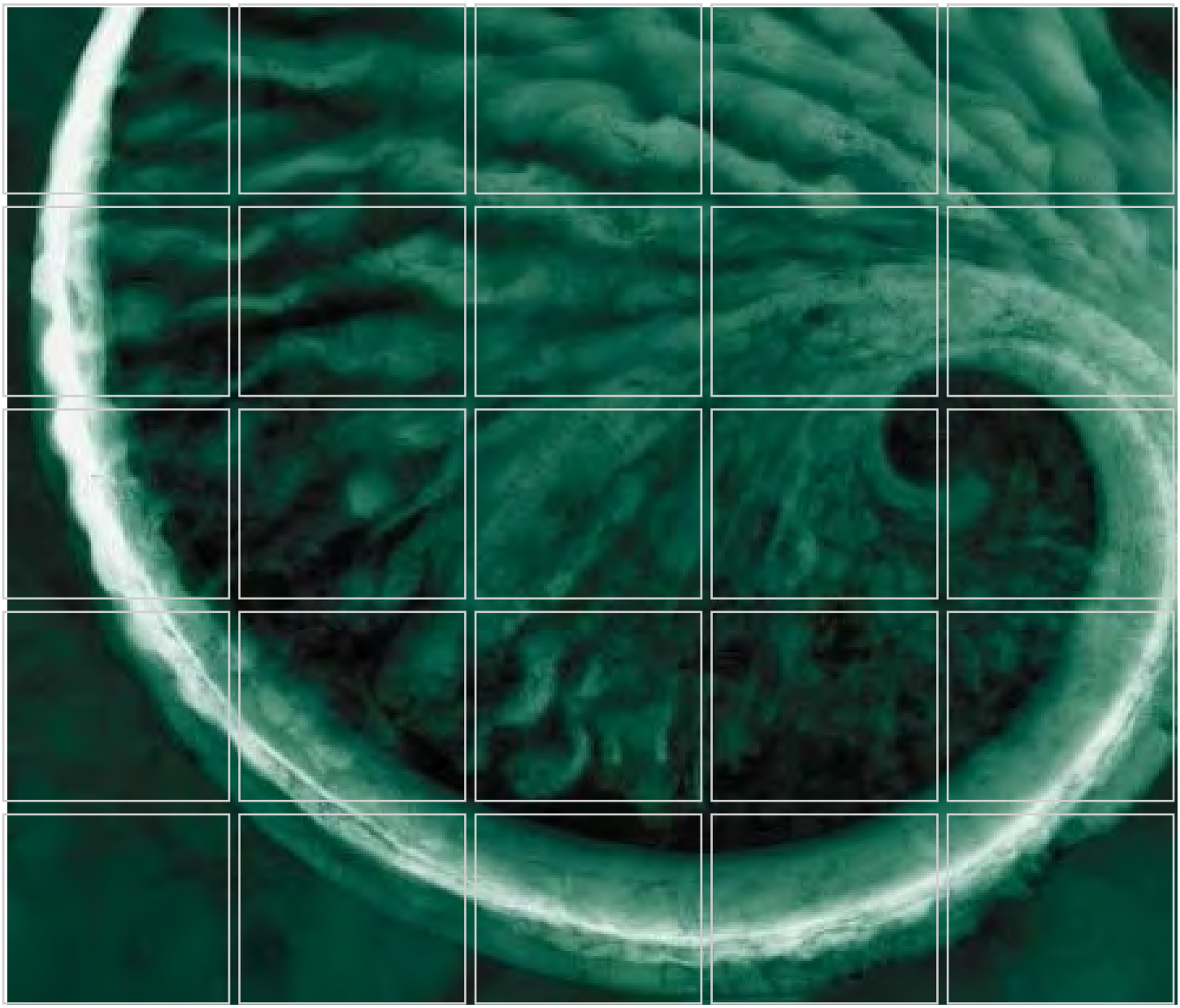

Revised Final Performance Monitoring Plan – Groundwater Source Control Measure

Premier Edible Oils
Portland, Oregon

Prepared for:
MMGL LLC

April 2017

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MMGL LLC.

Revised Final Performance Monitoring Plan – Groundwater Source Control Measure

Premier Edible Oils, Portland, Oregon

April 2017

Project No. 0382181

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ERM	ERM-West, Inc.
FS	Feasibility study
GWBW	Groundwater barrier wall
GW SCM	Groundwater source control measure
JSCS	Joint Source Control Strategy
LNAPL	Light non-aqueous phase liquid
ODEQ	Oregon Department of Environmental Quality
PMP	Performance Monitoring Plan
RI	Remedial Investigation
RG	Remediation Goal
SCM	Source control measure
TZW	Transition zone water
USEPA	United States Environmental Protection Agency
WQS	Water Quality Standard

1.0 INTRODUCTION

On behalf of MMGL LLC as successor by merger to MMGL Corp. (MMGL), ERM-West, Inc. (ERM) has prepared this Performance Monitoring Plan (PMP) for the former Premier Edible Oils (PEO) facility located at 10400 North Burgard Way in Portland, Oregon (the site) (Figure 1). This PMP has been prepared pursuant to the Voluntary Agreement for Upland Remedial Investigation (RI)/Feasibility Study (FS) and Source Control Measures issued by the Oregon Department of Environmental Quality (ODEQ) and signed 6 March 2001 (ODEQ ECDVC-NWR-01-06) (Voluntary Agreement), and revised based on comments received from the ODEQ in a letter dated 8 March 2017.

The purpose of this PMP is to present the monitoring requirements for the implementation of a groundwater source control measure (GW SCM) at the site.

1.1 BACKGROUND

The site is an industrial property located on the Portland Harbor waterfront along the east bank of the Willamette River. The upland portion of the site has been used for a number of industrial operations since approximately 1940, including bulk petroleum storage, ship building, production of dry cell battery materials, and refining of edible oils. PEO operated the site from 1972 to 1997 for the refining of edible oils, such as palm and cottonseed oils, and packaging and shipping the oils for use in the processed food industry. PEO operations were discontinued in approximately 1996, and the remaining buildings are vacant. A number of environmental investigations have been performed at the site since 2001 under the ODEQ Voluntary Cleanup Program.

The facility is bounded by the Willamette River on the south and west, exporting and metal recycling facilities to the east, and the Time Oil Co. Northwest Terminal (ECSI Site ID: 170) to the north.

Detailed historical land use and potential contaminant of interest sources were presented in the Phase II Remedial Investigation Work Plan (Treadwell & Rollo, A Langan Company [Treadwell & Rollo] 2014) and are summarized in this section.

1.1.2 Groundwater Source Control Measure Development

The Joint Source Control Strategy (JSCS) was developed by the ODEQ and the United States Environmental Protection Agency (USEPA) to identify, evaluate, and control sources of contamination that may impact the Willamette River in a manner that is consistent with the objective and schedule for the Portland Harbor Superfund Site RI/FS (ODEQ and USEPA 2005). The goal of the JSCS is to achieve timely upland source control to prevent the risk of significant recontamination after the Portland Harbor cleanup is complete.

Based on the results of previous investigations, a GW SCM consisting of a slurry wall in conjunction with an oxygenation/biobarrier system has been determined as a feasible alternative for controlling the potential migration of light non-aqueous phase liquid (LNAPL) into transition zone water (TZW) and mitigating dissolved phase impacts to the Willamette River (Treadwell & Rollo 2014; ODEQ 2014a).

The GW SCM consists of a groundwater barrier wall (GWBW) to physically separate the affected upland portions of the site from the Willamette River, and an upgradient oxygenation/biobarrier system to oxygenate the aquifer and promote degradation and stabilization of LNAPL and dissolved phase contaminants. The GW SCM is currently being implemented and the GWBW was installed in Fall 2015, as approved by the ODEQ and comments provided by the USEPA. The approximate GWBW alignment is shown on Figure 2. A detailed description of the GWBW installation was presented in the Groundwater Barrier Wall Construction Completion Report (ERM 2016).

The installation of a GWBW is expected to significantly reduce the mass of contaminants leaving the uplands portion of the site and flow of oxygenated water under and around the GWBW will increase the current ongoing natural attenuation. An objective of GWBW is to provide a barrier to groundwater to allow for greater treatment on the upgradient side of the GWBW with the oxygenation/biobarrier system (the second phase of the GW SCM).

A Pilot Study Workplan for the second phase of the GW SCM, the installation of the oxygenation/biobarrier phase, will be submitted under separate cover.

1.2 REPORT OBJECTIVE

The objective of this PMP is to present the monitoring scope and rationale for evaluating the performance of the GW SCM in controlling the potential migration of LNAPL to TZW and mitigating any potential dissolved phase impacts to the Willamette River.

1.3 REPORT ORGANIZATION

The remainder of the PMP is organized as follows:

- Section 2.0 presents the information on the monitoring well locations, groundwater elevation and sample collection and analysis;
- Section 3.0 presents the adaptive management process and reporting of performance monitoring results; and
- Section 4.0 lists the references cited.

2.0 GROUNDWATER MONITORING

Key long term objectives of the GW SCM are to control the potential migration of LNAPL into TZW and to mitigate potential dissolved phase impacts to the Willamette River. The proposed performance monitoring approach will consist of regular monitoring of the hydraulic and geochemical conditions in the vicinity of the GW SCM. The monitoring results will then be used to evaluate the performance of the GW SCM.

2.1 PROPOSED MONITORING WELL LOCATIONS

The proposed performance monitoring well network will consist of a combination of existing and new groundwater wells. Clusters of wells with shallow and deep screen intervals will be installed to evaluate groundwater conditions (e.g. groundwater flow, LNAPL, and dissolved phase impacts) across the site and in the vicinity of the GW SCM.

A total of 16 new groundwater monitoring wells are proposed to be installed. Proposed monitoring locations are shown on Figure 3 and construction details provided in Table 1. Actual locations and construction may be revised depending on accessibility and field conditions encountered. The monitoring wells in the vicinity of the GWBW will be used to evaluate horizontal and vertical flow adjacent to the GWBW.

Additional upgradient wells will be used to evaluate site-wide groundwater flow conditions, LNAPL extent, and dissolved phase impacts to groundwater (Figure 3).

Shallow wells will generally be constructed so that the well screen interval intersects the anticipated water table throughout the year (generally observed at 21 feet below ground surface). Deep wells will generally be constructed with the screen interval at or below the depth of the GWBW (35 feet bgs).

Existing wells MW-26 and MW-27 were previously installed with a screened interval below the proposed depth of the GWBW (depths of 34 to 39 and 35 to 40 ft bgs, respectively) and will be incorporated into the performance monitoring program to evaluate groundwater conditions at the top of bank.

2.2 GROUNDWATER CONCENTRATION PERFORMANCE EVALUATION CRITERIA

The purpose of the GW SCM is to protect ecological and human health receptors that are impacted by any contamination potentially passing from groundwater through the TZW (i.e. porewater) and into the surface water column in the Willamette River. The ODEQ has promulgated water quality standards (WQSs) for both aquatic protection and protection of human health (through ingestion of organisms and water) in the surface water column. EPA approved the WQSs on October 17, 2011, making these effective under the Clean Water Act. Additionally, EPA published Remediation Goals (RGs) in the Record of Decision for the Portland Harbor Superfund Site, dated January 2017 (USEPA 2017), for specific Remedial Action Objectives (RAOs) that are intended to be protective of the proposed sediment remedy for Portland Harbor Superfund site.

MMGL will evaluate performance of the GW SCM by monitoring groundwater at the top of bank. The groundwater concentrations will be compared to Oregon WQSs for both aquatic protection and protection of human health (through direct contact and ingestion of organisms). Although the ultimate goal of the GW SCM is to protect the beneficial use of the Willamette River when groundwater discharges through the TZW to the water column, the effectiveness of the GW SCM will initially be evaluated by comparing groundwater concentrations at the top of the bank to these performance evaluation criteria. The EPA RGs are applicable to the TZW compliance point. The performance evaluation criteria for site specific constituents of concern (COCs) are presented in Table 2. In cases where the performance evaluation criteria are below the laboratory method detection limit, the performance evaluation criteria are taken to be

equal to the method detection limit. The performance evaluation criteria were selected using the following rationale for priority:[1]

1. Oregon WQSs from Table 30 – Aquatic Life Criteria Summary or Table 40 – Human Health Water Quality Criteria for Toxic Pollutants;
2. If an Oregon WQS is not available, the lower of RGs for Portland Harbor RAOs 3, 4, 7, and 8 was used.

Applying the above as performance monitoring criteria is conservative and so, if these criteria are met, should ensure that the applicable WQS and RGs are achieved in TZW and surface water. However, if the applicable WQS and RGs cannot be achieved at the top bank performance monitoring wells within a reasonable timeframe, it may be necessary to assess COCs closer to or at the TZW. MMGL notes that recent TZW sampling results indicated that applicable WQS and RGs are currently being achieved in the TZW (ERM 2016).

As described below, the performance monitoring wells are located upland of the top of the bank. For hardness dependent criteria, specifically manganese, the average upland groundwater hardness of 159 mg/L (ERM 2016) was used to develop the performance evaluation criteria. If the hardness dependent criteria are achieved in upland groundwater, the RAOs will also be achieved in the TZW when the TZW or surface water hardness is applied, as dilution of both the hardness and manganese occurs simultaneously as groundwater moves from upland to the TZW.

[1] MMGL objects particularly to applying a human health WQS or a RG intended for the protection of human health in surface water to groundwater, based on either direct contact or the consumption of fish and surface water, because those exposure pathways do not exist in the groundwater that will be monitored. However, MMGL will accept use of human health WQSs and RGs intended for the human health pathways (RGs based on RAOs 3 and 4) for use as performance evaluation criteria, subject to further assessment as to the risk posed if the criteria are exceeded.

2.3 PERFORMANCE EVALUATION

The USEPA has published the guidance document *A Systematic Approach for Evaluation of Capture Zones at Pump and Treat Systems* (USEPA 2008) for the development of hydraulic containment performance monitoring programs and this guidance has been generally followed to develop the monitoring program described in this PMP. Although hydraulic containment is not the objective of the GW SCM at the site, this guidance was utilized to frame the evaluation of performance of the GW SCM.

Per the guidance, six steps have been identified for systematic evaluation of the GW SCM:

1. Review site data, site conceptual model, and remedy objectives;
2. Define site-specific target zone of influence of the GW SCM;
3. Interpret water levels using potentiometric surface maps;
4. Perform calculations, and numerical modeling (if necessary);
5. Evaluate concentration trends; and
6. Interpret actual zone of influence of the GW SCM, and assess uncertainties and data gaps.

The steps outlined above encompass an approach for evaluating the performance of the GW SCM. An adaptive management approach, described in Section 3.0, will be used to determine if changes to the operation or design of the GW SCM are required to achieve the RAOs within a reasonable timeframe.

Steps 1 and 2 have been performed as part of the development of the GW SCM.

2.4 GROUNDWATER ELEVATIONS

The purpose of the groundwater elevation monitoring described in this section is to provide sufficient data to perform Step 3 (as described above), the interpretation of water levels, and Step 4, the calculations and numerical modeling, if needed.

Water level data will be used to prepare potentiometric surface maps. Manual water level measurement and potentiometric surface mapping

will be conducted periodically and will be used to evaluate the groundwater flow direction in the vicinity of the GWBW. To reduce the tidal influence of the Willamette River on the potentiometric surface map data, groundwater level data will be collected over the shortest time-frame possible, increasing the accuracy of the potentiometric surface conditions presented.

Groundwater modeling may be used as a tool, if necessary, to evaluate the groundwater flow in the vicinity of the GW SCM. Simple horizontal analyses may be performed to estimate the total groundwater flux as a line of evidence in the performance evaluation. The potentiometric surface maps will be used to evaluate flow paths and estimate overall groundwater flux.

Water level data will also be collected using transducers. Select monitoring points will have water level transducers installed to allow frequent (e.g., 1-hour intervals) measurement of groundwater elevations in designated locations. Transducers will be checked monthly and compared to manual water level measurements to assess accuracy. The groundwater elevations in these monitoring wells are likely to be influenced by seasonal and tidal fluctuations of the river. Proposed well locations for transducers are presented in Table 1.

LNAPL thickness monitoring will be initiated immediately following installation of the proposed groundwater monitoring wells. The monitoring well network will be used to evaluate potential changes to LNAPL distribution around the ends of the GWBW.

The water level and LNAPL thickness measurement frequency will be as follows:

- Construction Phase and Year 1 – Manual water level and LNAPL thickness measurements will be completed monthly to evaluate variability of groundwater flow throughout the year.
- Year 2 and beyond – Manual water level and LNAPL thickness measurements will be conducted quarterly if the corresponding groundwater elevation contour maps from Year 1 demonstrate relatively consistent seasonal conditions.

Manual water level measurements will be completed monthly for the first year of operation of the GW SCM to evaluate performance variability throughout the year and to make potential changes to optimize GW SCM

performance. Following installation of the oxygenation/biobarrier phase, the appropriate long-term water level monitoring schedule will be determined based on the first year of system operation and optimization performance data.

Performance monitoring points are indicated on Table 1.

2.4.1 Water Level Data Filtering

The water levels outside of the GWBW are anticipated to fluctuate with the tides on a daily basis. The water levels inside the GWBW are anticipated to fluctuate with a much lower amplitude and significant time lag compared to the points closer to the Willamette River. The Willamette River experiences a tidal influence from the Pacific Ocean, which produces a progressive pressure wave that propagates inland, causing groundwater levels, and therefore hydraulic gradients, to fluctuate on a daily basis.

In a system with tidal fluctuations of groundwater levels, mathematic filtering methods are used to more accurately determine groundwater elevations by filtering tidal fluctuations using a 3-day moving average (Serfes 1991). In order to provide an accurate value for the calculation of the long-term hydraulic gradient, the Serfes filtering method will be applied to all data from electronic monitoring locations (wells equipped with level transducers).

Water level data collected continuously with transducers at select wells will be used to generate hydrographs indicating changes in water elevation over long-term time intervals. Wells with transducers include three pairs of co-located shallow and deep wells outside of the GWBW and three pairs of co-located shallow and deep wells inside the GWBW. Prior to plotting hydrographs, data from each well will be processed using the Serfes filter method to correct for tidal influences. Hydrographs will be used to evaluate the potential for groundwater flux towards the river in support of the key long-term GW SCM objective, control of potential migration of groundwater contaminants to the Willamette River. The evaluation will include an assessment of vertical hydraulic gradients at the co-located shallow and deep wells.

The Serfes filtering method applies the central limit theorem to groundwater elevation measurements collected on an hourly basis, where 72 consecutive hourly measurements are available. The data will be

recorded by level transmitters and monitored remotely. These 1-hour interval readings will be used to calculate a mean hydraulic gradient. A description of the Serfes filtering method is described below:

1. 48 separate moving average elevations are calculated from sets of 24 measurements starting with measurement 1. Example: the first moving average is based on measurements 1-24, the second would be 2-25 and the 48th and last average is based on measurements 49 through 72.

2. Using the 48 mean values generated in step 2, the same process is repeated, generating 25 "means of means". Example: the first "mean of means" is based on mean value 1 through mean value 24 generated in step 2, the second mean is based on mean values 2 through 25, and the last mean is based on mean values 25-48 generated in step 2.

3. In the last step, the mean of the 25 mean values generated in step 3 is calculated. This resulting value represents the filtered mean water elevation for the 72 hour period at hour 36.

After the initial calculation, hourly mean values are generated by the method, rolling forward in time. The mean hydraulic gradient will be calculated based on the filtered water levels.

Variations (i.e., too large or too small) in the water level monitoring data will be confirmed by manual measurement of the water level. Spurious data, caused by water level sensor malfunction or calibration drift, will not be used for gradient evaluation. In these cases, the water level sensors will be replaced or repaired as necessary to monitor the long-term hydraulic gradient. Transducers will be calibrated, repaired, or replaced based on the manufacturer's guidance for accuracy and tolerance. Transducers will also be compared to manual water level measurements at least once per month.

The results of monthly potentiometric surface mapping will be used to evaluate gradient during the first 12 months of operation, in accordance with the adaptive management plan.

2.5 *GROUNDWATER SAMPLE COLLECTION AND ANALYSIS*

The installed GWBW is expected to increase the travel time of groundwater flow, which will allow for greater treatment time on the upgradient side of the GWBW following implementation of the proposed oxygenation/biobarrier phase of the GW SCM. The flow of oxygenated water under and around the GWBW is expected to increase the current ongoing natural attenuation. Monitoring the dissolved phase concentrations will be conducted to evaluate contaminant distribution and assess, in combination with the depth to water data, whether they are being impacted by the GW SCM.

Pending approval and the completion of the proposed well installation activities, quarterly groundwater samples will be collected to evaluate seasonal conditions. Quarterly groundwater sampling will continue a minimum of two years beyond the installation of the second phase of the GW SCM. A reduction to semi-annual groundwater sampling is proposed for the third year after completion of the second phase of the GW SCM. MMGL will notify the ODEQ of changes to the proposed monitoring schedule.

Performance monitoring points are indicated on Table 1. Performance evaluation criteria for each COC are presented above and on Table 2. A Sampling and Analysis Plan (SAP) is included in Appendix A.

Reductions in COC concentrations in source area monitoring wells upgradient of the GWBW are anticipated to be observed within a short time (i.e. within the first year of operation). Reductions in COC concentrations at top of bank wells are anticipated to take longer to be observed, as these wells are located downgradient of the GWBW and source area treatment locations.

Trend analyses and an evaluation of the groundwater flows will be performed after the first year of monitoring and used to assess an expected timeline when performance evaluation criteria will be achieved at the top of bank monitoring wells.

2.5.1 *Field Parameters*

Field parameters, including dissolved oxygen, conductivity, and oxidation reduction potential will be collected.

2.5.2 *Analytical Parameters*

The groundwater samples will be analyzed for the parameters identified below and presented on Table 2:

- Semi-Volatile Petroleum Products Method for Soil and Water, Modified for Silica Gel Treatment - NW-TPH-Dx (Diesel Range);
- Volatile Petroleum Products Method for Soil and Water for the Northwest - NW-TPH-Gx (Gasoline Range);
- TPH analysis for diesel and gasoline, aliphatic C10-C12 hydrocarbons by method NWTPH-EPH;
- Arsenic and Manganese by USEPA Method 6020_LL;
- Benzene, Toluene, Ethylbenzene, and Total Xylenes (BTEX) by USEPA Method 8260C Low Level;
- PAHs by USEPA Method 8270D or 8270D SIM;
- Hardness by USEPA Method 130.1;
- Total Alkalinity by USEPA Method 2320;
- Nitrate by USEPA Method E353.2; and
- Sulfate by USEPA Method SW 9056.

2.5.3 *Quality Control*

Quality control samples will be collected during the sampling events. One of each of the following control samples will be collected and analyzed for all of the parameters listed above for each sampling event:

- Equipment rinsate;
- Matrix spike/matrix spike duplicate; and
- Field (blind) duplicate.

Quality control and data validation will be conducted in general accordance with the project Quality Assurance Project Plan presented in the Phase II Remedial Investigation Work Plan (Treadwell & Rollo 2014). The Treadwell & Rollo 2014 QAPP is included in Appendix B. Petroleum product analyses (NW-TPH-Dx, NW-TPH-Gx, and NWTPH-EPH) will be conducted in accordance with Analytical Methods for Petroleum

Hydrocarbons, Washington State Department of Ecology, dated June 1997, included as Appendix C.

3.0 ADAPTIVE MANAGEMENT PROCESS AND REPORTING

This section describes the adaptive management process and performance monitoring reporting scope and schedule. The reporting phase is intended to fulfill Steps 5 and 6 of the performance evaluation.

3.1 ADAPTIVE MANAGEMENT

An adaptive management flow process has been established to verify the objectives are being achieved. The adaptive management process is summarized in Figure 4. The adaptive management process is intended to distinguish between potential system design issues and maintenance issues, as well as evaluate whether the performance evaluation criteria are appropriate for determining whether RAOs are achievable.

The adaptive management process also includes a contingency response in the event contaminant migration towards the ends of the GWBW is observed. Groundwater near both ends of the GWBW will be monitored for mounding, LNAPL, and dissolved-phase contaminants before and during oxygen delivery. During full-scale startup the oxygen delivery rate will be increased slowly. For the first month, oxygen delivery will be increased on a weekly basis until the optimum injection rate specified in the full-scale pre-final design is achieved. Targeted oxygen delivery rates will be 20% of the optimum rate the first week, 40% of the optimum the second week, 60% of the optimum the third week, and 80% of the optimum the fourth week. Oxygen delivery will be increased to 100% of the optimum oxygen delivery rate at the end of the fourth week. If LNAPL is observed migrating towards either end of the GWBW, oxygen delivery will either be decreased or stopped completely in the adjacent area in order to control migration, and the ODEQ will be notified within 24 hours of the observation. This stepwise oxygen delivery rate increase and contingency response is included in Figure 4.

Solutions to specific problems associated with equipment performance after the initial start-up period will be developed through an equipment operation and maintenance (O&M) plan. The equipment O&M plan will be submitted within six months of installation of the full scale oxygenation/biobarrier system.

3.2 PERFORMANCE MONITORING REPORTING

The results of GW SCM performance monitoring will be reported to the ODEQ quarterly following installation of the proposed groundwater monitoring wells. Monitoring results will continue to be reported to the ODEQ quarterly for the first 12 months following installation of the second phase of the GW SCM (oxygenation/biobarrier phase). These quarterly reports will include water levels, potentiometric surface maps, and evaluation of concentration trends. Long-term monitoring requirements will be determined under an adaptive management approach. MMGL will request approval from the ODEQ before modifying the reporting frequency.

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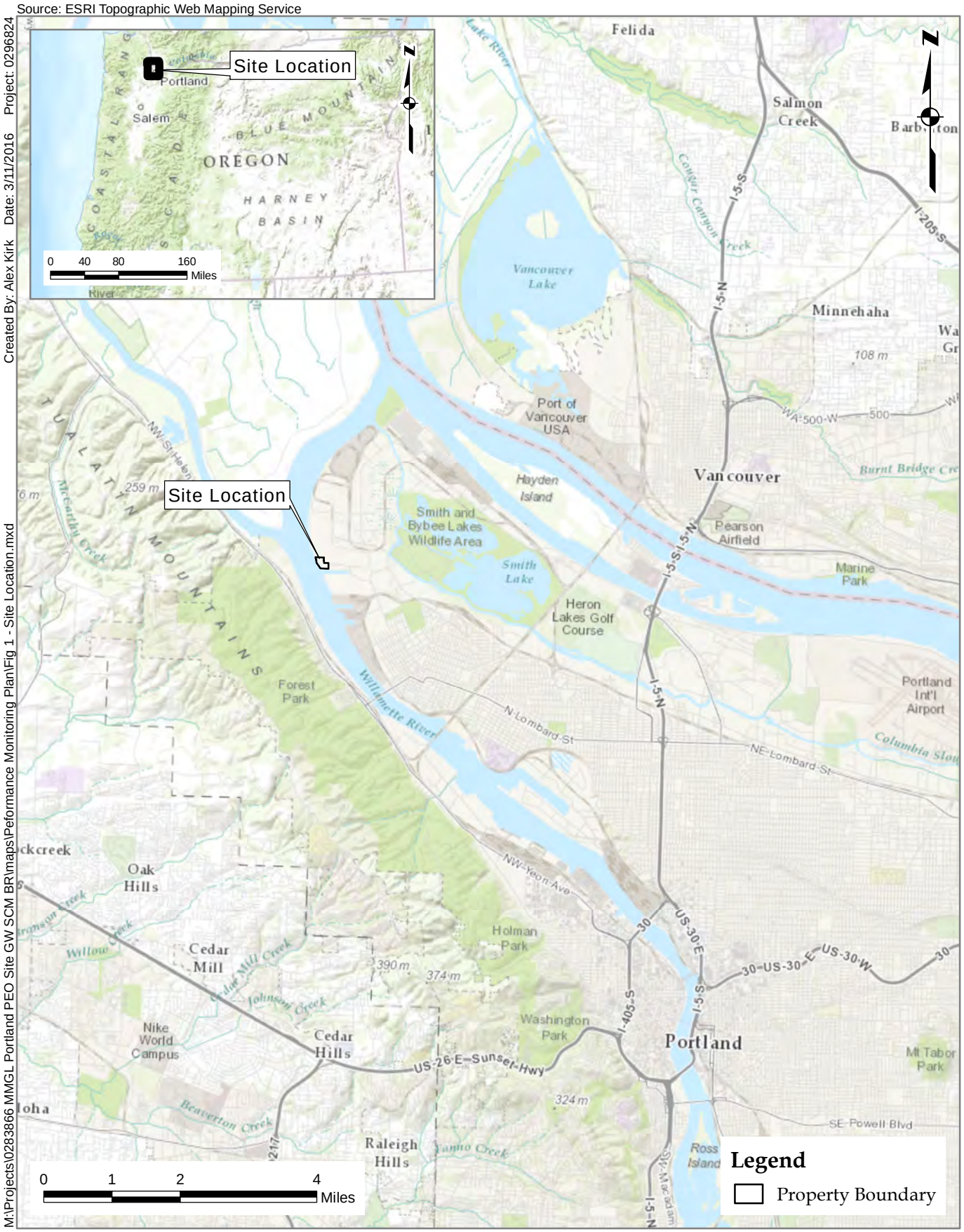

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Portland, Oregon 97204

Figure 1
Site Location
Performance Monitoring Plan
Southern PEO Site
Portland, Oregon

Legend

- Recovery Well
- Monitoring Well
- LNAPL Detected (6/3/15)
- Barrier Wall Alignment
- Ordinary High Water (20.5 ft)
- Top of Bank
- Approx. Direction of Groundwater Flow (August 2012)
- Conceptual Oxygenation/ Biobarrier Area
- Historic LNAPL Extent (Approx.)
- Historical Structures (Source: Treadwell & Rollo, 2014)
- Property Boundary
- Parcels

Notes:
LNAPL: Light Non-Aqueous Phase Liquid
ND: Not Detected
NS: Not Sampled
Monitoring occured on 6/3/2015, except for wells MW-26 and MW-27 which were sampled on 6/7/2015
Elevation data given in NAVD88
Aerial Imagery: City of Portland, flown 7/7/2012

Figure 2
GW SCM Layout
Performance Monitoring Plan
Southern PEO Site
Portland, Oregon

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Portland, Oregon 97204

ERM

Project: 0296824 Date: 9/19/2016 Created By: Alex Kirk

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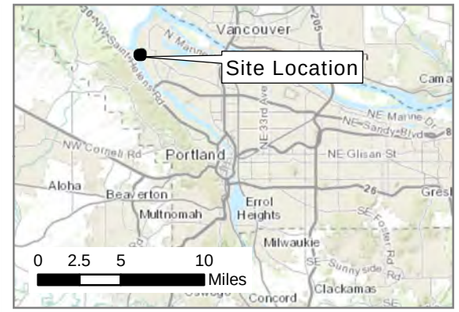

Legend

- Proposed Shallow Monitoring Well
- Proposed Deep Monitoring Well
- Shallow Monitoring Well
- Deep Monitoring Well
- Recovery Well
- Abandoned Monitoring Well
- Barrier Wall Alignment
- Ordinary High Water (20.5 ft)
- Top of Bank
- Conceptual Oxygenation/Biobarrier Area
- Property Boundary
- Parcels

Notes:
Elevation data given in NAVD88
Aerial Imagery: City of Portland, flown 7/7/2012

Figure 3
Proposed Performance Monitoring Network Layout
Performance Monitoring Plan
Southern PEO Site
Portland, Oregon

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Project: 0296824
Date: 6/27/2016
Created By: Alex Kirk
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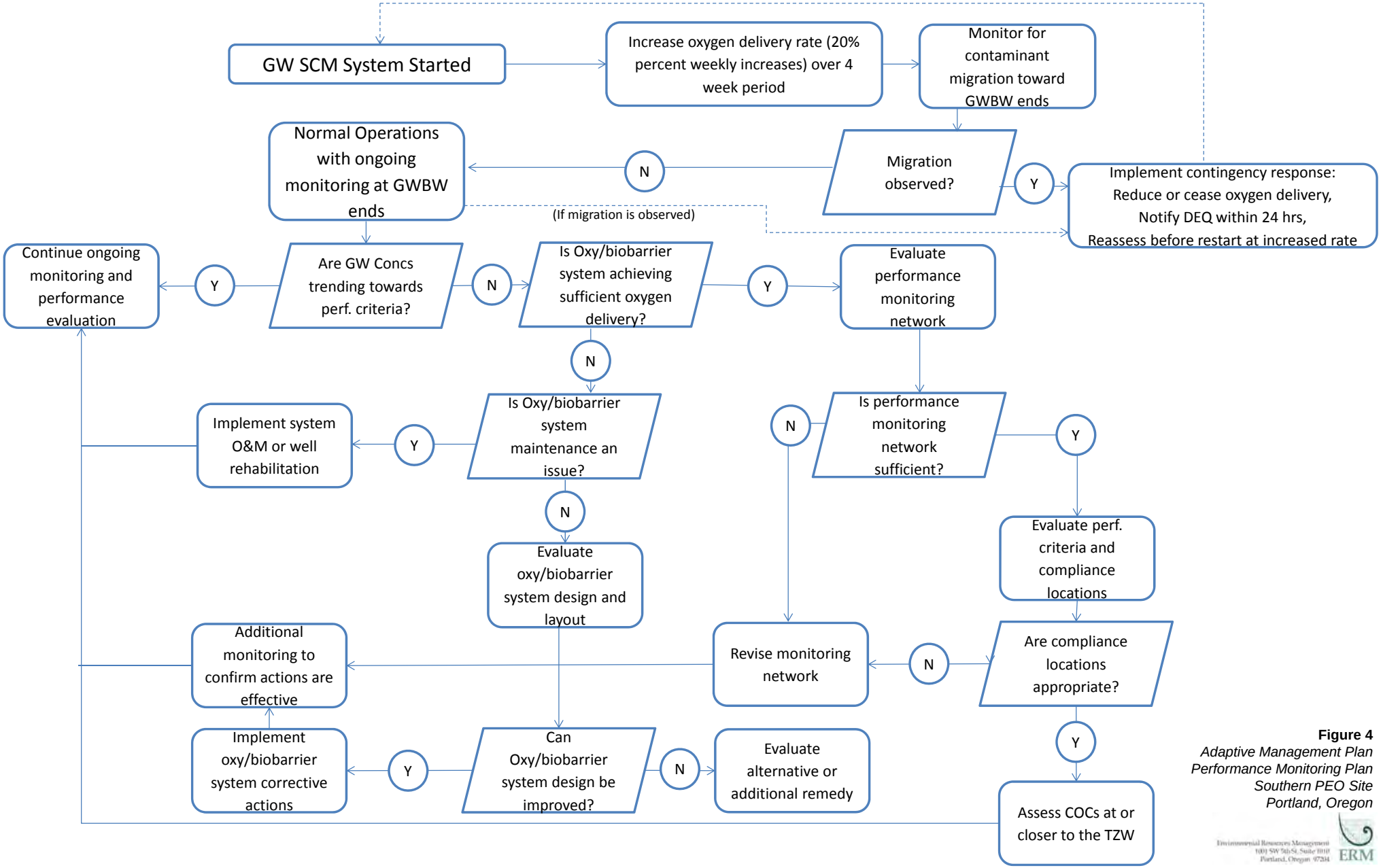

