of the known value. The spike addition should produce a minimum level of 10x and a maximum of 100x the IDL. If the spike is not recovered within the specified limits, a matrix effect should be suspected.

12.4.6.2 If the sample results are greater than four times the amount added, the Serial Dilution is the only valid assessment of interferences.

12.5 TCLP requirements:

12.5.1 If the concentration of any TCLP extract is within ±20% of the regulatory limit, or if the recovery rate of the matrix spike is <50%, a four-point method-of-standard additions is required (40 CFR 261)

12.5.1.1 Four aliquots are required; See supervisor for instructions on preparation of MSA aliquots.

12.5.1.2 The observed concentrations of the four aliquots will be entered into the Excel MSA_CALC.XLS worksheet which will subject the data to linear regression of external-calibration-derived concentrations (y-axis). The absolute value of the x-intercept is the MSA-corrected concentration.

12.6 Corrective Actions for Batch QC

12.6.1 The initial investigation of the problem involves:

12.6.1.1 Checking sample concentrations for being high compared to the spike concentration.

12.6.1.2 Checking calculations and raw data transcriptions (both QC and sample).

12.6.1.3 Checking sample concentrations and analytical sequence for influences such as carryover and mislabeling.

12.6.1.4 Reviewing the notes in the prep worksheet and analytical notebook for signs of problems or heterogeneity.

12.6.2 When control limits are not met for any batch QC, the analytical replicates are checked to verify that the out-of-control result is not a function of analytical variability.

12.6.3 Method Blank Corrective Actions

12.6.3.1 Method Blank results greater than the laboratory reporting limits it will be investigated. If a sample is greater than the reporting limit and less than ten times the Method Blank results, the sample will be re-prepped and reanalyzed. For DoD projects all analytes must be <½ RL. Any sample associated with a blank not meeting the criteria shall be re-prepped and re-analyzed, except when the sample analysis resulted in a non-detect.

12.6.4 Laboratory Control Sample Corrective Actions

12.6.4.1 Control limits which are not met for recovery in the LCS requires the batch to be prepared and reanalyzed

12.6.5 Post-Digestion Spike Corrective Actions

12.6.5.1 If the control limits are not met in the Post-Digestion Spike sample, a chemical or physical interference must be suspected, and the data for all affected analytes in the sample with that Post-Digestion Spike must be flagged according to AMRO policy.

12.6.6 Serial Dilution Corrective Actions

12.6.6.1 If the control limits are not met in the Serial Dilution sample, a chemical or physical interference must be suspected, and the data for all affected analytes in the sample with that Serial Dilution must be flagged according to AMRO policy.

12.6.7 Matrix Spike Corrective Actions

12.6.7.1 Control limits which are not met for a majority of elements in the Matrix Spike, but are met in both the Post-Digestion Spike and the Serial Dilution may indicate analyst error during preparation or sample-specific interferences during preparation; re-preparation and reanalysis are required.

12.6.7.2 If the original results are confirmed after re-preparation, the results indicate sample-specific interferences during preparation. The original data are reported and flagged according to AMRO policy.

12.6.8 Relative Percent Difference Corrective Actions

12.6.8.1 Control limits which are not met for Relative Percent Difference in the Duplicate Sample indicates sample heterogeneity. Check sample observations for documentation of visible heterogeneity. If no sign of failure of the total analytical system, including sample preparation, is found, the result will be flagged according to AMRO policy. A sign of failure of the analytical system requires re-prepping of the entire batch.

12.6.9 DoD Projects the laboratory will follow all requirements as indicated in Attachment 1.

13.0 DATA REVIEW AND STORAGE

13.1 All related preparation information and observations will be noted in the digestion logbook.

13.2 All cross-outs must be a single line and should be initialed and dated. Analyst review is documented on the ICP Data Review Checklist (Attachment 2).

13.3 All data from each run will be properly labeled and placed in the central file in an appropriate storage area. Each central file will include a run log, sample digestion worksheet, and all original data.

13.4 The Optima has the capability to generate analytical results with multiple lines. This is an extremely valuable feature. For example, the primary Antimony line at 206.833 has a serious spectral interference from Chromium. With the Optima, Antimony can be simultaneously determined at the 217.582 line and the results compared. Multiple wavelengths are utilized as much as practical in all methods to ensure accurate results (it's rare that two wavelengths would have the same spectral interference, for example) and to provide the analyst and the data reviewers a "reality check."

13.5 Another use of multiple lines is to extend linearity. The most sensitive Manganese line is 257.610 and it has a Linear Range of 20,000 µg/L. The slightly weaker Manganese line at 293.0000 can extend the Linear Range to 50,000µg/L.

13.6 Data Transfer

13.6.1 At the completion of the run, go to File/Utilities/and select WinLab32 – Off-Line.
13.6.2 Go to File/Utilities and select Data Manager.
13.6.3 Select a files (or files) to transfer and click on COPY.
13.6.4 Click BROUSE to locate the Limsimport on 'AMRO2' and click to open. Double click results to open the database and click OK to transfer the data.
13.6.5 After transferring, close the Data Manager (WinLab32 can stay open and run in the background).

14.0 REFERENCES

14.1 Optima 3000 Family Hardware Guide Operator's Manual, Perkin-Elmer (0993-06251 Release B), August, 1997.

14.2 ICP WinLab Software Guide, Perkin-Elmer (0993-8966, Rev E), June, 1997.

14.3 Inductively Coupled Plasma-Atomic Emission Spectrometry,6010B, Test Methods for Evaluating Solid Waste, SW-846, Edition, Revision 2, US EPA, December 1996.

14.4 40 CFR Part 136. Appendix B.

14.5 Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 3, January 2006.

Table 1. Primary Wavelengths

SW-846 Methods 6010B/3010A

ELEMENT	Wavelength, nm	RL (µg/L)	Linear Range(µg/L)
Aluminum	308.215	200	500,000
Antimony	206.833	20	20,000
Arsenic	188.979	50	10,000
Barium	233.527	200	60,000
Beryllium	313.107	4	2,000
Boron	208.957	250	40,000
Cadmium	214.44	5	10,000
Calcium	227.546	2500	500,000
Chromium	267.716	10	20,000
Cobalt	228.616	50	20,000
Copper	324.452	25	20,000 (Carryover Likely)
Iron	273.955	100	320,000
Lead	220.353	12	100,000
Lithium	670.784 (Radial)	100	50,000
Magnesium	279.077	2500	500,000
Manganese	257.61	15	20,000
Molybdenum	202.03	25	20,000
Nickel	213.604	40	40,000
Potassium	766.474 (Radial)	2500	500,000
Selenium	196.026	80	10,000
Silver	328.068	7	2,000
Sodium	589.592 (Radial)	2500	500,000
Strontium	421.522 (Radial)	10	12,500
Thallium	190.5	50	10,000
Tin	189.933	250	20,000
Titanium	368.519	100	20,000
Vanadium	292.402	50	40,000
Zinc	206.2	20	40,000

Table 2. Secondary Wavelengths

SW-846 Methods 6010B/3010A

ELEMENT	(Secondary Wavelength, nm)	RL (µg/L)	Linear Range (µg/L)
Aluminum	237.335	200	100,000
Aluminum	394.401	200	100,000
Antimony	217.582	20	20,000
Arsenic	193.696	50	10,000
Barium	230.425	200	60,000
Beryllium	234.861	4	10,000
Boron	182.527	250	40,000
Boron	249.677	250	40,000
Cadmium	226.502	5	10,000
Cadmium	228.802	5	10,000
Calcium	315.887	2500	100,000
Calcium	317.933	2500	100,000
Chromium	205.56	10	20,000
Copper	224.7	25	20,000 (Carryover Likely)
Copper	327.393	25	20,000 (Carryover Likely)
Iron	238.204	100	10,000
Iron	238.616	100	320,000
Lead	217	12	100,000
Lead	283.306	12	100,000
Manganese	260.568	15	20,000
Manganese	293.305	15	50,000
Nickel	227.022	40	40,000
Nickel	341.476	40	40,000
Silver	338.289	7	2,000
Sodium	330.237 (Axial)	NA	500,000 (Reference Only)
Strontium	407.771 (Radial)	10	5,000
Titanium	334.903 (Radial)	100	20,000
Titanium	337.279	100	20,000
Vanadium	270.093	50	40,000
Vanadium	290.88	50	40,000
Zinc	202.548	20	20,000
Zinc	213.857	20	20,000

Table 3. Primary Wavelengths

SW-846 Methods 6010B/3050A

ELEMENT	Wavelength, nm	RL (mg/Kg)	Linear Range(µg/L)
Aluminum	308.215	20	500,000
Antimony	206.833	4	20,000
Arsenic	188.979	5	10,000
Barium	233.527	20	60,000
Beryllium	313.107	0.5	2,000
Boron	208.957	25	40,000
Cadmium	214.44	0.5	10,000
Calcium	227.546	250	500,000
Chromium	267.716	1	20,000
Cobalt	228.616	5	20,000
Copper	324.452	2.5	20,000 (Carryover Likely)
Iron	273.955	10	320,000
Lead	220.353	2.5	100,000
Lithium	670.784 (Radial)	10	50,000
Magnesium	279.077	250	500,000
Manganese	257.61	1.5	20,000
Molybdenum	202.03	2.5	20,000
Nickel	213.604	4	40,000
Potassium	766.474 (Radial)	250	500,000
Selenium	196.026	12	10,000
Silver	328.068	1.4	2,000
Sodium	589.592 (Radial)	250	500,000
Strontium	421.522 (Radial)	1	12,500
Thallium	190.5	5	10,000
Tin	189.933	25	20,000
Titanium	368.519	10	20,000
Vanadium	292.402	5	40,000
Zinc	206.2	4	40,000

Table 4. Secondary Wavelengths

SW-846 Methods 6010B/3051A

ELEMENT	Wavelength, nm	RL (mg/Kg)	Linear Range(µg/L)
Aluminum	308.215	20	500,000
Antimony	206.833	4	20,000
Arsenic	188.979	5	10,000
Barium	233.527	20	60,000
Beryllium	313.107	0.5	2,000
Boron	208.957	25	40,000
Cadmium	214.44	0.5	10,000
Calcium	227.546	250	500,000
Chromium	267.716	1	20,000
Cobalt	228.616	5	20,000
Copper	324.452	2.5	20,000 (Carryover Likely)
Iron	273.955	10	320,000
Lead	220.353	2.5	100,000
Lithium	670.784 (Radial)	10	50,000
Magnesium	279.077	250	500,000
Manganese	257.61	1.5	20,000
Molybdenum	202.03	2.5	20,000
Nickel	213.604	4	40,000
Potassium	766.474 (Radial)	250	500,000
Selenium	196.026	8	10,000
Silver	328.068	1.4	2,000
Sodium	589.592 (Radial)	250	500,000
Strontium	421.522 (Radial)	1	12,500
Thallium	190.5	5	10,000
Tin	189.933	25	20,000
Titanium	368.519	10	20,000
Vanadium	292.402	5	40,000
Zinc	206.2	2	40,000