Figure 4
Adaptive Management Plan
Performance Monitoring Plan
Southern PEO Site
Portland, Oregon

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Tables

Table 1
Well Construction and Monitoring Summary
Performance Monitoring Plan
Premier Edible Oils
Portland, Oregon

Well Identification	Co-Located Shallow and Deep Well-Pair	Status	Well Depth (ft)	Screen Interval (ft bgs)		Screened Zone	Performance Monitoring Point	Transducer[1]	Compliance Point	Rationale
				Top	Bottom					
MW-02		Active	26	11	26	GW Surface	Y	N	N	LNAPL and dissolved phase impacts near GW SCM
MW-03		Active	26	11	26	GW Surface	Y	N	N	Background and upgradient conditions
MW-06		Active	27	12	27	GW Surface	Y	N	N	Background and upgradient conditions
MW-07		Active	27	12	27	GW Surface	Y	N	N	Background and upgradient conditions
MW-08		Active	27	12	27	GW Surface	Y	Y	N	Background and upgradient conditions
MW-09		Abandoned	27	12	27	-	-	N	N	-
MW-10		Abandoned	52.4	41.7	52.4	-	-	-	N	-
MW-11		Active	27	12	27	GW Surface	Y	Y	N	LNAPL and dissolved phase impacts near GW SCM
MW-12		Abandoned	27	12	27	-	-	N	N	-
MW-13		Abandoned	27	12	27	-	-	N	N	-
MW-18	X	Active	27	12	27	GW Surface	Y	Y	Y	LNAPL and dissolved phase impacts around end of GW SCM
MW-33		Installed	40	35	40	Below GWBW	Y	Y	Y	Below GWBW conditions
MW-19		Active	27	12	27	GW Surface	Y	N	N	Background and upgradient conditions
MW-21		Active	27	12	27	GW Surface	Y	Y	N	Background and upgradient conditions
MW-22		Abandoned	27	12	27	-	-	N	N	-
MW-23		Abandoned	27	12	27	-	-	N	N	-
MW-24A	X	Active	27	12	27	GW Surface	Y	Y	N	LNAPL and dissolved phase impacts around end of GW SCM
MW-42		Installed	40	35	40	Below GWBW	Y	Y	Y	Below GWBW conditions
MW-25		Active	NA	NA	NA	GW Surface	Y	N	N	Background and upgradient conditions
MW-43	X	Installed	27	12	27	GW Surface	Y	N	Y	LNAPL and dissolved phase impacts near GW SCM
MW-26		Active	39	34	39	Below GWBW	Y	N	Y	Below GWBW conditions
MW-36	X	Installed	27	12	27	GW Surface	Y	Y	Y	LNAPL and dissolved phase impacts near GW SCM
MW-27		Active	40	35	40	Below GWBW	Y	Y	Y	Below GWBW conditions
MW-28		Installed	27	12	27	GW Surface	Y	Y	N	Upgradient of GW SCM
MW-29		Installed	27	12	27	GW Surface	Y	N	N	LNAPL and dissolved phase impacts near GW SCM
MW-30	X	Installed	27	12	27	GW Surface	Y	Y	N	LNAPL and dissolved phase impacts around end of GW SCM
MW-32		Installed	40	35	40	Below GWBW	Y	Y	N	Below GWBW conditions
MW-31		Installed	27	12	27	GW Surface	Y	N	Y	LNAPL and dissolved phase impacts around end of GW SCM
MW-34	X	Installed	27	12	27	GW Surface	Y	Y	N	LNAPL and dissolved phase impacts near GW SCM
MW-35		Installed	40	35	40	Below GWBW	Y	Y	N	Below GWBW conditions
MW-38	X	Installed	27	12	27	GW Surface	Y	N	N	LNAPL and dissolved phase impacts near GW SCM
MW-37		Installed	40	35	40	Below GWBW	Y	N	N	Below GWBW conditions
MW-39		Installed	27	12	27	GW Surface	Y	N	Y	LNAPL and dissolved phase impacts near GW SCM
MW-41	X	Installed	27	12	27	GW Surface	Y	Y	N	LNAPL and dissolved phase impacts around end of GW SCM
MW-40		Installed	40	35	40	Below GWBW	Y	Y	N	Below GWBW conditions

Notes: NA = not available

1 = Wells without transducers will be gauged manually.

GWBW = Groundwater Barrier Wall

Table 2
Proposed Analytes for Groundwater Sampling
Final Performance Monitoring Plan
Premier Edible Oils
Portland, Oregon

Constituent	RAO 4	RAO 8	Portland Harbor RG[6]	Oregon WQS Table 30 Aquatic Protection	Oregon WQS Table 40 Human Health Organism Only	DEQ WQS	Selected GW SCM Performance Evaluation Criteria	Laboratory Information[5]		
	Reduce migration of COCs in groundwater to sediment and surface water such that levels are acceptable in sediment and surface water for human exposure	Reduce migration of COCs in groundwater to sediment and surface water such that levels are acceptable in sediment and surface water for ecological exposure						Analytical Method	Method Reporting Limit	Method Detection Limit
Media	Groundwater	Pore Water								
Units	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L	µg/L		µg/L	µg/L
VOCs										
Benzene	0.44	130	0.44	-	1.4	1.4	**1.4**	8260B	0.2	0.025
Ethylbenzene	68	7.3	7.3	-	210	210	**210**	8260B	0.2	0.03
Toluene	57	9.8	9.8	-	1500	1500	**1500**	8260B	0.2	0.025
Xylenes	10,000	13	13	-	-	-	**13**	8260B	0.5	0.06
Petroleum Hydrocarbons										
TPH C10-C12 Aliphatic		2.6	2.6				**2.6 (<4.1)**	NWTPH-EPH	50	4.1
TPH Gx							**1000**	NWTPHGX	50	14
TPH Dx							**1000**	NWTPHDX	110	19
SVOCs/PAHs										
2-Methylnaphthalene		2.1	-	-	-	-	**-**	8270D SIM	0.026	0.006
Anthracene		0.73	0.73	-	4000	4000	**4000**	8270D SIM	0.02	0.002
Benzo(a)anthracene	0.0012	0.03	0.0012		0.0018	0.0018	**0.0018 (<0.006)**	8270D SIM	0.02	0.006
Benzo(a)pyrene	0.00012	0.01	0.00012		0.0018	0.0018	**0.0018 (<0.006)**	8270D SIM	0.02	0.006
Benzo(b)fluoranthene	0.0012	0.7	0.0012		0.0018	0.0018	**0.0018 (<0.006)**	8270D SIM	0.02	0.006
Benzo(k)fluoranthene	0.0013	0.6	0.0013		0.0018	0.0018	**0.0018 (<0.006)**	8270D SIM	0.02	0.006
Chrysene	0.0013	2	0.0013		0.0018	0.0018	**0.0018 (<0.006)**	8270D SIM	0.02	0.006
Dibenzo(a,h)anthracene	0.00012	0.3	0.00012		0.0018	0.0018	**0.0018 (<0.003)**	8270D SIM	0.02	0.003
Fluorene		-	-		530	530	**530**	8270D SIM	0.02	0.006
Indeno(1,2,3-cd)pyrene	0.0012	0.3	0.0012		0.0018	0.0018	**0.0018 (<0.006)**	8270D SIM	0.02	0.006
Naphthalene		12	12				**12**	8270D SIM	0.02	0.003
Phenanthrene			-			-	**-**	8270D SIM	0.02	0.006
Benzo(a)Pyrene Equivalent (Calculated ND=0)	0.00012		0.00012		0.0018	-	**-**	Calculated	0.02	0.002
Metals										
Arsenic	0.018	150	0.018	360	2.1	2.1/3[1]	**2.1**	6020LL	0.2	0.1
Manganese	430	1433[2]	1433[2]		1925[3]	1925[3]	**1925**	6020LL	0.5	0.25
Additional Parameters										
Hardness			-		-	-	**-**	130.1	2000	2000
Total Alkalinity			-		20,000	20,000	**20,000**	2320	5000	5000
Nitrate					10,000	10,000	**10,000**	353.2	200	20
Sulfate					-	-	**-**	9056	1200	260

Notes:

1 = Alternate screening level to background level concentration. Site specific background level to be developed. Per personal communication with DEQ&EPA (9 September 2015), regional background levels are expected to be in the range of 0.003 – 0.005 mg/L.

2 = Alternate screening level, screened to RAO 8 for ecological exposure.

3 = Revised RAO calculated using risk based hardness-dependent critieria [RAO = $e^{(0.3331[\ln(\text{hardness})]+5.8743)}$]. Average upland groundwater hardness of 159 µg/L used to calculate RAO.

4 = Alternate screening level. Criterion is applied as hexavalent chromium.

5 = Laboratory information provided by TestAmerica

6 = Remediation goals (RGs) are from the Record of Decision, Portland Harbor Superfund Site, US EPA, January 2017

Abbreviations:

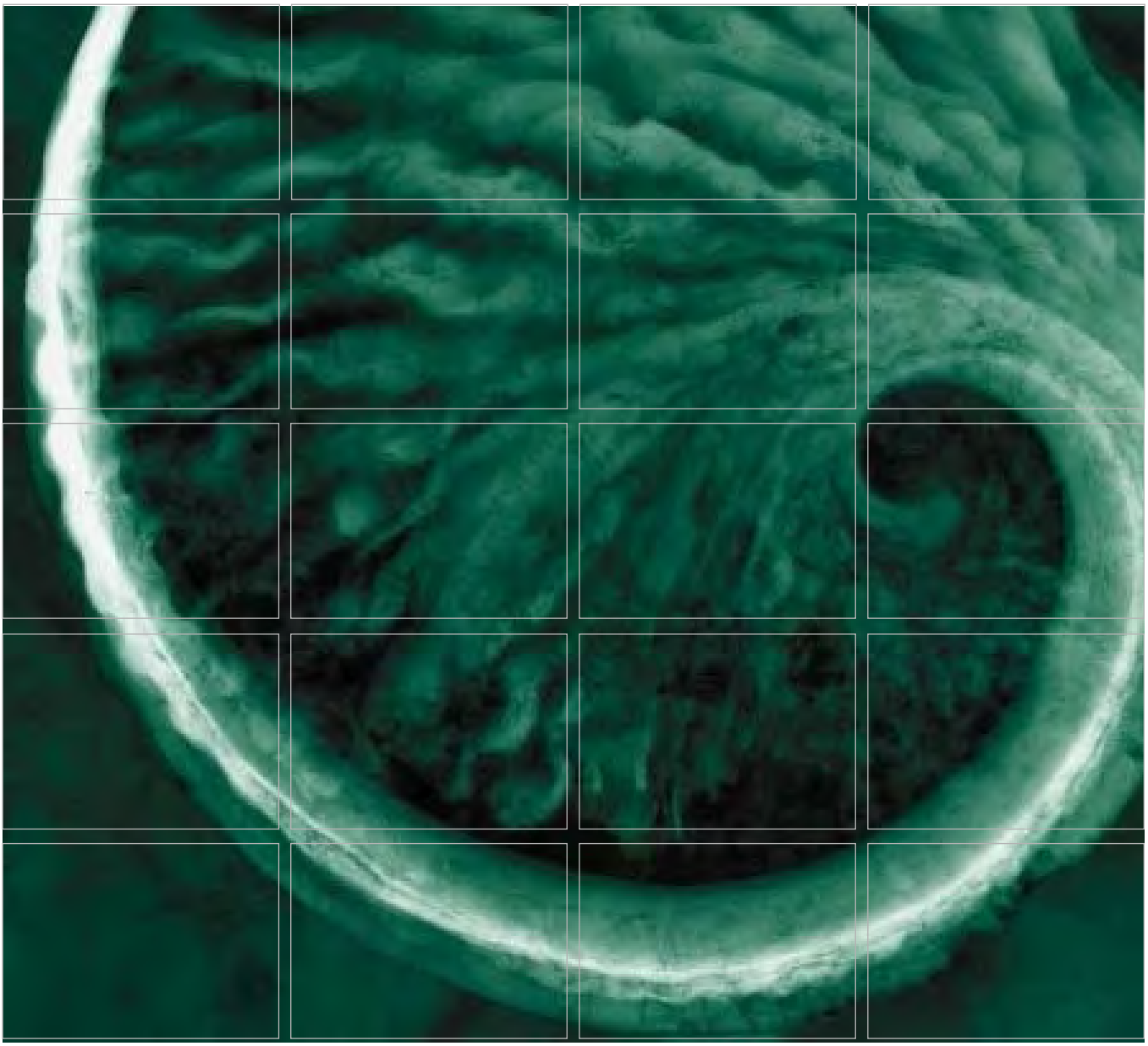
COC = Contaminant of Concern

RAO = Remedial Action Objective

< = Less than method detection limit indicated

WQS = Water Quality Standard

Appendix A
- Sampling and Analysis Plan



Sampling and Analysis Plan

Prepared for:
MMGL LLC

Premier Edible Oils
Portland, Oregon

October 2016

www.erm.com

The business of sustainability

ERM

MMGL LLC

Sampling and Analysis Plan
Premier Edible Oils
Portland, Oregon

October 2016

Project # 0283866

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TABLE OF CONTENTS

ATTACHMENTS

Attachment A - ERM Standard Operating Procedures

1.0 INTRODUCTION

ERM-West, Inc. (ERM) prepared this Sampling and Analysis Plan (SAP) in support of the Performance Monitoring Plan (PMP) for the former Premier Edible Oils (PEO) facility located at 10400 North Burgard Way in Portland, Oregon (the site). This SAP has been prepared pursuant to the Voluntary Agreement for Upland Remedial Investigation (RI)/Feasibility Study (FS) and Source Control Measures issued by the Oregon Department of Environmental Quality (ODEQ) and signed 6 March 2001 (ODEQ ECDVC-NWR-01-06) (Voluntary Agreement).

The objective of the scope of work described in this document is to evaluate groundwater conditions following the implementation of a groundwater source control measure (GW SCM) at the site.

2.0 SAMPLING AND ANALYSIS PLAN

This *Sampling and Analysis Plan* (SAP) defines procedures and data gathering methods to ensure that the data collected over the course of the project are of known quality to meet their intended use, and that all components of data acquisition are thoroughly documented, verifiable, and defensible.

A qualified ERM consultant will be onsite during all field activities. The ERM personnel will maintain a field notebook to document all work activities during the project. The field notebook will be a weather-resistant, bound, survey-type field book.

Standard Operating Procedures (SOPs) for various tasks discussed in this SAP are provided in Attachment A.

This section is organized as follows:

- Section 2.1 outlines the field program;
- Section 2.2 summarizes equipment decontamination procedures;
- Section 2.3 summarizes the field quality assurance/quality control (QA/QC) sample collection and analysis program;
- Section 2.4 describes the field documentation, sample handling, and sample analysis procedures;
- Section 2.5 outlines the project data QA/QC requirements; and
- Section 2.6 describes the containment, handling, and disposal of investigation-derived wastes.

2.1 FIELD PROGRAM

The field site characterization effort at the subject property will consist of the following investigative tasks:

- Completion of an underground utility location survey at each planned subsurface exploration location to confirm that no utilities or underground structures will be encountered or damaged during the investigation activities;
- Installation of 16 monitoring wells and collection of groundwater samples from each well; and

- Completion of a location and elevation survey of each installed well location.

A summary of each of these tasks is included in the subsections below.

2.1.1 Underground Utility Location

Prior to initiating ground disturbance activities at the subject property that have the potential for damaging underground utilities or other subsurface infrastructure, ERM will complete underground utility screening to minimize the potential encountering and damaging underground features. The screening will be completed at each planned soil disturbance location and will include the following tasks:

- Review of utility locations on existing facility as-built plans;
- Notification of the public utility "one-call" hotline at least 72 hours prior to digging;
- Completion of a private utility locate at each exploration location, to include standard utility location practices and a sweep of the area using a ground-penetrating radar (GPR) device; and
- Complete a pilot boring using hand tools or an air knife to a depth of 5 to 8 feet bgs.

2.1.2 Monitoring Well Installation and Groundwater Sample Collection

The boreholes for the monitoring wells will be advanced using hollow stem auger drilling methods, as described in the Drilling and Soil Boring SOP (Attachment A). The monitoring wells will be constructed using 2-inch diameter, threaded, Schedule 40 PVC screen and casing. Shallow wells will generally be constructed with the screen interval near the water table so that it is intersected throughout the year (generally observed at 21 feet below ground surface). Deep wells will generally be constructed with the screen interval at or below the depth of the GWBW (35 feet bgs). A sand pack consisting of 10/20 silica sand or equivalent, will be placed around the screen and extend approximately 2 feet above the top of the screen. A bentonite chip seal will be placed in the three feet above the sand pack and a cement-bentonite grout will be placed on top of the bentonite chips to a depth of approximately 2 to 3 feet bgs. The remainder of the monitoring well borehole annulus will be filled with concrete and finished with a flush-with-grade protective monument. The wells will be equipped with expandable locking caps.

Following construction, the wells will be developed to settle the soil around the screened interval and remove particulates from the sand pack. The procedures

for well development are outlined in ERM's Monitoring Well Development SOP (Attachment A).

Groundwater samples will be collected from the monitoring wells at least 24 hours after development activities are completed using the low-flow methods outlined in ERM's SOP Groundwater Monitoring in Attachment A. The low-flow groundwater purging/sampling technique employs the use of a flow-through cell, equipped with a meter for measuring groundwater quality parameters such as pH, temperature, specific conductivity, dissolved oxygen, and oxidation/reduction potential.

2.1.3 Location and Elevation Survey

After completion of the borings and monitoring wells, a licensed land surveyor will survey the location and ground surface elevation of each boring and well, and top of casing elevation of each well. The ground surface elevation and horizontal position of each of the wells, and the elevation of the highest point on the rim of each well casing, will be determined utilizing the local NAVD88 vertical system and horizontal NAV83 system. Horizontal locations will be established to an accuracy of 0.1 foot and elevations will be established to an accuracy of 0.01 inch.

2.2 DECONTAMINATION PROCEDURES

Decontamination of non-disposable sampling equipment that comes in contact with samples (such as pumps) will be performed to prevent the introduction of extraneous material into samples, and to prevent cross contamination between samples. All non-disposable (non-dedicated) sampling equipment will be decontaminated by washing with an aqueous solution of a non-phosphate detergent such as Liquinox™ or equivalent. Drilling equipment, such as drill rods and augers, will be decontaminated by high-pressure steam washing.

Decontamination water will be collected in appropriate 55-gallon drums and will be handled and disposed along with the other investigation-derived wastes as described below.

2.3 QUALITY ASSURANCE/QUALITY CONTROL SAMPLES

Field QA/QC samples will be collected and analyzed during the project to assess the consistency and performance of the sampling program. Field QA/QC samples for this project will include trip blanks, field blanks, field duplicates, and equipment rinsate blanks. Additionally, accommodation for laboratory preparation and analysis of matrix spikes/matrix spike duplicate (MS/MSD) sample analyses will be made. The laboratory will also measure the temperature

of the samples in each cooler upon delivery to the laboratory. Field QA/QC samples will be collected from the subject property during each groundwater sampling event, in general accordance with the *DEQ's Quality Assurance Policy for the Environmental Cleanup Programs (last updated July 31, 2015*).

2.3.1 Trip Blanks Samples

Trip blank samples will be provided by the laboratory with each sample container shipment and will consist of two or three 40-milliliter volatile organic analysis (VOA) vials filled with deionized water. The purpose of trip blank samples is to evaluate the potential for *ex situ* volatile organic vapors to affect sample results for gasoline-range petroleum hydrocarbons (TPH-G) and VOCs. One trip blank will be included in each cooler containing samples slated for TPH-G or VOC analysis.

2.3.2 Field Duplicate samples

Field duplicate samples consist of duplicate samples of the same matrix as the original samples collected at the same time and location, to the extent possible, using the same sampling techniques. The purpose of field duplicate samples is to evaluate the variability of the in the laboratory analytical method results. Field duplicate samples will be analyzed for the same constituents as the associated original samples.

2.3.3 Equipment Rinsate Blank Samples

Equipment rinsate blank samples are used to evaluate the effectiveness of the decontamination procedure and to identify potential cross-contamination during sampling events. Equipment rinsate samples will be collected by pouring deionized water over the decontaminated sampling equipment (e.g., pump) and collecting the rinsate directly into the sample containers. The rinsate samples will be analyzed for the same constituents as the associated samples collected with that sampling tool.

2.3.4 Matrix Spike and Matrix Spike Duplicate

The laboratory will analyze a MS/MSD. Sufficient sample volumes will be collected and submitted to the laboratory to allow the laboratory to select an appropriate sample from each batch for MS/MSD evaluation.

2.3.5 Temperature Blanks

The laboratory will measure and record the temperature of the samples in the cooler immediately upon receipt of the samples.

2.4 *FIELD DOCUMENTATION, SAMPLE HANDLING, AND SAMPLE ANALYSIS*

This section describes the procedures for documentation and sample management in the field, including field documentation, sample documentation, sample packaging and shipping procedures, and sample analysis.

The groundwater samples will be analyzed for the parameters as summarized in the PMP, using the analytical methods indicated. Groundwater samples collected from the subject property will be packed in coolers with ice and transported under standard chain-of-custody protocol to an accredited analytical laboratory. The laboratory analysis reports will be returned to ERM on a standard schedule, which is typically 10 to 15 business days, depending on the laboratory selected.

Specific documentation, handling, and analysis procedures related to the samples are described in the subsections below.

2.4.1 *Sample Identification, Numbering, and Labeling*

Sample labels will be filled out with indelible ink and affixed to each sample container. If non waterproof labels are used, then each sample label will be covered with clear tape to keep it dry. Sample containers will be placed in re-sealable plastic bags to protect the sample from moisture during transportation to the laboratory. Each sample container will be labeled with the following, at a minimum.

- Sample identification;
- Sample collection date;
- Time of collection;
- Project number;
- Sampler's initials;
- Analysis to be performed;
- Preservative used (if any); and
- Project location.

Samples submitted to the analytical laboratory will be uniquely identified based on the name of the location from which it is collected.

2.4.2 Field Documentation

Data collection activities performed at the subject property will be documented in field notebooks and/or on COC records using waterproof, indelible ink. Entries will be as detailed and as descriptive as possible so that a particular situation can be recalled without relying solely on the sampler's memory. Field log entries will be dated and signed. Information entered in the field notebook will include, at a minimum, the following items:

- Project name and number;
- Dates and times of entries;
- Weather conditions;
- Names of personnel performing the activities;
- Subcontractors and vendors on site;
- A description of daily activities;
- A description of sample locations, including sample name and type;
- Depths of samples if relevant;
- Sample descriptions (including odor and staining);
- Sample collection methods;
- Preservatives (if appropriate);
- Parameters for analysis;
- Field instrument calibration information;
- Field instrument readings; and
- Health and safety information.

Field notebooks will be stored in ERM's project file when not in use. In addition, digital photographs will be taken to document field activities.

At the beginning of each daily entry, the date, start time, weather, names of all sampling team members present, and the signature of the person making the entry will be entered. The names of visitors to the subject property, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Whenever a sample is collected, a detailed description of the sample collection location shall be recorded. A description of any photographs taken at the subject property will also be noted.

The equipment used to collect samples will be noted, along with the time of the sampling, sample description, volume, and number of containers. Decontamination procedures will also be recorded. Field QC samples collected will also be recorded documenting the location and time of the sample collection.

2.4.3 Chain-of-Custody Procedures

A chain-of-custody (COC) record will be completed for every sample. In addition to providing a custody exchange record for the samples, the COC record serves as a formal request for sample analyses. After completion of the COC, one copy will be retained by the sample coordinator for project files and the original will be sent to the analytical laboratory with the sample shipment.

The COC record will be the controlling document to ensure that sample custody is maintained. The COC record will be initiated in the field by sampling personnel upon collecting a sample. Each individual who has the sample(s) in his/her possession will sign the COC. Each time the sample custody is transferred, the former custodian will sign the COC on the "Relinquished by" line, and the new custodian will sign the COC on the "Received by" line. The date, time, and the name of their project or company affiliation will accompany each signature.

After the laboratory receives the samples, the sample custodian will inventory each shipment before signing for it, and note on the original COC record any discrepancy in the number of samples, temperature of the cooler or broken samples. The ERM Project Manager will be notified immediately of any problems identified with shipped samples, and will determine the appropriate course of action. The shipping container will be secured with a custody seal, thereby allowing for custody to be maintained by the shipping personnel until receipt by the laboratory.

The laboratory will initiate an internal COC that will track the sample within the various areas of the laboratory and subcontracted laboratories. The relinquishing signature of the sample custodian and the custody acceptance signature of the laboratory personnel transfer custody of the sample. This procedure is followed each time a sample changes hands.

2.4.4 Sample Preservation, Packaging, and Shipment

After collection, samples will be immediately labeled and stored in a chilled cooler with ice or a frozen ice pack to maintain a temperature of 4°C ±2°C.

The shipping of samples to the analytical laboratory, by land delivery services, will be performed according to the DOT regulations. Transportation methods will be selected to ensure that the samples arrive at the laboratory in time to permit testing according to established holding times and project schedules. No samples will be accepted by the receiving laboratory without a properly prepared COC record and properly labeled and sealed shipping container(s).

Packaging of sample containers will be based on the level of protection a sample will require during handling, shipping, and storage. Protection may vary according to sample type, sample media, suspected amount of hazardous substances, required testing, and handling and storage conditions. Proper packaging will include:

- **Inner packing**: plastic bags, shock-absorbing packing material, and ice for preservation;
- **Over packing**: Metal or plastic coolers;
- **Over pack sealing**: Strapping tape and custody seals; and
- **Marking and labeling of over pack**: Laboratory address, any appropriate DOT Hazard Class Labels, and handling instructions.

Before sample collection, sample labels will be affixed to each sample container. If non-waterproof labels are used, each sample label will be covered with clear tape to keep the label dry. Sample bottles will be placed in a re-sealable plastic bag to keep the container dry. Glass sample containers will be protected with bubble wrap or other protective packaging material. A temperature blank will be placed in every cooler with samples.

The COC will be filled out and a copy of the COC form will be retained for documentation. Samples will be packed in a sample cooler with ice in sufficient quantity to keep the samples cooled to 4 ^{o}C ±2 ^{o}C for the duration of the shipment to the laboratory. Saturday deliveries will be coordinated in advance with the laboratory.

The COC form will then be taped to the inside of the sample cooler lid. Cooler drain spouts will be taped from the inside and outside of the cooler to prevent any leakage. The cooler will be taped shut with strapping tape, and a custody seal will be taped across the cooler lid. Clear tape will be applied to the custody seals to prevent accidental breakage during shipping. The samples will then be

shipped to the analytical laboratory. A copy of the courier air bill will be retained for documentation.

2.5 PROJECT DATA QUALITY ASSURANCE AND QUALITY CONTROL REQUIREMENTS

This section summarizes the protocols put in place to ensure the quality of the field and laboratory data generated as part of this project.

2.5.1 Laboratory Data Validation/Review

ERM will perform a level II data review of the analytical data reports. The data review will be in accordance with the USEPA Contract Laboratory Program National Functional Guidelines for Data Review and the QA/QC criteria specified in this document. Data will be reviewed and flagged with the appropriate data qualifiers.

Based on laboratory data validation/review, a qualified ERM scientist will determine if the QA criteria have been met, and will establish and document data usability.

2.5.2 Record Keeping

The project file will include copies of the *Sampling and Analysis Plan* which document the proposed collection and sample analytical approaches. Additionally, records that document any departures from the plan (such as site logbooks) will be maintained in the project file. The results of all analyses, including laboratory reports and summary tables or interpretive reports, will also be retained.

The contract laboratory will submit analytical data in both hard-copy format and as electronic data deliverables (EDD).

2.6 INVESTIGATION-DERIVED WASTE

Investigation-derived waste (i.e., development, purge and decontamination water, and soil cuttings) from the drilling and sampling activities will be collected in 55-gallon drums. The drums will be labelled with the content source and type, date of accumulation, and ERM contact information. ERM will develop disposal recommendation based on the analytical results for the associated samples and will assist MMGL in disposal of the waste in accordance with the State and Federal regulations.

Attachment A
ERM Standard Operating Procedures

EQUIPMENT DECONTAMINATION

Equipment decontamination is the process by which potential contaminants are removed from reusable equipment between sampling locations and between sites to minimize the potential for cross-contamination of sampled media.

TASK-SPECIFIC EQUIPMENT AND SUPPLIES

- Plastic sheeting.
- Three 5-gallon buckets.
- Brush.
- Tap water.
- Distilled or deionized water.

STANDARD EQUIPMENT DECONTAMINATION AREA SETUP

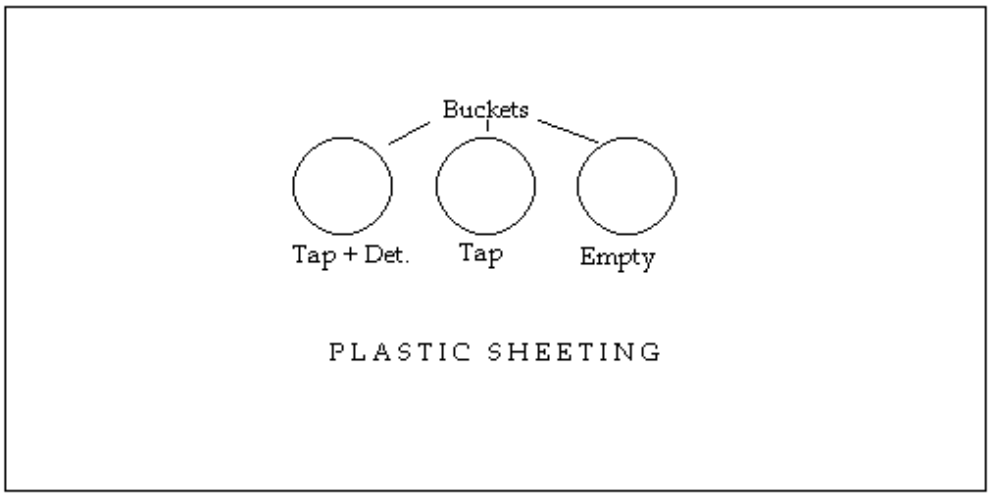
Different projects may have different equipment decontamination requirements and procedures; however, the method described below should be considered a minimum standard for reusable equipment decontamination when procedures are not specified in the proposal, work plan, or field briefing documentation. Note that single-use supplies, such as disposable bailers or tubing, do not require decontamination prior to use.

Larger equipment, such as power drilling tools and excavator buckets, are typically decontaminated using high-pressure, hot water wash (i.e., steam cleaning) supplied by the operating contractor. Procedures for this decontamination method are summarized in a separate section below.

- Place plastic sheeting on the ground or floor in the decontamination area. The sheeting should cover an area approximately 6 feet by 10 feet.
- Fill two buckets with an appropriate amount of tap water (warm or hot water, if possible).
- Place an appropriate amount of laboratory-grade detergent in a bucket containing tap water.

- Place the second tap-water containing bucket to the right of the bucket with the detergent.

- Place the third, empty, bucket to the right of the second bucket. The decontamination area should look something like this:



- Place equipment to be decontaminated to the left of the Tap + Detergent bucket.

- Complete equipment wash and rinse using the following procedures:

    - Wash equipment in tap + detergent water, using brushes to remove soil and residue;

    - Rinse equipment in tap water in second bucket; and

    - Hold equipment over third bucket and pour distilled/deionized water over the equipment for a final rinse, taking care not to let the equipment contact the water collected in the bucket.

- Place decontaminated equipment to the right of the third bucket.

- Dispose used decontamination water to an approved container, such as a DOT-approved drum. Container should be labeled on the side (not lid) with

date(s) of accumulation, contents, source of contents (e.g., "decontamination water"), and a unique number in sequence with other investigation-derived waste containers at the site. This information should be recorded in a drum log in the field notes.

- Change wash and rinse water regularly, especially if water becomes turbid from soil.

SPECIAL CONSIDERATIONS

- Decontamination procedures may require some modification of personal protective equipment to address issues such as splash. Evaluate work conditions and take appropriate steps to protect yourself from potential exposure routes specific to the task at hand.

- Equipment that has come into contact with high concentrations of contaminants or free product should not be decontaminated in the same decontamination process area as equipment that does not come into contact with high contaminant concentrations. Equipment that has come into contact with high concentrations of contaminants should be reported to the project manager, and should not be re-used until an appropriate procedure has been implemented to mitigate the increased chance of cross-contamination related to use of that equipment. Except for oil/water interface probes, equipment that comes into contact with high levels of contamination should not be used on other sites without the approval of both the project manager for the site at which exposure occurred and the manager of the subsequent project site.

LARGE EQUIPMENT DECONTAMINATION PROCEDURES

Drillers and heavy equipment subcontractors typically use high-pressure hot water wash, or steam cleaning, to decontaminate equipment such as auger flights, drill casing, and backhoe/excavator buckets. This decontamination is typically completed by subcontractor personnel, but should be observed and documented by the ERM site representative to ensure that the coverage and duration of the steam cleaning effort is sufficient to remove possible contaminants from the portions of the equipment that may contact subsequent soil, groundwater, or other sampled media.