Table 5. Standard and QC Concentrations

scp_STDS_2009

PERKIN-ELMER OPTIMA 3300DV ICP
OCTOBER 2009 to 2010

Element	AMRO S1 ug/L 1.0_>1L	AMRO S2 ug/L 5mL->1L	AMRO S3 ug/L 5mL->0.5L	AMRO S4 ug/L 100->1L 4->1L	AMRO ICV ug/L VHG Jul-09	AMRO RL4 ug/L 0.25->1L	AMRO RL1 ug/L 0.5->1L	AMRO RL2 ug/L 1.0->1L	AMRO CCV ug/L 5mL->1L	AMRO LCS/SPK ug/L 0.20mL->0.95L	AMRO ICSA ug/L 100->1L 4->1L	AMRO ICSAB ug/L 100->1L 10->1L 4->1L	(from CRI) AMRO PDS ug/L 5.0->0.05L 0.10->5.0mL	(from CRI) (+ Mix) AMRO IDL/MDL ug/L 0.5->50 MIX
Al	404.1	10,000	20,000	500,000	1,000	101.03	202.1	404.1	10,000	8,000	500,000	501,000	792	40.4
Sb	40.65	2,525	5,050		1,000	10.163	20.33	40.65	2,525	2,020		253	80	4.07
As	101.8	2,535	5,070		1,000	25.45	50.9	101.8	2,535	2,028		254	200	50.2
Ba	403.1	5,040	10,080		1,000	100.78	201.6	403.1	5,040	4,032		504	790	40.3
Be	9.99	995	1,990		1,000	2.4975	5.0	9.99	995	796		100	20	1.00
Cd	8.07	1,002	2,004		1,000	2.0175	4.0	8.07	1,002	802		100	16	0.81
Ca	5011	25,045	50,090	500,000	21,006	1253	2506	5011	25,045	20,036	500,000	502,505	9,825	501
Cr	20.13	4,985	9,970		1,000	5.0	10.07	20.13	4,985	3,988		499	39	2.01
Co	100.5	5,030	10,060		1,000	25.125	50.25	100.5	5,030	4,024		503	197	10.1
Cu	50.1	2,515	5,030		1,000	12.525	25.05	50.1	2,515	2,012		252	98	5.01
Fe	200.1	5,015	10,030	300,864	1,000	50.025	100.1	200.1	5,015	4,012	299,860	300,111	392	20.0
Pb	50.2	2,505	5,010		1,000	12.55	25.1	50.2	2,505	2,004		251	98	5.02
Mg	4999	25,005	50,010	500,000	20,998	1249.8	2500	4999	25,005	20,004	500,000	502,501	9,802	500
Mn	30.09	5,030	10,060		1,000	7.5225	15.05	30.09	5,030	4,024		503	59	3.01
Mo	50.4	2,505	5,010		1,000	12.6	25.2	50.4	5,010	4,008		501	99	5.04
Ni	80.3	5,045	10,090		1,000	20.075	40.15	80.3	5,045	4,036		505	157	8.03
K	5000	25,090	50,180		30,000	1250	2500	5000	25,090	20,072		2,509	9,804	500
Se	120.6	2,007	4,013		1,000	30.15	60.3	120.6	2,007	1,605		201	236	52.1
Ag	14.11	502	1,004		500	3.5275	7.1	14.11	502	402		50	28	1.41
Na	5030	25,025	50,050		21,000	1257.5	2515	5030	25,025	20,020		2,503	9,863	503
Sr	20.19	2,525	5,050		1,000	5.0	10.1	20.19	2,525	2,020		253	40	2.02
Tl	100.3	2,010	4,019		1,000	25.075	50.15	100.3	2,010	1,608		201	197	50.0
Ti	198.7	4,975	9,950		1,000	49.675	99.35	198.7	4,975	3,980		498	390	19.9
V	99.9	4,950	9,900		1,000	24.975	49.95	99.9	4,950	3,960		495	196	10.0
Zn	40.28	5,025	10,050		1,000	10.07	20.14	40.28	5,025	4,020		503	79	4.03
Li	201.7	2,525	5,050		1,000	50.4	100.9	201.7	2,525	2,020		253	395	20.2
B	499	4,970	9,940		1,000	124.75	249.5	499	4,970	3,976		497	978	49.9
Sn	499.3	2,497	4,993		1,000	124.83	249.7	499.3	2,497	1,997		250	979	69.9

Page 1 of 1

Attachment 1

TABLE D-19. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7471 SOLID MATRIX[28]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	95	5	80	120	75	120
Antimony	96	5	80	120	75	120
Arsenic	95	4	80	120	80	120
Barium	98	3	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	97	4	80	120	80	120
Calcium	97	4	80	120	80	120
Chromium	99	5	80	120	80	120
Cobalt	98	4	80	120	80	120
Copper	97	3	80	120	80	120
Iron	100	4	80	120	80	120
Lead	95	4	80	120	80	120
Magnesium	96	3	80	120	80	120
Manganese	97	4	80	120	80	120
Mercury	100	6	80	120	No ME	No ME
Molybdenum	96	5	80	120	75	120
Nickel	97	4	80	120	80	120
Potassium	96	4	80	120	80	120
Selenium	93	4	80	120	75	120
Silver	96	7	75	120	70	125
Sodium	96	4	80	120	80	120
Thallium	94	4	80	120	80	120
Vanadium	99	3	80	120	80	120
Zinc	95	5	80	120	75	120

TABLE D-18. LCS CONTROL LIMITS FOR METALS SW-846 METHODS 6010 AND 7470 WATER MATRIX[28]

Analyte	Mean	Standard Deviation	Lower Control Limit	Upper Control Limit	Lower ME Limit	Upper ME Limit
Aluminum	97	5	80	120	80	120
Antimony	98	4	80	120	80	120
Arsenic	98	4	80	120	80	120
Barium	99	4	80	120	80	120
Beryllium	99	4	80	120	80	120
Cadmium	100	4	80	120	80	120
Calcium	98	4	80	120	80	120
Chromium	100	4	80	120	80	120
Cobalt	99	3	80	120	80	120
Copper	99	3	80	120	80	120
Iron	102	4	80	120	80	120
Lead	99	4	80	120	80	120
Magnesium	98	4	80	120	80	120
Manganese	100	4	80	120	80	120
Mercury	100	5	80	120	No ME	No ME
Molybdenum	95	5	80	120	75	120
Nickel	100	4	80	120	80	120
Potassium	98	4	80	120	80	120
Selenium	98	6	80	120	75	120
Silver	97	5	80	120	75	120
Sodium	99	4	80	120	80	120
Thallium	97	4	80	120	80	120
Vanadium	99	4	80	120	80	120
Zinc	100	4	80	120	80	120

Attachment 2

AMRO Environmental
Laboratories Corporation

111 Herrick Street
Merrimack, NH 03054
(603) 424-2022

ICP DATA REVIEW CHECKLIST

LAB ORDER: _____ CLIENT: _____ /

Method:

Analysis:

A.	GENERAL			
1.	Copy of Digestion Log?	Y	N	NA
2.	Copy of ICP Checklist?	Y	N	NA
3.	Copy of LIMS Checklist?	Y	N	NA
4.	All samples met digestion and/or analysis holding time criteria?	Y	N	NA
5.	Initial Calibration Verification (ICV) meets method 6010 criteria: ±10%?	Y	N	NA
6.	Initial Calibration Verification (ICV) meets method 200.7 criteria: ±5%?	Y	N	NA
7.	Is the RL1 check QC at ±20%?	Y	N	NA
8.	Initial Calibration Blank (ICB) free of contamination?(<½RL)	Y	N	NA
9.	Continuing Calibration Verification(CCV) meets method 6010 criteria: ±10% ?	Y	N	NA
10.	Initial Performance Calibration(IPC) meets method 200.7 criteria: ±5% ?	Y	N	NA
11.	Continuing Calibration Blank(CCB) free of contamination?(<½RL)	Y	N	NA
12.	Are CCV and CCB analyzed after every 10 samples ?	Y	N	NA
13.	Is the Interference Check Solution %R between 80-120%?	Y	N	NA
14.	Was the serial dilution performed? If yes was %R= 90-110?	Y	N	NA
15.	Was post digestion performed? If yes was %R=75-125?	Y	N	NA
16.	Was the MSA performed?	Y	N	NA
17.	Method Blank- contaminant free(<½RL)?	Y	N	NA
18.	Are all reported results within linear range?	Y	N	NA
19.	LCS % Recovery criteria met for all compounds?	Y	N	NA
20.	MS/MSD %R's and %RPD's are met for all compounds?	Y	N	NA
21.	%RPD criteria met between sample and duplicate?	Y	N	NA
22.	Are all data exported to LIMS, calculated and verified?	Y	N	NA

COMMENTS

Analyst (s) _____ DATE: _____

REVIEWER: _____ DATE OF REVIEW: _____

Attachment 3

AMRO Environmental
Laboratories Corporation

111 Herrick Street
Merrimack, NH 03054
(603) 424-2022

QUALIFICATION CARD FOR SW-846 METHOD 6010B

Trainee: _____ Signature: _____

Trainer: _____ Signature: _____

I. Knowledge Factors:

SOP Title: Analysis of Metals by Inductively Coupled Plasma Revision Number/Date: _____

1. Did the Trainee read and understand the above SOP?	Yes ☐	No ☐	NA ☐
2. Was the Trainee instructed on how to properly fill out the documentation?	Yes ☐	No ☐	NA ☐
3. Has the Trainee been instructed on the proper procedures for addressing potential problems?	Yes ☐	No ☐	NA ☐

II. Practical Factors:

1. Has the Trainee completed a Precision and Accuracy Study?	Yes ☐	No ☐	NA ☐
2. Has the Trainee completed the Bi-Weekly maintenance schedule followed by a successful IEC update?	Yes ☐	No ☐	NA ☐
3. Has the Trainee demonstrated the ability to review and document Instrument QC and Batch QC problems?	Yes ☐	No ☐	NA ☐