Note that equipment should be completely free of residual soil or staining prior to initiating drilling, excavation, or sampling at each specific location.

GROUNDWATER SAMPLING – MONITORING WELLS

APPLICATION

Groundwater sampling at monitoring wells is typically completed to evaluate contaminant concentrations, groundwater geochemistry, and/or groundwater physical properties to provide data helpful to understand the nature and extent of contamination, to evaluate contaminant fate and transport, and to assist in the selection and design of appropriate remedial measures. Groundwater samples are typically collected after a prescribed volume of water has been removed ("purged") from the well, or after groundwater physical parameters (at a minimum, temperature, specific conductivity, and pH) have stabilized within established limits.

TASK-SPECIFIC EQUIPMENT AND SUPPLIES

While not a complete list of everything you might need for a specific project, the equipment and supplies listed below are a minimum required for completion of groundwater monitoring. Additional materials may be required to complete tasks as indicated in the proposal, work plan, and/or field briefing.

Minimum required equipment and supplies for groundwater monitoring:

- Health and safety equipment and supplies per the project HASP
- Table or surface for sample handling
- Plastic sheeting
- Heavy-duty paper towels
- Heavy-duty trash bags
- Sealable plastic bags ("Ziplocs")
- Fine-tipped permanent markers ("Sharpies")
- Water level indicator
- Well purging and sampling forms
- 5-gallon plastic buckets

- Laboratory-prepared sample containers
- Distilled/deionized water
- Primary sampling apparatus (submersible pump, peristaltic pump, or bailers) and associated equipment:
    - Submersible or peristaltic pump – Power source, power cables, discharge tubing (Note: Low-flow sampling requires a pump with an adjustable flow rate)
    - Bailers and nylon rope
- Backup sampling apparatus, at a minimum bailers and nylon rope
- Water quality meter(s) with a minimum of temperature, specific conductivity, and pH capability
- Water quality meter field calibration supplies
- In-line water filters, if necessary
- Tools for opening and cleaning well protective casings, including a tool to remove water from flush-mount casings (an empty plastic soda bottle works well for most protective casings)
- Well lock keys
- 55-gallon drums to contain waste water
- Summary of well construction details
- Sample packing equipment and supplies (see Sample Handling and Shipping SOP)
- Decontamination supplies (see Equipment Decontamination SOP)

PRE-SAMPLING PROCEDURES

Prior to initiating groundwater sampling, the following procedures apply:

- Comply with ERM health and safety procedures and the requirements in the HASP (e.g., communicate risks to subcontractors, complete daily safety briefings, evaluate equipment conditions, etc).

- Confirm well and sample collection locations per the proposal, work plan, and/or field briefing.

- Use available data to determine an appropriate sequence for well monitoring, from least to most contaminated, and follow this sequence when collecting water levels and when sampling.

- Confirm access to monitoring well locations.

- Measure water levels in all site wells, including those not slated for sampling. Water level measurements should be completed in a single day with as little interruption as possible.

- Calibrate water quality monitoring instruments and record equipment calibration procedures and results in the field notes or on an equipment calibration form.

GROUNDWATER SAMPLING PROCEDURES

Groundwater sampling is typically completed in two stages, well purging and sample collection. Well purging involves removing water from the well casing to allow water entrained in the formation surrounding the well to flow into the casing. Sample collection involves filling appropriate containers with groundwater for laboratory analysis.

Well Purging

Two general types of well purging are typically used prior to sample collection – standard and low-flow. Standard sampling involves the removal of at least three well casing volumes of groundwater prior to sample collection, while low-flow sampling relies on laminar flow of groundwater out of the formation to minimize the volume of water evacuated from the well prior to sample collection. Both purging methods are described below. The well purging method should be confirmed by the ERM project manager, or described in the proposal, work plan, or field briefing. If a purging method is not specified, standard purging procedures should be used.

For both methods, plastic sheeting should be placed on the ground surface in the area that sampling equipment and supplies will be staged and used to limit the potential for inadvertent contamination by contact with the ground surface.

Standard Purging Method

Standard groundwater purging prior to groundwater sample collection is completed to ensure that the sample is collected from formation water that is not affected by potential local groundwater geochemical anomalies caused by the well (e.g., increased dissolved oxygen). This method is typically applicable for sampling any groundwater monitoring well. Unless otherwise indicated in the proposal, work plan, or field briefing, the following procedures apply to standard purging in preparation for groundwater sample collection:

- Calculate and record the volume of water in each well casing using the following equation:

  $$V_c = (D_c - D_w) * (V_f)$$

  Where:
  V_c = Volume of water in the well casing
  D_c = Depth of well from the top of the well casing
  D_w = Depth of groundwater from the top of the well casing
  V_f = Volume of water per foot of well casing

  Note: 2-inch-diameter wells = 0.16 gallons per foot
  4-inch-diameter wells = 0.65 gallons per foot

- Prepare sampling equipment by decontamination of reuseable materials per the Equipment Decontamination SOP and by dedicating fresh, unused disposable supplies, such as tubing or a bailer and rope.

- If used, attach flow-through cell for groundwater parameter measurement to the discharge tubing.

- Using a submersible pump, peristaltic pump, or bailer, remove at least three well casing volumes of water from the monitoring well, discharging the purge water into 5-gallon buckets or other container. Bailers and pumps should be lowered gently into the well to minimize groundwater disturbance. If a pump is used, the intake should be set at approximately the midpoint of the well screen, except in low-yield wells. For low-yield wells (i.e., wells where groundwater recovery is interrupted and water level does not readily recover) follow the procedure in the next bullet.

- For low-yield wells (typically producing less than 1 liter per minute), the pump intake should be lowered as deep as possible into the well without disturbing sediment at the bottom of the well casing. The well should be purged until groundwater is no longer available for purging. The well should then be allowed to recover as much as possible (but not longer than 8 hours). After the water level in the well has recovered, the required samples will be collected with the pump intake placed near the middle of the screened interval.

- At least once each well casing volume, measure and record, at a minimum, temperature, specific conductivity, and pH of the water. Note that project-specific groundwater parameter monitoring requirements should be followed, if available. (Note: Dissolved oxygen and reduction/oxidation potential measurements should never be completed on an ex-situ sample that has been exposed to ambient air, and should only be completed using a downwell probe or a flow-through cell where the purge water is not exposed to air).

- After three well casing volumes of water have been purged from the well, confirm that the following parameters have stabilized to within the following ranges during the most recent two measurements:

  - Temperature: + 1 degree Celsius;

  - pH: + 0.1 units; and

  - Specific conductance: + 10 percent.

  If parameters are not stabilized, purge an additional well volume of water from the well and measure the parameters again. Repeat this process until groundwater parameters are stable within the indicated range.

- Record in the field notes the time of start and end of purging, the volume of water purged, and any observations made during sampling (e.g., well damage, water odor or color).

Low-Flow Purging Method

This procedure describes the standard method and equipment used to perform low-flow groundwater sampling. The techniques described in this procedure are in general agreement with the procedures outlined in the United States Environmental Protection Agency publication entitled "Low-Flow (Minimal Drawdown) Ground-Water Sampling Procedures" (Puls and Barcelona 1995).

Certain states also have state-specific low-flow groundwater sample procedures, which should also be consulted prior to conducting low-flow sampling for any application where results may be reported to a state regulatory agency.

The low-flow (minimal drawdown) groundwater sampling procedures are used to facilitate the collection of representative groundwater samples and offer the following advantages over the standard procedure described above:

- The water column in the well experiences minimal disturbance during the purging and sampling procedure;

- The volume of purge water to achieve stabilization parameters is greatly reduced; and

- The work effort associated with field decontamination of sampling equipment is greatly reduced.

Note that low-flow sampling techniques should not be used in the following conditions:

- Groundwater yield is primarily from segregated and discontinuous zones across the screened interval (e.g., bedrock fractures or coarse-grained beds within a glacial till unit); and/or

- Groundwater yield will not allow sustained pumping at a minimum of 100 milliliters per minute with a resultant water level drawdown of less than 20 centimeters.

Unless otherwise indicated in the proposal, work plan, or field briefing, the following procedures apply to low-flow purging in preparation for groundwater sample collection:

- Attach a fresh length of disposable polyethylene (or equivalent) tubing to the outlet of the decontaminated pump.

- Attach flow-through cell for groundwater parameter measurement to the discharge tubing.

- Lower the pump slowly into the well to minimize the mixing of casing water and the suspension of any silt at the bottom of the well, placing the pump intake near the middle or slightly above the middle of the screened interval.

- Purge the well at 100 to 500 milliliters per minute; the goal is to minimize drawdown in the well (ideally less than 10 centimeters drawdown). Remove air bubbles from the pump discharge tubing or in-line flow cell as soon as possible after purging begins.

- During purging, monitor, using the in-line flow cell, purge water temperature, specific conductance, and pH every 3 to 5 minutes. Note that these parameters are a minimum requirement. Confirm project-specific monitoring requirements with project documentation and/or ERM project manager.

- Stop purging when the following parameters have stabilized for three successive readings:

  - Temperature: + 1 degree Celsius;

  - pH: + 0.1 units; and

  - Specific conductance: + 10 percent.

  If parameters are not stabilized, continue purging water from the well and measure parameters until stabilization is verified within the indicated ranges.

- Record in the field notes the time of start and end of purging, the volume of water purged, and any observations made during sampling (e.g., well damage, water odor or color).

Groundwater Sample Collection

After purging has been completed as outlined in the section above, groundwater samples should be collected from the monitoring well using the following procedure:

- Fill out sample labels completely on laboratory-provided sample containers using a fine felt-tipped marker (IMPORTANT: Do not use ball-point pen, as the ink may wash off).

- Collect groundwater samples into laboratory-provided sample containers directly from the bailer or pump discharge tubing. If an in-line flow cell is used, detach flow cell prior to sample collection. Take care not contact the sample containers with any part of the sampling equipment or supplies (e.g., don't place the pump discharge tubing end in the sample container). Note

that sampling for volatile organic compounds using a peristaltic pump should be avoided.

- Place the groundwater samples in sealable plastic bags, and then into a cooler and log them on the chain-of-custody (COC) form. See the Sample Handling SOP for details on managing, documenting, handling, and shipping of the samples.

- Record the time of sampling in the field notes.

Note that for filtered samples, the filter should be threaded into the end of the pump discharge for sample collection. If a filtered sample is required for a well sampled using a bailer, the bailed water should be placed in a clean laboratory-supplied glass container (e.g., a 1-liter amber bottle) and transferred through a filter using a peristaltic pump. Note that the glass container should not be reused.

Sample Preservation

Note that sample bottles for various analyses will be provided by the laboratory with preservative materials (e.g., hydrochloric acid or nitric acid) included in the container. Take care not to spill the preservative from the bottle prior to sampling, and minimize any bottle overfills that may dilute the preservative. Also, if a bottle contains or has contained preservative, it should never be used to collect a sample that should be unpreserved (e.g., an unfiltered dissolved metals sample destined for laboratory filtration).

If sample preservatives are not included in the sample bottles and must be added in the field, the procedure should be established in project documentation or by discussions with the ERM project manager prior to the field effort.

EQUIPMENT DECONTAMINATION AND WASTE HANDLING

After sample collection is complete at each well, follow the following procedures to complete equipment decontamination and waste containment:

- After completion of sampling at each well, discard disposable equipment and complete reusable equipment decontamination per the Equipment Decontamination SOP or project-specific requirements.

- Place purge water and decontamination water in an appropriate container (55-gallon drum). Container should be labeled on the side (not lid) with date(s) of accumulation, contents, source of contents (e.g., boring number),

and a unique number in sequence with other investigation-derived waste containers at the site. Note that for some sites may require segregation of water from certain wells into specific drums because of disposal requirements or other considerations. Confirm appropriate waste segregation requirements before placing waste in holding containers.

- Complete a drum log in the field notes that includes the unique number, contents, dates of accumulation, and sources of contents.

HOLLOW-STEM AUGER DRILLING

Hollow-stem (HSA) drilling is completed using a truck- or track-mounted rotary drilling rig operated by professional drilling subcontractor. Boreholes are advanced using a power head that rotates flighted augers while simultaneously applying downward pressure. A drill bit at the bottom of the augers breaks up the soil, and the auger flights return the soil to the surface.

Soil samples are typically collected by driving a split-spoon sampler ahead of the drill bit once the drill bit is advanced to the planned sampling depth. The split-spoon sampler consists of a cylinder of metal halved in the long axis, held together during sampling by a threaded drive shoe at one end and the threaded drill rod attachment at the other. The sampler is lowered on drill rod through the hollow center of the auger, and a power hammer is used to drive the sampler into the soil. During drilling, a metal plug is threaded onto the end of the drill rod to prevent soil from filling the auger interior.

APPLICATION

HSA drilling is typically completed in unconsolidated soils to evaluate subsurface conditions and install monitoring wells. HSA drilling should be avoided in the following conditions:

- Bedrock, cobbles, and/or boulders are present or expected in soils within the target drilling interval;
- Target drilling depth is greater than 100-150 feet below ground surface (bgs);
- Drilling will penetrate aquitards or other features that are isolating shallower contaminated soils and/or groundwater from deeper intervals; or
- An artesian aquifer will be encountered.

TASK-SPECIFIC EQUIPMENT AND SUPPLIES

While not a complete list of everything you might need, the equipment and supplies listed below are a minimum required for completion of HSA soil borings. Additional materials may be required to complete tasks as indicated in the proposal, work plan, and/or field briefing.

Minimum required equipment and supplies for HSA drilling:

- Health and safety equipment and supplies per the project HASP
- Table or surface for sample handling
- Plastic sheeting
- Heavy-duty paper towels
- Heavy-duty trash bags
- Photoionization detector (PID) and calibration equipment
- Water level indicator
- Soil knife or small trowel
- Sealable plastic bags ("Ziplocs")
- Fine-tipped permanent marker ("Sharpies")
- Unified Soil Classification System summary
- Drill logs and well completion forms
- 5-gallon plastic bucket for waste soil
- Shovel or tool for collecting soils from utility clearance excavation
- Laboratory sample containers
- Distilled/deionized water
- Sample packing equipment and supplies (see Sample Handling and Shipping SOP)
- Decontamination supplies (see Equipment Decontamination SOP)

Also, it is important to ensure that the drillers are bringing the proper equipment and supplies for the tasks to be completed, such as:

- Proper length and diameter split-spoons or other soil samplers;

- Sufficient drums for soil and decontamination water;
- Proper boring backfill material and/or well construction materials;
- Concrete coring equipment or a coring subcontractor; and
- Equipment for moving drums to the storage area.

PRE-DRILLING PROCEDURES

Prior to mobilization to the site, appropriate notifications for underground utility location must be completed. Once on the site, the following procedures apply:

- Comply with ERM health and safety procedures and the requirements in the HASP (e.g., communicate risks to subcontractors, complete daily safety briefings, evaluate equipment conditions, etc). Ensure that all site workers know where the emergency shutoff switches are for the rig.
- Confirm that drilling equipment brought onto the site is free of leaks from the hydraulic, lubrication, fuel or other systems and that the switches, gages, and other electrical, mechanical, pneumatic, and hydraulic systems are in a safe and operable condition.
- Confirm drilling locations per the proposal, work plan, and/or field briefing.
- Confirm drill rig access to drilling locations.
- Confirm utility notifications and clearance per ERM, HASP, client, and facility requirements.
- Complete concrete or asphalt coring or cutting as necessary.
- Complete pre-drilling utility clearance excavation. Typically, an air knife/vacuum system is required because of the minimum diameter requirements of the clearance hole.
- If present, measure water levels in nearby onsite monitoring wells to confirm the expected depth to water.
- Prepare soil handling table or surface by covering with fresh plastic sheeting prior to drilling at each borehole.

- Calibrate PID and other equipment and record calibration details in the field notes, prepare soil handling area, prepare sample coolers, set up waste collection area (bucket and trash bag).

- Ensure that the drillers have appropriately decontaminated the augers, rods, and samplers prior to beginning drilling.

- Ensure that the drillers have an appropriate decontamination area set up adjacent to the drilling area for sampling equipment.

- Confirm that driller is aware of critical drilling details, such as sampling intervals, total depth, expected depth to water, etc.

- Log soils and conditions within the interval excavated for utility clearance. Use a narrow shovel or other tool to collect soils for observation.

Additionally, it is recommended that a few minutes be spent prior to drilling each hole to arrange the sample handling area so that the necessary equipment and supplies are ready to go and within arm's reach prior to samples arriving at the table. The samples can come fairly rapidly, especially in the first several feet of borehole, and a bit of organization beforehand will cut the frustration factor (and the time required to finish the boring) significantly.

DRILLING PROCEDURES

Effective management of the drilling process typically requires mindfulness of several issues simultaneously while also maintaining focus on properly handling and processing the soil samples as they come out of the boring. Attention must be paid to both soil sample processing as well as the operation of the drill rig in order to maintain a complete and accurate account of the drilling effort.

Soil Sample Processing

Soil samples are typically collected by driving a split spoon sampler below the drill bit using a hammer mounted on the rig. Unless otherwise indicated in the proposal, work plan, and/or field briefing, soil samples should be driven at least once each 2.5 feet of drilling. The split spoon is driven the length of the sampler (typically 18 or 24 inches), and the driller keeps track of the number of hammer blows required to advance the sampler each 6 inches of depth (blow counts). The sampler is then removed from the boring and the drillers open the sampler for soil sample processing by ERM personnel. A sample boring log is attached that illustrates the logging procedures indicated below. Once the soil sample is available from the drillers, the following procedures should be applied in order:

- Get the blow counts from the driller and record them on the drilling log.

- Confirm the depth interval from which the sample was collected and record the interval, time of sampling, and inches of recovered soil (do not include sloughed soils as recovery) on the drilling log.

- Collect all planned or potential laboratory analytical soil samples from the split spoon. If soils critical for description are identified in the sample interval, place a small amount of that soil to the side for describing later, and note the depth of any contacts that may be interrupted by sampling activities. It is critical, however, to collect soil samples for laboratory analysis as soon as possible upon opening of the split spoon. Note laboratory sample intervals on the drilling log.

- Place the soil samples in the cooler and log them on the chain-of-custody (COC) form.

- Collect a portion of the remaining soil in a sealable plastic bag for headspace organic vapor screening.

- Note soil contacts, depth of the contacts, and a description of the soil sample using the Unified Soil Classification System (USCS) and appropriate modifiers and descriptions of miscellaneous conditions (e.g., bedding, minor interbeds, presence of brick fragments, etc.). Refer to Soil Logging SOP for further details.

- If ground water is encountered, note depth at which wet soil was first encountered, and measure depth to water using a water level indicator once borehole is completed.

- Dump excess soil in waste bucket, return sampler parts to driller for decontamination, and wipe down table with a paper towel before the next sample interval is delivered.

- Once drilling is completed, place waste soils in appropriate container (55-gallon drum). On contaminated or potentially-contaminated sites, do not return soils to the borehole. Container should be labeled on the side (not lid) with date(s) of accumulation, contents, source of contents (e.g., boring number), and a unique number in sequence with other investigation-derived waste containers at the site. This information should be recorded in a drum log in the field notes.

- Between boreholes, use the PID to complete headspace organic vapor screening of soil sample intervals collected in sealable plastic bags and record the results on the boring log.

- Between boreholes, decontaminate soil knife by washing with a laboratory-grade detergent solution and rinsing with distilled/deionized other procedure as specified in the proposal, work plan, and/or field briefing.

Drilling Observations

In addition to soil sample processing, ERM personnel should also monitor drilling operations are the borehole is advanced to confirm data recorded as part of soil sample processing, collect additional data, and ensure that the drillers are following proper procedures. The following procedures should be followed during drilling:

- Keep track of the number of auger sections (typically 5 feet in length) in the ground to confirm sampling and total drilling depths.

- Confirm that soil samplers are removed from the borehole immediately after the samples are driven. The sampler should not remain in the auger during drilling through the sampled interval or deeper, as sample loss and/or contamination by deeper soils or ground water could result.

- Confirm that drillers are appropriately decontaminating samplers between sample collection intervals (at least a detergent/water wash, a tap water rinse, and a second tap water rinse).

- Be aware of changes in drilling effort, such as changes in engine sound, rig movement, or speed of auger advancement, that may indicate changes in lithology. If a contact is not clear from the soil record, ask the driller if they noticed any drilling change in the depth interval in question.

- DO NOT allow the driller to place any water or other fluids in the borehole during drilling without prior approval from the ERM project manager. Note that drillers will commonly want to add water if sand is heaving up into the augers below the water table.

Borehole Abandonment

After drilling and sampling are completed, the borehole should be properly abandoned. This is completed by the licensed well driller with appropriate regard to the regulations; however, ERM personnel should provide clear

instructions to the driller regarding the placement of bentonite in the borehole, as well as the restoration of grade conditions at the borehole.

Boreholes are typically abandoned using bentonite chips. The bentonite chips should be poured through the interior of the augers as the augers are removed, taking care that no open space is available between the top of the accumulating bentonite and the bottom of the drill bit (i.e., the bentonite chips are added incrementally as the augers are raised). This is important to minimize the caving of soils into the borehole, which may lead to the formation of a preferential pathway for downward migration of contaminants.

In some cases, the driller may want to remove the augers from the borehole, then pour bentonite chips into the open borehole. While this may be acceptable for shallow borings in cohesive unsaturated soils, bentonite chips should be placed through the augers if there is any concern that soils may cave into an open borehole.

Bentonite-cement grout may be used on some projects, especially where boreholes are advanced below the water table. Boreholes abandoned with bentonite-cement grout should be abandoned in a similar manner as bentonite chips (placed through the auger). Grout should be placed into the borehole using a tremie pipe extended to the bottom of the borehole and raised incrementally as grout is added.

Surface conditions should be returned to as close a condition as possible to conditions prior to drilling. For borings completed in lawn areas, a sod patch should be cut out of the drilling location and then replaced after abandonment is complete. Borings completed through asphaltic pavement should be finished with asphalt cold patch or blackened concrete. Borings completed through concrete should be finished with concrete. The patch material used to finish borehole abandonment should be at least as thick as the penetrated slab.

Note that for stiff or hard fine-grained soils, bentonite chips may push up on the borehole plug, and displace sod or pavement as the bentonite swells. In these conditions, this effect is best alleviated by using bentonite-cement grout for well abandonment. If equipment and supplies for grouting are not available, this situation can be less favorably remedied by placing about 18 inches of sand between the top of the bentonite chips and the bottom of the borehole finish plug. The sand provides pore space into which the bentonite can expand, alleviating the upward pressure on the borehole finish plug.

SAMPLE HANDLING AND SHIPPING

This Standard Operating Procedure details the minimum requirements for handling environmental media samples during collection activities and preparing and packing the samples for shipping to the analytical laboratory. Note that different or additional procedures may be required for specific projects as indicated in the proposal, work plan, or field briefing.

APPLICATION

Environmental media samples are commonly collected to evaluate conditions, such as contaminant concentrations, that cannot be ascertained by observations and screening in the field. All samples require proper identification and documentation as part of the collection process, intermediate handling procedures as sample collection activities are ongoing, and steps to protect the integrity of the sample during shipment to the analytical laboratory, especially when shipping is completed by a third party other than ERM or the laboratory.

Note that equipment, supplies, and procedures in italics below are only necessary when samples must stay cold during shipment. Those indicated with an asterisk (*) are not required when samples are delivered directly to the laboratory by the ERM representative responsible for the samples, but must be completed if a laboratory representative or other courier is picking up the samples for delivery to the laboratory.

TASK-SPECIFIC EQUIPMENT AND SUPPLIES

- Sample containers
- Sample labels
- Coolers
- Sample packing material. If glass or other fragile containers (e.g., vacuum canisters) are being shipped, sheet- or bag-type bubble wrap is preferred. Avoid loose packing material ("peanuts") or any material that loses strength when wet (e.g., cardboard).
- Fine-tipped permanent markers ("Sharpies") for labeling samples
- Ball-point pens for filling out chain-of-custody (COC) forms

- COC forms
- Sealable plastic bags ("Ziplocs"), various sizes
- *Large heavy-duty plastic bags (garbage can liners)*
- *Crushed Ice, 7- or 10-pound bags*
- *COC seals
- *Packing tape
- *"Fragile" and "This Side Up" Stickers*
- *Shipping labels

SAMPLE HANDLING PROCEDURES

During Sample Collection

During ongoing sample collection activities, the following sample handling procedures apply:

- *Prepare coolers prior to sample collection by lining them with heavy-duty plastic bags.*
- *Prior to sample collection, place crushed ice on the bottom of the cooler and maintain additional bagged ice in the cooler to chill samples as they are collected.*
- Record pertinent project information in the header of the COC form prior to initiating sampling efforts (See attached COC form) to include:
    - Company name (ERM);
    - Project name;
    - Project number;
    - Sampler name; and
    - Receiving laboratory name and location.

- Using a fine-tipped permanent marker (never use ball point pen), put a label on sample containers and record on that label the following information immediately prior to sample collection:

  - Unique sample identification number;

  - Date and time of sample collection;

  - Site name;

  - Project number; and

  - Sampler's name or initials.

  Alternately, sample information may be recorded on the sample label after collection of dry samples, such as soil.

- Place samples in a sealable plastic bag (to protect the sample and maintain data integrity if the label falls off). Note that sample jars (especially soil samples) aren't always waterproof, so ensure that there is a good seal on the bag to prevent sample contamination (e.g., from melting ice or contents of sample bottles broken during handling/shipping).

- Place sample in cooler, taking care to arrange the samples and temporary packing material to prevent breakage *while maximizing contact with ice to cool the samples as rapidly as possible*.

- Record sample information on the chain of custody form as the samples are collected and placed in the cooler:

  - Unique sample identification number;

  - Date and time of sample collection.

  - Sampling method (e.g., split spoon, trowel, bailer, bladder pump, etc.)

  - Sample preservatives used or present;

  - Number of containers collected for each sample; and

  - Sampled media (e.g., soil or groundwater).

- Note that the above procedures should be completed for each sample prior to collection of the subsequent sample.

- Replace ice as necessary to maintain sample temperatures between 2 and 4 degrees Celsius while samples are handled by ERM personnel.

- Unattended, unsealed sample coolers should be locked in a vehicle or otherwise secured in a manner that allows access by the sampler only.

- Separate COC forms must be completed for each sample cooler.

Transport/Shipping Preparation

In order to prepare the samples for shipping, the following procedures apply:

- Inventory samples to confirm that the COC form data match the samples in the cooler.

- Complete the analysis request portion of the COC form.

- *Remove heavy-duty plastic bag, partially-melted ice, and meltwater from the cooler.*

- *Completely and redundantly plug any drain holes that could leak water during sample shipment.*

- *Line the cooler with a new heavy-duty plastic bag.*

- *Place the loose contents of one bag of crushed ice (at least 7 pounds) in the bottom of the plastic bag/cooler.*

- Place samples in cooler using packing material for protection during shipping.

- *Place loose contents of at least one bag of crushed ice (at least 7 pounds) over the top of the samples in the plastic bag/cooler (Note that the weight/volume of ice should be greater than the weight/volume of packed samples, with a minimum of 7 pounds of ice for each 12 hours of expected time between sample packing and delivery to the laboratory).*

- *Gather the top of the heavy-duty plastic bag together, twist/seal the top, and stow the loose end so that the lid of the cooler can be closed with the entire bag remaining inside the cooler.*

- Sign "Relinquished by" portion of COC form, add date and time, retain the ERM copies of the COC forms and place the laboratory copies in a sealable plastic bag placed on top of (not inside) the heavy-duty plastic bag so that it is readily visible and accessible when the cooler is opened;

- *Complete COC seals for the coolers with the following information:

  - Company name (ERM);

  - Project name;

  - Project number; and

  - Sampler name or initials.

- *Close the cooler and place one COC seal across the cooler lid/body interface on the side opposite the hinges; or, if the lid is completely removable, place one COC seal each across the cooler lid/body interface on two opposite sides of the cooler.

- *Seal the cooler generously with duct tape, taking care to preserve and protect the integrity of the COC seals, and paying special attention to further securing any cooler drain ports.

- Place "Fragile" and "This Side Up" stickers in prominent locations on the top of the cooler.

- *Complete and securely attach shipping label on top of the cooler.

Note that the person who signs the COC form is responsible for sample integrity until the samples are received by the lab, including during transport by third parties such as overnight couriers. Pack and arrange shipment of samples accordingly. If the samples are shipped via overnight courier, it is strongly recommended that the ERM person listed on the COC form deliver the samples directly to the courier offices.

If the sampler transfers the responsibility for handling/packing/shipping the samples to another ERM employee, that transfer must be to another technical person with adequate training and experience, and the transfer should be recorded on the COC form in the "Relinquished By/ Received By" section. It is not acceptable to assign a non-technical person (e.g., administrative assistant) to sample shipping tasks.

SOIL LOGGING

Soil logging is the procedure by which subsurface conditions are systematically described in order to provide an understanding of the different lithologic conditions present in the subsurface, and the spatial relationship of each of those units. Soil logging should be completed through the entire depth of any borehole or excavation completed as part of an environmental investigation or remediation effort.

TASK-SPECIFIC EQUIPMENT AND SUPPLIES

- Soil log appropriate to the type of exploration (e.g., boring or trench logs).
- Unified Soil Classification System (USCS) summary.

Other optional equipment and supplies, such as a Munsell color chart, may be required to meet project requirements.

SOIL LOGGING PROCEDURE

- Note soil contacts, depth of the contacts, and a description of the soil sample using the USCS and appropriate modifiers and descriptions of miscellaneous conditions (e.g., bedding, minor interbeds, presence of brick fragments, etc.). Soils should be described as follows:

  (Color) (modifying grain size*) (PRIMARY GRAIN SIZE), (minor grain size*), (density/stiffness), (plasticity**), (moisture), (miscellaneous conditions*)

  * designates descriptor elements that may not apply in all cases.
  ** for primarily fine-grained soils only.

  For example, a soil might be described as: Gray silty fine to medium SAND, trace fine gravel, medium dense, loose, moist, wood fragments, slight petroleum odor.

  If a contact is present but is not intercepted by the recovered soil samples, use other information (e.g., listen for changes in rig torque while turning augers) that might provide a clue to the depth of lithological changes. In the absence of available data, put a diagonal line on the log between the deepest confirmed depth of the upper lithology and the shallowest depth of the deeper lithology (see attached example drill log).

The objective of soil logging is providing as complete a description as possible of soil conditions over the entire length of the borehole, not just over the intervals from which soil samples were recovered. When logging soils, especially in soil borings, it is important to log the soil column as a whole using all available information, not just the separate individual samples that are recovered.

A summary of USCS description criteria and modifiers is attached, as is an example of a completed boring log for reference.

- Note the two-letter USCS soil designator for each unit encountered on the boring or excavation log.

- For trenches or excavations, develop a log that shows any variation in lithologic contact elevations or other lateral variation along each wall of the excavation.

SOIL DESCRIPTORS

Soil Particle Size Description

Descriptor	**Grain Diameter/Observations**
Boulder	> 12″
Cobble	3″ to 12″
Coarse Gravel	¾″ to 3″
Fine Gravel	4.76 mm to ¾″
Coarse Sand	2 mm to 4.76 mm
Medium Sand	0.42 mm to 2 mm
Fine Sand	0.074 mm to 0.42 mm
Silt and Clay	Smaller than 0.074 mm

Soil Particle Distribution

Descriptor	**Percentage by Volume of Total Grain Sizes Present**
Trace	<2%
Little	2% to 12%
Some	12% to 25%
(Modifier)	25% to 49%

Density (Predominantly coarse soils)

Descriptor	**Blow Count (Sum of 6″-12″ and 12″-18″ Intervals)**
Very Loose	<4
Loose	4 - 10
Medium Dense	11 - 30
Dense	31 - 50
Very Dense	>50

Stiffness (Predominantly fine soils)

Descriptor	**Blow Count (Sum of 6″-12″ and 12″-18″ Intervals)**
Very Soft	<2
Soft	2 - 4
Medium Stiff	5 - 8
Stiff	9 - 15
Very Stiff	16 - 30
Hard	>30

Plasticity

Descriptor	**Condition Observed**
Nonplastic	Soil cannot be rolled into a ½″ ball without crumbling
Slightly Plastic	Soil can be rolled into a ½″ ball with some care
Medium Plastic	Soil easily rolled into ½″ ball
Highly Plastic	Soil can be kneaded without rupturing

Moisture

Descriptor	Condition Observed
Dry	Soil crumbles, visible dust, powdery, none or very little evidence of water present, below liquid limit
Moist	Soil contains enough water to wet all grains, but no free water is present. At or above plastic limit but below liquid limit.
Wet	Free water is visible. Silty soils are dilatent (free water appears when soils are disturbed).

Appendix B
- Quality Assurance Project Plan

APPENDIX B
QUALITY ASSURANCE PROJECT PLAN

PHASE II RI WORK PLAN
10400 NORTH BURGARD WAY
Portland, Oregon

Oregon Department of Environmental Quality
Portland, Oregon

October 2013
Project No. 750608605

Treadwell&Rollo
A LANGAN COMPANY

Appendix B
Phase II RI Work Plan
Premier Edible Oils
Portland, Oregon

Treadwell&Rollo
A LANGAN COMPANY

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APPENDIX B
Quality Assurance Project Plan

B1.0 MANAGEMENT

B1.1 Introduction

This Quality Assurance Project Plan (QAPP) is prepared on behalf of MMGL Corp. (MMGL) by Treadwell and Rollo, A Langan Company (T&R), to support the Phase II Remedial (Phase II RI) Investigation Work Plan (Work Plan) at the Premier Edible Oils property at 10400 North Burgard Way in Portland, Oregon (the Site). The contents of this QAPP are consistent with previously-approved quality control procedures for the Site (BridgeWater Group, 2001). The Site is an 18.5-acre industrial property located on the Portland Harbor waterfront adjacent to the Willamette River. Over the previous century, the site has been used for bulk petroleum storage and handling, logistical support for a World War II federal shipbuilding facility, production of materials used in batteries and soaps, and processing and storage of edible oils.