Completion Date: _____ Trainer's Signature: _____

I _____ have read, understood and follow the above method/SOP, which is in use at AMRO Laboratories for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

Section Manager/ Supervisor: _____ Date: _____

QA Manager: _____ Date: _____

APPENDIX D: DETAILED COST ESTIMATE TABLE

Urban Reserve
102417-R
DETAILED FEE JUSTIFICATION & SCOPE DETAILS

Staff Type	Name	Rate Per Unit	Unit	Amount	Subtotal		Scope Details
Task 1 - Preparation of SSQAPP							Prepare a SSQAPP for approval by VT DEC and USEPA.
Professional Services							
Senior Professional 1		$115.00 / hour	3	$345			
Project Professional 1		$90.00 / hour	10	$900			
Staff Professional 2		$75.00 / hour	24	$1,800			
TASK SUBTOTAL						**$3,045**	
Task 2 - Surface Soil Assessment (Area A - Bike Path)							26 surface soil samples + 2 duplicates for analysis of PAHs, TPH (DRO), PCBs, Herbicides, and RCRA 8 metals. Preparation of letter report documenting field procedures and results, with applicable tables and figures.
Professional Services - Field Work							
Staff Professional 2		$75.00 / hour	16	$1,200			
Technician 2		$65.00 / hour	16	$1,040			
Professional Services Summary			*32*		*$2,240*		
Stone Field Services							
Geoprobe 6610DT		$1,500 / day	1.5	$2,250			
Drilling Consumables		$200 / ls	1	$200			
Mobe / Demobe		$300.00 / ls	1	$300			
Summary					*$2,750*		
Subcontractor - AMRO							
8270 SIM (for PAHs)		$161.00 / sample	28	$4,959			
PCBs manual soxhlet extraction		$70.00 / sample	28	$2,156			
Herbicides		$200.00 / sample	28	$6,160			
TPH (8015-DRO)		$56.00 / sample	28	$1,725			
RCRA 8 Metals		$91.00 / sample	28	$2,803			
Subcontractor Summary			*140*		*$17,802*		
Expenses							
Asphalt Patch		$15 / bag	2	$33			
Expense Summary					*$33*		
Professional Services - Reporting							
Senior Professional 1		$115.00 / hour	2	$230			
Project Professional 1		$90.00 hour	6	$540			
Staff Professional 1		$75.00 / hour	10	$750			
Labor Summary					*$1,520*		
TASK SUBTOTAL						**$24,345**	
PROJECT TOTAL						***$27,390***	

Stone Environmental's standard mark-up on all Subcontractor expenses is 10%.

APPENDIX E: HEALTH AND SAFETY PLAN

STONE ENVIRONMENTAL INC

SITE SPECIFIC HEALTH AND SAFETY PLAN

SECTION 1: GENERAL INFORMATION AND DISCLAIMER		PROJECT NUMBER:	102417-R/01
PROJECT NAME:	Urban Reserve Phase II ESA	CLIENT NAME:	Chittenden County Regional Planning Commission
PROJECT MANAGER:	Dan Voisin	SITE SAFETY OFFICER	Lee Rosberg
PREPARED BY:	Lee Rosberg	DATE:	12/17/12

NOTE: This site specific Health and Safety Plan - Short Form (HASP-SF) has been prepared for use by **Stone Environmental, Inc. (Stone)** employees for work at this site / facility. **The HASP-SF has been written for the specific site / facility conditions, purposes, tasks, dates and personnel specified, and must be amended and reviewed by those personnel named in Section 4 if these conditions change.** Stone Environmental, Inc., is not responsible for its use by others.

Subcontractors shall be solely responsible for the health and safety of their employees and shall comply with all applicable laws and regulations. In accordance with 1910.120(b)(1)(iv) and (v), Stone Environmental, Inc. will inform subcontractors of the site / facility emergency response procedures, and any potential fire, explosion, health, safety or other hazards by making this Site Specific Health and Safety Plan and site information obtained by others available during regular business hours. All contractors and subcontractors are responsible for: (1) developing their own Health and Safety Plan, including a written Hazard Communication Program and any other written hazard specific or safety programs required by federal, state and local laws and regulations, that details subcontractor tasks, potential or actual hazards identified as a result of a risk analysis of those tasks, and the engineering controls, work practices and personal protective equipment to be utilized to minimize or eliminate employee exposure to the hazard; (2) providing their own personal protective equipment; (3) providing documentation that their employees have been health and safety trained in accordance with applicable federal, state and local laws and regulations; (4) providing evidence of medical surveillance and medical approvals for their employees; and (5) designating their own site safety officer responsible for ensuring that their employees comply with their own Health and Safety plan and taking any other additional measures required by their site activities.

Providing a copy of this Stone Environmental, Inc. HASP-SF to subcontractors, does not establish, nor is it intended to establish a "joint employer" relationship between the Contractor and Stone Environmental, Inc. This allowance does not establish, nor is it intended to establish, a direct or indirect employer/employee relationship with subcontractor's employees.

SECTION 2: EMERGENCY INFORMATION

(A) LOCAL RESOURCES	SERVICE NUMBER	TELEPHONE NUMBER
EMERGENCY MEDICAL SERVICES	Burlington Fire Department Emergency Medical Services Division	911/802-864-4554
HOSPITAL (Map attached)	Fletcher Allen Health Care	911/802-847-0000
FIRE DEPARTMENT	Burlington Fire Department	911/802-658-7660
POLICE / SECURITY	Burlington Police Department	911/802-658-2704
HAZMAT/ SPILL / OTHER RESPONSE	VT State HAZMAT Response Team	911/1-800-641-5005

(B) CORPORATE RESOURCES	STONE ENVIRONMENTAL, INC.	INCIDENT TELEPHONE NUMBERS	
MAIN LINE NUMBER (802) 229-4541		OFFICE	CELL PHONE
CORPORATE HEALTH & SAFETY	Kim Watson	(802) 229-2196	(802) 249-7753
PROJECT OFFICER	Seth Pitkin	(802) 229-2192	(802) 249-8700
PROGRAM MANAGER	Seth Pitkin	(802) 229-2192	(802) 249-8700
PROJECT MANAGER	Dan Voisin	(802) 229-1875	(802) 279-8174
SITE SAFETY OFFICER	Lee Rosberg	(802) 229-5378	(802) 309-1629

SECTION 3: PROJECT INFORMATION

(A) SITE / FACILITY INFORMATION:

SITE NAME:	**Urban Reserve**	SITE CLIENT CONTACT:	**Dan Albrecht**
ADDRESS		PHONE NUMBER:	**802-846-4490 x29**
TOWNSHIP / COUNTY:	**Burlington**	SITE SAFETY CONTACT:	
STATE	**Vermont**		

CLIENT TYPE ☐ FEDERAL ☐ STATE ☒ MUNICIPAL / REGIONAL ☐ PRIVATE

(B) SITE / FACILITY TYPE:

☐ HAZARDOUS (RCRA)	☒ UST / LUST	☐ WTP / WWTP
☒ HAZARDOUS (CERCLA / STATE)	☒ BROWNFIELD	☐ FIFRA
☒ CONSTRUCTION	☐ CHEMICAL PLANT	☐ OTHER:
☐ LANDFILL (NON-HAZARDOUS)	☐ REFINERY	
☐ ACTIVE	☒ INACTIVE	

(C) INVESTIGATION TYPE:

☒ HAZARDOUS WASTE	☐ SOLID WASTE	☐ WASTE WATER
☐ HYDROGEOLOGY	☒ ENVIRONMENTAL	☐ WASTE STREAM
☐ WASTE WATER	☐ AUDIT	☐ AIR/ODOR
☐ PRE-JOB VISIT	☒ CONSTRUCTION	☐ SEDIMENT
☐ (SUB) CONTRACTOR OVERSIGHT	☐ LANDFILL	☒ SURFACE SOIL
☐ CONSTRUCTION MGMT	☐ AIR	☐ OTHER
☐ INSPECTION	☐ SURFACE WATER	
☐ INVESTIGATION SURVEY	☐ GROUND WATER	

DATE(S) OF FIELD ACTIVITIES: **TBD**

(D) FIELD TASKS

STONE ENVIRONMENTAL TASKS (List field tasks to be performed by Stone Environmental, Inc. staff)

S1 **Mobilization/Demobilization**

S2 **Surface Soil Assessment**

S3

S4

S5

TASKS PERFORMED BY OTHERS (List field tasks to be performed by client, subcontractors, or contractors)

O1

O2

SECTION 4: PROJECT SAFETY ORGANIZATION, HEALTH AND SAFETY TRAINING, AND MEDICAL MONITORING

(A) PROJECT HEALTH AND SAFETY ROLES, RESPONSIBILITIES AND COORDINATION

PROJECT OFFICER: **Seth Pitkin**	The Project Officer (PO) is ultimately responsible for project performance. The PO seeks and gets appropriate approvals for risk management decisions (e.g. from Regional/Practice Director(s), Legal Council, Corporate Health and Safety), and selects an effective and qualified project team. The PO supports the Project Manager or Deputy Project Manager with appropriate resources.
PROGRAM MANAGER: **Seth Pitkin**	The Program Manager is the chief liaison with the client and will coordinate Stone Environmental, Inc Project Managers and Resources to respond to the client's needs.
PROJECT MANAGER: **Dan Voisin**	The Project Manager (PM) has the responsibility for executing the project in accordance with the scope of work and good engineering practice. The PM will supervise the allocation of resources and staff to implement specific aspects of this HASP-SF and may delegate authority to expedite and facilitate any application of the program. The PM implements and executes an effective program of site-specific personnel protection and accident prevention. The Project Manager reports to the Program Manager.
CORPORATE HEALTH & SAFETY: **Kim Watson**	Corporate Health and Safety is responsible for Stone Environmental, Inc.'s overall Health and Safety Program and provides project guidance on air monitoring methodology, data interpretation and assistance in determining appropriate project engineering controls, work practices, and personal protective equipment. Corporate Health and Safety also reviews and approve HASPs in accordance with Section 1.
SITE SAFETY OFFICER: **Lee Rosberg** ALTERNATE SITE SAFETY OFFICER (S): **David Linari**	The Site Safety Officer (SSO) is responsible for interpreting and implementing the site health and safety provisions set out in this HASP-SF, and will guide the efforts of field team personnel in their day-to-day compliance with this HASP-SF. The SSO has the ability and authority to make necessary changes or additions to this HASP-SF and provide technical assistance to field team personnel on problems relating to worksite safety. The SSO has the authority to correct safety-related deficiencies in materials or practice and to call a Project STOP in the most serious cases. Alternate Site Safety Officer (ASSO) is assigned all duties and responsibilities of the Site Safety Officer in his/her absence.
FIELD TEAM PERSONNEL: **Lee Rosberg, David Linari**	Field personnel have the following health and safety responsibilities: • Implement the procedures set forth in the HASP-SF; • Take all reasonable precautions to prevent injury to themselves and their fellow employees; and • Perform only those tasks that they believe they can do safely, and immediately report any accidents and/or unsafe conditions in accordance with Section 1.

PROJECT TEAM - The above Stone Environmental, Inc. personnel are designated to carry out the stated project job functions on site. THE SITE SAFETY OFFICER OR A DESIGNATED ALTERNATE WILL BE ON-SITE DURING **ALL** SITE ACTIVITIES. (NOTE: One person may carry out more than one job function.)

The following subcontractor(s) and governmental agencies have been informed by Stone Environmental, Inc. of emergency response procedures, and any potential fire, explosion, health, safety or other hazards of the site / facility by making this Site Specific Health and Safety Plan and site information obtained by others available during regular business hours. Subcontractors and governmental agencies shall be solely responsible for the health and safety of their employees and shall comply with all applicable laws and regulations as described in **Section 1** of this plan.

SUBCONTRACTOR: _____ FEDERAL AND STATE AGENCY REPS: _____

OTHER AGENCY REPS: _____

(A) HEALTH AND SAFETY TRAINING, MEDICAL MONITORING, AND FIT TESTING PROGRAM

The following project staff members are included in the Stone Environmental, Inc. Health and Safety Training and Medical Monitoring programs. The details of these programs can be found in the Health and Safety Policies and Written Programs.

	HAZWOPER TRAINING		MEDICAL MONITORING	
NAME	INITIAL 40HR (DATE)	8HR (DATE)	MEDICAL (DATE)	FIT TEST (DATE)
Lee Rosberg	**3/1/2007**	**1/12/12**	**10/16/12**	**NA**
David Linari	**4/4/2003**	**12/10/12**	**10/8/12**	**NA**

SECTION 5: HAZARD ANALYSIS

(A) ACTUAL OR POTENTIAL PHYSICAL HAZARDS – (Check all that apply to Stone Environmental, Inc. activities)

☒ ANIMALS / PLANTS	☐ ELECTRICAL	☐ HUNTING SEASON	☐ POWERED PLATFORMS
☐ ASBESTOS / LEAD	☐ EXCAVATIONS (See Section 13)	☐ IMMERSION	☐ POOR VISIBILITY
☐ CHEMICAL EXPOSURE (See Section 5B/5C)	☒ EXTREME COLD (See Section 10)	☐ IONIZING RADIATION	☐ ROLLING OBJECTS
☐ CONFINED SPACE (See section 5b/5c)	☐ FALL >6' VERTICAL	☐ LIGHT RADIATION (i.e., Welding, High Intensity)	☐ SCAFFOLDING
☐ DEMOLITION	☐ FALLING OBJECTS	☐ LIMITED CONTACT	☐ SHARP OBJECTS
☒ DRILLING	☐ HEAT STRESS	☐ MOVING PARTS (LO / TO)	☐ TRAFFIC (STRUCK BY)
☐ DRUM HANDLING	☒ HEAVY EQUIPMT	☒ NOISE (> 85 dB)	☐ STEEP / UNEVEN TERRAIN
☒ DUST, HARMFUL	☒ HEAVY LIFTING	☐ NON-IONIZING RADIATION	☐ OTHER:
☒ DUST, NUISANCE	☐ HOT WORK	☐ OVERHEAD OBJECTS	

(B) **PRESENCE OF HAZARDOUS MATERIALS STORED OR USED ON SITE**

TYPE	CURRENTLY ☐ YES	FORMERLY ☒ YES	☐ NO
☐ EXPLOSIVES	☒ FLAMMABLE/ COMBUSTIBLE LIQUIDS	☐ OXDIZERS	☐ CORROSIVE
☐ COMPRESSED GASES	☐ FLAMMABLE/ REACTIVE SOLIDS	☐ TOXIC/INFECTIOUS	☐ MISCELLANEOUS
		☐ RADIOACTIVE	☒ HAZARDOUS WASTE (STORED)

(C) **CHEMICAL HAZARDS OF CONTAMINANTS INFORMATION**

(1) IDENTIFIED CONTAMINANTS - Known or suspected hazardous/toxic materials (attach historical information, physical description, map of contamination and tabulated data, if available)

Characteristics: CA (corrosive, acid), CC (corrosive, caustic), IG (ignitable), RA (radioactive), VO (volatile), TO (toxic), RE (reactive), BIO (infectious), UN (unknown), OT (other, describe)

Media types: GW (ground water), SW (surface water), WW (wastewater), AIR (air), SG (soil, gas), SL (soil), SD (sediment), WL (waste, liquid), WS (waste, solid), WD (waste, sludge), WG (waste, gas), OT (other, describe).

SUBSTANCES INVOLVED	CHARACTERISTICS	MEDIA	ESTIMATED CONCENTRATIONS	LOWEST PEL, or TLV	
					☐ PPM
Herbicides	**TO**	**SL**	**Unknown**		☐ mg/m^3
					☐ PPM
Heavy Metals	**TO**	**SL**	**7.4 mg/Kg**	**0.050**	☒ mg/m^3
					☐ PPM
PCBs	**OT (Carcinogen)**	**SL**	**Unknown**	**0.001**	☒ mg/m^3
					☐ PPM
TPH-DRO	**IG, VO, TO**	**SL**	**>200 ppm**	**100**	☒ mg/m^3
					☐ PPM
PAHs	**TO**	**SL**	**>200 ppm**	**10**	☒ mg/m^3

(2) DESCRIBE POTENTIAL FOR CONTACT WITH EACH MEDIA TYPE FOR EACH OF THE MPI TASKS LISTED IN SEC 3 (E):

MPI TASK	ROUTE OF EXPOSURE (INHAL/INGEST/CONTACT/ABSORB)	POTENTIAL FOR CONTACT (HIGH / MEDIUM / LOW)	METHOD OF CONTROL
S1	**NA**	**NA**	**NA**
S2	**Inhalation, ingestion, contact**	**High**	**PPE**
S3			
S4			
S5			

The Site Safety Officer will brief the field team on symptoms and signs of overexposure to chemical hazards

SECTION 6: SITE CONTROL MEASURES

(A) **WORK ZONES – EXCAVATIONS, DRILLING OPERATIONS AND HEAVY EQUIPMENT**

Lee Rosberg has been designated to coordinate access control and security for operations on site.

(B) **WORK ZONES – CONTAMINATION**

The prevailing wind conditions are to the East. A wind direction indicator is used to determine daily wind direction. The Command Post is located from the Exclusion Zone or at sufficient distance to prevent exposure should a release occur. Control boundaries are identified by cones.

NO UNAUTHORIZED PERSON SHOULD BE WITHIN THIS AREA

SECTION 7: SAFETY PROCEDURES/EQUIPMENT REQUIRED

Identify all procedures and equipment needed to eliminate or minimize exposure to hazards identified in Section 5.

☐ AIR MONITORING EQUIPMENT (see Section 9)	☒ FIRST AIDKIT	☐ EMERGENCY AIR HORN
☒ BARRIER TAPE	☐ FLOTATION DEVICE (USCG)	☒ PPE – PHYSICAL HAZARDS (see section 15)
☒ COMMUNICATIONS - ONSITE	☐ GFCI EXTENSION CORDS	☐ RESPIRATORY PROTECTION PROGRAM & EQUIPMENT (APR) (see Section 15)
☒ COMMUNICATIONS – OFFSITE (i.e. cell/digital phones if no other means)	☐ HARNESS(S) / LIFELINE(S)	☒ TRAFFIC CONES
☐ CONFINED SPACE PROGRAM & EQUIP (see Section 12)	☐ INSECT/TICK REPELLENT	☐ MSDS (Site Specific)
☐ VENTILATION EQUIPMENT	☒ SAFETY VESTS	☐ MSDS (Stone Environmental)
☐ EMERGENCY SHOWERS	☐ LADDER(S)	
☐ EYE WASH	☐ LIGHTING – HANDHELD	☐ OTHER: _____
☐ FALL PROTECTION PROGRAM & EQUIPMENT	☐ LIGHTING – FIXED EMERGENCY	
☒ FIRE EXTINGUISHER(S) - ABC	☐ LOCKOUT/TAGOUT PROGRAM & EQUIPMENT	

SECTION 8: COMMUNICATIONS AND SAFE WORK PRACTICE

(A) COMMUNICATIONS - ONSITE

Whenever possible, communications between site personnel should be face-to-face. When verbal communications is not possible, radio communications shall be established.

In case of radio communications failure, or when respiratory protection is in use, the following hand signals will be used:

OK; I AM ALL RIGHT; I UNDERSTAND	= THUMBS UP
NO; NEGATIVE	= THUMBS DOWN
NEED ASSISTANCE	= BOTH HANDS ON TOP OF HEAD
DANGER - NEED TO LEAVE AREA, NO QUESTIONS	= GRIP PARTNERS WRIST WITH BOTH HANDS
HAVING DIFFICULTY BREATHING	= HANDS TO THROAT

(B) COMMUNICATIONS – OFF SITE

If applicable, telephone communication to the Home Office should be established as soon as practical.

Telephone numbers that can be used to reach the command post are: **(802) 229-4541** and **800 959-9987**

(C) SAFE WORK PRACTICES

1. A "BUDDY SYSTEM" IN WHICH ANOTHER WORKER IS CLOSE ENOUGH TO RENDER IMMEDIATE AID WILL BE IN EFFECT. CLIENTS AND/OR CONTRACTORS MAY SERVE AS A "DESIGNATED BUDDY."
2. WHERE THE EYES OR BODY MAY BE EXPOSED TO CORROSIVE MATERIALS, SUITABLE FACILITIES FOR QUICK DRENCHING OR FLUSHING SHALL BE AVAILABLE FOR IMMEDIATE USE (SEE SECTION 7).
3. IF DRILLING EQUIPMENT IS INVOLVED, HAVE A CURRENT UTILITY SURVEY, AND KNOW WHERE THE 'KILL SWITCH' IS.
4. CONTACT WITH SAMPLES, EXCAVATED MATERIALS, OR OTHER CONTAMINATED MATERIALS MUST BE MINIMIZED.
5. ALL ELECTRICAL EQUIPMENT USED IN OUTSIDE LOCATIONS, WET AREAS OR NEAR WATER MUST BE PLUGGED INTO GROUND FAULT CIRCUIT INTERRUPTER (GFCI) PROTECTED OUTLETS (SEE SECTION 7).