B1.2 Project Organization Roles and Responsibilities

The activities at the Site, as described in the Investigation Workplan, will include the following organizations:

- The Oregon Department of Environmental Quality (DEQ) staff
- T&R (environmental contractor), and
- Analytical laboratory contractors

B1.2.1 DEQ Staff

We understand that staff in DEQ's offices will perform the following duties:

- Review and approve the site-specific Workplan, Sampling and Analysis Plan (SAP), and Quality Assurance Project Plan (QAPP) prepared by T&R;
- Review T&R's overall field implementation of the Workplan;
- Review the results of the Workplan and the subsequent reporting at the Site;
- Update DEQ's Environmental Cleanup Site Information (ECSI) database in a timely

manner, if needed.

B1.2.2 Treadwell and Rollo, A Langan Company

T&R, as the prime environmental contractor conducting field work at the Site, will perform the following tasks:

- Develop a site-specific Workplan, SAP, and QAPP;
- Assemble project teams, implement field work, and coordinate sample analyses;
- Verify the proper functioning of equipment before beginning field activities;
- Verify that the proper number, type, and quantity of sample containers, including preservation requirements, are available for field activities;
- Follow sampling protocols as defined in the SAP (Appendix A);
- Record field data in the manner specified in this QAPP; and
- Following applicable SAP procedures check that samples are collected, preserved, labeled, packaged, and shipped to laboratories in an appropriate manner.

B1.2.3 Analytical and Geotechnical Laboratory Contractors

Contract laboratories analyzing and reporting on samples collected at the Site will:

- Understand and follow procedures outlined in this QAPP and SAP;
- Perform requested analyses using appropriate test methods specified in the QAPP and SAP;
- Satisfy all laboratory and analytical QA/QC
- Objectives and activities;
- Prepare laboratory reports for T&R, including all relevant data and QC reports;

- Communicate analytical problems, issues, or concerns to T&R in a timely manner; and
- Initiate corrective action when deficiencies in sample collection, preservation, handling, test methods, or documentation are identified internally, by the contract laboratory, or by DEQ or T&R.

B1.3 Project Task Description

The objective of QA activities described in this QAPP is to improve the likelihood that data obtained from investigation at the Site are of known quality, represent actual site conditions, and are adequate and appropriate for making informed environmental decisions. The data obtained under this QAPP will be used to evaluate the nature, magnitude, and extent of contamination at the Site to complete human health and ecological risk assessment and to evaluate remedial alternatives for mitigating risks of Site impacts to human and ecological health.

Media to be sampled under this QAPP include:

- Soil;
- Soil Gas; and
- Groundwater.

Categories of contaminants for which these media will be analyzed include:

- Polycyclic Aromatic Hydrocarbons (PAHs);
- Metals (total and dissolved);
- Total Petroleum Hydrocarbons (TPHs);
- Volatile Organic Compounds (VOCs);
- Chlorinated volatile organic compounds;
- Nitrate, sulfate and chloride;
- Iron (II);
- Alkalinity;
- Organochlorine Pesticides; and

- Dissolved oxygen (DO), temperature, pH, oxygen-reduction-potential (ORP) and conductivity;

B1.4 Quality Objectives and Criteria for Measurement Data

The purpose of this section is to check that qualitative and quantitative guidelines are followed and used to define goals contained in this QAPP. Field personnel and laboratories analyzing samples must record and retain sufficient notes and QC documentation to demonstrate and support the level of data quality required for these projects. Laboratories analyzing samples must have a quality system that meets the requirements in the standards developed by The NELAC Institute and adopted by the National Environmental Laboratory Accreditation Program (NELAP) (http://www.nelac-institute.org). Appendix B-1 includes the Quality Management Plan (QMP) for the likely analytical laboratory, Columbia Analytical Services (CAS). If CAS is not used, a laboratory with an equivalent QMP will be used.

Section 2 of this QAPP contains site-specific QC measures to be implemented in conjunction with the SAP (Appendix A) during the field activity, including the following elements:

- Sampling procedures;
- Field documentation and procedures;
- Field equipment calibration and analyses;
- The number and type of QC samples to be collected and submitted for analysis (e.g., trip and rinsate blanks, duplicate samples, etc.);
- The analytical methods and minimum detection limits that laboratories analyzing the samples must achieve;
- The analytical QC elements (e.g., laboratory blanks, laboratory replicates, fortified samples, etc.) and assessment criteria that the laboratories must meet, if these differ from those described in the laboratories' QMP; and
- Reporting requirements and formats for laboratory data (e.g., reporting units, electronic or printed formats, data flagging, etc.); Laboratory data must be accompanied by supporting QC data.

B1.5 Special Training Requirements and Certifications

The field activities proposed for the Site pose certain risks. Field personnel and contractors are required to have appropriate Occupational Health and Safety Administration (OSHA) health and safety training for hazardous waste sites (Hazardous Waste Operations and Emergency Response [HAZWOPER] training), supplemented by annual refresher courses. Contractors are responsible for ensuring that their personnel are informed about and trained on relevant OSHA guidelines.

Field staff with questions about risks they might be dealing with should use existing resources (e.g., Material Safety Data Sheets [MSDS], literature, and laboratory staff) and contact the appropriate authority (e.g., T&R project managers, contractor project managers, and laboratory managers).

B1.6 Documentation and Records

Documents and records produced during the Site investigation must be properly managed. Documents and records produced may include, but are not limited to the following items:

- Workplan, SAP, and QAPP;
- Field notes and records;
- Chain-of-custody forms;
- Laboratory analytical reports;
- Field and laboratory QC data;
- Photographs; and
- Records of communication such as phone logs, memos, emails, or other written correspondence.

Documents associated with the Site investigation will be filed in T&R's electronic project folder. Project records will be maintained for a period of no less than five years. Electronic records, wherever possible, will be maintained in write-protected formats such as the Portable Document Format (.pdf).

B1.6.1 Laboratory Documentation

The analytical laboratory will follow the sample acceptance policy as described in their QMP

(Appendix B-1). This policy describes the minimum data elements for samples submitted to the laboratory for analyses.

Samples failing to meet the criteria described above may be analyzed, depending on the circumstances, but the data will be clearly flagged when reported as having been compromised because of a deficiency in one or more of the elements listed above. Release of data from compromised samples will be deferred, awaiting the necessary documentation.

Documentation of missing information or instructions may be furnished to the laboratory in writing at any time up to the release of the data by the laboratory. When all sample acceptance criteria are met, the qualifying data flag will be expunged from the report provided the quality of the data has not been compromised.

Complete sample documentation must be provided, including:

- Unique sample identification;
- Sample location;
- Sample matrix (e.g., liquid, solid, soil gas, sludge, sediment);
- Sample classification (grab, discrete composite);
- Date and time of collection;
- Sampler's names;
- Analytes to be analyzed and, when appropriate, the specific analytical method; and
- Special remarks describing the sample, if appropriate.

B2.0 DATA GENERATION AND ACQUISTION

B2.1 Sampling Process Design

The purpose of sampling at the Site is to evaluate the nature, magnitude, and extent of contamination at the Site, to complete human health and ecological risk assessment, delineate potential areas of soil

excavation and to evaluate remedial alternatives for mitigating risks of Site impacts to human and ecological health.

Procedures for groundwater, soil, and soil gas sampling are included in the SAP (Appendix A). Those procedures describe methods for assembly and method of use for sampling equipment, use of personal protective equipment, collection and labeling of the sample, and decontamination.

B2.2 Sampling Methods

Sampling of soil, soil gas, and groundwater will be performed using the methods described in the SAP (Appendix A). These methods were selected such that sampling will be collected in a manner consistent with the media being sampled and the site-specific analytes.

The use of proper sample containers and appropriate preservation techniques when collecting samples is important. Samples should be collected in containers supplied by the analyzing laboratory. This helps check that the container has been properly cleaned and that the analyzing laboratory will have sufficient sample material to conduct the requested test. Samples must also be properly preserved according the laboratory protocol. Table A-1 summarizes required sample containers, preservation techniques, and holding times for analytes of interest for this Site investigation.

B2.3 Sample Handling and Custody

Sample handling and custody will be performed in accordance with the QMP for the likely chemical analytical laboratory (Appendix B-1).

B2.4 Analytical Methods

Analytical methods have been selected in order to provide the most relevant and accurate analytical results relative to screening level values applicable at the Site. The list of analytical methods is included in the SAP (Appendix A). A summary of method detection, quantitation, and reporting limits for soil and groundwater samples provided by the analytical laboratory is included as Appendix B-2. If needed, to obtain adequate detection limits, additional laboratory methods can be employed to reduce the results of matrix interferences, including cleanup of samples using silica gel and targeting of specific compounds using selective ion monitoring.

B2.5 Quality Control

Environmental decision making at the Site requires data and information of the highest possible quality. Procedural aspects, from project planning, sample collection, laboratory analysis, and data assessment impart a significant bearing on the decisions to be made. This section describes quality-control procedures for the site investigation.

B2.5.1 Field Quality Control

Field instruments and equipment used for measurement data will be operated and calibrated according to manufacturer's guidelines and recommendations. Calibration records will include the following information (whenever available and appropriate for the specific instrument or equipment):

- Calibration date;
- Test method;
- Instrument;
- Analysis date;
- Each analyte's name;
- Analyst's initials or signature;
- Concentration and response; and
- Calibration curve or response factor.

Personnel properly trained in these procedures will operate and calibrate the instruments. Calibration records will be maintained in T&R's electronic project file.

B2.5.2 Laboratory Quality Control

Routine laboratory QA activities are documented in the analyzing laboratory's QMP (Appendix B-1), which conform to NELAP requirements.

B2.5.3 Data Assessment

Data processing, verification, and validation are the quality management tools used to determine whether project data meet the planned data quality objectives (DQOs) and requirements defined in the SAP and QAPP. Procedures of these items will conform to the analytical laboratory's QMP (Appendix B-1), which confirmed to general USEPA and DEQ requirements.

B2.6 Instrument/Equipment Testing, Inspection and Maintenance

Field and laboratory analytical instruments and equipment will be tested, inspected, and maintained according to the manufacturer's guidelines and recommendations. Data collected from improperly functioning equipment will not be used. The equipment testing, inspection, and maintenance logs for contractor equipment will be maintained in T&R's electronic project file.

B2.7 Inspection and Acceptance Requirements for Supplies and Consumables

Supplies and consumables will be examined for damage or other characteristics that would otherwise compromise data quality. Procedures for inspecting and accepting supplies and consumables at the analytical laboratory are included in their QMP (Appendix B-1).

B2.8 Non-Direct Measurements

Data collected from non-measurement sources, such as computer databases or scientific publications, will approved by the T&R project manager before use and will be cited accordingly.

B2.9 Data Management

Field data from the site investigation will be recorded on a field data sheet. Field data is reported to the Project Manager through submission of field notebooks or field sampling data sheets. Laboratory analytical data will be submitted by the laboratory to T&R in both printed and electronic form. Field, laboratory, or other data received for this project will be maintained in T&R's electronic project file.

B3.0 ASSESSMENT AND OVERSIGHT

The T&R project manager will confer daily with field personnel to discuss problems, and check that planned samples are being collected. The T&R project manager will also review field logs and data

weekly, and arrange re-sampling if needed to correct deficiencies. Contract laboratories will participate in Performance Evaluation studies twice yearly and satisfy NELAC requirements. Personnel responsible for data assessment will check the results of every sampling event for precision and completeness. Technical or quality system audits of environmental or laboratory contractors may be initiated on an as-needed basis in response to identified or suspected problems. Assessment and response actions will be documented maintained in T&R's electronic project file. Identified deficiencies will be followed up by written corrective action plans.

B4.0 DATA VALIDATION AND USABILITY

B4.1 Data Review, Verification and Validation

Data review, verification, and validation procedures and requirements will follow though outlines in the analytical laboratory's QMP (Appendix B-1).

B4.2 Verification and Validation Methods

Verification and validation procedures and requirements will follow though outlines in the analytical laboratory's QMP (Appendix B-1).

B4.3 Reconciliation with User Requirements

The T&R project manager will check that data collected during the Site investigation will meet the needs for environmental decision-making. Moreover, the T&R project manager will check that environmental and laboratory contractors satisfy requirements specified in the SAP and QAPP. The laboratory conducting sample analyses will submit appropriate QC data identified in this QAPP with its analytical data.

Appendix B-1
Laboratory Quality Management Plan

QUALITY ASSURANCE MANUAL

Columbia Analytical Services, Inc.

1317 South 13th Avenue
Kelso, Washington 98626
(360) 577-7222
Effective Date: November 1, 2011

Approved by:

Laboratory Director/Technical Director: _____ 11/11/11
Jeff Grindstaff — Date

Quality Assurance Program Manager: _____ 11/11/11
Julie Gish — Date

Technical Director – Microbiology: _____ 11/11/11
Chris Kerksieck — Date

Technical Director – Metals: _____ 11/11/11
Jeff Coronado — Date

Technical Director – General Chemistry: _____ 11/11/11
Harvey Jacky — Date

Technical Director –Orgaincs GC: _____ 11/11/11
Aqilla Kamawal — Date

Technical Director – Organics GC/MS HPLC: _____ 11/11/11
Jon James — Date

DOCUMENT CONTROL

NUMBER: _____

Initials: _____ Date: _____

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2.0 INTRODUCTION AND COMPANY QUALITY ASSURANCE POLICY

Columbia Analytical Services, Inc. (CAS) is a professional analytical services laboratory which performs chemical and microbiological analyses on a wide variety of sample matrices, including drinking water, groundwater, surface water, wastewater, soil, sludge, sediment, tissue, industrial and hazardous waste, air, and other material. Columbia Analytical Services operates a network of laboratory facilites located in Arizona, California, Florida, New York, Texas, and Washington.

We recognize that quality assurance requires a commitment to quality by everyone in the organization - individually, within each operating unit, and throughout the entire laboratory. Laboratory management is committed to ensuring the effectiveness of its quality systems and to ensure that all tests are carried out in accordance to customer requirements. Key elements of this commitment are set forth in the Columbia Analytical Services, Inc. Quality and Ethics Policy Statement, September 2010 (Appendix A) and in this Quality Assurance Manual (QAM). Columbia Analytical Services, Inc. is committed to operate in accordance with these requirements and those of regulatory agencies, accrediting authorities, and certifying organizations. The laboratory also strives for improvement through varying continuous improvement initiatives and projects.

Quality Management Systems are established, implemented and maintained by management. Policies and procedures are established in order to meet requirements of accreditation bodies and applicable programs, such as the Department of Defense (DOD) Environmental Laboratory Accreditation Program, as well as client s quality objectives. Systems are designed so that there will be sufficient Quality Assurance (QA) activities conducted in the laboratory to ensure that all analytical data generated and processed will be scientifically sound, legally defensible, of known and documented quality, and will accurately reflect the material being tested. Quality Systems are applicable to all fields of testing in which the laboratory in involved.

Quality Control (QC) procedures are used to continually assess performance of the laboratory and quality systems. Columbia Analytical maintains control of analytical results by adhering to written standard operating procedures (SOPs), using analytical control parameters with all analyses, and by observing sample custody requirements. All analytical results are calculated and reported in units consistent with project specifications to allow comparability of data.

This QAM is applicable to the facility listed on the title page. The information in this QAM has been organized according to requirements found in the National Environmental Laboratory Accreditation Program (NELAP) Quality Systems Standards (2003 and 2009), the EPA Requirements for Quality Assurance Project Plans, EPA QA/R-5, USEPA, 2001; and *General Requirements for the Competence of Testing and Calibration Laboratories*, ISO/IEC 17025:2005.

3.0 PROGRAM DESCRIPTION

The purpose of the QA program at Columbia Analytical is to ensure that our clients are provided with analytical data that is scientifically sound, legally defensible, and of known and documented quality. The concept of Quality Assurance can be extended, and is expressed in the mission statement of Columbia Analytical:

> "The mission of Columbia Analytical Services, Inc. is to provide high quality, cost-effective, and timely professional testing services to our customers. We recognize that our success as a company is based on our ability to maintain customer satisfaction. To do this requires constant attention to customer needs, maintenance of state-of-the-art testing capabilities and successful management of our most important asset - our people - in a way that encourages professional growth, personal development and company commitment."

3.1 Quality Management Systems

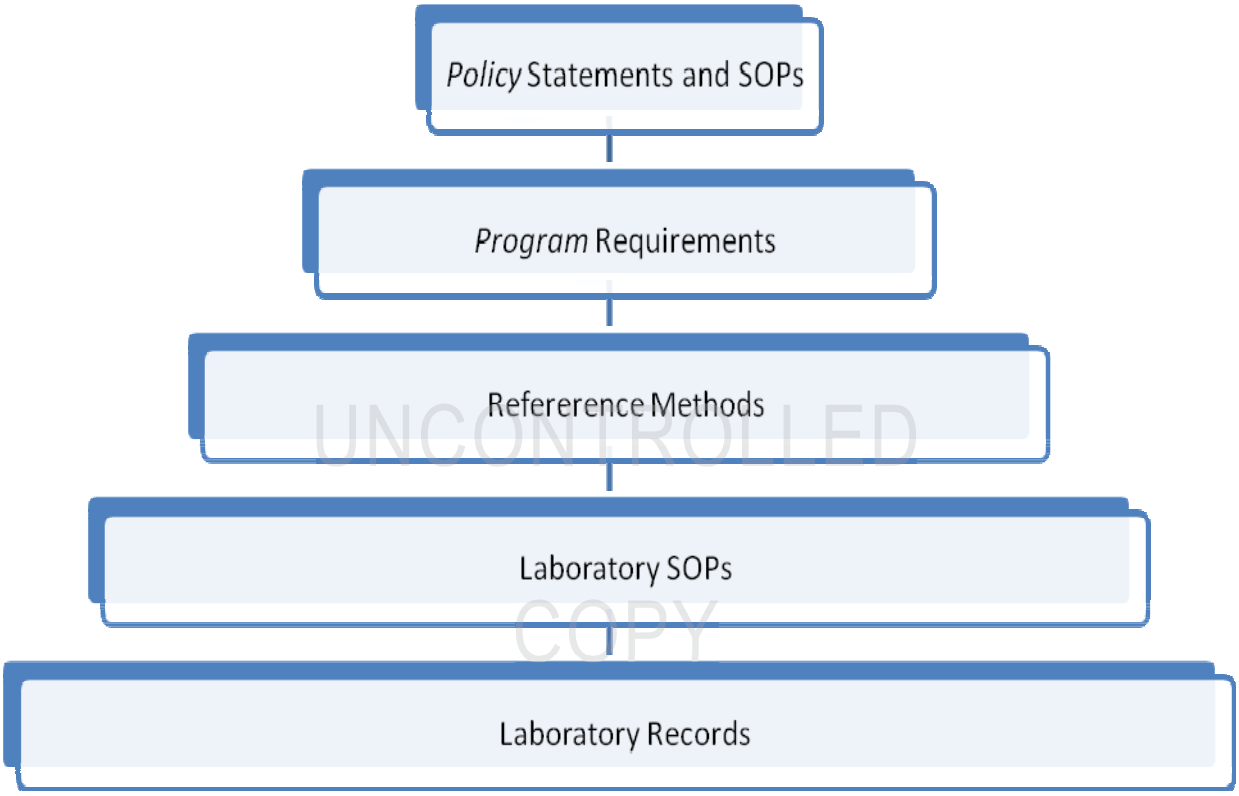
In support of this mission, the laboratory has developed a Quality Management System to ensure all products and services meet our client s needs. The system is implemented and maintained by the Quality Assurance Program Manager (QA PM) with corporate oversight by the Chief Quality Officer (CQO). These systems are based upon ISO 17025:2005 standards, upon which fundamental programs (NELAC 2003, 2009 and DoD QSM) are based. Implementation and documentation against these standards are communicated in corporate policy statements, this QAM, and SOPs. Actual procedures, actions and documentation are defined in both administrative and technical SOPs. Figure 3-1 shows the relationships of the quality systems and associated documentation. Quality systems include:

- Standard Operating Procedures
- Sample Management and Chain of Custody procedures
- Statistical Control Charting
- Standards Traceability
- Ethics Training
- Document Control
- Corrective Action Program
- Management Reviews
- Demonstration of Capability

The effectiveness of the quality system is assessed in several ways, including:

- Internal and External Audits covering all aspects of the organization
- Annual Management Reviews
- Analysis of Customer Feedback
- Internal and External Proficiency Testing

Figure 3-1
Relationships of Quality Management Systems and Documentation



3.2 Facilities, Equipment, and Security

Columbia Analytical features over 45,000 square feet of laboratory and administrative workspace. The laboratory has been designed and constructed to provide safeguards against cross-contamination of samples and is arranged according to work function, which enhances the efficiency of analytical operations. The ventilation system has been specially designed to meet the needs of the analyses performed in each work space. Also, Columbia Analytical minimizes laboratory contamination sources by employing janitorial and maintenance staff to ensure that good housekeeping and facilities maintenance are performed. In addition, the segregated laboratory areas are designed for safe and efficient handling of a variety of sample types. These specialized areas (and access restrictions) include:

- Shipping and Receiving/Purchasing
- Sample Management Office, including controlled-access sample storage areas
- Inorganic/Metals Sample Preparation Laboratories (2)
- Inorganic/Metals clean room sample preparation laboratory
- ICP-AES Laboratory
- ICP-MS Laboratory
- AA Laboratory
- Water Chemistry General Chemistry Laboratories (3)
- Semi-volatile Organics Sample Preparation Laboratory
- Gas Chromatography/High Performance Liquid Chromatography Laboratory
- Gas Chromatography/Mass Spectrometry Laboratory (2)
- Semi-volatile Organics Drinking Water Laboratories (2)
- Volatile Organics Laboratory
    - Separate sample preparation laboratory
    - Access by semi-volatile sample preparation staff only after removing lab coat and solvent-contaminated gloves, etc.
- Microbiology Laboratory
- Laboratory Deionized Water Systems (2)
- Laboratory Management, Client Service, Report Generation and Administration
- Data Archival, Data Review and support functions areas
- Information Technology (IT) and LIMS

In addition, the designated areas for sample receiving, refrigerated sample storage, dedicated sample container preparation and shipping provide for the efficient and safe handling of a variety of sample types. Figure 3-2 shows the facility floor plan. The laboratory is equipped with state-of-the-art analytical and administrative support equipment. The equipment and instrumentation are appropriate for the procedures in use. Appendix C lists the major equipment, illustrating the laboratory's overall capabilities and depth.

3.3 Technical Elements of the Quality Assurance Program

The laboratory s technical procedures are based upon procedures published by various agencies or organizations (See Section 17). The Quality Assurance Program provides to the laboratory organization, procedures, and policies by which the laboratory operates. The necessary certifications and approvals administered by external agencies are maintained by the QA department. This includes method approvals and audit administration. In addition, internal audits are performed to assess compliance with policies and procedures. SOPs are maintained for technical and administrative functions. A document control system is used for SOPs, as well as laboratory notebooks, and this QA Manual. A list of QA Program documents is provided in Appendix A and SOPs in Apppendix F.

Acceptable calibration procedures are defined in the SOP for each test procedure. Calibration procedures for other laboratory equipment (balances, thermometers, etc.) are also defined. Quality Control (QC) procedures are used to monitor the testing performed. Each analytical procedure has associated QC requirements to be achieved in order to demonstrate data quality. The use of method detection limit studies, control charting, technical training and preventive maintenance procedures further ensure the quality of data produced. Proficiency Testing (PT) samples are used as an external means of monitoring the quality and proficiency of the laboratory. PT samples are obtained from qualified vendors and are performed on a regular basis. In addition to method proficiency, documentation of analyst training is performed to ensure proficiency and competency of laboratory analysts and technicians. Sample handling and custody procedures are defined in SOPs. Procedures are also in place to monitor the sample storage areas. The technical elements of the QA program are discussed in further detail in later sections of this QA manual.

3.4 Operational Assessments and Service to the Client

The laboratory uses a number of systems to assess its daily operations. In addition to the routine quality control (QC) measurements, the senior laboratory management examines a number of other indicators to assess the overall ability of the laboratory to successfully perform analyses for its clients including; on-time performance, customer complaints, training reports and non-conformity reports. A frequent, routine assessment must also be made of the laboratory s facilities and resources in anticipation of accepting an additional or increased workload.

Columbia Analytical utilizes a number of different methods to ensure that adequate resources are available for service demands. Senior staff meetings, tracking of outstanding proposals and an accurate, current synopsis of incoming work all assist the senior staff in properly allocating sufficient resources. All Requests for Proposal (RFP) documents are reviewed by the Project Manager and appropriate managerial staff to identify any project specific requirements that differ from the standard practices of the laboratory. Any requirements that cannot be met are noted and communicated to the client, as well as requesting the client to provide any project specific Quality Assurance Project Plans (QAPPs) if available. Status/production meetings are also conducted regularly with the laboratory and Project Managers to inform the staff of the status of incoming work, future projects, or project requirements.

When a customer requests a modification to an SOP, policy, or standard specification the Project Manager will discuss the proposed deviation with the Client Services Manager, Laboratory Director, and department manager to obtain approval for the deviation. The QA PM may also be involved. All project-specific requirements must be on-file and with the service request upon logging in the samples. The modification or deviation must be documented. A Project-Specific Communication Form, Form V, or similar, may be used to document such deviations.

The laboratory shall afford clients cooperation to clarify the client s request and to monitor the laboratory s performance in relation to the work performed, provided that the laboratory ensures confidentiality to other clients. The laboratory maintains and documents timely communication with the client for the purposes of seeking feedback and clarifying customer requests. Feedback is used and analyzed to improve the quality of services. The *SOP for Handling Customer Feedback* (ADM-FDBK) is in place for these events.

3.5 Document Control and Records

Procedures for control and maintenance of documents are described in the *SOP for Document Control (ADM-DOC_CTRL).* The requirements of the SOP apply to all laboratory logbooks (standards, maintenance, run logbooks, etc), certificates of analysis, SOPs, QAMs, quality assurance project plans (QAPPs), Environmental Health Safety (EHS) manuals, and other controlled Columbia Analytical documents.

Each controlled copy of a controlled document will be released only after a document control number is assigned and the recipient is recorded on a document distribution list. Filing and distribution is performed by the QA PM, or designee, and ensure that only the most current version of the document is distributed and in use. A document control number is assigned to logbooks. Completed logbooks that are no longer in use are archived in a master logbook file. Logbook entries are standardized following the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). The entries made into laboratory logbooks are reviewed and approved at a regular interval (quarterly).

A records system is used which ensures all laboratory records (including raw data, reports, and supporting records) are retained and available. The archiving system is described in the *SOP for Data Archiving (ADM-ARCH).*

External documents relative to the management system are managed by the QA PM. To prevent the use of invalid and/or outdated external documents, the laboratory maintains a master list of current documents and their availability. The list is reviewed before making the documents available. External documents are not issued to personnel.

3.6 Subcontracting

Analytical services are subcontracted when the laboratory needs to balance workload or when the requested analyses are not performed by the laboratory. Subcontracting is only done with the knowledge and approval of the client and to qualified laboratories. Subcontracting to another Columbia Analytical laboratory is preferred over external-laboratory subcontracting. Further, subcontracting is done using capable and qualified laboratories. Established procedures are used to qualify external subcontract laboratories. These procedures are

described in the *SOP for Qualification of Subcontract Laboratories (ADM-SUBLAB).* The Corporate Quality Assurance staff is responsible for maintaining a list of qualified subcontract laboratories.

3.7 Procurement

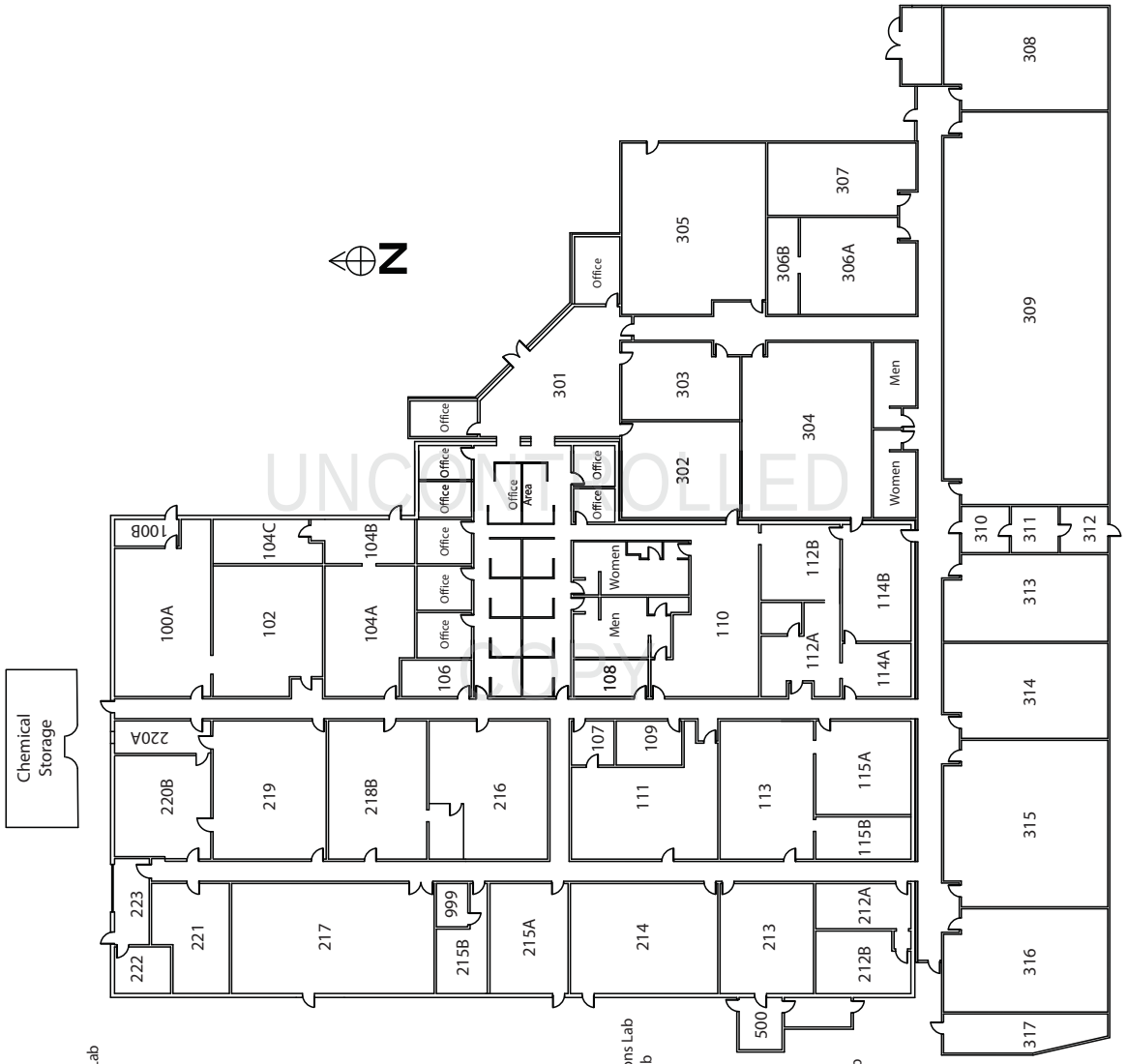
The quality level of reagents and materials (grade, traceability, etc.) required is specified in analytical SOPs. Department supervisors ensure that the proper materials are purchased. Inspection and verification of material ordered is performed at the time of receipt by receiving personnel. The receiving staff labels the material with the date received. Expiration dates are assigned as appropriate for the material. Storage conditions and expiration dates are specified in the analytical SOP. The corporate Policy for Standards and Reagents Expiration Dates provides default expiration requirements. Supplies and services that are critical in maintaining the quality of laboratory testing are procured from pre-approved vendors. The policy and procedure for purchasing and procurement are described in the *SOP for Purchasing and Approval of Vendors* (ADM-PUR). Also, refer to section 9.4 for a discussion of reference materials.

Receipt procedures include technical review of the purchase order/request to verify that what was received is identical to the item ordered. The laboratory checks new lots of reagents for unacceptable levels of contamination prior to use in sample preservation, sample preparation, and sample analysis by following the *SOP for Checking New Lots of Chemicals for Contamination* (ADM-CTMN).

3.8 Review of Requests, Tenders and Contracts (Procedures for Accepting New Work)

Requests for new work are reviewed prior to signing any contracts or otherwise agreeing to perform the work. The specific methods to be used are agreed upon between the laboratory and the client. A capability review is performed to determine if the laboratory has or needs to obtain certification to perform the work, to determine if the laboratory has the resources (personnel, equipment, materials, capacity, skills, expertise) to perform the work, and if the laboratory is able to meet the client s required reporting and QC limits. The results of this review are communicated to the client and any potential conflict, deficiency, lack of appropriate accreditation status, or concerns of the ability to complete the client s work are resolved. Any differences between the request or tender and the contract shall be resolved before any work commences. The client should be notified at this time if work is expected to be subcontracted. Each contract shall be acceptable both to the laboratory and the client. Records are maintained of pertinent discussions with a client relating to the client s requirements or the results of the work. If a contract needs to be amended after work has commenced, the contract review process is repeated and any amendments are communicated to all affected personnel. Changes in accreditation status affecting ongoing projects must be reported to the client.