6. IN THE EVENT OF TREACHEROUS WEATHER-RELATED WORKING CONDITIONS (I.E., THUNDERSTORM, LIMITED VISIBILITY, EXTREME COLD OR HEAT) FIELD TASKS WILL BE SUSPENDED UNTIL CONDITIONS IMPROVE OR APPROPRIATE PROTECTION FROM THE ELEMENTS IS PROVIDED.

7. SMOKING, EATING, CHEWING GUM OR TOBACCO, OR DRINKING ARE FORBIDDEN EXCEPT IN CLEAN OR DESIGNATED AREAS.

8. USE OF CONTACT LENSES NEAR CHEMICALS OR DURING USE OF RESPIRATORY PROTECTION IS PROHIBITED AT ALL TIMES.

9. GOOD HOUSEKEEPING PRACTICES ARE TO BE MAINTAINED.

10. SITE / FACILITY SPECIFIC SAFE WORK PRACTICES: (example, hard hat, steel toe work shoes; protective eyewear)

SECTION 9: ENVIRONMENTAL MONITORING ☒ THIS SECTION NOT APPLICABLE TO SITE ACTIVITIES

(A) The following environmental monitoring instruments shall be used on site at the specified intervals and recorded in the site logbook. (NOTE: If monitoring period is "OTHER", monitoring schedule will be attached to this plan.)

EQUIPMENT	MONITORING PERIOD	ACTION LEVEL
☐ Combustible Gas Indicator	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____
☐ O_2 Meter	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____
☐ Toxics: ☐ CO ☐ H_2S	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____
☐ Other: _____	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____
☐ PID (Lamp _____ eV)	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____
☐ FID		
☐ Colorimetric tubes:		
_____	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____
_____	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____
☐ Radiation: ☐ α ☐ β ☐ γ	☐ **x Day** ☐ Hourly x Day ☐ Other	_____
☐ Respirable Dust Meter	☐ Continuous ☐ Hourly x Day ☐ Other	_____
☐ Noise Meter	☐ Continuous ☐ Hourly x Day ☐ Other	_____
☐ Other: _____	☐ Continuous ☐ Hourly x Day ☐ Other	_____
_____	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____
_____	☐ Continuous ☐ Hourly ☐ x Day ☐ Other	_____

(B) Monitoring equipment is calibrated according to manufacturers' instructions. Record calibration data and air concentrations in the Health and Safety on-site log book.

(C) Recommended Action Levels for Upgrade or Downgrade of Respiratory Protection, or Site Shutdown and Evacuation. These are average values. Consideration should be given to the potential for release of highly toxic compounds from the waste or from reaction by-products. Levels are for persistent (> 10 min) breathing zone measurements in non-confined spaces. **For unexpected conditions, stop all work and contact Corporate Health and Safety.**

Oxygen Levels	
Less than 19.5%	Level B necessary for work to start / continue. Consider toxicity potential.
19.5% to 23.5%	Work may start / continue. Investigate changes. Continuous monitoring.
Greater than 23.5%	PROHIBITED WORK CONDITION
Flammability / Explosive Hazards	
Less than 10% of LEL	Work may start / continue. Consider toxicity potential.
10% to 25% of LEL	Work may start / continue. Continuous monitoring.
Greater than 25% of LEL	PROHIBITED WORK CONDITION.
Uncharacterized Airborne Organic Vapors or Gases	
Background*	Work may start / continue. Continue to monitor conditions.
Up to 5 meter units (m.u. or "ppm") above background	Level C necessary for work to start / continue. Continuous monitoring. Use Colorimetric tubes to characterize vapors.
Up to 50 m.u. above background	Level B necessary for work to start / continue. Continuous monitoring.
Greater than 50 m.u.	PROHIBITED WORK CONDITION.
*** Off-site clean air measurement**	
Characterized Airborne Organic Vapors or Gases**	
Up to 50% of TLV, or PEL or REL	Work may start / continue. Continue to monitor conditions.
Up to 25 times the TLV, or PEL or REL	Level C necessary for work to start / continue. Continuous monitoring.
Up to 500 times the TLV, or PEL or REL	Level B necessary for work to start / continue. Continuous monitoring.
Greater than 500 times the TLV, or PEL or REL	PROHIBITED WORK CONDITION.

** **Use mixture calculations (% allowed = $\sum C_N EL_N$) if more than one contaminant is present.**

Radiation	
Less than 0.5 mR/Hour (500 μR)	Work may start / continue. Continue to monitor conditions.
Up to 1 mR/Hour above background	Work may start / continue with Radiation Safety Officer present on site.
Greater than 1 mR/Hour above background	PROHIBITED WORK CONDITION.

SECTION 10: PERSONAL MONITORING ☒ THIS SECTION IS NOT APPLICABLE TO SITE ACTIVITIES

(A) PERSONAL EXPOSURE SAMPLING (Consider if high levels of noise or high concentrations of lead, mercury or arsenic are present)

The following personal monitoring will be in effect on site: _____

A copy of personal monitoring results is to be sent to Corporate Health and Safety for inclusion in the Employee's Confidential Exposure Record File.

(B) HEAT / COLD STRESS MONITORING

The expected air temperature range will be _____°F. If it is determined that heat stress or cold stress monitoring is required (mandatory for heavy exertion in PPE at temperatures over 70°F, or at temperatures under 40°F or wind chill equivalent), the following procedures shall be followed (describe procedures in effect, for heat stress i.e., monitoring body temperature, body weight, pulse rate; for cold stress i.e., appropriate clothing, shelter breaks): _____

SECTION 11: HAZARD COMMUNICATION PROGRAM

☐ THIS SECTION IS NOT APPLICABLE TO SITE ACTIVITIES

If chemicals are introduced to the site by Stone Environmental, Inc. (e.g., decontamination liquids, preservatives, etc.), a copy of the Stone Environmental, Inc. Hazard Communication Program and Material Safety Data Sheets (MSDSs) of chemicals introduced by Stone Environmental, Inc. to the site is attached to this plan. The Site Safety Officer will review this information with all field personnel prior to the start of the project, and will inform other employers (e.g., Owner, Contractor and Subcontractors) the availability and location of this information. The Comprehensive List of Chemicals introduced by Stone Environmental, Inc. to this site is:

_____ _____

_____ _____

All chemicals being introduced to the site, hazardous/potentially hazardous samples prepared at the site, and/or any hazardous materials previously sent to the site, that will be stored at the site or will be transported from the site by common carrier, will be packaged, labeled and identified as hazardous materials in accordance with U.S. Department of Transportation (DOT) and/or International Air Transport Association (IATA) regulations by a trained HazMat employee. *(NOTE: At multi-employer sites, the Site Safety Officer will obtain information, if applicable, on hazardous chemicals other employers may produce or introduce to the job site to which Stone Environmental, Inc. employees may be exposed, including the location of their written hazard communication program(s), labeling program(s), and Material Safety Data Sheet(s).*

SECTION 12: CONFINED SPACE ENTRY

☒ THIS SECTION NOT APPLICABLE TO SITE ACTIVITIES

If a permit-required confined space entry will be made on site, a copy of the Stone Environmental, Inc. Confined Space Entry Program, and a completed Stone Environmental, Inc. Confined Space Pre-Entry Inspection Check List will be attached to this plan. A Confined Space Entry Permit must be completed and posted outside the confined space prior to entry, and the entry will follow the Stone Environmental, Inc. Confined Space Entry written program. Permits are to be saved and logged with project documentation.

SECTION 13: EXCAVATION SAFETY

☒ THIS SECTION NOT APPLICABLE TO SITE ACTIVITIES

Excavations being created in order to accomplish Stone Environmental, Inc. tasks or in progress during Stone Environmental, Inc. inspection of other activities or tasks, shall be shored or slopped or otherwise protected to prevent accidental collapse prior to entry, in accordance with Subpart F of 29 CFR 1926. It is Stone Environmental, Inc. policy that Stone Environmental, Inc. personnel will not enter trench or excavated areas without approval of Corporate Health and Safety. If an entry into an excavation by Stone Environmental, Inc. personnel is necessary, a Competent Person will be designated by the Corporate Health and Safety Officer. Excavations will not be left open overnight unless absolutely necessary.

COMPETANT PERSON

_____	_____	_____
Print Name	**Signature**	**Date**

SECTION 14: DECONTAMINATION PROCEDURES

☐ THIS SECTION NOT APPLICABLE TO SITE ACTIVITIES

Personnel and equipment leaving the Exclusion Zone shall be thoroughly decontaminated. The Site Safety Officer is responsible for monitoring adherence with this decontamination plan.

A _____ decontamination protocol shall be used with the following decontamination stations:

(1) _____

(2) _____

(3) _____

(Other) _____

The following decontamination equipment is required:

☐ Decon Pad (Plastic Sheet) _____ ☐ Dry Brushes ☐ Buckets Other _____

☐ Trash Cans/Bags ☐ Wet Brushes ☐ Hose / Spray _____

_____ Will be used as the decontamination solution

SECTION 15: PERSONAL PROTECTIVE EQUIPMENT

1 - List all that apply, i.e., FF w/ OV/AG/P

2 - Use same codes for clothing and boots of same material

TASK *	RESPIRATORS & CARTRIDGE[1]	USE ** (See Section 16)	CLOTHING	GLOVES	BOOTS	OTHER
S1	**NA**	**NA**	**NA**	**NA**	**NA**	**NA**
S2	**NA**	**NA**	**N/S**	**N**	**SL**	**HH, G, HP**
S3						
S4						
S5						

* Same as Section 3E

**UP = Upgrade
CONT = Continuous

NOTE: PPE use will be in accordance with Stone Environmental, Inc.'s Health and Safety Policy and Written Programs

CODES:

RESPIRATORS[1]
HF = Half Face APR
FF = Full Face APR
ESCBA = Escape Bottle
SAR = Airline
SCBA = SCBA

CARTRIDGES[1]
P = Particulate
OV = Organic Vapors
AG = Acid Gas
Multi = Multi-Gas/Vapor
Other

CLOTHING
N/S = No Special
C = Coveralls
T = Tyvek
Sx = Saranex
PT = PE Tyvek

GLOVES[2]
Co = Cotton
Le = Leather
L = Latex
N = Nitrile
B = Butyl
Neo = Neoprene
V = Viton
PVC = Polyvinyl Chloride
PVA = Polyvinyl Alcohol
T = Teflon
Other: _____

BOOTS
SL = Leather Safety
H = Hip (Fireman)
O = Latex overboots
CHM = Chemical Resistant

OTHER
HH = Hard Hat
G = Safety Glasses
GP = Glare Protection
Gl = Goggles - Impact
GS = Goggles - Splash
FS = Face Shield
HP = Hearing Protection
PFD=Personal Flotation Device

SECTION 16: PERSONAL PROTECTIVE EQUIPMENT (CONTINUED)

Respiratory protection will be upgraded under the following conditions:

NA

The following cartridge change out schedule is to be followed onsite (attach any calculations to plan):

NA

SECTION 17: EMERGENCY ACTION PLAN

The following standard emergency response procedures will be used by onsite personnel. The Site Safety Officer shall be notified of any onsite emergencies and be responsible for ensuring that the appropriate procedures are followed.