Figure 3-2
Columbia Analytical Services-Kelso Laboratory Floor Plan



Room #	
100A	Semivolatile Organics GC Instrument Lab
100B	Electrical Room
102	Semivolatile Organics GC Office
104 A - C	Organics LC Labs and Offices
106	Deionized Water System Room
107	Oven Room
108	Zero Headspace Extraction Lab
109	Storeroom
110	Lunch Room
111	General Chemistry Lab
112A	Microbiology Lab
112B	Copy Center
113	ICP MS Lab
114A	Grain Size
114B	Total Solids/Oven Room
115A	ICP Lab
115B	Metals Reporting
212A	Mercury and Flame AA Lab
212B	Low Level Mercury Lab
213	General Chemistry Lab
214	General Chemistry Lab
215A	Drinking Water Lab
215B	Information Technology
216	Data Review & Storage
217	Sample Storage
218	Volatiles Organics Lab
219	Semivolatile Organics GC/MS Lab
220A	Semivolatile Organics GC/MS Office
220B	Semivolatile Organics GC/MS Extractions Lab
221	Drinking Water Sample Preparation Lab
222	Purchasing Office
223	General Receiving
301	Reception Lobby
302	Lunch Room
303	Conference Room
304	Sample Storage
305	Sample Receiving
306A	Pharmaceutical Metals
306B	Pharmaceutical Microbiology
307	Pharmaceutical Instrument Lab
308	Pharmaceutical General Chemistry Lab
309	Organics Sample Preparation Lab
310	Communication Room
311	Electrical Room
312	Fire Room/Water System
313	Glass Wash Room
314	Clean Room
315	Metals Digestion Room
316	Tissue Preparation
317	Mechanical
500	Janitorial Room
999	Computer Room

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4.0 PROFESSIONAL CONDUCT, DATA INTEGRITY, AND ETHICS

One of the most important aspects of the success of CAS is the emphasis placed on the integrity of the data provided and the services rendered. This success is reliant on both the professional conduct of all employees within CAS as well as established laboratory practices. All personnel involved with environmental testing and calibration activities must familiarize themselves with the quality documentation and implement the policies and procedures in their work.

4.1 Professional Conduct

To promote quality, CAS requires certain standards of conduct and ethical performance among employees. The following examples of documented CAS policy are representative of these standards, and are not intended to be limiting or all-inclusive:

- Under no circumstances is the willful act of fraudulent manipulation of analytical data condoned. Such acts are to be reported immediately to senior management for appropriate corrective action.
- Unless specifically required in writing by a client, alteration, deviation or omission of written contractual requirements is not permitted. Such changes must be in writing and approved by senior management.
- Falsification of data in any form will not be tolerated. While much analytical data is subject to professional judgment and interpretation, outright falsification, whenever observed or discovered, will be documented, and appropriate remedies and punitive measures will be taken toward those individuals responsible.
- It is the responsibility of all Columbia Analytical employees to safeguard sensitive company information, client data, records, and information; and matters of national security concern should they arise. The nature of our business and the well being of our company and of our clients is dependent upon protecting and maintaining proprietary company/client information. All information, data, and reports (except that in the public domain) collected or assembled on behalf of a client is treated as confidential. Information may not be given to third parties without the consent of the client. Unauthorized release of confidential information about the company or its clients is taken seriously and is subject to formal disciplinary action. All employees sign a confidentiality agreement upon hire to protect the company and client s confidentiality and proprietary rights.

4.2 Prevention and Detection of Improper, Unethical or Illegal Actions

It is the intention of CAS to proactively prevent and/or detect any improper, unethical or illegal action conducted within the laboratory. This is performed by the implementation of a program designed for not only the detection but also prevention. Prevention consists of educating all laboratory personnel in their roles and duties as employees, company policies, inappropriate practices, and their corresponding implications as described here.

In addition to education, appropriate and inappropriate practices are included in SOPs such as manual integration, data review and specific method procedures. Electronic and hardcopy data audits are performed regularly, including periodic audits of chromatographic electronic data. Requirements are described in the Policy for Internal Quality Assurance Audits and details are listed in laboratory admininstrative SOPs. All aspects of this program are documented and retained on file according to the company policy on record retention.

The CAS Employee Handbook also contains information on the CAS ethics and data integrity program, including mechanisms for reporting and seeking advice on ethical decisions.

4.3 Laboratory Data Integrity and Ethics Training

Each employee receives in-depth (approximately 6-8 hour) core Data Integrity/Ethics Training. New employees are given a QA and Ethics orientation within the first month of hire, followed by the the core training within 1 year of hire. On an ongoing basis, all employees receive semi-annual ethics refresher training. Topics covered are documented in writing and all training is documented. It is the responsibility of the QA PM to ensure that the training is conducted as described.

Key topics covered are the organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting, how and when to report data integrity issues and record keeping. Training includes discussion regarding all data integrity procedures, data integrity training documentation, in-depth data monitoring and data integrity procedure documentation.

Trainees are required to understand that any infractions of the laboratory data integrity procedures will result in a detailed investigation that could lead to very serious consequences including immediate termination, or civil/criminal prosecution.

The training session includes many concepts and topics, numerous examples of improper actions (defined by DoD as deviations from contract-specified or method-specified analytical practices and may be intentional or unintentional), legal and liability implications (company and personal), causes, prevention, awareness, and reporting mechanisms.

4.4 Management and Employee Commitment

Columbia Analytical makes every attempt to ensure that employees are free from any commercial, financial, or other undue pressures that might affect their quality of work. Related policies are described in the Columbia Analytical Employee Handbook. This includes:

- CAS Open Door Policy (CAS Employee Handbook) Employees are encouraged to bring any work related problems or concerns to the attention of local management or their Human Resources representative. However, depending on the extent or sensitivity of the concern, employees are encouraged to directly contact any member of upper management.

- CAS Corporate Ethics Point Program An anonymous and confidential reporting system available to all employees that is used to communicate misconduct and other concerns. The program shall help minimize negative morale, promote a positive work place, and encourage reporting suspected misconduct without retribution. Associated upper management is notified and the investigations are documented.
- Use of flexible work hours. Within reason and as approved by supervisors, employees are allowed flexible work hours in order to help ease schedule pressures which could impact decision-making and work quality.
- Operational and project scheduling assessments are continually made to ensure that project planning is performed and that adequate resources are available during anticipated periods of increased workloads. Procedures for subcontracting work are established, and within the Columbia Analytical laboratory network additional capacity is typically available for subcontracting, if necessary.
- Gifts and Favors (CAS Employee Handbook) To avoid possible conflict of interest implications, employees do not receive unusual gifts or favors to, nor accept such gifts or favors from, persons outside the Company who are, or may be, in any way concerned with the projects on which the Company is professionally engaged.

All employees are required to sign and adhere to the requirements set forth in the Columbia Analytical *Confidentiality and Conflicts of Interest Employee Agreement* and the Columbia Analytical *Commitment to Excellence in Data Quality* (see Appendix A).

5.0 ORGANIZATION AND RESPONSIBILITIES

The Columbia Analytical/Kelso staff, consisting of approximately 150 employees, includes chemists, technicians and support personnel. They represent diverse educational backgrounds and experience, and provide the comprehensive skills that the laboratory requires. During seasonal workload increases, additional temporary employees may be hired to perform specific tasks.

CAS is committed to providing an environment that encourages excellence. All employees share the responsibility for maintaining and improving the quality of our analytical services. The responsibilities of key personnel within the laboratory are described below. Table 5-1 lists the Columbia Analytical Kelso personnel assigned to these key positions. Managerial staff members are provided the authority and resources needed to perform their duties. An organizational chart of the laboratory, as well as the resumes of these key personnel, can be found in Appendix B.

- The role of the **Laboratory Director** is to provide technical, operational, and administrative leadership through planning, allocation and management of personnel and equipment resources. The Laboratory Director provides leadership and support for the QA program and is responsible for overall laboratory efficiency and the financial performance of the (Location) facility. The Laboratory Director has the authority to stop work in response to quality problems. The Laboratory Director also provides resources for implementation of the QA program, reviews and approves this QA Manual, reviews and approves standard operating procedures (SOPs), and provides support for business development by identifying and developing new markets through continuing support of the management of existing client activities.
- The **Quality Assurance Program Manager (QA PM)** has the authority and responsibility for implementing, maintaining, and improving the quality system. This includes coordination of QA activities within the laboratory, ensuring that all personnel understand their contributions to the quality system, ensuring communication takes place at all levels within the laboratory regarding the effectiveness of the quality system, evaluating the effectiveness of training; and monitor trends and continually improve the quality system. Audit and surveillance results, control charts, proficiency testing results, data analysis, corrective and preventive actions, customer feedback, and management reviews can all are used to support quality system implementation. The QA PM is responsible for ensuring compliance with NELAC standards (and ISO, DoD QSM, etc. as applicable). The QA PM works with laboratory staff to establish effective quality control and assessment plans and has the authority to stop work in response to quality problems. The QA PM is responsible for maintaining the QA Manual and performing an annual review of it; reviewing and approving SOPs and ensuring the annual review of technical SOPs; maintaining QA records such as metrological records, archived logbooks, PT results, etc.; document control; conducting PT sample studies; approving nonconformity and corrective action reports; maintaining the laboratory s certifications and approvals; and performing internal QA audits.

The QA PM reports directly to the Laboratory Director and also reports indirectly to the Chief Quality Officer. It is important to note that when evaluating data, the QA PM does so in an objective manner and free of outside, or managerial, influence.

The Chief Quality Officer (CQO) is responsible for the overall QA program at all the Columbia Analytical laboratories. The CQO is responsible for oversight of QA PMs regulatory compliance efforts (NELAC, ISO, DOD, etc). The CQO performs annual internal audits at each laboratory; maintains a database of laboratory certification/accreditation programs; approves company-wide SOPs; maintains a database of approved subcontract laboratories; provides assistance to the laboratory QA staff and laboratory managers; prepares a quarterly QA activity report; etc.

- In the case of absence of the Laboratory Director or QA PM, deputies are assigned to act in that role. Default deputies for these positions are the Client Services Manager or Metals Department Manager (for the Laboratory Director) and the CQO or Laboratory Director (for the QA PM).
- In the event that work is stopped in response to quality problems, only the Laboratory Director or Quality Assurance Program Manager has the authority to resume work.

- The **Environmental Health and Safety Officer** (EH S) is responsible for the administration of the laboratory health and safety policies. This includes the formulation and implementation of safety policies, the supervision of new-employee safety training, the review of accidents, incidents and prevention plans, the monitoring of hazardous waste disposal and the conducting of departmental safety inspections. The EH S officer is also designated as the Chemical Hygiene Officer. The EH S Officer has a dotted-line reporting responsibility to Columbia Analytical s EH S Director.

- The **Client Services Manager** is responsible for the Client Services Department defined for the laboratory (i.e. Project Managers, electronic deliverables, etc.) and the sample management office/bottle preparation sections. The Client Services Department provides a complete interface with clients from initial project specification to final deliverables. Sample management handles all activities associated with receiving, storage, and disposal of samples. The Client Services Manager has the authority to stop subcontractor work in response to quality problems.
- The **Project Manager** is a scientist assigned to each client to act as a technical liaison between the client and the laboratory. The Project Manager is responsible for ensuring that the analyses performed by the laboratory meet all project, contract, and regulatory-specific requirements. This entails coordinating with the Columbia Analytical laboratory and administrative staff to ensure that client-specific needs are understood and that the services Columbia Analytical provides are properly executed and satisfy the requirements of the client.
- The Analytical Laboratory is divided into operational units based upon specific disciplines. Each department is responsible for establishing, maintaining and documenting a QC program meeting department needs. Each **Department Manager and Supervisor** has the responsibility to ensure that QC functions are carried out as planned, and to guarantee the production of high quality data. Managers and bench-level supervisors monitor the day-to-day operations to ensure that productivity and data quality objectives are met. A department manager has the authority to stop work in response to quality problems in their area. Analysts have the responsibility to carry out testing according to prescribed methods, SOPs, and quality control guidelines particular to the laboratory in which he/she is working.
- The **Sample Management Office** plays a key role in the laboratory QA program by maintaining documentation for all samples received by the laboratory, and by assisting in the archival of all

laboratory results. The sample management office staff is also responsible for the proper disposal of samples after analysis.

- **Information Technology** (IT) staff is responsible for the administration of the Laboratory Information Management System (LIMS) and other necessary support services. Other functions of the IT staff include laboratory network maintenance, IT systems development and implementation, education of analytical staff in the use of scientific software, Electronic Data Deliverable (EDD) generation, and data back-up, archival and integrity operations.

Table 5-1
Summary of Technical Experience and Qualifications

Personnel	Years of Experience	Project Role
Jeff Grindstaff, B.S.	22	Laboratory Director
Julie Gish, M.S.	19	Quality Assurance Program Manager
Lynda Huckestein, B.S.	22	Client Services Manager Sample Management Office Manager
Jeff Coronado, B.S.	21	Metals Department Manager
Harvey Jacky, B.S.	22	General Chemistry Department Manager
Aqilla Kamawal, B.A.	11	Semi-Volatile Organics Department Manager
Jon James, B.A.	20	HPLC, GC/MS Organics Department Manager
Christina Kerksieck, B.S.	3	Microbiology Technical Manager
Elieen Arnold, B.A.	29	Environmental Health and Safety Officer
Mike Sullivan, B.S.	11	Information Technology
Jeff Christian, B.S.	32	Chief Operations Officer
Lee Wolf, B.S.	26	Chief Quality Officer/Quality Assurance Director

6.0 INFORMATION MANAGEMENT

The generation, compilation, reporting, and archiving of electronic data is a critical component of laboratory operations. In order to generate data of known and acceptable quality, the quality assurance systems and quality control practices for electronic data systems must be complete and comprehensive and in keeping with the overall quality assurance objectives of the organization. CAS management provides the tools and resources to implement electronic data systems and establishes information technology standards and policies. Appendix C lists major computing equipment.

6.1 Software Quality Assurance Plan

Columbia Analytical has defined practices for assuring the quality of the computer software used throughout all laboratory operations to generate, compile, report, and store electronic data. These practices are described in the *CAS Software Quality Assurance Plan (SQAP)*. The purpose of the SQAP is to describe the policies and practices for the procurement, configuration management, development, validation and verification, data security, maintenance, and use of computer software. The policies and practices described in the plan apply to purchased computer software as well as to internally developed computer software. Key components of this plan are policies for software validation and control.

6.2 IT Support

The local Columbia Analytical Information Technology (IT) department is established to provide technical support for all computing systems. The IT department staff continually monitors the performance and output of operating systems. The IT department oversees routine system maintenance and data backups to ensure the integrity of all electronic data. A software inventory is maintained. Additional IT responsibilities are described in the SQAP.

In addition to the local IT department, Columbia Analytical corporate IT provides support for network-wide systems. Columbia Analytical also has personnel assigned to information management duties such as development and implementation of reporting systems; data acquisition, and Electronic Data Deliverable (EDD) generation.

6.3 Information Management Systems

Columbia Analytical has various systems in place to address specific data management needs. The Columbia Analytical Laboratory Information Management System (LIMS) is used to manage sample information and invoicing. Access is controlled by password. This system defines sample identification, analysis specifications, and provides a means of sample tracking. This system is used during sample login to generate the internal service request. Included on the service request is a summary of client information, sample identification, required analyses, work instructions, deliverable requirements. The LIMS is used to track the status of a sample and is important in maintaining internal chain of custody.

Where possible, instrument data acquired locally is immediately moved to a server (Microsoft Windows2003 domain). This provides a reliable, easily maintained, high-volume acquisition and storage system for electronic data files. With password entry, users may access the system from many available computer stations, improving efficiency and flexibility. The server is also used for data reporting, EDD generation, and administrative functions. Access to these systems is controlled by password. A standardized EDI (electronic data interchange) format is used as a reporting platform, providing functionality and flexibility for end users. With a common standardized communication platform, the EDI provides data reporting in a variety of hardcopy and electronic deliverable formats, including Staged Electronic Data Deliverable (SEDD) format.

6.4 Backup and Security

Columbia Analytical laboratory data is either acquired directly to the centralized acquisition server or acquired locally and then transferred to the server. All data is eventually moved to the centralized data acquisition server for reporting and archiving. Differential backups are performed on all file server information once per day, Sunday through Thursday. Full backups are performed each Friday night. Tapes are physically stored in a locked media cabinet within a locked, temperature controlled computer room, with every other full backup also securely stored offsite.

Access to sample information and data is on a need-to-know basis. Access is restricted to the person s areas of responsibility. Passwords are required on all systems. No direct external, non- Columbia Analytical access is allowed to any of our network systems.

The external e-mail system and Internet access is established via a single gateway to discourage unauthorized entry. Columbia Analytical uses a closed system for company e-mail. Files, such as electronic deliverables, are sent through the external e-mail system only via a trusted agent. The external messaging system operates through a single secure gateway. Email attachments sent in and out of the gateway are subject to a virus scan. Because the Internet is not regulated, we use a limited access approach to provide a firewall for added security. Virus screening is performed continuously on all network systems.

7.0 SAMPLE MANAGEMENT

7.1 Sampling and Sample Preservation

The quality of analytical results is highly dependent upon the quality of the procedures used to collect, preserve and store samples. Columbia Analytical recommends that clients follow sampling guidelines described in 40 CFR 136, 40 CFR 141, USEPA SW-846, and state-specific sampling guidelines, if applicable. Sampling factors that must be taken into account to insure accurate, defensible analytical results include:

- Amount of sample taken
- Type of container used
- Type of sample preservation
- Sample storage time
- Proper custodial documentation

Columbia Analytical uses the sample preservation, container, and holding-time recommendations published in a number of documents. The primary documents of reference are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IV for hazardous waste samples; USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, and Supplements; EPA 40CFR parts 136 and 141; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples (see Section 18 for complete citations). The container, preservation and holding time information for these references is summarized in Table 7-1 for soil, water, and drinking water. The current EPA CLP Statement of Work should be referred to for CLP procedures. Where allowed by project sampling and analysis protocols (such as Puget Sound Protocols) the holding time for sediment, soil, and tissue samples may be extended for a defined period when stored frozen at -20°C.

Columbia Analytical routinely provides sample containers with appropriate preservatives for our clients. Containers are purchased as precleaned to a level 1 status, and conform to the requirements for samples established by the USEPA. Certificates of analysis for the sample containers are available to clients if requested. Reagent water used for sampling blanks (trip blanks, etc.) and chemical preservation reagents are tested by the laboratory to ensure that they are free of interferences and documented. Our sample kits typically consist of foam-lined, precleaned shipping coolers, (cleaned inside and out with appropriate cleaner, rinsed thoroughly and air-dried), specially prepared and labeled sample containers individually wrapped in protective material, (VOC vials are placed in a specially made, foam holder), chain-of-custody (COC) forms, and custody seals. Container labels and custody seals are provided for each container.

Figure 7-1 shows the chain-of-custody form routinely used at Columbia Analytical and included with sample kits. For large sample container shipments, the containers may be shipped in their original boxes. Such shipments will consist of several boxes of labeled sample containers and sufficient materials (bubble wrap, COC forms, custody seals, shipping coolers, etc.) to allow the sampling personnel to process the sample containers and return them to Columbia Analytical. The proper preservative is added to the sample containers prior to shipment, unless otherwise instructed by the client.

If any returning shipping cooler exhibits an odor or other abnormality after receipt and subsequent decontamination by laboratory personnel, a second, more vigorous decontamination process is employed. Containers exhibiting an odor or abnormality after the second decontamination process are promptly and properly discarded. Columbia Analytical keeps client-specific shipping requirements on file and utilizes major transportation carriers to guarantee that sample shipping requirements (same-day, overnight, etc.) are met. Columbia Analytical also provides courier service that makes regularly scheduled trips to the Greater Portland, Oregon Metropolitan area.

When Columbia Analytical ships environmental samples to other laboratories for analysis each sample bottle is wrapped in protective material and placed in a plastic bag (preferably Ziploc) to avoid any possible cross-contamination of samples during shipping. The sample management office (SMO) follows formalized procedures (SMO-GEN) for maintaining the samples chain of custody, packaging and shipment. Dry ice gel ice is the only temperature preservative used by Columbia Analytical, unless otherwise specified by the client or receiving laboratory.

7.2 Sample Receipt and Handling

Standard Operating Procedures (SMO-GEN) are established for the receiving of samples into the laboratory. These procedures ensure that samples are received and properly logged into the laboratory, and that all associated documentation, including chain of custody forms, is complete and consistent with the samples received.

Once samples are delivered to the Columbia Analytical sample management office (SMO), a Cooler Receipt and Preservation Check Form (CRF - See Figure 7-2 for an example) is used to assess the shipping cooler and its contents as received by the laboratory personnel. Verification of sample integrity includes the following activities:

- Assessment of custody seal presence/absence, location and signature;
- Temperature of sample containers upon receipt;
- Chain of custody documents properly used (entries in ink, signature present, etc.);
- Sample containers checked for integrity (broken, leaking, etc.);

- Sample is clearly marked and dated (bottle labels complete with required information);
- Appropriate containers (size, type) are received for the requested analyses;
- The minimum amount of sample material is provided for the analysis.
- Sample container labels and/or tags agree with chain of custody entries (identification, required analyses, etc.);
- Assessment of proper sample preservation (if inadequate, corrective action is employed); and
- VOC containers are inspected for the presence/absence of bubbles. (Assessment of proper preservation of VOC containers is performed by lab personnel).

Samples are logged into a Laboratory Information Management System (LIMS). Any anomalies or discrepancies observed during the initial assessment are recorded on the CRF and COC documents. Potential problems with a sample shipment are addressed by contacting the client and discussing the pertinent issues. When the Project Manager and client have reached a satisfactory resolution, the login process may continue and analysis may begin. During the login process, each sample container is given a unique laboratory code and a service request form is generated. The LIMS generates a Service Request that contains client information, sample descriptions, sample matrix information, required analyses, sample collection dates, analysis due dates and other pertinent information. The service request is reviewed by the appropriate Project Manager for accuracy, completeness, and consistency of requested analyses and for client project objectives.

Samples are stored as per method requirements until they undergo analysis, unless otherwise specified, using various refrigerators or freezers, or designated secure areas. Columbia Analytical has five walk-in cold storage units which house the majority of sample containers received at the laboratory. In addition, there are four additional refrigerators, including dedicated refrigerated storage of VOC samples. The dedicated storage areas for VOC samples are monitored using storage blanks, as described in the *SOP for VOA Storage Blanks (VOC-BLAN)*. Columbia Analytical also has nine sub-zero freezers capable of storing samples at -10 to -30° C primarily used for tissue and sediment samples requiring specialized storage conditions. The temperature of each sample storage unit is monitored real time with an electronic temperature monitoring system.

Columbia Analytical adheres to the method-prescribed or project-specified holding times for all analyses. The sampling date and time are entered into the LIMS system at the time of sample receipt and login. Analysts then monitor holding times by obtaining analysis-specific reports from the LIMS. These reports provide holding time information on all samples for the analysis, calculated from the sampling date and the holding time requirement. To document holding time compliance, the date and time analyzed is printed or written on the analytical raw data. For analyses with a holding time prescribed in hours it is essential that the sample collection time is provided, so holding time compliance can be demonstrated. If not, the sample collection time is assumed as the earliest in the day (i.e. the most conservative). Unless other arrangements have been made in advance, upon completion of all analyses and submittal of the final report, aqueous samples and sample extracts are retained at ambient temperature for 30 days, soil samples are retained at ambient temperature for 60 days, and tissue samples are retained frozen for 3 months. Upon expiration of these time limits, the samples are either returned to the client or disposed of according to approved disposal practices. All samples are characterized according to hazardous/non-hazardous waste criteria and are segregated accordingly. All hazardous waste samples are disposed of according to formal procedures

outlined in the *CAS Environmental Health and Safety Manual.* All waste produced at the laboratory, including the laboratory s own various hazardous waste streams, is treated in accordance with applicable local and Federal laws. Documentation is maintained for each sample from initial receipt through final disposal to ensure that an accurate history of the sample from cradle to grave is available.

7.3 Sample Custody

Sample custody transfer at the time of sample receipt is documented using chain-of-custody (COC) forms accompanying the samples. During sample receipt, it is also noted if custody seals were present. This is described in the *SOP for Sample Receiving (SMO-GEN)*. Figure 7-1 is a copy of the chain-of-custody form routinely used at Columbia Analytical.

Facility security and access is important in maintaining the integrity of samples received at Columbia Analytical-Kelso. Access to the laboratory facility is limited by use of locked exterior doors with a coded entry, except for the reception area and sample receiving doors, which are manned during business hours and locked at all other times. In addition, the sample storage area within the laboratory is a controlled access area with locked doors with a coded entry. The Columbia Analytical facility is equipped with an alarm system and Columbia Analytical employs a private security firm to provide nighttime and weekend security.

A barcoding system is used to document internal sample custody. Each person removing or returning samples from/to sample storage while performing analysis is required to document this custody transfer. The system uniquely identifies the sample container and provides an electronic record of the custody of each sample. For sample extracts and digestates the analyst documents custody of the sample extract or digestate by signing on the benchsheet, or custody record, that they have accepted custody. The procedures are described in the *SOP for Sample Tracking and Internal Chain of Custody (SMO-SCOC).*

7.4 Project Setup

The analytical method(s) used for sample analysis are chosen based on the client s requirements. Unless specified otherwise, the most recent versions of reference methods are used. For SW-846 methods, some projects may require the most recent *promulgated* version, and some projects may require the most recent *published* version. The Project Manager will ensure that the correct method version is used. LIMS codes are chosen to identify the analysis method used for analysis. The Project Manager ensures that the correct methods are selected for analysis, deliverable requirements are identified, and due dates are specified on the service request. To communicate and specify project-specific requirements, a Tier V form (Figure 7-3) is used and accompanies the service request form.

Table 7-1
Sample Preservation and Holding Times

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Bacterial Tests				
Coliform, Colilert (SM 9223)	W, DW	P, Bottle or Bag	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Coliform, Fecal and Total (SM 9221, 9222D)	W, S, DW	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Fecal Streptococci (SM 9230B)	W	P,G	Cool, 4°C, 0.008% $Na_2S_2O_3$[d]	6-24 hours[e]
Inorganic Tests				
Acidity (SM 2310B)	W	P,G	Cool, 4°C	14 days[EPA]
Alkalinity (SM 2320B)	W, DW	P,G	Cool, 4°C	14 days[EPA]
Ammonia (SM 4500NH3)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH 2	28 days
Biochemical Oxygen Demand(SM 5210B)	W	P,G	Cool, 4°C	48 hours
Bromate (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	28 days
Bromide (EPA 300.1)	W, DW	P,G	None Required	28 days
Chemical Oxygen Demand (SM 5220C)	W	P,G	Cool, 4°C, H_2SO_4 to pH 2	28 days
Chloride (EPA 300.0)	W, DW	P,G	None Required	28 days
Chloride (EPA 9056)	W, S	P,G	Cool, 4°C	28 days
Chlorine, Total Residual (SM 4500 Cl F)	W,S	P,G	None Required	24 hours
Chlorite (EPA 300.1)	W, DW	P,G	50mg/L EDA, cool to 4°C	14 days
Chlorophyll-A (SM 11200H)	W	G Amber	Cool, 4°C	Analyze immediately
Chromium VI (EPA 7196A)	W	P,G	Cool, 4°C	24 hours
Color (SM 2120B)	W, DW	P,G	Cool, 4°C	48 hours
Cyanide, Total and Amenable to Chlorination (EPA 335.4, 9010, 9012) (SM 4500CN E,G)	W, S,DW	P,G	Cool, 4°C, NaOH to pH>12, plus 0.6 g Ascorbic Acid	14 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Inorganic Tests				
Cyanide, Weak Acid Dissociable (SM 4500CN I)	W,S	P,G	Cool, 4°C, NaOH to pH >12	14 days
Ferrous Iron (CAS SOP)	W, D	G Amber	Cool, 4°C	24 hours
Fluoride (EPA 300.0, SM 4500 F-C)	W,S	P,G	Cool, 4°C	28 days
Fluoride (EPA 9056)	W,S	P,G	Cool, 4°C	Analyze immediately
Formaldehyde (ASTM D6303)	W	G Amber	Cool, 4°C	48 hours
Hardness (SM 2340C)	W, DW	P,G	HNO_3 to pH 2	6 months
Hydrogen Ion (pH) (SM 4500H B)	W, DW	P,G	None Required	Analyze immediately
Kjeldahl and Organic Nitrogen (ASTM D3590-89)	W	P,G	Cool, 4°C, H_2SO_4 to pH 2	28 days
Nitrocellulose	S	G	Cool, 4°C	28 days
Nitrate (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrate (EPA 353.2)	W, S	P,G	Cool, 4°C, H_2SO_4 to pH 2	48 hours
Nitrate (EPA 9056)	W,S	P,G	Cool, 4°C	Analyze immediately
Nitrate-Nitrite (EPA 353.2)	W, DW	P,G	Cool, 4°C, H_2SO_4 to pH 2	28 days
Nitrite (EPA 300.0)	W, DW	P,G	Cool, 4°C	48 hours
Nitrite (EPA 353.2)	W, S	P,G	Cool, 4°C, H_2SO_4 to pH 2	48 hours
Nitrite (EPA 9056)	W,S	P,G	Cool, 4°C	Analyze immediately
Orthophosphate (SM 4500 P-E)	W, DW	P,G	Cool, 4°C	Analyze immediately
Oxygen, Dissolved (Probe) (SM 4500O G)	W, DW	G, Bottle and Top	None Required	Analyze immediately

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Inorganic Tests				
Oxygen, Dissolved (Winkler)	W, DW	G, Bottle and Top	Fix on Site and Store in Dark	8 hours
Phenolics, Total (EPA 420.1,9056)	W, S	G Amber	Cool, 4°C, H_2SO_4 to pH 4	28 days
Perchlorate (EPA 314.0)	W, DW,S	P,G	Protect from temp. extremes	28 days
Phosphorus, Total (EPA 365.3)	W	P,G	Cool, 4°C, H_2SO_4 to pH 2	28 days
Residue, Total (SM 2540B)	W	P,G	Cool, 4°C	7 days
Residue, Filterable (TDS) (SM2540C)	W	P,G	Cool, 4°C	7 days
Residue, Nonfilterable (TSS) (SM 2540D)	W	P,G	Cool, 4°C	7 days
Residue, Settleable (SM 2540F)	W	P,G	Cool, 4°C	48 hours
Residue, Volatile (EPA 160.4)	W	P,G	Cool, 4°C	7 days
Silica (SM 4500SiO2 C)	W	P Only	Cool, 4°C	28 days
Specific Conductance(SM 2510 B)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 300.0)	W, DW	P,G	Cool, 4°C	28 days
Sulfate (EPA 9056)	W, S	P,G	Cool, 4°C	28 days
Sulfide (SM 4500S2 D)	W	P,G	Cool, 4°C, Add Zinc Acetate,plus Sodium Hydroxide to pH>9	7 days
Sulfide (SM 4500S2 F)	W	P,G	Cool, 4°C, Add Zinc Acetate,plus Sodium Hydroxide to pH>9	7 days
Sulfide (9030/934)	W, S	P,G	Cool, 4°C, Add Zinc Acetate,plus Sodium Hydroxide to pH>9	7 days
Sullfides, Acid Voaltile	S	G	Cool, 4°C	14 days
Sulfite (SM 4500SO3 B)	W	P,G	None Required	24 hours
Surfactants (MBAS) (SM 5540 C)	W	P,G	Cool, 4°C	48 hours

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Inorganic Tests				
Tannin and Lignin (SM 5550B)	W	P,G	Cool, 4°C	28 days
Turbidity (EPA 180.1)	W, DW	P,G	Cool, 4°C	48 hours
Oil and Grease, Hexane Extractable Material (EPA 1664)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 or HCL to pH 2	28 days
Organic Carbon, Total (9060 SM 5310 C)	W	P,G	Cool, 4°C, H_2SO_4 to pH 2	28 days
Organic Carbon, Total (ASTM-D4129)	S	P,G	Cool, 4°C	28 days
Organic Halogens, Total (EPA 9020)	W	G, Teflon-Lined Cap	Cool, 4°C, H_2SO_4 to pH 2, No headspace	28 days
Organic Halogens, Adsorbable (EPA 1650B)	W	G, Teflon-Lined Cap	Cool, 4°C, HNO_3 to pH 2	6 months

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Metals				
Chromium VI (EPA 7195/7191)	W	P,G	Cool, 4°C	24 hours
Metals (200.7, 200.8, 200.9, 6010, 6020)	W,DW	P,G	HNO_3 to pH 2	6 months
Metals (200.7, 200.8, 200.9, 6010, 6020)	S	G, Teflon-Lined cap	Cool, 4°C	6 months
Mercury (EPA 245.1, 7470, 7471)	W, DW	P,G	HNO_3 to pH 2	28 days
Mercury (7471)	S	P,G	Cool, 4°C	28 days
1631E	W	F	Cool, 4°C, HCl or H_2SO_4 to pH 2	90 days
1631E	S	F	Freeze -15 C	1 Yr
Methyl Mercury 1630	W,S,T	F	HCL to pH 2	6 months
Arsenic Species 1632	W	G	HCL to pH 2, Cool 4 C	28 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Gasoline Range Organics (8015, NWTPH-Gx)	W	G, Teflon-Lined, Septum Cap	Cool, 4°C, HCl to pH 2, No headspace	14 days
Gasoline Range Organics (8015, NWTPH-Gx)	S	G, Teflon-Lined Cap	Cool, 4 C, Minimize Headspace	14 days
Purgeable Halocarbons (624, 8021, 8260)	W	G, Teflon-Lined, Septum Cap	**No Residual Chlorine Present**: HCl to pH 2, Cool, 4°C, No Headspace	14 days
Purgeable Halocarbons (624, 8021, 8260)	W	G, Teflon-Lined, Septum Cap	**Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH 2, Cool, 4°C	14 days
Purgeable Halocarbons (8021, 8260)	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
Purgeable Halocarbons (8021, 8260)	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4C	48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Halocarbons (8021, 8260)	S	Method 5035	Sodium Bisulfate Cool, 4°C	48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	W	G, Teflon-Lined,Septum Cap, No Headspace	**No Residual Chlorine Present**: HCl to pH 2, Cool, 4°C, No Headspace	14 days
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	W	G, Teflon-Lined,Septum Cap, No Headspace	**Residual Chlorine Present**: 10% $Na_2S_2O_3$, HCl to pH 2, Cool 4°C	14 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Volatile Organics				
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	S	G, Teflon-Lined Cap	Cool, 4°C, Minimize Headspace	14 days
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	S	Method 5035	Encore, Freeze at -20°C Methanol, Cool, 4C	48 hrs to prepare from Encore, 14 days after preparation.
Purgeable Aromatic Hydrocarbons (including BTEX and MTBE 624, 8021, 8260)	S	Method 5035	Sodium Bisulfate Cool, 4°C	48 hrs to prepare from Encore, 14 days after preparation.
Acrolein, Acrylonitrile, Acetonitrile (624, 8260)	W	G, Teflon-Lined, Septum Cap	Adjust pH to 4-5, Cool, 4°C, No headspace	7 days
EDB and DBCP (EPA 8260)	W,S	G, Teflon-Lined Cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	28 days
Vinyl chloride,styrene, 2-chloroethyl vinyl ether (8260)	W	G, Teflon-Lined, Septum Cap	Cool, 4°C, Minimize Headspace	7 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Nonyl Phenols	W	G, Teflon-Lined Cap	H2SO4 to pH 2, Cool, 4°C	28 days
Organotins (CAS SOP)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7[f] days until extraction;40 days after extraction
Otto Fuel	W	G, Teflon-Lined Cap	Cool, 4°C	7[f] days until extraction;40 days after extraction
Resin and Fatty Acids (NCASI 85.02)	W	G, Teflon-Lined Cap	NaOH to pH >10, Cool, 4°Cg	30 days until extraction; 30 days after extraction
Methanol in Process Liquid NCASI 94.03	L	G, Teflon-Lined Cap	Cool, 4°C	30 days
HAPS Condensates NCASI 99.01		G, Teflon-Lined Cap	Cool, 4°C	14/30 days
HAPS Impinger/Canisters NCASI 99.02			Cool, 4°C	21 days
Perfluorinated Compounds HPLC/MS/MS	W	P	Cool, 4°C	14 days until extraction; 40 days after extraction
PBDE/PBB ROHS GC/MS	W,S,T	G	Cool, 4°C	40 days after extraction
Pharma Personal Care Products 1694	W	Amber G, Teflon-Lined Cap	Cool, 6°C	7[f] days until extraction;30 days after extraction
Nitroaromatics and Nitramines 8330B	W,S	G, Teflon-Lined Cap	Cool, 4°C	S 14, W 7 days until extraction; 40 days after extraction
Nitroaromatics/Nitoramines HPLC/MS/MS	W,S,T	G	Cool, 4°C Tissues -10 C	S 14, W 7 days until extraction; 40 days after extraction
Organic acids HPLC/MS/MS	W	G, Teflon-Lined, Septum Cap	H2SO4 to pH 2, Cool, 4°C	14 days