(A) EVACUATION

All work activities are suspended and the site is to be EVACUATED IMMEDIATELY, when there is a threat to life or health as determined by individual good judgment, i.e. fire, hazardous chemical spill, dangerous gas leak, severe weather (i.e., tornado); or when notified by other site / facility staff and local fire or police officials.

- If an evacuation is called for, the emergency alarm system for weather-related, medical, fire and other evacuation emergencies is:
- 3 short blasts on a compressed air horn
- Evacuation from the Exclusion Zone should whenever possible occur through the decontamination line. In those situations where egress in this manner cannot occur, the following emergency escape routes have been designated (document on map if possible):

Once off site, all staff should gather at the bike path which is a minimum of 250 feet away from the incident

(B) FIRE OR EXPLOSION

Upon discovery of a fire or an explosion, the above-designated emergency signal shall be sounded and all personnel shall assemble on the bike path. The fire department is to be notified and all personnel moved to a safe distance (minimum 250') from the involved area.

If a person's clothing should catch fire, burning clothing may be extinguished by having the individual drop to the floor and roll. If necessary, physically restrain the person and roll them around on the floor to smother the flames. Use a fire blanket or extinguisher if one is readily available and you have been trained in its use. Call emergency medical services if not already done so.

If a person's clothing should become saturated with a chemical, douse the individual with water from the nearest safety shower if available. Consult the chemical Material Safety Data Sheets (MSDSs) for further information. Call emergency medical services if indicated by the MSDSs.

NEVER RE-ENTER THE SITE / FACILITY until the emergency has been declared over and permission to re-enter has been given by site / facility health and safety staff or local fire or police officials. If any staff is unaccounted for, notify an individual in charge.

(C) MEDICAL EMERGENCY

If you discover a medical emergency and are by yourself, CALL OUT FOR HELP. When someone arrives, tell them to call for help. If no one comes or you know you are alone, provide whatever care you can for 1 minute, and then make the call yourself. (See Section 2)

Upon notification of an injury the SSO or alternate should evaluate the nature of the injury and shall initiate the appropriate first aid, and contact should be made for an ambulance (and other emergency services as needed) and with the designated medical facility (if required). No persons shall reenter the Exclusion Zone until the cause of the injury or a symptom is determined.

The hospital is **Seven Minutes** from the site. Ambulance response time is **Unknown**. When IDLH conditions exist, arrangements should be made for onsite standby of emergency services. A map for directions to the nearest hospital is attached to this plan.

(D) FOLLOW UP

In all situations, when an on site / facility emergency results in evacuation of the work area, or a "large spill" has occurred, staff shall not resume work until:

- The conditions resulting in the emergency have been corrected;
- The hazards reassessed by the SSO and Corporate Health and Safety;
- The HASP has been reviewed by the SSO and Corporate Health and Safety; and
- Site personnel have been briefed on any changes in the HASP by the SSO.

SECTION 18: SPILL CONTAINMENT / CONTROL ☐ THIS SECTION NOT APPLICABLE TO SITE ACTIVITIES

For most chemicals introduced to the worksite, or under control of Stone Environmental, Inc. employees, spills of chemicals would be considered incidental and would be controlled in the immediate area of the spill. Such spills shall be handled utilizing precautions appropriate for the chemical characteristics specified in the MSDS for the chemical including spill control methods and selection and use of minimum personal protective equipment.

For chemicals introduced to the worksite, or under control of Stone Environmental, Inc. employees, that would cause a "large spill" (greater than 55 gallons), a copy of the appropriate Emergency Response Guidebook (ERG) guide shall be attached to this plan, and a spill response contractor shall be identified in Section 2.

SECTION 19: EMPLOYEE ACKNOWLEDGEMENTS

PLAN REVIEWED BY:	NAME	DATE
Project Officer:	**Seth Pitkin**	
Project Manager:	**Dan Voisin**	
Site Safety Officer:	**Lee Rosberg**	
Corporate H & S	**Kim Watson**	

I acknowledge that I have read the information on this HASP-SF, attached Material Safety Data Sheets (MSDSs), DOT Emergency Response Guides, and Health and Safety Programs.
I understand the site / facility hazards as described and agree to comply with the contents of the plan.

EMPLOYEE (Print Name/Signature/Date)

_____ / _____ / _____ _____ / _____ / _____

_____ / _____ / _____ _____ / _____ / _____

VISITORS (Print Name/Signature/Date)

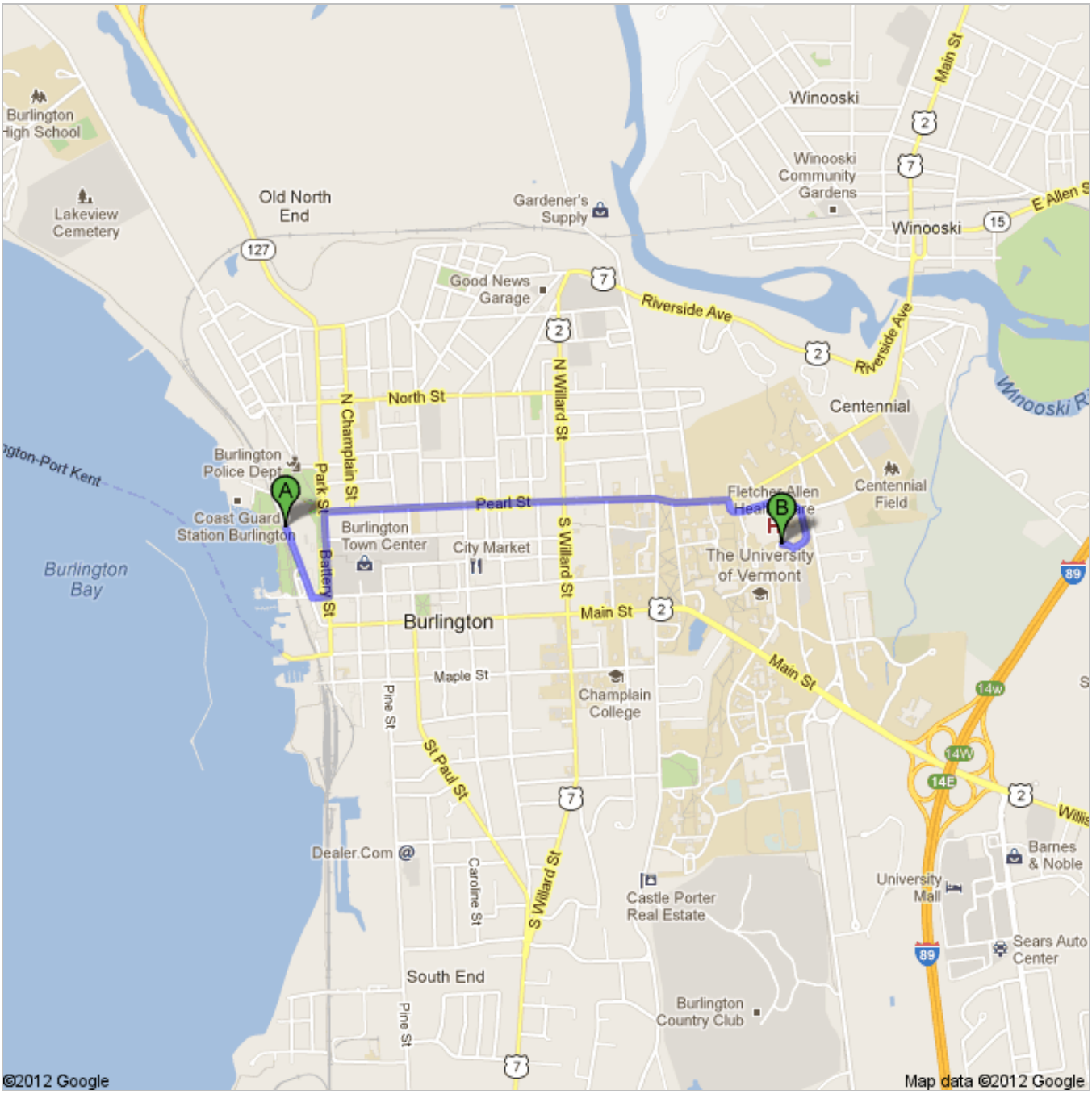
_____ / _____ / _____ _____ / _____ / _____

_____ / _____ / _____ _____ / _____ / _____

ATTACHED DOCUMENTS

☒ MSDS(s)	☐ Hazard Communication Written Program	☐ Confined Space Entry Written Program	☐ Respiratory Protection Program
☐ Site Map	☐ Personal Protective Equipment Written Program	☐ DOT ERG Guides	☐ Task/Job Hazard Analysis Forms
☒ Hospital Directions	☐ Emergency Action Plan	☒ H&S Daily Site Sheet	☐ Other _____

Directions to FLETCHER ALLEN HEALTHCARE-MED CTR CMPS
111 Colchester Avenue, Burlington, VT 05401
2.0 mi – about **7 mins**



Lake St, Burlington, VT 05401

1.	Head **south** on **Lake St** toward **Island Line Trail**	go 0.2 mi total 0.2 mi
2.	Take the 1st left onto **College St**	go 243 ft total 0.2 mi
3.	Turn left onto **Battery St** About 58 secs	go 0.2 mi total 0.5 mi
4.	Turn right onto **Pearl St** About 3 mins	go 0.9 mi total 1.4 mi
5.	Continue onto **Colchester Ave**	go 0.2 mi total 1.6 mi
6.	Turn right About 1 min	go 0.4 mi total 1.9 mi
7.	Turn right Destination will be on the right	go 174 ft total 2.0 mi

FLETCHER ALLEN HEALTHCARE-MED CTR CMPS
111 Colchester Avenue, Burlington, VT 05401

These directions are for planning purposes only. You may find that construction projects, traffic, weather, or other events may cause conditions to differ from the map results, and you should plan your route accordingly. You must obey all signs or notices regarding your route.

Map data ©2012 Google

Directions weren't right? Please find your route on maps.google.com and click "Report a problem" at the bottom left.

Safety Data Sheet

Material Name: Diesel Fuel, All Types

SDS No. 9909
US GHS

Synonyms: Ultra Low Sulfur Diesel; Low Sulfur Diesel; No. 2 Diesel; Motor Vehicle Diesel Fuel; Non-Road Diesel Fuel; Locomotive/Marine Diesel Fuel

* * * Section 1 - Product and Company Identification * * *

Manufacturer Information

Hess Corporation
1 Hess Plaza
Woodbridge, NJ 07095-0961

Phone: 732-750-6000 Corporate EHS
Emergency # 800-424-9300 CHEMTREC
www.hess.com (Environment, Health, Safety Internet Website)

* * * Section 2 - Hazards Identification * * *

GHS Classification:

Flammable Liquids - Category 3
Skin Corrosion/Irritation – Category 2
Germ Cell Mutagenicity – Category 2
Carcinogenicity - Category 2
Specific Target Organ Toxicity (Single Exposure) - Category 3 (respiratory irritation, narcosis)
Aspiration Hazard – Category 1
Hazardous to the Aquatic Environment, Acute Hazard – Category 3

GHS LABEL ELEMENTS

Symbol(s)



Signal Word

DANGER

Hazard Statements

Flammable liquid and vapor.
Causes skin irritation.
Suspected of causing genetic defects.
Suspected of causing cancer.
May cause respiratory irritation.
May cause drowsiness or dizziness.
May be fatal if swallowed and enters airways.
Harmful to aquatic life.

Precautionary Statements

Prevention

Keep away from heat/sparks/open flames/hot surfaces. No smoking
Keep container tightly closed.
Ground/bond container and receiving equipment.

Use explosion-proof electrical/ventilating/lighting/equipment.
Use only non-sparking tools.
Take precautionary measures against static discharge.
Wear protective gloves/protective clothing/eye protection/face protection.
Wash hands and forearms thoroughly after handling.
Obtain special instructions before use.
Do not handle until all safety precautions have been read and understood.
Avoid breathing fume/mist/vapours/spray.

Response
In case of fire: Use water spray, fog or foam to extinguish.
IF ON SKIN (or hair): Wash with plenty of soap and water. Remove/Take off immediately all contaminated clothing and wash it before reuse. If skin irritation occurs: Get medical advice/attention.
IF INHALED: Remove person to fresh air and keep comfortable for breathing. Call a poison center/doctor if you feel unwell.
If swallowed: Immediately call a poison center or doctor. Do NOT induce vomiting.
IF exposed or concerned: Get medical advice/attention.

Storage
Store in a well-ventilated place. Keep cool.
Keep container tightly closed.
Store locked up.

Disposal
Dispose of contents/container in accordance with local/regional/national/international regulations.

* * * Section 3 - Composition / Information on Ingredients * * *

CAS #	Component	Percent
68476-34-6	Fuels, diesel, no. 2	100
91-20-3	Naphthalene	<0.1

A complex mixture of hydrocarbons with carbon numbers in the range C9 and higher.

* * * Section 4 - First Aid Measures * * *

First Aid: Eyes
In case of contact with eyes, immediately flush with clean, low-pressure water for at least 15 min. Hold eyelids open to ensure adequate flushing. Seek medical attention.

First Aid: Skin
Remove contaminated clothing. Wash contaminated areas thoroughly with soap and water or with waterless hand cleanser. Obtain medical attention if irritation or redness develops. Thermal burns require immediate medical attention depending on the severity and the area of the body burned.

First Aid: Ingestion
DO NOT INDUCE VOMITING. Do not give liquids. Obtain immediate medical attention. If spontaneous vomiting occurs, lean victim forward to reduce the risk of aspiration. Monitor for breathing difficulties. Small amounts of material which enter the mouth should be rinsed out until the taste is dissipated.

First Aid: Inhalation

Remove person to fresh air. If person is not breathing, provide artificial respiration. If necessary, provide additional oxygen once breathing is restored if trained to do so. Seek medical attention immediately.

* * * Section 5 - Fire Fighting Measures * * *

General Fire Hazards

See Section 9 for Flammability Properties.

Vapors may be ignited rapidly when exposed to heat, spark, open flame or other source of ignition. When mixed with air and exposed to an ignition source, flammable vapors can burn in the open or explode in confined spaces. Being heavier than air, vapors may travel long distances to an ignition source and flash back. Runoff to sewer may cause fire or explosion hazard.

Hazardous Combustion Products

Carbon monoxide, carbon dioxide and non-combusted hydrocarbons (smoke).

Extinguishing Media

SMALL FIRES: Any extinguisher suitable for Class B fires, dry chemical, CO2, water spray, fire fighting foam, and other gaseous agents.

LARGE FIRES: Water spray, fog or fire fighting foam. Water may be ineffective for fighting the fire, but may be used to cool fire-exposed containers.

Unsuitable Extinguishing Media

None

Fire Fighting Equipment/Instructions

Small fires in the incipient (beginning) stage may typically be extinguished using handheld portable fire extinguishers and other fire fighting equipment. Firefighting activities that may result in potential exposure to high heat, smoke or toxic by-products of combustion should require NIOSH/MSHA- approved pressure-demand self-contained breathing apparatus with full facepiece and full protective clothing. Isolate area around container involved in fire. Cool tanks, shells, and containers exposed to fire and excessive heat with water. For massive fires the use of unmanned hose holders or monitor nozzles may be advantageous to further minimize personnel exposure. Major fires may require withdrawal, allowing the tank to burn. Large storage tank fires typically require specially trained personnel and equipment to extinguish the fire, often including the need for properly applied fire fighting foam.

* * * Section 6 - Accidental Release Measures * * *

Recovery and Neutralization

Carefully contain and stop the source of the spill, if safe to do so.

Materials and Methods for Clean-Up

Take up with sand or other oil absorbing materials. Carefully shovel, scoop or sweep up into a waste container for reclamation or disposal. Caution, flammable vapors may accumulate in closed containers.

Emergency Measures

Evacuate nonessential personnel and remove or secure all ignition sources. Consider wind direction; stay upwind and uphill, if possible. Evaluate the direction of product travel, diking, sewers, etc. to confirm spill areas. Spills may infiltrate subsurface soil and groundwater; professional assistance may be necessary to determine the extent of subsurface impact.

Personal Precautions and Protective Equipment

Response and clean-up crews must be properly trained and must utilize proper protective equipment (see Section 8).

Environmental Precautions

Protect bodies of water by diking, absorbents, or absorbent boom, if possible. Do not flush down sewer or drainage systems, unless system is designed and permitted to handle such material. The use of fire fighting foam may be useful in certain situations to reduce vapors. The proper use of water spray may effectively disperse product vapors or the liquid itself, preventing contact with ignition sources or areas/equipment that require protection.

Prevention of Secondary Hazards

None

* * * Section 7 - Handling and Storage * * *

Handling Procedures

Handle as a combustible liquid. Keep away from heat, sparks, excessive temperatures and open flame! No smoking or open flame in storage, use or handling areas. Bond and ground containers during product transfer to reduce the possibility of static-initiated fire or explosion.

Special slow load procedures for "switch loading" must be followed to avoid the static ignition hazard that can exist when higher flash point material (such as fuel oil) is loaded into tanks previously containing low flash point products (such as this product) - see API Publication 2003, "Protection Against Ignitions Arising Out Of Static, Lightning and Stray Currents."

Storage Procedures

Keep away from flame, sparks, excessive temperatures and open flame. Use approved vented containers. Keep containers closed and clearly labeled. Empty product containers or vessels may contain explosive vapors. Do not pressurize, cut, heat, weld or expose such containers to sources of ignition.

Store in a well-ventilated area. This storage area should comply with NFPA 30 "Flammable and Combustible Liquid Code". Avoid storage near incompatible materials. The cleaning of tanks previously containing this product should follow API Recommended Practice (RP) 2013 "Cleaning Mobile Tanks In Flammable and Combustible Liquid Service" and API RP 2015 "Cleaning Petroleum Storage Tanks."

Incompatibilities

Keep away from strong oxidizers.

* * * Section 8 - Exposure Controls / Personal Protection * * *

Component Exposure Limits

Fuels, diesel, no. 2 (68476-34-6)

ACGIH: 100 mg/m3 TWA (inhalable fraction and vapor, as total hydrocarbons, listed under Diesel fuel)
Skin - potential significant contribution to overall exposure by the cutaneous route (listed under Diesel fuel)

Naphthalene (91-20-3)

ACGIH: 10 ppm TWA
15 ppm STEL
Skin - potential significant contribution to overall exposure by the cutaneous route
OSHA: 10 ppm TWA; 50 mg/m3 TWA
NIOSH: 10 ppm TWA; 50 mg/m3 TWA
15 ppm STEL; 75 mg/m3 STEL

Engineering Measures

Use adequate ventilation to keep vapor concentrations of this product below occupational exposure and flammability limits, particularly in confined spaces.

Personal Protective Equipment: Respiratory

A NIOSH/MSHA-approved air-purifying respirator with organic vapor cartridges or canister may be permissible under certain circumstances where airborne concentrations are or may be expected to exceed exposure limits or for odor or irritation. Protection provided by air-purifying respirators is limited.

Use a positive pressure, air-supplied respirator if there is a potential for uncontrolled release, exposure levels are not known, in oxygen-deficient atmospheres, or any other circumstance where an air-purifying respirator may not provide adequate protection.

Personal Protective Equipment: Hands

Gloves constructed of nitrile, neoprene, or PVC are recommended.

Personal Protective Equipment: Eyes

Safety glasses or goggles are recommended where there is a possibility of splashing or spraying.