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Semivolatile Organics				
Petroleum Hydrocarbons, Extractable (Diesel-Range Organics) (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7[f] days until extraction;40 days after extraction
Alcohols and Glycols (EPA 8015)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7[f] days until extraction;40 days after extraction
Acid Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7[f] days until extraction;40 days after extraction
Base/Neutral Extractable Semivolatile Organics (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7[f] days until extraction;40 days after extraction
Chlorinated Herbicides (EPA 8151)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7[f] days until extraction;40 days after extraction
Chlorinated Phenolics (EPA 1653)	W	G, Teflon-Lined Cap	H2SO4 to pH 2, Cool, 4°Cg	30 days until extraction; 30 days after extraction
Polynuclear Aromatic Hydrocarbons (EPA 625, 8270)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Darkg	7[f] days until extraction;40 days after extraction
Organochlorine Pesticides and PCBs (EPA 608, 8081, 8082, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C	7[f] days until extraction;40 days after extraction
Organophosphorus Pesticides (EPA 8141, GC/MS/MS)	W,S	G, Teflon-Lined Cap	Cool, 4°C, Store in Darkg	7[f] days until extraction;40 days after extraction
Nitrogen- and Phosphorus-Containing Pesticides (EPA 8141)	W,S	G, Teflon-Lined Cap	Cool, 4°Cg	7[f] days until extraction;40 days after extraction

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Drinking Water Organics				
Purgeable Organics (EPA 524.2)	DW	G, Teflon-Lined, Septum cap	Ascorbic Acid, HCl to pH_2, Cool, 4°C, No Headspace	14 days
EDB, DBCP, and TCP (EPA 504.1)	DW	G, Teflon-Lined, Septum cap	Cool, 4°C, 3 mg $Na_2S_2O_3$, No Headspace	14 days
Carbamates, Carbamoyloximes (EPA 531.1)	DW	G, Amber, Teflon-Lined Cap	1.8 mL monochloroacetic acid to pH 3; 80 mg/L $Na_2S_2O_3$ if Res.Cl.; Cool, 4°C	28 days
Chlorinated Herbicides (EPA 515.4)	DW	G, Amber, Teflon-Lined Cap	If Res.Cl, 2mg/4omL NaS; Cool , 6 C	14 days until extraction; 21 days after extraction
Chlorinated Pesticides (EPA 508.1, 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH_ 2;Cool 4 C	14 days until extraction; 30 days after extraction
Diquat and Paraquat (EPA 549.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$ if Res.Cl.Cool 4 C	7days until extraction; 21 days after extraction
Endothall (EPA 548.1)	DW	G, Amber, Teflon-Lined Cap	Cool, 4°C	7 days until extraction; 14 days after extraction
Glyphosate (EPA 547)	DW	G, Amber, Teflon-Lined Cap	100 mg/L $Na_2S_2O_3$,Cool, 4°C	14 days
Haloacetic Acids (EPA 552.2)	DW	G, Amber, Teflon-Lined Cap	100 mg/L NH_4Cl,Cool, 4°C	14 days until extraction; 7 days after extraction
Semivolatile Organics (EPA 525.2)	DW	G, Amber, Teflon-Lined Cap	50 mg/L NaS, HCl to pH_ 2;Cool, 4°C	14 days until extraction; 30 days after extraction
Nitrosoamines (EPA 521)	DW	G, Amber, Teflon-Lined Cap	Dechlorinate at collection[g]	14 days until extraction; 28 days after extraction
Selected Pesticides and Flame Retardants (EPA 527)	DW	G, Amber, Teflon-Lined Cap	See Method, Cool, 4°C	14 days until extraction; 28 days after extraction

Table 7-1 (continued)
Sample Preservation and Holding Times[a]

DETERMINATION[a]	MATRIX[b]	CONTAINER[c]	PRESERVATION	MAXIMUM HOLDING TIME
Toxicity Characteristic Leaching Procedure (TCLP)				
Semivolatile Organics (EPA 1311/8270)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C, Store in Dark[g]	14 days until TCLP ext'n;
			TCLP extract: Cool, 4°C, Store in Dark[g]	7 days until extraction; 40 days after extraction
Organochlorine Pesticides (EPA 1311/8081)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C	14 days until TCLP ext'n;
			TCLP extract: Cool, 4°C	7 days until extraction; 40 days after extraction
Chlorinated Herbicides (EPA 1311/8151)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C	14 days until TCLP ext'n;
			TCLP extract: Cool, 4°C	7 days until extraction; 40 days after extraction
Mercury(EPA 1311/7470)	HW	P,G	Sample: Cool, 4°C	28 days until extraction
			TCLP extract: HNO_3 to pH 2	28 days after extraction
Metals, except Mercury (EPA 1311/6010)	HW	P,G	Sample: Cool, 4°C	180 days until extraction;
			TCLP extract: HNO_3 to pH 2	14 days until TCLP ext'n;
Volatile Organics (EPA 1311/8260)	HW	G, Teflon-Lined Cap	Sample: Cool, 4°C , Minimize Headspace	14 days until TCLP ext'n;
			Extract: Cool 4°C, HCL to pH,2, No Headspace	14 days after extraction

a For EPA SW-846 methods the method number is listed generically, without specific revision suffixes.

b DW = Drinking Water, W = Water; S = Soil or Sediment; HW = Hazardous Waste

c P = Polyethylene; G = Glass, F- Fluoropolymer

d For chlorinated water samples

e The maximum holding time is dependent upon the geographical proximity of sample source to the laboratory.

f Fourteen days until extraction for soil, sediment, and sludge samples.

g If the water sample contains residual chlorine, 10% sodium thiosulfate is used to dechlorinate.

Figure 7-1
Chain of Custody Form

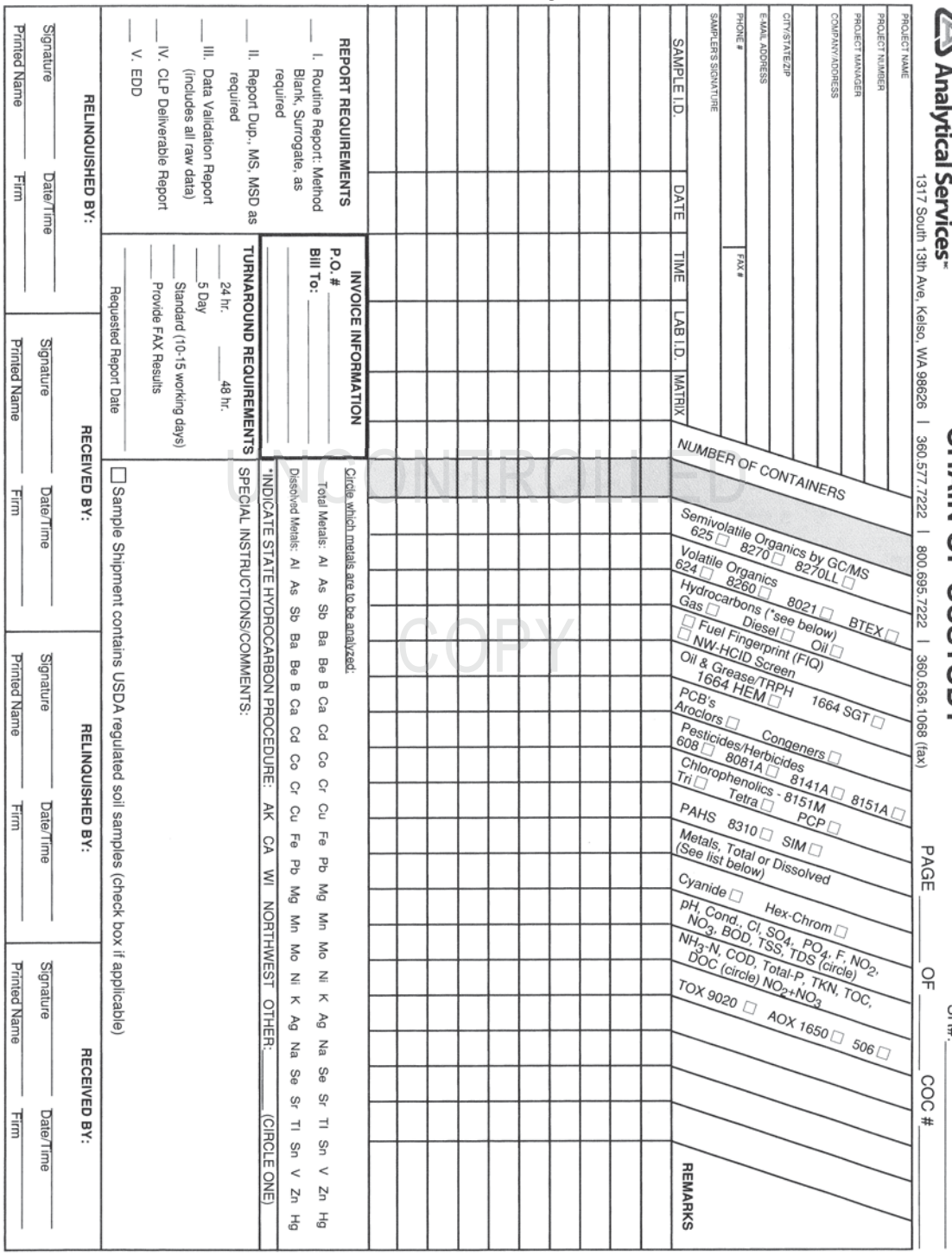


Figure 7-2

Columbia Analytical Services, Inc.
Cooler Receipt and Preservation Form

PC_____

Client / Project:_____ Service Request *K11*_____

Received:_____ Opened:_____ By:_____ Unloaded:_____ By:_____

1. Samples were received via? ***Mail*** ***Fed Ex*** ***UPS*** ***DHL*** ***PDX*** ***Courier*** ***Hand Delivered***
2. Samples were received in: (circle) ***Cooler*** ***Box*** ***Envelope*** ***Other***_____ ***NA***
3. Were custody seals on coolers? NA Y N If yes, how many and where?_____
   If present, were custody seals intact? Y N If present, were they signed and dated? Y N

Cooler Temp °C	Temp Blank °C	Thermometer ID	Cooler/COC ID	NA	Tracking Number	NA	Filed

7. Packing material used. ***Inserts*** ***Baggies*** ***Bubble Wrap*** ***Gel Packs*** ***Wet Ice*** ***Sleeves*** ***Other***_____
8. Were custody papers properly filled out (ink, signed, etc.)? NA Y N
9. Did all bottles arrive in good condition (unbroken)? *Indicate in the table below.* NA Y N
10. Were all sample labels complete (i.e analysis, preservation, etc.)? NA Y N
11. Did all sample labels and tags agree with custody papers? *Indicate major discrepancies in the table on page 2.* NA Y N
12. Were appropriate bottles/containers and volumes received for the tests indicated? NA Y N
13. Were the pH-preserved bottles (*see SMO GEN SOP*) received at the appropriate pH? *Indicate in the table below* NA Y N
14. Were VOA vials received without headspace? *Indicate in the table below.* NA Y N
15. Was C12/Res negative? NA Y N

Sample ID on Bottle	Sample ID on COC	Identified by:

Sample ID	Bottle Count Bottle Type	Out of Temp	Head-space	Broke	pH	Reagent	Volume added	Reagent Lot Number	Initials	Time

Notes, Discrepancies, & Resolutions:_____

*Page 1 of*_____

Figure 7-2 cont.

Columbia Analytical Services Inc.
Cooler Receipt and Preservation Form

Client / Project: _____ Service Request *K11* _____

Sample ID on Bottle	Sample ID on COC	Identified by:

Sample ID	Bottle Count Bottle Type	Out of Temp	Head-space	Broke	pH	Reagent	Volume added	Reagent Lot Number	Initials	Time

Notes, Discrepancies & Resolutions:

_____

_____

_____

_____

_____

Figure 7-3

Tier V Form

Client: Project Chemist:
Project Name: Service Request:
Project Number: LIMS Template ID:
Project Description:

QAPP/SOW Information:

Reporting

TierLevel: PFD: Report to:
In results field use:
Flagging Requirements:
Other Requirements:

Sample Considerations:

Sample Limitations:
Sample Prep/Analysis:
Non-Standard Holdtimes:
Historical Data:
Comments:

8.0 ANALYTICAL PROCEDURES

Columbia Analytical employs methods and analytical procedures from a variety of external sources. The primary method references are: USEPA SW-846, Third Edition and Updates I, II, IIA, IIB, III, IVA, IVB, and online updates for hazardous waste samples, and USEPA 600/4-79-020, 600/4-91-010, 600/4-82-057, 600/R-93/100, 600/4-88-039, 600/R-94-111, EPA 40CFR parts 136 and 141, and Supplements; and *Standard Methods for the Examination of Water and Wastewater* for water and wastewater samples. Complete citations for these references can be found in Section 17.0. Other published procedures, such as state-specific methods, program-specific methods (such as Puget Sound Protocols), or in-house methods may be used. Several factors are involved with the selection of analytical methods to be used in the laboratory. These include the method detection limit, the concentration of the analyte being measured, method selectivity, accuracy and precision of the method, the type of sample being analyzed, and the regulatory compliance objectives. The implementation of methods by Columbia Analytical is described in SOPs specific to each method. A list of NELAP-accredited methods is given in Appendix G. Further details are described below.

8.1 Standard Operating Procedures (SOPs) and Laboratory Notebooks.

Columbia Analytical maintains SOPs for use in both technical and administrative functions. SOPs are written following standardized format and content requirements as described in the *SOP for Preparation of Standard Operating Procedures*. Each SOP is reviewed and approved by a minimum of two managers (the Laboratory Director and/or Department Manager and the Quality Assurance Program Manager). All SOPs undergo a documented annual review to make sure current practices are described. The QA PM maintains a comprehensive list of current SOPs. The document control process ensures that only the most currently prepared version of an SOP is being used. The QA Manual, QAPPs, SOPs, standards preparation logbooks, maintenance logbooks, et al., are controlled documents. The procedures for document control are described in the *SOP for Document Control* (ADM-DOC_CTRL). In addition to SOPs, each laboratory department maintains a current file, accessible to all laboratory staff, of the current methodology used to perform analyses. Laboratory notebook entries are standardized following the guidelines in the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY). Entries made into laboratory notebooks are reviewed and approved by the appropriate supervisor at a regular interval.

8.2 Deviation from Standard Operating Procedures

When a customer requests a modification to an SOP (such as a change in reporting limit, addition or deletion of target analyte(s), etc.), the Project Manager handling that project must discuss the proposed deviation with the department manager in charge of the analysis and obtain their approval to accept the project. The Project Manager is responsible for documenting the approved or allowed deviation from the SOP by placing a detailed description of the deviation attached to the quotation or in the project file and also providing an appropriate comment on the service request when the samples are received.

For circumstances when a deviation or departure from company policies or procedures involving any non-technical function is found necessary, approval must be obtained from the appropriate supervisor, manager, the laboratory director, or other level of authority. Frequent departure from policy is not encouraged. However, if frequent departure from any policy is noted, the laboratory director will address the possible need for a change in policy.

8.3 Modified Procedures

Columbia Analytical strives to perform published methods as described in the referenced documents. If there is a material deviation from the published method, the method is cited as a Modified method in the analytical report. Modifications to the published methods are listed in the standard operating procedure. Standard operating procedures are available to analysts and are also available to our clients for review, especially those for Modified methods. Client approval is obtained for the use of Modified methods prior to the performance of the analysis.

8.4 Analytical Batch

The basic unit for analytical quality control is the analytical batch. The definition that Columbia Analytical has adopted for the analytical batch is listed below. The overriding principle for describing an analytical batch is that all the samples in a batch, both field samples and quality control samples are to be handled exactly the same way, and all of the data from each analysis is to be manipulated in exactly the same manner. The minimum requirements of an analytical batch are:

1) The number of (field) samples in a batch is not to exceed 20.
2) All (field) samples in a batch are of the same matrix.
3) The QC samples to be processed with the (field) samples include:
   a) Method Blank (a.k.a. Laboratory Reagent Blank)

      Function: Determination of laboratory contamination.

   b) Laboratory Control Sample

      Function: Assessment of method performance

   c) Matrix Spiked (field) Sample (a.k.a. Laboratory Fortified Sample Matrix)*

      Function: Assessment of matrix bias

   d) Duplicate Matrix Spiked (field) Sample or Duplicate (field) Sample (a.k.a. Laboratory Duplicate)*

      Function: Assessment of batch precision

   * A sample identified as a field blank, an equipment blank, or a trip blank is not to be matrix spiked or duplicated.
4) A single lot of reagents is used to process the batch of samples.

5) Each operation within the analysis is performed by a single analyst, technician, chemist, or by a team of analysts/technicians/chemists.

6) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours between the start of processing of the first and last sample of the batch.

7) Samples are analyzed in a continuous manner over a timeframe not to exceed 24-hours.

8) (Field) samples are assigned to batches commencing at the time that sample processing begins. For example: for analysis of metals, sample processing begins when the samples are digested. For analysis of organic constituents, it begins when the samples are extracted.

9) The QC samples are to be analyzed in conjunction with the associated field samples prepared with them. However, for tests which have a separate sample preparation step that defines a batch (digestion, extraction, etc.), the QC samples in the batch do not require analysis each time a field sample within the preparation batch is analyzed (multiple instrument sequences to analyze all field samples in the batch need not include re-analyses of the QC samples).

10) The batch is to be assigned a unique identification number that can be used to correlate the QC samples with the field samples.

11) Batch QC refers to the QC samples that are analyzed in a batch of (field) samples.

12) Project-specific requirements may be exceptions. If project, program, or method requirements are more stringent than these laboratory minimum requirements, then the project, program, or method requirements will take precedence. However, if the project, program, or method requirements are less stringent than these laboratory minimum requirements, these laboratory minimum requirements will take precedence.

8.5 Specialized Procedures

Columbia Analytical not only strives to provide results that are scientifically sound, legally defensible, and of known and documented quality; but also strives to provide the best solution to analytical challenges. Procedures using specialized instrumentation and methodology have been developed to improve sensitivity (provide lower detection limits), selectivity (minimize interferences while maintaining sensitivity), and overall data quality for low concentration applications. Examples are trace-level Mercury and Methylmercury analyses, reductive precipitation metals analysis, specialized GC/MS analyses, LC/MS analyses, and ultra-low level organics analyses (including PAHs, pesticides and PCBs).

8.6 Sample Cleanup

Columbia Analytical commonly employs several cleanup procedures to minimize known common interferences prior to analysis. EPA methods (3620, 3630, 3640, 3660, and 3665) for cleanup of sample extracts for organics analysis are routinely used to minimize or eliminate interferences that may adversely affect sample results and data usability.

9.0 CALIBRATION PROCEDURES

All equipment and instruments used at Columbia Analytical are operated, maintained and calibrated according to the manufacturer's guidelines and recommendations, as well as to criteria set forth in the applicable analytical methodology. Operation and calibration are performed by personnel who have been properly trained in these procedures. Documentation of calibration information is maintained in appropriate reference files. Brief descriptions of the calibration procedures for our major laboratory equipment and instruments are described below. Calibration verification is performed according to the applicable analytical methodology. Calibration verification procedures and criteria are listed in laboratory Standard Operating Procedures. Documentation of calibration verification is maintained in appropriate reference files. Records are maintained to provide traceability of reference materials.

Laboratory support equipment (thermometers, balances, and weights) are routinely verified on an annual basis by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standards. All analytical measurements generated at Columbia Analytical are performed using materials where possible and/or processes that are traceable to a reference material. Metrology equipment (analytical balances, thermometers, etc.) is calibrated using reference materials traceable to the National Institute of Standards and Technology (NIST). These primary reference materials are themselves recertified on an annual basis. Vendors used for metrology support are required to verify compliance to International Standards by supplying the laboratory with a copy of their scope of accreditation.

Equipment subjected to overloading or mishandling, or has been shown by verification to be defective; is taken out of service until it is repaired. When an instrument is taken out of service, an *Out of Service* sign is placed by the laboratory on the instrument. The equipment is placed back in service only after verifying, by calibration, that the equipment performs satisfactorily.

9.1 Temperature Control Devices

Temperatures are monitored and recorded each day for all of the temperature-regulating support equipment such as sample refrigerators, freezers, and standards refrigerators/freezers. Temperatures are recorded in either laboratory logbook or through Check Point Wireless Monitoring System. During weekends and holidays a min/max thermometer may be used.

Laboratory records contain the recorded temperature, identification and location of equipment, acceptance criteria and the initials of the technician who performed the checks. The procedure for performing these measurements is provided in the *SOP for Support Equipment Monitoring and Calibration (SOP ADM-SEMC).* The SOP also includes the use of acceptance criteria and correction factors.

Where the operating temperature is specified as a test condition (such as ovens, incubators, evaporators) the temperature is recorded on the raw data. All thermometers are identified according to serial number, and the calibration is checked annually against a National Institute of Standards and Technology (NIST) certified thermometer. The NIST thermometer is

recertified by a vendor accredited to A2LA or ISO/IEC 17025:2005 International Standard on an annual basis.

9.2 Analytical Balances

The calibration of each analytical balance is checked by the user each day of use with three Class S or S-1 weights, which assess the accuracy of the balance at low, mid-level and high levels bracketing the working range. Records are kept which contain the recorded measurements, identification of the balance, acceptance criteria, and the initials of user who performed the check. The procedure for performing these measurements and use of acceptance criteria is described in the SOP ADM-SEMC. The weights are recertified using NIST traceable standards by an accredited metrology organization on an annual basis.

As needed, the balances are recalibrated using the manufacturers recommended operating procedures. Analytical balances are serviced on a semi-annual basis by an accredited metrology organization.

9.3 Water Purification Systems

Columbia Analytical uses two independent water purification systems is designed to produce deionized water meeting method specifications. One system consists of a series of pumps, filters, and resin beds designed to yield deionized water meeting the specifications of ASTM Type II water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Activated carbon filters are also in series with the demineralizers to produce "organic-free" water. A second system consists of pumps, filters, and treatment components designed to yield deionized water meeting the specifications of ASTM Type I water, and *Standard Methods for the Examination of Water and Wastewater* (SM1080, 20th Ed.) *High Quality* water. Following a written SOP, the status of each system is monitored continuously for conductivity and resistivity with an on-line meter and indicator light, and readings recorded daily in a bound record book. The meter accuracy is verified annually. Deionizers are rotated and replaced on a regular schedule. Microbiology water is checked on a daily basis at a point downstream of the purification system at a tap in the laboratory.

9.4 Source and Preparation of Standards and Reference Materials

Consumable reference materials routinely purchased by the laboratories (e.g., analytical standards) are purchased from nationally recognized, reputable vendors. All vendors where possible have fulfilled the requirements for 9001 certification and/or are ISO 17025 accredited. Columbia Analytical Service relies on a primary vendor for the majority of its analytical supplies. Consumable primary stock standards are obtained from certified commercial sources or from sources referenced in a specific method. Supelco, Ultra Scientific, AccuStandard, Chem Services, Inc., Aldrich Chemical Co., Baker, Spex, etc. are examples of the vendors used. Reference material information is recorded in the appropriate logbook(s) and materials are stored under conditions that provide maximum protection against deterioration and contamination. The logbook entry includes such information as an assigned logbook identification code, the source of the material (i.e. vendor identification), solvent (if applicable) and concentration of analyte(s), reference to the certificate of analysis and an assigned expiration date. The date that the

standard is received in the laboratory is marked on the container. When the reference material is used for the first time, the date of usage and the initials of the analyst are also recorded on the container.

Stock solutions and calibration standard solutions are prepared fresh as often as necessary according to their stability. All standard solutions are properly labeled as to analyte concentration, solvent, date, preparer, and expiration date; these entries are also recorded in the appropriate notebook(s) following the *SOP for Reagent Login and Tracking* (SOP ADM-RTL). Prior to sample analysis, all calibration reference materials are verified with a second, independent source of the material (see section 11.3.5).

9.5 Inductively Coupled Plasma-Atomic Emission Spectrograph (ICP-AES)

Each emission line on the ICP is calibrated daily against a blank and against standards whose concentrations fall within the instruments linear range. Analyses of calibration standards, initial and continuing calibration verification standards, and inter-element interference check samples are carried out as specified in the applicable method SOP and analytical method (i.e. EPA 200.7, 6010B, 6010C, CLP SOW, etc.).

9.6 Inductively Coupled Plasma-Mass Spectrometer (ICP-MS)

Each element of interest is calibrated for using a blank and a single standard. Prior to calibration, a short-term stability check is performed on the system. Following calibration, an independent check standard is analyzed, and a continuing calibration verification standard (CCV) is analyzed with every ten samples.

9.7 Atomic Absorption Spectrophotometers (AAS)

These instruments are calibrated daily using a minimum of four standards and a blank. Calibration is validated using reference standards, and is verified at a minimum frequency of once every ten samples. Initial calibration points cannot be dropped from the resulting calibration curve.

9.8 GC/MS Systems

All GC/MS instruments are calibrated at multiple concentration levels for the analytes of interest (unless specified otherwise) using procedures outlined in Standard Operating Procedures and/or appropriate USEPA method citations. All reference materials used for this function are vendor-certified standards. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method. For isotope dilution procedures, the internal standard response(s) and labeled compound recovery must meet method criteria. Method-specific instrument tuning is regularly checked using bromofluorobenzene (BFB) for volatile organic chemical (VOC) analysis, or decafluorotriphenylphosphine (DFTPP) for semi-volatile analysis. Mass spectral peaks for the tuning compounds must conform both in mass numbers and in relative intensity criteria before analyses can proceed. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

9.9 Gas Chromatographs and High Performance Liquid Chromatographs

Calibration and standardization follow SOP guidelines and/or appropriate USEPA method citations. All GC and HPLC instruments are calibrated at a minimum of five different concentration levels for the analytes of interest (unless specified otherwise). The lowest standard is equivalent to the method reporting limit; additional standards define the working range of the GC or LC detector. Results are used to establish response factors (or calibration curves) and retention-time windows for each analyte. Calibration is verified at a minimum frequency of once every ten samples, unless otherwise specified by the reference method. *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

9.10 LC/MS Systems

Calibration and tuning procedures are included in analytical SOPs written specifically for these tests. In general, multiple concentration levels for the analytes of interest are used to generate calibration curves. All reference materials used for this function are vendor-certified standards. Calibration and tuning verification is performed at SOP-defined intervals. Any other system performance checks are described in the applicable SOP. Calibration policies for organics chromatographic analyses are described in the *SOP for Calibration of Instruments for Organics Chromatographic Analyses (SOP SOC-CAL).*

9.11 UV-Visible Spectrophotometer (manual colorimetric analyses)

Routine calibrations for colorimetric and turbidimetric analyses involve generating a 5-point calibration curve including a blank. Initial calibration points cannot be dropped from the resulting calibration curve. Correlation coefficients must meet method or SOP specifications before analysis can proceed. Independent calibration verification standards (ICVs) are analyzed with each batch of samples. Continuing calibration is verified at a minimum frequency of once every ten samples. Typical UV-Visible spectrophotometric methods at Columbia Analytical include total phenolics, phosphates, surfactants and tannin-lignin.

9.12 Flow Injection Analyzer (automated colorimetric analysis)

A minimum of six standards and a blank are used to calibrate the instrument for cyanide analysis. A blank and (minimum of) five standards are used to calibrate the instrument for all other automated chemistries. Initial calibration points cannot be dropped from the resulting calibration curve. Standard Columbia Analytical acceptance limits are used to evaluate the calibration curve prior to sample analysis.

9.13 Discrete Auto-Analyzer (automated absorbance analysis)

A minimum of five standards and a blank are used to calibrate the instrument. Initial calibration points cannot be dropped from the resulting calibration curve. Method specific acceptance limits are used to evaluate the calibration curve prior to sample analysis.

9.14 Ion Chromatographs

Calibration of the ion chromatograph (IC) involves generating a calibration curve with the method-specified number of points (or more). Initial calibration points cannot be dropped from the resulting calibration curve. A correlation coefficient of $\geq$ 0.995 for the curve is required before analysis can proceed. Quality Control (QC) samples that are routinely analyzed include blanks and laboratory control samples. The target analytes typically determined by the IC include nitrate, nitrite, chloride, fluoride, sulfate and drinking water inorganic disinfection byproducts. Calibration verification is performed at method-specified intervals following the procedures in the SOP and reference method.

9.15 Turbidimeter

Calibration of the turbidimeter requires analysis of three Nephelometric Turbidity Unit (NTU) formazin standards. Quality Control samples that are routinely analyzed include blanks, *Environmental Resource Associates* QC samples (or equivalent) and duplicates.

9.16 Ion-selective electrode

The method-prescribed numbers of standards are used to calibrate the electrodes before analysis. The slope of the curve must be within acceptance limits before analysis can proceed. Quality Control samples that are routinely analyzed include blanks, LCSs and duplicates.

9.17 Pipets

The calibration of pipets and autopipettors used to make critical-volume measurements is verified following the *SOP Checking Volumetric Labware (ADM-VOLWARE)*. Both accuracy and precision verifications are performed, at intervals applicable to the pipet and use. The results of all calibration verifications are recorded in bound logbooks.

9.18 Other Instruments

Calibration for the total organic carbon (TOC), total organic halogen (TOX), and other instruments is performed following manufacturer's recommendations and applicable SOPs.

10.0 QUALITY CONTROL

A primary focus of Columbia Analytical s QA Program is to ensure the accuracy, precision and comparability of all analytical results. Prior to using a procedure for the analysis on field samples, acceptable method performance is established by performing demonstration of capability analyses. Performance characteristics are established by performing method detection limit studies and assessing accuracy and precision according to the reference method. Columbia Analytical has established Quality Control (QC) objectives for precision and accuracy that are used to determine the acceptability of the data that is generated. These QC limits are either specified in the test methodology or are statistically derived based on the laboratory's historical data. Quality Control objectives are defined below.

10.1 Quality Control Objectives

10.1.1 Demonstration of Capability - A demonstration of capability (DOC) is made prior to using any new test method or when a technician is new to the method. This demonstration is made following regulatory, accreditation, or method specified procedures. In general, this demonstration does not test the performance of the method in real world samples, but in the applicable clean matrix free of target analytes and interferences.

A quality control sample material may be obtained from an outside source or may be prepared in the laboratory. The analyte(s) is (are) diluted in a volume of clean matrix (for analytes which do not lend themselves to spiking, e.g., TSS, the demonstration of capability may be performed using quality control samples). Where specified, the method-required concentration levels are used. Four aliquots are prepared and analyzed according to the test procedure. The mean recovery and standard deviations are calculated and compared to the corresponding acceptance criteria for precision and accuracy in the test method or laboratory-generated acceptance criteria (if there are not established mandatory criteria). All parameters must meet the acceptance criteria. Where spike levels are not specified, actual Laboratory Control Sample results may be used to meet this requirement, provided acceptance criteria is met.

10.1.2 Accuracy - Accuracy is a measure of the closeness of an individual measurement (or an average of multiple measurements) to the true or expected value. Accuracy is determined by calculating the mean value of results from ongoing analyses of laboratory-fortified blanks, standard reference materials, and standard solutions. In addition, laboratory-fortified (i.e. matrix-spiked) samples are also measured; this indicates the accuracy or bias in the actual sample matrix. Accuracy is expressed as percent recovery (% REC.) of the measured value, relative to the true or expected value. If a measurement process produces results whose mean is not the true or expected value, the process is said to be biased. Bias is the systematic error either inherent in a method of analysis (e.g., extraction efficiencies) or caused by an artifact of the measurement system (e.g., contamination).

Columbia Analytical utilizes several quality control measures to eliminate analytical bias, including systematic analysis of method blanks, laboratory control samples and independent calibration verification standards. Because bias can be positive or negative, and because several types of bias can occur simultaneously, only the net, or total, bias can be evaluated in a measurement.

10.1.3 Precision - Precision is the ability of an analytical method or instrument to reproduce its own measurement. It is a measure of the variability, or random error, in sampling, sample handling and in laboratory analysis. The American Society of Testing and Materials (ASTM) recognizes two levels of precision: repeatability - the random error associated with measurements made by a single test operator on identical aliquots of test material in a given laboratory, with the same apparatus, under constant operating conditions, and reproducibility - the random error associated with measurements made by different test operators, in different laboratories, using the same method but different equipment to analyze identical samples of test material.

"Within-batch" precision is measured using replicate sample or QC analyses and is expressed as the relative percent difference (RPD) between the measurements. The "batch-to-batch" precision is determined from the variance observed in the analysis of standard solutions or laboratory control samples from multiple analytical batches.

10.1.4 Control Limits - The control limits for accuracy and precision originate from two different sources. For analyses having enough QC data, control limits are calculated at the 99% confidence limits. For analyses not having enough QC data, or where the method is prescriptive, control limits are taken from the method on which the procedure is based. If the method does not have stated control limits, then control limits are assigned method-default or reasonable values. Control limits are reviewed each year and may be updated if new statistical limits are generated for the appropriate surrogate, laboratory control sample, and matrix spike compounds (typically once a year) or when method prescribed limits change. The updated limits are reviewed by the QA PM. The new control limits replace the previous limits and data is assessed using the new values. Current acceptance limits for accuracy and precision are available from the laboratory. For inorganics, the precision limit values listed are for laboratory duplicates. For organics, the precision limit values listed are for duplicate laboratory control samples or duplicate matrix spike analyses. Procedures for establishing control limits are found in the *SOP for Control Limits* (ADM-CTRL_LIM).

10.1.5 Representativeness - Representativeness is the degree to which the field sample, being properly preserved, free of contamination, and analyzed within holding time, represents the overall sample site or material. This can be extended to the sample itself, in that representativeness is the degree to which the subsample that is analyzed represents the entire field sample submitted for analysis. Columbia Analytical has sample handling procedures to ensure that the sample used for analysis is representative of the entire sample. These include the *SOP for Subsampling and Compositing of Samples* (GEN-SUBS) and the *SOP for Tissue Sample Preparation (MET-TISP)*. Further, analytical SOPs specify appropriate sample handling and sample sizes to further ensure the sample aliquot that is analyzed is representative in entire sample.

10.1.6 Comparability Comparability expresses the confidence with which one data set can be compared to another and is directly affected by data quality (accuracy and precision) and sample handling (sampling, preservation, etc). Only data of known quality can be compared. The objective is to generate data of known quality with the highest level of comparability, completeness, and usability. This is achieved by employing the quality controls listed below and standard operating procedures for the handling and analysis of all samples. Data is reported in units specified by the client and using Columbia Analytical or project-specified data qualifiers.

10.2 Method Detection Limits, Method Reporting Limits, and Limits of Detection/Quantitation

Method Detection Limits (MDL) for methods performed at Columbia Analytical/(Location) is determined during initial method set up and if any significant changes are made. If an MDL study is not performed annually, the established MDL is verified by performing a limit of detection (LOD) verification on every instrument used in the analysis. The MDLs are determined by following the *SOP for Performing Method Detection Limits Studies and Establishing Limits of Detection and Quantitation (ADM-MDL),* which is based on the procedure in 40 CFR Part 136, Appendix B. As required by NELAP and DoD protocols, the validity of MDLs is verified using LOD verification samples.

The Method Reporting Limit (MRL) is the lowest amount of an analyte in a sample that can be quantitatively determined with stated, acceptable precision and accuracy under stated analytical conditions (i.e. limit of quantitation- LOQ). LOQ are analyzed on an annual basis and cannot be lower than the lowest calibration standard. Current MDLs and MRLs are available from the laboratory.

10.3 Quality Control Procedures

The specific types, frequencies, and processes for quality control sample analysis are described in detail in method-specific standard operating procedures and listed below. These sample types and frequencies have been adopted for each method and a definition of each type of QC sample is provided below.

10.3.1 Method Blank (a.k.a. Laboratory Reagent Blank)

The method blank is an analyte-free matrix (water, soil, etc.) subjected to the entire analytical process. When analyte-free soil is not available, anhydrous sodium sulfate, organic-free sand, or an acceptable substitute is used. The method blank is analyzed to demonstrate that the analytical system itself does not introduce contamination. The method blank results should be below the Method Reporting Limit (MRL) or, if required for DoD projects, MRL for the analyte(s) being tested. Otherwise, corrective action must be taken. A method blank is included with the analysis of every sample preparation batch, every 20 samples, or as stated in the method, whichever is more frequent.

10.3.2 Calibration Blanks

For some methods, calibration blanks are prepared along with calibration standards in order to create a calibration curve. Calibration blanks are free of the analyte of interest and, where applicable, provide the zero point of the calibration curve. Additional project-specific requirements may also apply to calibration blanks.

10.3.3 Continuing Calibration Blanks

Continuing calibration blanks (CCBs) are solutions of analyte-free water, reagent, or solvent that are analyzed in order to verify the system is contamination-free when CCV standards are analyzed. The frequency of CCB analysis is either once every ten samples or as indicated in the method, whichever is greater. Additional project-specific requirements may also apply to continuing calibration blanks.

10.3.4 Calibration Standards

Calibration standards are solutions of known concentration prepared from primary standard or stock standard materials. Calibration standards are used to calibrate the instrument response with respect to analyte concentration. Standards are analyzed in accordance with the requirements stated in the particular method being used.

10.3.5 Initial (or Independent) Calibration Verification Standards

Initial (or independent) calibration verification standards (ICVs) are standards that are analyzed *after* calibration but *prior to* sample analysis, in order to verify the validity and accuracy of the standards used in for calibration. Once it is determined that there is no defect or error in the calibration standard(s), standards are considered valid and may be used for subsequent calibrations and quantitative determinations (as expiration dates and methods allow). The ICV standards are prepared from materials obtained from a source independent of that used for preparing the calibration standards (second-source). ICVs are also analyzed in accordance with method-specific requirements.

10.3.6 Continuing Calibration Verification Standards

Continuing calibration verification standards (CCVs) are midrange standards that are analyzed in order to verify that the calibration of the analytical system is still acceptable. The frequency of CCV analysis is either once every ten samples, or as indicated in the method.

10.3.7 Internal Standards

Internal standards are known amounts of specific compounds that are added to each sample prior to instrument analysis. Internal standards are generally used for GC/MS and ICP-MS procedures to correct sample results that have been affected by changes in instrument conditions or changes caused by matrix effects. The requirements for evaluation of internal standards are specified in each method and SOP.

10.3.8 Surrogates

Surrogates are organic compounds which are similar in chemical composition and chromatographic behavior to the analytes of interest, but which are not normally found in environmental samples. Depending on the analytical method, one or more of these compounds is added to method blanks, calibration and check standards, and samples (including duplicates, matrix spike samples, duplicate matrix spike samples and laboratory control samples) prior to extraction and analysis in order to monitor the method performance on each sample. The percent recovery is calculated for each surrogate, and the recovery is a measurement of the overall method performance.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

10.3.9 Laboratory Control Samples

The laboratory control sample (LCS) is an aliquot of analyte-free water or analyte-free solid (or anhydrous sodium sulfate or equivalent) to which known amounts of the method analyte(s) is (are) added. A reference material of known matrix type, containing certified amounts of target analytes, may also be used as an LCS. An LCS is prepared and analyzed at a minimum frequency of one LCS per 20 samples, with every analytical batch or as stated in the method, whichever is more frequent. The LCS sample is prepared and analyzed in exactly the same manner as the field samples.

The percent recovery of the target analytes in the LCS is compared to established control limits and assists in determining whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required reporting limit. Comparison of batch-to-batch LCS analyses enables the laboratory to evaluate batch-to-batch precision and accuracy.

Recovery (%) = (M/T) x 100

Where: M = The measured concentration of analyte,
T = The theoretical concentration of analyte added.

10.3.10 Laboratory Fortified Blanks - LFB

A laboratory blank fortified at the MRL used to verify the minimum reporting limit. The LFB is carried through the entire extraction and analytical procedure. A LFB is required with every batch of drinking water samples.

10.3.11 Matrix Spikes (a.k.a. Laboratory Fortified Sample Matrix)

Matrix spiked samples are aliquots of samples to which a known amount of the target analyte (or analytes) is (are) added. The samples are then prepared and analyzed in the same analytical batch, and in exactly the same manner as are routine samples. For the appropriate methods, matrix spiked samples are prepared and analyzed and at a minimum frequency of one spiked sample (and one duplicate spiked sample, if appropriate) per twenty samples. The spike recovery measures the effects of interferences caused by the sample matrix and reflects the accuracy of the method for the particular matrix in question. Spike recoveries are calculated as follows:

$$Recovery\ (\%) = (S - A) \times 100 \div T$$

Where: S = The observed concentration of analyte in the spiked sample,
A = The analyte concentration in the original sample, and
T = The theoretical concentration of analyte added to the spiked sample.

10.3.12 Laboratory Duplicates and Duplicate Matrix Spikes

Duplicates are additional replicates of samples that are subjected to the same preparation and analytical scheme as the original sample. Depending on the method of analysis, either a duplicate analysis (and/or a matrix spiked sample) or a matrix spiked sample and duplicate matrix spiked sample (MS/DMS) are analyzed. The relative percent difference between duplicate analyses or between an MS and DMS is a measure of the precision for a given method and analytical batch. The relative percent difference (RPD) for these analyses is calculated as follows:

$$Relative\ Percent\ Difference\ (RPD) = (S1 - S2) \times 100 \div S_{ave}$$

Where S1 and S2 = The observed concentrations of analyte in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike, and

S_{ave} = The average of observed analyte concentrations in the sample and its duplicate, or in the matrix spike and its duplicate matrix spike.

Depending on the method of analysis, either duplicates (and/or matrix spikes) or MS/DMS analyses are performed at a minimum frequency of one set per 20 samples. If an insufficient quantity of sample is available to perform a laboratory duplicate or duplicate matrix spikes, duplicate LCSs will be prepared and analyzed.

10.3.13 Interference Check Samples

An interference check sample (ICS) is a solution containing both interfering and analyte elements of known concentration that can be analyzed to verify background and interelement correction factors in metals analyses. The ICS is prepared to contain known concentrations (method or program specific) of elements that will provide an adequate test of the correction factors. The ICS is analyzed at the beginning and end of an analytical run or at a method-specified frequency. Results must meet method criteria and any project-specific criteria.

10.3.14 Post Digestion Spikes

Post digestion spikes are samples prepared for metals analyses that have an analyte spike added to determine if matrix effects may be a factor in the results. The spike addition should produce a method-specified minimum concentration above the method reporting limit. A post digestion spike is analyzed with each batch of samples and recovery criteria are specified for each method.

10.3.15 Control Charting

The generation of control charts is routinely performed at Columbia Analytical. Surrogate, Matrix Spike and LCS recoveries are all monitored and charted. In addition, the laboratory also monitors the Relative Percent Difference (RPD) measurement of precision. Control charts are available to each individual laboratory unit to monitor the data generated in its facility using control charts that have been programmed to identify various trends in the analytical results. If trends in the data are perceived, various means of corrective action may then be employed in order to prevent future problems with the analytical system(s). Finally, data quality reports using control charts are generated for specific clients and projects pursuant to contract requirements. The control charting procedure is described in the SOP for *Control Charting Quality Control Data* (ADM-CHRT).

10.3.16 Glassware Washing

Glassware washing and maintenance play a crucial role in the daily operation of a laboratory. The glassware used at Columbia Analytical undergoes a rigorous cleansing procedure prior to every usage. A number of SOPs have been generated that outline the various procedures used at Columbia Analytical; each is specific to the end-use of the equipment as well as to the overall analytical requirements of the project. In addition, other equipment that may be routinely used at the laboratory is also cleaned following instructions in the appropriate SOP.

11.0 DATA PROCESSING, VALIDATION, AND REPORTING

Columbia Analytical reports the analytical data produced in its laboratories to the client via the certified analytical report. This report includes a transmittal letter, a case narrative, client project information, specific test results, quality control data, chain of custody information, and any other project-specific support documentation. The following procedures describe our data reduction, validation and reporting procedures.

11.1 Data Reduction and Review

Results are generated by the analyst who performs the analysis and works up the data. All data is initially reviewed and processed by analysts using appropriate methods (e.g., chromatographic software, instrument printouts, hand calculation, etc.). Equations used for calculation of results are found in the applicable analytical SOPs. The resulting data set is either manually entered (e.g., titrimetric or microbiological data) into an electronic report form or is electronically transferred into the report from the software used to process the original data set (e.g., chromatographic software). Once the complete data set has been transferred into the proper electronic report form(s), it is then printed. The resulting hardcopy version of the electronic report is then reviewed by the analyst for accuracy. Once the primary analyst has checked the data for accuracy and acceptability, the hardcopy is forwarded to the supervisor or second qualified analyst, who reviews the data for errors. Where calculations are not performed using a validated software system, the reviewer rechecks a minimum of 10% of the calculations. When the entire data set has been found to be acceptable, a final copy of the report is printed and signed by the laboratory supervisor, departmental manager or designated laboratory staff. The entire data package is then placed into the appropriate service request file, and an electronic copy of the final data package is forwarded to the appropriate personnel for archival. Data review procedures are described in the *SOP for Laboratory Data Review Process (ADM-DREV).*

Policies and procedures for manual editing of data are established. The analyst making the change must initial and date the edited data entry, without obliteration of the original entry. The policies and procedures are described in the *SOP for Making Entries into Logbooks and onto Benchsheets* (ADM-DATANTRY).

Policies and procedures for electronic manual integration of chromatographic data are established. The analyst performing the integration must document the integration change by printing both the before and after integrations and including them in the raw data records. The policies and procedures are described in the *SOP for Manual Integration of Chromatographic Peaks* (ADM-INT).

11.2 Confirmation Analysis

11.2.1 Gas Chromatographic and Liquid Chromatographic Analyses

For gas chromatographic (GC) and liquid chromatographic (LC) analyses, all positive results are confirmed by a second column, a second detector, a second wavelength (HPLC/UV), or by GC/MS analysis, unless exempted by one of the following situations:

- The analyte of interest produces a chromatogram containing multiple peaks exhibiting a characteristic pattern, which matches appropriate standards. This is limited to petroleum hydrocarbon analyses (e.g., gasoline and diesel) and does not include polychlorinated biphenyls.
- The sample meets all of the following requirements:
    1. All samples (liquid or solid) come from the same source (e.g., groundwater samples from the same well) for continuous monitoring. Samples of the same matrix from the same site, but from different sources (e.g., different sampling locations) are not exempt.
    2. All analytes have been previously analyzed in sample(s) from the same source, identified and confirmed by a second column or by GC/MS. The chromatogram is largely unchanged from the one for which confirmation was carried out. The documents indicating previous confirmation must be available for review.

11.2.2 Confirmation Data

Confirmation data will be provided as specified in the method. Identification criteria for GC, LC or GC/MS methods are summarized below:

- GC and LC Methods
    1. The analyte must fall within plus or minus three times the standard deviation (established for the analyte/column) of the retention time of the daily midpoint standard in order to be qualitatively identified. The retention-time windows will be established and documented, as specified in the appropriate Standard Operating Procedure (SOP).
    2. When sample results are confirmed by two dissimilar columns or detectors, the agreement between quantitative results must be evaluated. The relative percent difference between the two results is calculated and evaluated against SOP and/or method criteria.
- GC/MS Methods - Two criteria are used to verify identification:
    1. Elution of the analyte in the sample will occur at the same relative retention time (RRT) as that of the analyte in the standard.
    2. The mass spectrum of the analyte in the sample must, in the opinion of a qualified analyst or the department manager, correspond to the spectrum of the analyte in the standard or the current GC/MS reference library.

11.3 Data Review and Validation of Results

The integrity of the data generated is assessed through the evaluation of the sample results, calibrations, and QC samples (method blanks, laboratory control samples, sample duplicates, matrix spikes, trip blanks, etc.). A brief description of the evaluation of these analyses is described below, with details listed in applicable SOPs. The criteria for evaluation of QC samples are listed within each method-specific SOP. Other data evaluation measures may include (as necessary) a check of the accuracy check of the QC standards and a check of the system sensitivity. Data transcriptions and calculations are also reviewed.

Note: Within the scope of this document, all possible data assessment requirements for various project protocols cannot be included in the listing below. This listing gives a general description of data evaluation practices used in the laboratory in compliance with NELAP Quality Systems requirements. Additional requirements exist for certain programs, such as projects under the DoD QSM protocols, and project-specific QAPPs.

- Method Calibration Following the analysis of calibration blanks and standards according to the applicable SOP the calibration correlation coefficient, average response factor, etc. is calculated and compared to specified criteria. If the calibration meets criteria analysis may continue. If the calibration fails, any problems are isolated and corrected and the calibration standards reanalyzed. Following calibration and analysis of the independent calibration verification standard(s) the percent difference for the ICV is calculated. If the percent difference is within the specified limits the calibration is complete. If not, the problem associated with the calibration and/or ICV are isolated and corrected and verification and/or calibration is repeated.
- Continuing Calibration Verification (CCV) Following the analysis of the CCV standard the percent difference is calculated and compared to specified criteria. If the CCV meets the criteria analysis may continue. If the CCV fails, routine corrective action is performed and documented and a 2nd CCV is analyzed. If this CCV meets criteria, analysis may continue, including any reanalysis of samples that were associated with a failing CCV. If the routine corrective action failed to produce an immediate CCV within criteria, then either acceptable performance is demonstrated (after additional corrective action) with two consecutive calibration verifications or a new initial calibration is performed.
- Method Blank Results for the method blank are calculated as performed for samples. If results are less than the MRL (MRL for DoD projects), the blank may be reported. If not, associated sample results are evaluated to determine the impact of the blank result. If possible, the source of the contamination is determined. If the contamination has affected sample results the blank and samples are reanalyzed. If positive blank results are reported, the blank (and sample) results are flagged with an appropriate flag, qualifier, or footnote.

- Sample Results (Inorganic) Following sample analysis and calculations (including any dilutions made due to the sample matrix) the result is verified to fall within the calibration range. If not, the sample is diluted and analyzed to bring the result into calibration range. When sample and sample duplicates are analyzed for precision, the calculated RPD is compared to the specified limits. The sample and duplicate are reanalyzed if the criteria are exceeded. The samples may require re-preparation and reanalysis. For metals, additional measures as described in the applicable SOP may be taken to further evaluate results (dilution tests and/or post-digestion spikes). Results are reported when within the calibration range, or as estimates when outside the calibration range. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL s including alternative analysis.
- Sample Results (Organic) For GC/MS analyses, it is verified that the analysis was within the prescribed tune window. If not, the sample is reanalyzed. Following sample analysis and calculations (including any dilutions made due to the sample matrix) peak integrations, retention times, and spectra are evaluated to confirm qualitative identification. Internal standard responses and surrogate recoveries are evaluated against specified criteria. If internal standard response does not meet criteria, the sample is diluted and reanalyzed. Results outside of the calibration range are diluted to within the calibration range. For GC and HPLC tests, results from confirmation analysis are evaluated to confirm positive results and to determine the reported value. The procedure to determine which result to report is described in the SOP for *Confirmation Procedure for GC and HPLC Analysis (SOC-CONF).* If obvious matrix interferences are present, additional cleanup of the sample using appropriate procedures may be necessary and the sample is reanalyzed. When dilutions are performed the MRL is elevated accordingly and qualified. Efforts are made to meet the project MRL s including additional cleanup.
- Surrogate Results (Organic) The percent recovery of each surrogate is compared to specified control limits. If recoveries are acceptable, the results are reported. If recoveries do not fall within control limits, the sample matrix is evaluated. When matrix interferences are present or documented, the results are reported with a qualifier that matrix interferences are present. If no matrix interferences are present and there is no cause for the outlier, the sample is reprepared and reanalyzed. However, if the recovery is above the upper control limit with non-detected target analytes, the sample may be reported. All surrogate recovery outliers are appropriately qualified on the report.
- Duplicate Sample and/or Duplicate Matrix Spike Results The RPD is calculated and compared to the specified control limits. If the RPD is within the control limits the result is reported. If not, an evaluation of the sample is made to verify that a homogenous sample was used. Despite the use of homogenizing procedures prior to sample preparation or analysis, the sample may not be homogenous or duplicate sample containers may not have been sample consistently. If non-homogenous, the result is reported with a qualifier about the homogeneity of the sample. Also, the results are compared to the MRL. If the results are less than five times the MRL, the results are reported with a qualifier that the high RPD is due to the results being near the MRL. If the sample is homogenous and results above five times the MRL, the samples and duplicates are reanalyzed. If re-analysis also produces out-of-control results, the results are reported with an appropriate qualifier.

- Laboratory Control Sample Results The LCS percent recovery is calculated and compared to specified control limits. If the recovery is within control limits, the analysis is in control and results may be reported. If not, this indicates that the analysis is not in control. Samples associated with the 'out of control LCS, shall be considered suspect and the samples re-extracted or re-analyzed or the data reported with the appropriate qualifiers. For analysis where a large number of analytes are in the LCS, it becomes more likely that some analytes (marginal exceedences) will be outside the control limits. The procedure described in the 2003 NELAC standards, Appendix D.1.1.2.1 are used to determine if the LCS is effective in validating the analytical system and the associated samples.
- Matrix Spike Results The MS percent recovery is calculated and compared to specified control limits. If the recovery is within control limits the results are reported. If not, and the LCS is within control limits, this indicates that the matrix potentially biases analyte recovery. It is verified that the spike level is at least five times the background level. If not, the results are reported with a qualifier that the background level is too high for accurate recovery determination. If matrix interferences are present or results indicate a potential problem with sample preparation, steps may be taken to improve results; such as performing any additional cleanups, dilution and reanalysis, or re-preparation and reanalysis. Results that do not meet acceptance limits are reported with an appropriate qualifier.

11.4 Data Reporting

When an analyst determines that a data package has met the data quality objectives (and/or any client-specific data quality objectives) of the method and has qualified any anomalies in a clear, acceptable fashion, the data package is reviewed by a trained analyst or chemist. Prior to release of the report to the client, the Project Manager reviews and approves the entire report for completeness and to ensure that any and all client-specified objectives were successfully achieved. The original raw data, along with a copy of the final report, is scanned and archived by service request number. Columbia Analytical maintains control of analytical results by adhering to standard operating procedures and by observing sample custody requirements. All data are calculated and reported in units consistent with project specifications, to enable easy comparison of data from report to report.

To the extent possible, samples shall be reported only if all QC measures are acceptable. If a QC measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). The *SOP for Data Reporting and Report Generation (ADM-RG)* addresses the flagging and qualification of data. The Columbia Analytical-defined data qualifiers, state-specific data qualifiers, or project-defined data qualifiers are used depending on project requirements. A case narrative may be written by the Project Manager to explain problems with a specific analysis or sample, etc.

For subcontracted analyses, the Project Manager verifies that the report received from the subcontractor is complete. This includes checking that the correct analyses were performed, the analyses were performed for each sample as requested, a report is provided for each analysis, and the report is signed. The Project Manager accepts the report if all verification items are complete. Acceptance is demonstrated by forwarding the report to the client.

11.5 Documentation

Columbia Analytical maintains a records system which ensures that all laboratory records of analysis data retained and available. Analysis data is retained for 5 years from the report date unless contractual terms or regulations specify a longer retention time. The archiving system is described in the *SOP for Data Archiving (ADM-ARACH)*.

12.5.1Documentation and Archiving of Sample Analysis Data

The archiving system includes the following items for each set of analyses performed:

- Benchsheets describing sample preparation (if appropriate) and analysis;
- Instrument parameters (or reference to the data acquisition method);
- Sample analysis sequence;
- Instrument printouts, including chromatograms and peak integration reports for all samples, standards, blanks, spikes and reruns;
- Logbook ID number for the appropriate standards;
- Copies of report sheets submitted to the work request file; and
- Copies of Nonconformity and Corrective Action Reports, if necessary.

Individual sets of analyses are identified by analysis date and service request number. Since many analyses are performed with computer-based data systems, the final sample concentrations can be automatically calculated. If additional calculations are needed, they are written on the integration report or securely stapled to the chromatogram, if done on a separate sheet.

For organics analysis, data applicable to all analyses within the batch, such as GCMS tunes, CCVs, batch QC, and analysis sequences; are kept using a separate documentation system. This system is used to archive data on a batch-specific basis and is segregated according to the date of analysis. This system also includes results for the most recent calibration curves, as well as method validation results.

11.6 Deliverables

In order to meet individual project needs, Columbia Analytical provides several levels of analytical reports. Standard specifications for each level of deliverable are described in Table 11-1. Variations may be provided based on client or project specifications. This includes (but is not limited to) the following specialized deliverables:

- DoD QSM Army Corp of Engineers, Air Force Center for Environmental Excellence, Navy
- Drinking water - State specific formats

When requested by the client or relevant to the validity of reported results, the estimation of measurement uncertainty will be provided to a client or regulatory agency. How the uncertainty will be reported may be dictated by the client s reporting specifications. Procedures for determining and reporting uncertainty are given in the *SOP for Estimation of Uncertainty of Measurements*.

When requested, Columbia Analytical provides Electronic Data Deliverables (EDDs) in the format specified by client need or project specification. Columbia Analytical is capable of generating EDDs with many different formats and specifications. The EDD is prepared by report production staff using the electronic version of the laboratory report to minimize transcription errors. User guides and EDD specification outlines are used in preparing the EDD. The EDD is reviewed and compared to the hard-copy report for accuracy.

Table 11-1
Descriptions of Columbia Analytical Standard Data Deliverables

Tier I. Routine Certified Analytical Report (CAR) includes the following:

1. Transmittal letter
2. Chain of custody documents and sample/cooler receipt documentation
3. Sample analytical results
4. Method blank results
5. Surrogate recovery results and acceptance criteria for applicable organic methods
6. Dates of sample preparation and analysis for all tests
7. Case narrative - **optional**

Tier II. In addition to the Tier I Deliverables, this CAR includes the following:

1. Matrix spike result(s) with calculated recovery and including associated acceptance criteria
2. Duplicate or duplicate matrix spike result(s) (as appropriate to method), with calculated relative percent difference
3. Laboratory Control Sample result(s) with calculated recovery and including associated acceptance criteria
4. Case narrative - **optional**

Tier III. Data Validation Package. In addition to the Tier II Deliverables, this CAR includes the following:

1. Case narrative - **required**
2. Summary forms for all associated QC and Calibration parameters, with associated control criteria/acceptance limits

Note: Other summary forms specified in QAPPs or project/program protocols, or those related to specialized analyses such as HRGC/MS will be included.

Tier IV. Full Data Validation Package.

1. All raw data associated with the sample analysis, including but not limited to:
   a. Preparation and analysis bench sheets and instrument printouts,
   b. For organics analyses, all applicable chromatograms, spectral, confirmation, and manual integration raw data. For GC/MS this includes tuning results, mass spectra of all positive hits, and the results and spectra of TIC compounds when requested.
   c. QC data,
   d. Calibration data (initial, verification, continuing, etc),
   e. Calibration blanks or instrument blanks (as appropriate to method).
2. If a project QAPP or program protocol applies, the report will be presented as required by the QAPP.

12.0 PERFORMANCE AND SYSTEM AUDITS

Quality audits are an essential part of Columbia Analytical/Kelso's quality assurance program. There are two types of audits used at the facility: System Audits are conducted to qualitatively evaluate the operational details of the QA program, while Performance Audits are conducted by analyzing proficiency testing samples in order to quantitatively evaluate the outputs of the various measurement systems.

12.1 System Audits

The system audit examines the presence and appropriateness of laboratory systems. External system audits of Columbia Analytical/Kelso are conducted regularly by various regulatory agencies and clients. Appendix G lists the certification and accreditation programs in which Columbia Analytical/Kelso participates. Programs and certifications are added as required. Additionally, internal system audits of Columbia Analytical/Kelso are conducted regularly under the direction of the Quality Assurance Program Manager. The internal audit procedures are described in the *SOP for Internal Audits (ADM-IAUD)*. The internal audits are performed as follows:

- Comprehensive lab-wide system audit performed annually. This audit is conducted such that systems, technical operations, hardcopy data, and electronic data are assessed.
- Technical/method audits minimum of 3 per quarter
- Hardcopy report audits minimum of 2 per quarter.
- Chromatographic electronic data audits each applicable instrument per quarter.

All audit findings, and corrective actions are documented. The results of each audit are reported to the Laboratory Director and Department Managers for review. Any deficiencies identified are summarized in the audit report. Managers must respond with corrective actions correcting the deficiency within a defined timeframe. Should problems impacting data quality be found during an internal audit, any client whose data is adversely impacted will be given written notification within the corrective action period (if not already provided).

Electronic data audits may be performed in conjunction with hardcopy data audits. The electronic audits focus on organic chromatographic data and include an examination of audit trails, peak integrations, calibration practices, GCMS tuning data, peak response data, use of appropriate files, and other components of the analysis. The audit also verifies that the electronic data supports the hardcopy reported data.

Additional internal audits or data evaluations may be performed as needed to address any potential data integrity issues that may arise.

12.2 Performance Audits

Columbia Analytical/Kelso also participates in the analysis of interlaboratory proficiency testing (PT) samples. Participation in PT studies is performed on a regular basis and is designed to evaluate all analytical areas of the laboratory. General procedures for these analyses are described in the SOP for *Proficiency Sample Testing Analysis (ADM-PTS)*. Columbia Analytical routinely participates in the following studies:

- Water Pollution (WP) and additional water parameters, 2 per year.
- Water Supply (WS) PT studies, 2 per year.
- Hazardous Waste/Soil PT studies, 2 per year.
- Underground Storage Tank PT studies, 2 per year.
- Microbiology (WS and WP) PT studies, 2 per year.
- Other studies as required for specific certifications, accreditations, or validations.

PT samples are processed by entering them into the LIMS system as samples (assigned Service Request, due date, testing requirements, etc.) and are processed the same as field samples. The laboratory sections handle samples the same as field samples, performing the analyses following method requirements and performing data review. The laboratory sections submit results to the QA Manager for subsequent reporting to the appropriate agencies or study provider. Results of the performance evaluation samples and audits are reviewed by the QA PM, Laboratory Director, the laboratory staff, and the Chief Quality Officer. For any results outside acceptance criteria, the analysis data is reviewed to identify a root cause for the deficiency, and corrective action is taken and documented through nonconformance (NCAR) procedures.

13.0 PREVENTIVE MAINTENANCE

Preventive maintenance is a crucial element of the Quality Assurance program. Instruments at Columbia Analytical (e.g., ICP/MS and ICP systems, GC/MS systems, atomic absorption spectrometers, analytical balances, gas and liquid chromatographs, etc.) are maintained under commercial service contracts or by qualified, in-house personnel. All instruments are operated and maintained according to the instrument operating manuals. All routine and special maintenance activities pertaining to the instruments are recorded in instrument maintenance logbooks. The maintenance logbooks used at Columbia Analytical contain extensive information about the instruments used at the laboratory.

An initial demonstration of analytical control is required on every instrument used at Columbia Analytical before it maybe used for sample analysis. If an instrument is modified or repaired, a return to analytical control is required before subsequent sample analyses can occur. When an instrument is acquired at the laboratory, the following information is noted in a bound maintenance notebook specifically associated with the new equipment:

- The equipment s serial number;
- Date the equipment was received;
- Date the equipment was placed into service;
- Condition of equipment when received (new, used, reconditioned, etc.); and
- Prior history of damage, malfunction, modification or repair (if known).

Preventive maintenance procedures, frequencies, etc. are available for each instrument used at Columbia Analytical. They may be found in the various SOPs for routine methods performed on an instrument and may also be found in the operating or maintenance manuals provided with the equipment at the time of purchase.

Responsibility for ensuring that routine maintenance is performed lies with the section supervisor. The supervisor may perform the maintenance or assign the maintenance task to a qualified bench level analyst who routinely operates the equipment. In the case of non-routine repair of capital equipment, the section supervisor is responsible for providing the repair, either by performing the repair themselves with manufacturer guidance or by acquiring on-site manufacturer repair. Each laboratory section maintains a critical parts inventory. The parts inventories include the items needed to perform the preventive maintenance procedures listed in Appendix D.

This inventory or parts list also includes the items needed to perform any other routine maintenance and certain in-house non-routine repairs such as gas chromatography/mass spectrometry jet separators and electron multipliers and ICP/MS nebulizer. When performing maintenance on an instrument (whether preventive or corrective), additional information about the problem, attempted repairs, etc. is also recorded in the notebook. Typical logbook entries include the following information:

- Details and symptoms of the problem;
- Repairs and/or maintenance performed;
- Description and/or part number of replaced parts;
- Source(s) of the replaced parts;
- Analyst's signature and date; and
- Demonstration of return to analytical control.

See the table in Appendix E for a list of preventive maintenance activities and frequency for each instrument.

14.0 CORRECTIVE AND PREVENTIVE ACTION

The laboratory takes all appropriate steps necessary to ensure all sample results are reported with acceptable quality control results. When sample results do not conform to established quality control procedures, responsible management will evaluate the significance of the nonconforming work and take corrective action to address the nonconformance.

Nonconforming events such as errors, deficiencies, deviations from SOP, proficiency (PT) failure or results that fall outside of established QC limits are documented using a *Nonconformity and Corrective Action Report* form (See Figure 14-1). The procedure and responsibilities for addressing nonconforming work is defined in the SOP ADM-CA *Corrective Action.* Nonconformances are reported to the client using various means (voice, email, narrative, etc). When a nonconformance occurs that casts doubt on the validity of the test results or additional client instructions are needed, the Project Manager notifies the client the same business day that the nonconformance is confirmed and reported. The QA PM reviews each problem, ensuring that appropriate corrective action has been taken by the appropriate personnel. The Nonconformity and Corrective Action Report (NCAR) is filed in the associated service request file and a copy is kept by the QA PM. The QA PM periodically reviews all NCARs looking for chronic, systematic problems that need more in-depth investigation and alternative corrective action consideration. In addition, the appropriate Project Manager is promptly notified of any problems in order to inform the client and proceed with any action the client may want to initiate.