Personal Protective Equipment: Skin and Body

Chemical protective clothing such as of E.I. DuPont TyChem®, Saranex® or equivalent recommended based on degree of exposure. Note: The resistance of specific material may vary from product to product as well as with degree of exposure. Consult manufacturer specifications for further information.

* * * Section 9 - Physical & Chemical Properties * * *

Appearance:	Clear, straw-yellow.	**Odor:**	Mild, petroleum distillate odor
Physical State:	Liquid	**pH:**	ND
Vapor Pressure:	0.009 psia @ 70 °F (21 °C)	**Vapor Density:**	>1.0
Boiling Point:	320 to 690 °F (160 to 366 °C)	**Melting Point:**	ND
Solubility (H2O):	Negligible	**Specific Gravity:**	0.83-0.876 @ 60°F (16°C)
Evaporation Rate:	Slow; varies with conditions	**VOC:**	ND
Percent Volatile:	100%	**Octanol/H2O Coeff.:**	ND
Flash Point:	>125 °F (>52 °C) minimum	**Flash Point Method:**	PMCC
Upper Flammability Limit (UFL):	7.5	**Lower Flammability Limit (LFL):**	0.6
Burning Rate:	ND	**Auto Ignition:**	494°F (257°C)

* * * Section 10 - Chemical Stability & Reactivity Information * * *

Chemical Stability

This is a stable material.

Hazardous Reaction Potential

Will not occur.

Conditions to Avoid

Avoid high temperatures, open flames, sparks, welding, smoking and other ignition sources.

Incompatible Products

Keep away from strong oxidizers.

Hazardous Decomposition Products

Carbon monoxide, carbon dioxide and non-combusted hydrocarbons (smoke).

* * * Section 11 - Toxicological Information * * *

Acute Toxicity

A: General Product Information

Harmful if swallowed.

B: Component Analysis - LD50/LC50

Naphthalene (91-20-3)

Inhalation LC50 Rat >340 mg/m3 1 h; Oral LD50 Rat 490 mg/kg; Dermal LD50 Rat >2500 mg/kg; Dermal LD50 Rabbit >20 g/kg

Potential Health Effects: Skin Corrosion Property/Stimulativeness

Practically non-toxic if absorbed following acute (single) exposure. May cause skin irritation with prolonged or repeated contact. Liquid may be absorbed through the skin in toxic amounts if large areas of skin are repeatedly exposed.

Potential Health Effects: Eye Critical Damage/ Stimulativeness

Contact with eyes may cause mild irritation.

Potential Health Effects: Ingestion

Ingestion may cause gastrointestinal disturbances, including irritation, nausea, vomiting and diarrhea, and central nervous system (brain) effects similar to alcohol intoxication. In severe cases, tremors, convulsions, loss of consciousness, coma, respiratory arrest, and death may occur.

Potential Health Effects: Inhalation

Excessive exposure may cause irritations to the nose, throat, lungs and respiratory tract. Central nervous system (brain) effects may include headache, dizziness, loss of balance and coordination, unconsciousness, coma, respiratory failure, and death.

WARNING: the burning of any hydrocarbon as a fuel in an area without adequate ventilation may result in hazardous levels of combustion products, including carbon monoxide, and inadequate oxygen levels, which may cause unconsciousness, suffocation, and death.

Respiratory Organs Sensitization/Skin Sensitization

This product is not reported to have any skin sensitization effects.

Generative Cell Mutagenicity

This material has been positive in a mutagenicity study.

Carcinogenicity

A: General Product Information

Suspected of causing cancer.

Studies have shown that similar products produce skin tumors in laboratory animals following repeated applications without washing or removal. The significance of this finding to human exposure has not been determined. Other studies with active skin carcinogens have shown that washing the animal's skin with soap and water between applications reduced tumor formation.

B: Component Carcinogenicity

Fuels, diesel, no. 2 (68476-34-6)

ACGIH: A3 - Confirmed Animal Carcinogen with Unknown Relevance to Humans (listed under Diesel fuel)

Naphthalene (91-20-3)

ACGIH: A4 - Not Classifiable as a Human Carcinogen
NTP: Reasonably Anticipated To Be A Human Carcinogen (Possible Select Carcinogen)
IARC: Monograph 82 [2002] (Group 2B (possibly carcinogenic to humans))

Reproductive Toxicity

This product is not reported to have any reproductive toxicity effects.

Specified Target Organ General Toxicity: Single Exposure

This product is not reported to have any specific target organ general toxicity single exposure effects.

Specified Target Organ General Toxicity: Repeated Exposure

This product is not reported to have any specific target organ general toxicity repeat exposure effects.

Aspiration Respiratory Organs Hazard

The major health threat of ingestion occurs from the danger of aspiration (breathing) of liquid drops into the lungs, particularly from vomiting. Aspiration may result in chemical pneumonia (fluid in the lungs), severe lung damage, respiratory failure and even death.

* * * Section 12 - Ecological Information * * *

Ecotoxicity

A: General Product Information

Keep out of sewers, drainage areas and waterways. Report spills and releases, as applicable, under Federal and State regulations.

B: Component Analysis - Ecotoxicity - Aquatic Toxicity

Fuels, diesel, no. 2 (68476-34-6)

Test & Species		Conditions
96 Hr LC50 Pimephales promelas	35 mg/L [flow-through]	

Naphthalene (91-20-3)

Test & Species		Conditions
96 Hr LC50 Pimephales promelas	5.74-6.44 mg/L [flow-through]	
96 Hr LC50 Oncorhynchus mykiss	1.6 mg/L [flow-through]	
96 Hr LC50 Oncorhynchus mykiss	0.91-2.82 mg/L [static]	
96 Hr LC50 Pimephales promelas	1.99 mg/L [static]	

96 Hr LC50 Lepomis macrochirus	31.0265 mg/L [static]
72 Hr EC50 Skeletonema costatum	0.4 mg/L
48 Hr LC50 Daphnia magna	2.16 mg/L
48 Hr EC50 Daphnia magna	1.96 mg/L [Flow through]
48 Hr EC50 Daphnia magna	1.09 - 3.4 mg/L [Static]

Persistence/Degradability
No information available.

Bioaccumulation
No information available.

Mobility in Soil
No information available.

* * * Section 13 - Disposal Considerations * * *

Waste Disposal Instructions
See Section 7 for Handling Procedures. See Section 8 for Personal Protective Equipment recommendations.

Disposal of Contaminated Containers or Packaging
Dispose of contents/container in accordance with local/regional/national/international regulations.

* * * Section 14 - Transportation Information * * *

DOT Information
Shipping Name: Diesel Fuel
NA #: 1993 **Hazard Class:** 3 **Packing Group:** III
Placard:



* * * Section 15 - Regulatory Information * * *

Regulatory Information

Component Analysis

This material contains one or more of the following chemicals required to be identified under SARA Section 302 (40 CFR 355 Appendix A), SARA Section 313 (40 CFR 372.65) and/or CERCLA (40 CFR 302.4).

Naphthalene (91-20-3)

CERCLA: 100 lb final RQ; 45.4 kg final RQ

SARA Section 311/312 – Hazard Classes

Acute Health	Chronic Health	Fire	Sudden Release of Pressure	Reactive
X	X	X	--	--

SARA SECTION 313 - SUPPLIER NOTIFICATION

This product may contain listed chemicals below the de minimis levels which therefore are not subject to the supplier notification requirements of Section 313 of the Emergency Planning and Community Right- To-Know Act (EPCRA) of 1986 and of 40 CFR 372. If you may be required to report releases of chemicals listed in 40 CFR 372.28, you may contact Hess Corporate Safety if you require additional information regarding this product.

State Regulations

Component Analysis - State

The following components appear on one or more of the following state hazardous substances lists:

Component	CAS	CA	MA	MN	NJ	PA	RI
Fuels, diesel, no. 2	68476-34-6	No	No	No	Yes	No	No
Naphthalene	91-20-3	Yes	Yes	Yes	Yes	Yes	No

The following statement(s) are provided under the California Safe Drinking Water and Toxic Enforcement Act of 1986 (Proposition 65):

WARNING! This product contains a chemical known to the state of California to cause cancer.

Component Analysis - WHMIS IDL

No components are listed in the WHMIS IDL.

Additional Regulatory Information

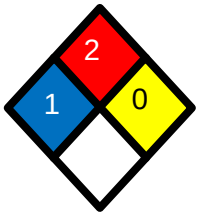
Component Analysis - Inventory

Component	CAS #	TSCA	CAN	EEC
Fuels, diesel, no. 2	68476-34-6	Yes	DSL	EINECS
Naphthalene	91-20-3	Yes	DSL	EINECS

* * * Section 16 - Other Information * * *

NFPA® Hazard Rating

Health	1
Fire	2
Reactivity	0



HMIS® Hazard Rating

Health	1*	Slight
Fire	2	Moderate
Physical	0	Minimal
		*Chronic

Key/Legend

ACGIH = American Conference of Governmental Industrial Hygienists; ADG = Australian Code for the Transport of Dangerous Goods by Road and Rail; ADR/RID = European Agreement of Dangerous Goods by Road/Rail; AS = Standards Australia; DFG = Deutsche Forschungsgemeinschaft; DOT = Department of Transportation; DSL = Domestic Substances List; EEC = European Economic Community; EINECS = European Inventory of Existing Commercial Chemical Substances; ELINCS = European List of Notified Chemical Substances; EU = European Union; HMIS = Hazardous Materials Identification System; IARC = International Agency for Research on Cancer; IMO = International Maritime Organization; IATA = International Air Transport Association; MAK = Maximum Concentration Value in the Workplace; NDSL = Non-Domestic Substances List; NFPA = National Fire Protection Association; NOHSC = National Occupational Health & Safety Commission; NTP = National Toxicology Program; STEL = Short-term Exposure Limit; TDG = Transportation of Dangerous Goods; TLV = Threshold Limit Value; TSCA = Toxic Substances Control Act; TWA = Time Weighted Average

Literature References

None

Other Information

Information presented herein has been compiled from sources considered to be dependable, and is accurate and reliable to the best of our knowledge and belief, but is not guaranteed to be so. Since conditions of use are beyond our control, we make no warranties, expressed or implied, except those that may be contained in our written contract of sale or acknowledgment.

Vendor assumes no responsibility for injury to vendee or third persons proximately caused by the material if reasonable safety procedures are not adhered to as stipulated in the data sheet. Additionally, vendor assumes no responsibility for injury to vendee or third persons proximately caused by abnormal use of the material, even if reasonable safety procedures are followed. Furthermore, vendee assumes the risk in their use of the material.

End of Sheet