If a quality control measure is found to be out of control, and the data is to be reported, all samples associated with the failed quality control measure shall be reported with the appropriate data qualifier(s). Failure to meet established analytical controls, such as the quality control objectives, prompts corrective action. Corrective action may take several forms and may involve a review of the calculations, a check of the instrument maintenance and operation, a review of analytical technique and methodology, and reanalysis of quality control and field samples. If a potential problem develops that cannot be solved directly by the responsible analyst, the supervisor, team leader, the department manager, and/or the QA PM may examine and pursue alternative solutions. In addition, the appropriate Project Manager is notified in order to ascertain if the client needs to be notified.

Part of the corrective action process involves determining the root cause. Identifying the root cause of a nonconformance can be difficult, but important for implementing effective corrective action. Root cause principles are used to determine assignable causes, which leads to corrective action taken to prevent recurrence. Various preventive action processes are used for eliminating a potential problem or averting a problem before it occurs. This is explained in the *SOP for Preventive Action* (ADM-PA).

In addition to internal communication of data issues, the laboratory also maintains a system for dealing with customer complaints. The person who initially receives the feedback (typically the Project Manager) is responsible for documenting the complaint. If the Project Manager is unable to satisfy the customer, the complaint is brought to the attention of the Client Services Manager, Laboratory Director, or QA PM for final resolution. The complaint and resolution are documented. The procedure is described in the *SOP for Handling Customer Feedback (ADM-FDBK)*.

Figure 14-1

Nonconformity and Corrective Action Report

NCAR No: *Assigned by QA*

PROCEDURE (SOP or METHOD): EVENT DATE: _____

EVENT:
- ☐ Missed Holding Time
- ☐ Method Blank Contamination
- ☐ Equipment Failure
- ☐ SOP Deviation
- ☐ QC Failure
- ☐ Login Error
- ☐ Unacceptable PT Sample Result
- ☐ Other (describe):
- ☐ Lab Error (spilled sample, spiking error, etc.)
- ☐ Project Management Error

INCLUDE NUMBER OF SAMPLES / PROJECTS / CUSTOMERS / SYSTEMS AFFECTED

DETAILED DESCRIPTION

ORIGINATOR: _____ DATE: _____

PROJECT MANAGER(S): _____ NOTIFIED BY: _____ DATE: _____

ROOT CAUSE OF NON-CONFORMITY (POTENTIAL CAUSES COULD BE TRAINING, COMMUNICATION, SPECIFICATIONS, EQUIPMENT, KNOWLEDGE)

What is the cause of the error or finding:

CORRECTIVE ACTION AND OUTCOME

Re-establishment of conformity must be demonstrated and documented. Describe the steps that were taken, or are planned to be taken, to correct the particular Nonconformity and prevent its reoccurrence. Include Project Manager Instructions here.

Is the data to be flagged in the Analytical Report with an appropriate qualifier? ☐ **No** ☐ **Yes**

APPROVAL AND NOTIFICATION

Supervisor Verification and Approval of Corrective Action _____ Date: _____
Comments:

QA PM Verification and Approval of Corrective Action _____ Date: _____
Comments:

Project Manager Verification and Approval of Corrective Action _____ Date: _____
Comments:

Customer Notified by ☐ Telephone ☐ Fax ☐ E-mail ☐ Narrative ☐ Not notified

(Attach record or cite reference where record is located.)

15.0 QUALITY ASSURANCE REPORTS AND MANAGEMENT REVIEW

Quality assurance requires an active, ongoing commitment by Columbia Analytical personnel at all levels of the organization. Communication and feedback mechanisms are designed so that analysts, supervisors and managers are aware of QA issues in the laboratory. Analysts performing routine testing are responsible for generating a data quality narrative or data review document with every analytical batch processed. This report also allows the analyst to provide appropriate notes and/or a narrative if problems were encountered with the analyses. A Nonconformance and Corrective Action Report (NCAR) (see Section 14.0) may also be attached to the data prior to review. Supervisors or qualified analysts review all of the completed analytical batches to ensure that all QC criteria have been examined and any deficiencies noted and addressed.

It is the responsibility of each laboratory unit to provide the Project Manager with a final report of the data, accompanied by signature approval. Footnotes and/or narrative notes must accompany any data package if problems were encountered that require further explanation to the client. Each data package is submitted to the appropriate Project Manager, who in turn reviews the entire collection of analytical data for completeness and to ensure that any and all client-specified objectives were successfully achieved. A case narrative is written by the Project Manager to explain any unusual problems with a specific analysis or sample, etc.

The QA PM provides overview support to the Project Managers as required (e.g., contractually specified, etc.). The QAM is also responsible for the oversight of all internal and external audits, for all proficiency testing sample and analysis programs, and for all laboratory certification/accreditation responsibilities. The QAM regularly communicates with the Laboratory Director to review the various QA/QC activities, priorities, and status of program implementation; including such topics as the following:

- Status, schedule, and results of internal and external audits;
- Status, schedule, and results of internal and external proficiency testing studies;
- Status of certifications, accreditations, and approvals;
- Status of QA Manual and SOP review and revision;
- Status of MDLs studies;
- Discussion of QC problems in the laboratory;
- Discussion of corrective action program issues;
- Status of staff training and qualification; and
- Other topics as appropriate.

An annual management review of the quality and testing systems is perfomed as described in the *SOP for Managerial Reviews of the Laboratory's Quality Systems and Testing Activities* (ADM-MGMTRVW). This is done to identify any necessary changes or improvements to the quality system or quality assurance policies. This review is documented in a Managerial Review of the Laboratory s Quality Systems and Testing Activities and sent to senior management.

16.0 PERSONNEL TRAINING

Technical position descriptions are available for all employees, regardless of position or level of seniority. These documents are maintained by the Human Resources personnel and are available for review. In order to assess the technical capabilities and qualifications of a potential employee, all candidates for employment at Columbia Analytical are evaluated, in part, against the appropriate technical description.

Training begins the first day of employment at Columbia Analytical when the company policies are presented and discussed. Safety and QA/QC requirements are integral parts of all technical SOPs and, consequently, are integral parts of all training processes at Columbia Analytical. Safety training begins with the reading of the *Environmental Health and Safety Manual.* Employees are also required to attend periodic safety meetings where additional safety training may be performed by the Environmental, Health and Safety Officer.

Employees are responsible for complying with the requirements of the QA Manual and QA/QC requirements associated with their function(s). Quality Systems training begins with Quality Assurance orientation for new employees and reading the Quality Assurance Manual. During the employees first year, the employee attends Core Ethics training and learns about Columbia Analytical Services quality systems. Each employee participates in annual Ethics Refresher training, which is part of the Columbia Analytical Improper Practices Prevention Program.

Columbia Analytical also encourages its personnel to continue to learn and develop new skills that will enhance their performance and value to the Company. Ongoing training occurs for all employees through a variety of mechanisms. The The corporate, company-wide training and development program, external and internal technical seminars and training courses, and laboratory-specific training exercises are all used to provide employees with professional growth opportunities.

All technical training is documented and records are maintained in the QA department. Training requirements and its documentation are described in the *SOP for Documentation of Training.* (ADM-TRANDOC). A training plan is developed whenever an employee starts a new procedure to new position. The training plan includes a description of the step-by-step process for training an employee and for initial demonstration of capability. Where the analyst performs the entire procedure, a generic training plan may be used.

16.1 Initial Demonstration of Capability (IDOC)

Training in analytical procedures typically begins with the reading of the Standard Operating Procedure (SOP) for the method. Hands-on training begins with the observation of an experienced analyst performing the method, followed by the trainee performing the method under close supervision, and culminating with independent performance of the method on quality control samples. Successful completion of the applicable Demonstration of Capability analysis qualifies the analyst to perform the method independently. Demonstration of Capability is performed by one of the following:

- Successful completion of an Initial Precision and Recovery (IPR) study (required where mandated by the method).
- Analysis of 4 consecutive Laboratory Control Samples, with acceptable accuracy and precision.
- Where spiking is not possible but QC standards are used (non-spiked Laboratory Control Samples), analysis of 4 consecutive Laboratory Control Samples with acceptable accuracy and precision.
- Where one of the three above is not possible, special requirements are as follows:
  - Total Settleable Solids: Successful single-blind PT sample analysis and duplicate results with RPD 10%.
  - Color: Four consecutive prepared LCSs with acceptable accuracy and precision of 10% RSD.
  - Physical Tests (Grain size, Corrosivity to Steel, etc.): Supervisor acknowledgement of training and approval.

A flowchart identifying the Demonstration of Proficiency requirements is given in Figure 16-1. The flowchart identifies allowed approaches to assessing Demonstration of Capability when a 4-replicate study is not mandated by the method, when spiking is not an option, or when QC samples are not readily available.

16.2 Continuing Demonstration of Proficiency

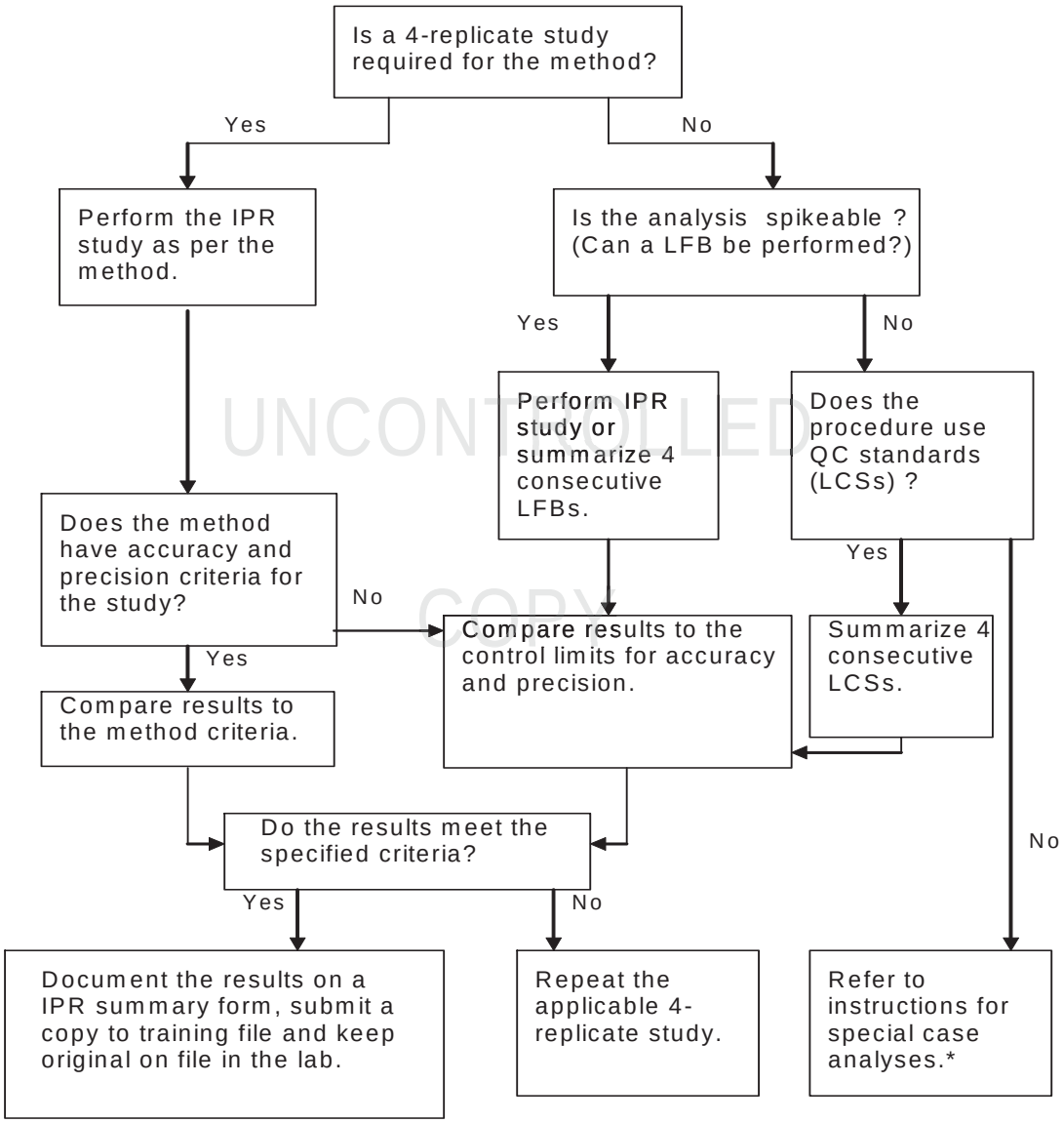
A periodic demonstration of proficiency is required to maintain continuing qualification. Continuing Demonstration of Proficiency is required each year, and may be performed one of the following ways:

- Successful performance on external (independent) single-blind sample analyses using the test method, or a similar test method using the same technology. I.e. PT sample or QC sample blind to the analyst.
- Performing Initial Demonstration of Capability as described above, with acceptable levels of precision and accuracy.
- Analysis of at least 4 consecutive LCSs with acceptable levels of accuracy and precision from in-control analytical batches.
- If the above cannot be performed, analysis of authentic samples with results statistically indistinguishable from those obtained by another trained analyst.
- For methods for which PT samples are not available and a spiked analysis (LFB, MDL, etc.) is not possible, analysis of field samples that have been analyzed by another analyst with statistically indistinguishable results.

16.3 Documentation of Training

Records are maintained to indicate the employee has the necessary training, education, and experience to perform their functions. Information of previously acquired skills and abilities for a new employee is maintained in Human Resources personnel files and Columbia Analytical resumes. QA maintains a database to record the various technical skills and training acquired while employed by Columbia Analytical. Information includes the employee s name, a description of the skill including the appropriate method and SOP reference, the mechanism used to document proficiency, and the date the training was completed. General procedures for documenting technical training are described in the *SOP for Documentation of Training* (ADM-TRANDOC).

Figure 16-1
Initial Demonstration of Capability Requirements



* Refer to the SOP for Documentation of Training for details.

17.0 REFERENCES FOR QUALITY SYSTEMS, EXTERNAL DOCUMENTS, MANUALS, STANDARDS, AND ANALYTICAL PROCEDURES

The analytical methods used at Columbia Analytical generally depend upon the end-use of the data. Since most of our work involves the analysis of environmental samples for regulatory purposes, specified federal and/or state testing methodologies are used and followed closely. Typical methods used at Columbia Analytical are taken from the following references:

- National Environmental Laboratory Accreditation Program (NELAP), 2003 Quality Standards.
- TNI Standard Environmental Laboratory Sector, Volume 1, *Management and Technical Requirements for Laboratories Performing Environmental Analysis,* EL-V1-2009.
- Quality Standards.American National Standard *General requirements for the competence of testing and calibration laboratories,* ANSI/ISO/IEC 17025:2005(E)
- *DoD Quality Systems Manual for Environmental Laboratories,* Version 4.2, 10/25/2010
- *Good Automated Laboratory Practices, Principles and Guidance to Regulations For Ensuring Data Integrity In Automated Laboratory Operations*, EPA 2185 (August 1995).
- *Manual for the Certification of Laboratories Analyzing Drinking Water*, 4th Edition, EPA 815-B-97-001 (March 1997).
- *Procedure Manual for the Environmental Laboratory Accredditation Program,* Washington Department of Ecology, 10-03-048, September 2010.
- *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*, SW-846, Third Edition, (September 1986) and Updates I (July 1992), II (September 1994), IIA (August 1993), IIB (January 1995), III (December 1996), Final Update IV (February 2007), and updates posted online at http://www.epa.gov/epaoswer/hazwaste/test/sw846.htm. See Chapters 1, 2, 3, and 4.
- *Methods for Chemical Analysis of Water and Wastes*, EPA-600/4-79-020, (Revised March 1983).
- *Methods for the Determination of Inorganic Substances in Environmental Samples*, EPA/600/R-93/100 (August 1993).
- *Methods for the Determination of Metals in Environmental Samples*, EPA/600/4-91/010 (June 1991) and Supplements.
- *Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater*, EPA 600/4-82-057 (July 1982) and 40 CFR Part 136, Appendix A.
- *Methods for the Determination of Organic Compounds in Drinking Water*, EPA/600/4-88/039 (December 1988) and Supplements.
- *Standard Methods for the Examination of Water and Wastewater*, 20th Edition (1998) and SM On-Line. See Introduction in Part 1000.

- 40 CFR Part 136, Guidelines for Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act.
- 40 CFR Part 141, National Primary Drinking Water Regulations.
- *Analytical Methods for Petroleum Hydrocarbons*, ECY 97-602, Washington State Department of Ecology, June 1997.
- State-specific total petroleum hydrocarbon methods for the analysis of samples for gasoline, diesel, and other petroleum hydrocarbon products (Alaska, Arizona, California, Oregon, Washington, Wisconsin, etc.).
- Annual Book of ASTM Standards, Part 31, Water.
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Organic Data Review*, EPA-540/R-94/012 (February 1993).
- *U. S. EPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review*, EPA-540/R-94/013 (February 1994).
- *Recommended Protocols for Measuring Selected Environmental Variables in Puget Sound*, for USEPA and USACE (March 1986), with revisions through April 1997.
- WDOE 83-13, *Chemical Testing Methods for Complying with the State of Washington Dangerous Waste Regulations* (March 1982) and as Revised (July 1983 and April 1991).
- *Identification and Listing of Hazardous Waste,* California Code of Regulations, Title 22, Division 4.5, Chapter 11.
- *Analytical Methods for the Determination of Pollutants in Pulp and Paper Industry Wastewater*, EPA 821-R-93-017 (October 1993).
- *Analytical Methods for the Determination of Pollutants in Pharmaceutical Manufacturing Industry Wastewaters*, EPA 821-B-98-016 (July 1998).
- National Council of the Pulp and Paper Industry for Air and Stream Improvement (NCASI).

APPENDIX A

Approved Signatories

QA Program Documents

Corporate Policies

Administrative Corporate SOP List

APPROVED SIGNATORIES FOR ANALYTICAL REPORTS

Columbia Analytical Services, Kelso, WA

ARNOLD, EILEEN
BAILEY, JOSH
CHAN, JIM
CORONADO, JEFFREY
DEGNER, CARL
DOMENIGHINI, LISA
GRINDSTAFF, JEFF
HADERLY, DOUGLAS
HARRIS, LISA
HOLMES, HOWARD
HUCKESTEIN, LYNDA
JACKY, HARVEY
JAMES, JON
KAMAWAL, AQUILLA
KENNEDY, LES
LEAF, CHRIS
MIHAI-LAZAR, CARMEN
MOORE, RACHEL
MURRY, SHANE
PORTWOOD, LOREN
REASONER, KAREN
SALATA, GREGORY
SENKBEIL, RANI
SHELDON, BRIAN
WALLACE, ED

QA Program Files

Program	Location
Quality Assurance Manual	Q:\QA Manual\QAM.rXX.DOC
Software Quality Assurance Plan	Corp IT
CAS-Kelso Certifications/Accreditations	Cert_kel.xls
Columbia Analytical Services MDL Tracking Spreadsheet	Q:\MDL Tracking\MDL_LIST.r1.XLS
Technical Training Summary Database	TrainDat.mdb
Approved Signatories List	QAM App A
Personnel resumes/qualifications	HR dept
Personnel Job Descriptions	HR Department
CAS/KELSO DATA QUALITY OBJECTIVES	CAS Kelso DQO 20XX.rX.xls
Master Logbook of Laboratory Logbooks	QA Masterlog-001
Standard Operating Procedure Database	Q:\ENVIRONMENTAL\1 SOP Policy Statements\1_ Kelso SOP.xls

Corporate Policies

POLICY TITLE	POLICY DATE	DATE APPROVED	DATE EFFECTIVE
CAS Quality and Ethics Policy Statement	September 2010	9/28/10	9/28/10
Policy for Data Review and Validation	September 2010	9/9/10	9/10/10
Policy for Internal Quality Assurance Audits	May 2009	5/5/09	7/1/09
Policy for Standards and Reagents Expiration Dates	September 2009	9/15/09	9/28/09
Policy for Use of Accreditation Organization s Name, Symbols, and Logos	September 2009	9/21/09	10/1/09
Policy for Conducting Research, Method Development, and Method Investigations	December 2009	12/15/09	12/17/09 Replaced by SOP 7/1/11

Corporate SOPs

SOP TITLE	SOP Code	Rev	SOP Date	Date of Last Review
SOP for Checking New Lots of Chemicals for Contamination	ADM-CTMN	5	5/2/11	5/4/11
SOP for Control Limits	ADM-CTRL_LIM	7	12/14/09	12/22/10
SOP for Corrective Action	ADM-CA	6	9/15/09	9/22/10
SOP for Data Recall	ADM-DATARECALL	0	9/21/07	11/22/10
SOP for Document Control	ADM-DOC_CTRL	8	9/15/09	9/22/10
SOP for Document Management Policy Implementation	ADM-DOC_MGMT	0	6/16/11	6/30/11
SOP for Documentation of Training	ADM-TRANDOC	12	4/28/11	5/15/11
SOP for Estimation of Uncertainty of Measurements	ADM-UNCERT	6	9/23/10	9/29/10
SOP for Handling Customer Feedback	ADM-FDBK	5	12/14/09	12/22/10
SOP for Making Entries into Logbooks and onto Analytical Records	ADM-DATANTRY	9	9/27/10	9/29/10
SOP for Managerial Review of the Laboratory s Quality Systems and Testing Activities	ADM-MGMTRVW	4	5/2/11	5/5/11
SOP for Manual Integration of Chromatographic Peaks	ADM-INT	4	10/5/10	10/9/10
SOP for Method Development, Investigation, and Transfer	ADM_MDEV	0	6/16/11	6/16/11
SOP for Performing Method Detection Limit Studies and Establishing Limits of Detection and Quantitation	ADM-MDL	9	9/8/09	9/21/10
SOP for Preparation of Electronic-data for Organic Analyses for Electronic-data Audits	ADM-E_DATA	3	8/29/07	11/22/10
SOP for Preparation of SOPs	ADM-SOP	10	12/20/10	12/22/10

SOP TITLE	SOP Code	Rev	SOP Date	Date of Last Review
SOP for Preventive Action	ADM-PA	1	12/14/09	12/22/10
SOP for Proficiency Testing Sample Analysis	ADM-PTS	3	9/22/10	9/29/10
SOP for Purchasing and Approval of Vendors	ADM-PUR	4	10/15/09	10/5/10
SOP for Qualification of Subcontract Laboratories	ADM_SUBLAB	5	9/15/09	9/22/10
SOP for Significant Figures	ADM-SIGFIG	8	1/28/09	1/13/10
SOP for Tape Backup and Tape Archiving	ADM-TAPEBU	0	10/3/11	10/15/11

Forms

FORM	FILE NAME	DATE
Audit Finding Response Form	Audit Finding Response Form	8/12/2010
CASED Employee Development Plan Template	CAS EDP Template_033011_form only.doc	3/30/11
Complaint Report	Complaint Report_r121509	12/15/09
Critical Job Function Authorization Statement	Critical Job Function Authorization Statement_r071206	7/12/06
Data Re-submittal Request Form	Data Resubmittal Request Form_r112107	11/21/07
Demonstration of Capability Certification Statement (no table version)	DOC Certification Statement_r071206-	7/12/06
Extraction Solvent Critical Consumables Evaluation	Extraction Solvent Critical Consumables Evaluation_r050311.doc	5/3/11
Laboratory Training Certification	LAB-TRNG_r092109	9/21/09

FORM	FILE NAME	DATE
Metals Critical Consumables Evaluation	Metals Critical Consumables Evaluation_r050311.doc	5/3/11
Method Detection Limit Study Calculation Spreadsheet	MDL_FORMR4_r030510	3/5/10
New Vendor Evaluation	Vendor Evaluation Form_r101509	10/15/09
Nonconformity and Corrective Action Report	NCAR09_r092109	9/21/09
Preventive Action Report	PA Report_r072108	7/21/08
Procedure Change Form	Procedure Change Form_r121610	12/16/10
Reagent/Consumable Critical Consumables Evaluation	Reagent Critical Consumables Evaluation_r050311.doc	5/3/11
Standard Operating Procedure Change Form	SOP Change Form_r092109	9/21/09
LOD Verification	H:\group\QA\QA_Forms\LOD Verification071610.xls	07/16/10
LOQ Verification	H:\group\QA\QA_Forms\LOQ Verification022410.xls	02/24/10
Various Training Plans	H:\group\QA\QA_Forms\Training Plans\	NA

APPENDIX B

ORGANIZATIONAL CHARTS and RESUMES OF KEY PERSONNEL

JEFFERY A. GRINDSTAFF 1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**LABORATORY DIRECTOR, KELSO LABORATORY –** 2010 to Present
Responsibilities	Responsible for all phases of laboratory operations at the Kelso (WA) facility, including project planning, budgeting, and quality assurance. Primary duties include the direct management of the Kelso laboratory
	Documentation of Demonstration of Capabilities is available for review.
Experience	**Technical Manager III, Pharmaceutical, GC/MS VOA And Semi-VOA Laboratories,** *Columbia Analytical Services, Inc., Kelso, Washington* **–** 1997-2010 Primary responsibilities include leadership of the Pharmaceutical, GC/MS VOA and Semi-VOA staff, management of method development, training, data review, tracking department workload, scheduling analyses. Responsible for ensuring data quality and timeliness. Also responsible for project management and coordination for pharmaceutical clients.
	Manager, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1997. Responsible for supervision of GC/MS VOA staff, method development, training, data review, tracking department workload, scheduling analyses, and general maintenance and troubleshooting of GC/MS systems.
	Scientist III, GC/MS VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1991-1994. Responsibilities included scheduling workload, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts. Additional supervisory duties included report generation and data review for GC analyses. Responsibilities also included project management and customer service.
	Chemist, *Enseco-CRL, Ventura, California,* 1990-1991. Established GC/MS department including inventory maintenance, preparation of state certification data packages, method development, SOPs, and extended data programs. Performed daily maintenance and troubleshooting of GC and GC/MS instrumentation. Scheduled and performed routine and non-routine VOA analyses.
	GC/MS Chemist, VOA Laboratory *Coast-to-Coast Analytical Service, San Luis Obispo, California,* 1990-1991. Responsible for standard preparation for VOA analyses, instrument calibration, tuning, and maintenance. Also implemented and further developed EPA methods for quantitative analysis of pesticides and priority pollutants.
Education	**Sampling and Testing of Raw Materials***, PTI International, 2004*. **Leadership Training,** *Richard Rogers Group, 1996* **Mass Selective Detector Maintenance,** *Hewlett Packard Education Center, 1993* **Interpretation of Mass Spectra I,** *Hewlett-Packard Analytical Education Center,* 1992. **B.S., Chemistry**, *California Polytechnic State University, San Luis Obispo, California,* 1989. **A.A., Liberal Arts,** *Allan Hancock College, Santa Maria, California.* 1986
Publications/ Presentations	*Mr. Grindstaff has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.*
Affiliations	American Chemical Society. 1989

JULIE GISH

1996 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

TECHNICAL MANAGER I, KELSO LAB QUALITY ASSURANCE MANAGER 2008 to Present

Responsibilities

Responsible for the overall implementation of the laboratory QA program. Oversees implementation of Quality management systems including: Quality Assurance Manual, Certifications, SOP Control, Proficiency Testing (PT), Non-Conformity, Preventative Actions, Internal Auditing, Control Charting, Documentation of Training, and Metrology. Conducts employee QA training including orientations, sop, and ethics. Maintains state, agency and program certifications/accreditations. Acts as primary point of contact during laboratory audits coordinates audit responses and corrective actions.

Experience

Scientist IV, Semi-Volatile Mass Spectrometry Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 2002-2008. Primary responsibilities were analysis, interpretation and report generation for semivolatile organics by GC/MS. Analyses included EPA 625, 8270, SIM, and other miscellaneous methodology.

Technical Manager I, Semi-Volatile GC Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1999-2002. Primary responsibilities include supervision and oversight of semi-volatile GC department. This includes initiating new methods, staff training, workload management, and instrument maintenance/troubleshooting. Duties include departmental compliance with CAS QA and Safety policies. Responsible for analysis, interpretation and report generation for pesticides and PCB s by EPA Methods 608, 8080, 8081, 8082, EPA 8141A, Organotins, and CLP Pesticides.

Scientist III, Semi-Volatile Organics Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-1999. Primary responsibilities were analysis, interpretation and report generation for pesticides and PCB s by EPA Methods 608, 8080, 8081, 8082, and CLP-Pesticides. Secondary responsibilities include organics semi-volatile sample preparation.

Scientist, Volatile Organics Sample Preparation, *Employer's Overload, Longview, Washington – assigned to the Columbia Analytical Services, Inc., Kelso, Washington facility,* 1996. Primary duties included the preparation of water, soil, sediment and tissue samples using EPA Methods 3510, 3520, 3540, 3550, and 3545. Other duties were the further clean up of extracts using EPA Methods 3620 (Florsil), 3610 (Alumina), 3630 (Silica gel), 3650 (Acid/Base Partitioning), and 3660 (Sulfur).

Organics Chemist and GC/MS Chemist, *Coffey Laboratories, Portland, Oregon,* 1990-1996. Primary responsibilities included sample preparation and analysis for EPA FID, ECD, and HPLC using various EPA SW-846 and 500-series methods, as well as other methodology. Later, moved to GC/MS position which included sample preparation, analysis, and associated instrument maintenance for EPA Methods 625, 8027, and 525 BNA s. Also responsible for data review and approval of data packages.

QC Manager/QC Supervisor and Product Manager, Corn Products, *Frito-Lay, Inc., Vancouver, Washington,* 1982-1990. Manager of the QC department overseeing three supervisors and approximately 30 technicians. Responsible for department cost, accuracy, timeliness of data and safety performance. Later, responsible for production oversight of brand name snacks. Responsible for cost, quality and safety performance over three shifts. Managed four supervisors directly and approximately 60 employees indirectly.

Food Technologist, QA Department, *Kraft, Inc., Buena Park, California*, 1978-1981. Responsible for audits, formulations, finished product evaluation, batch reviews and technical support.

Education

MS, Food Science, Minor in Industrial Engineering, *Oregon State Univ. Corvallis, Oregon,* 1978.
BS, Food Science, Minor in Business Administration, *Utah State University, Logan, Utah,* 1975

Affiliations Achievements

ASQ-American Society of Quality, ISO/IEC 17025:2005 Expanded Internal Auditor Course

Quality Improvement Team Leader, Coffey Laboratories, Portland, Oregon. 1991

Methods Improvement Program, Coffey Laboratories, Portland, Oregon. Seminars on Development and Implementation 1990.

Statistical Process Control and Total Quality Management, Frito-Lay, Vancouver, Washington. Routine Training Classes 1986-1988.

JEFFREY A. CORONADO

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER IV, INORGANICS DEPARTMENT MANAGER –** 2001 to Present
Responsibilities	Oversee the operation of the Metals Group. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Metals Department Manager,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-2001. Responsibilities included management of all aspects of the metal laboratory operation, including personnel training and evaluation, review of all metals data, and report generation. Also responsible for client service on a number of ongoing CAS accounts. Technical duties include primary analytical responsibility for trace level metals analysis by ICP/MS. Analyses range from routine water and soil analysis, to marine tissues, as well as industrial applications such as ultra-trace QA/QC work for various semiconductor clients. Also responsible for a number of specialized sample preparation techniques including trace metals in seawater by reductive precipitation, and arsenic and selenium speciation by ion-exchange chromatography. Developed methodology for performing mercury analysis at low part per trillion levels by cold vapor atomic fluorescence.. **Supervisor, GFAA Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1989-1992. Responsibilities included supervision of metals analysis by graphite furnace atomic absorption following SW-846 and EPA CLP methodologies. Duties include workload scheduling, data review, instrument maintenance, personnel training and evaluation.
Education	**Field Immunoassay Training Course**, *EnSys Inc*., 1995. **Winter Conference on Plasma Spectrochemistry**, *San Diego, California,* 1994. **ICP-MS Training Course**, *VG-Elemental*, 1992. **BS, Chemistry,** *Western Washington University, Bellingham, Washington,* 1988. **BA, Business Administration,** *Western Washington University, Bellingham, Washington,* 1985.

HARVEY L. JACKY

1999TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

TECHNICAL MANAGER II 2008 to Present

Responsibilities

Oversee the operation of the General Chemistry and Microbiology groups. Responsible for the quality and timeliness of the inorganic laboratories analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation.

Documentation of Demonstration of Capabilities is available for review.

Experience

Project Manager III, *Columbia Analytical Services, Inc., Kelso, WA*, 1999-2008. Responsible for technical project management, ensuring overall data quality and compliance with customer requirements, and providing technical support to clients regarding laboratory application to projects. Additionally, acts as a consultant to clients regarding industrial/environmental compliance issues; serving as liaison between clients and regulatory agencies.

Director of Project Management, *Coffey Laboratories, Portland, Oregon*, 1997-1999. Responsible for technical project management. Communicated with clients to determine needs and expectations. Monitored laboratory production and ensured the timely completion of analytical projects. Technical consultant for clients regarding environmental compliance. Supervised and managed other members of the project management team. Served as a member of the senior management team for oversight of general operations, strategic planning, finances, and policy.

Project Manager/Chemist, *Coffey Laboratories, Portland, Oregon*, 1997-1999. Served as primary liaison between Coffey Laboratories and major clients. Ensured that work was completed in a timely manner and done to client specifications. Served as technical consultant regarding environmental chemistry, soil remediation, and waste water industrial compliance. Clients included the Oregon Department of Transportation, Hazmat Unit, Portland, Oregon; Raythion Demilitarization Co., Umatilla, Oregon; Hydroblast - Wastewater Evaporator Systems, Vancouver, Washington; and Union Pacific Railroad, Northwest Region, Klamath Falls, Oregon.

Technical Sales Representative, *Coffey Laboratories, Portland, Oregon*, 1995-1997. Responsible for marketing and sales, including actively prospecting for new potential clients. Additional responsibilities included procurement and preparation of all major project bids; ensuring that client expectations were met; and maintaining customer satisfaction. Served as consultant regarding industrial compliance issues, environmental remediation projects, and hazardous waste management.

Senior Chemist/Laboratory Chemical Hygiene Officer, *Coffey Laboratories, Portland, Oregon*, 1988-1995. Performed analytical tests including Anions by Ion Chromatography (EPA 300.0), PAHs by HPLC (EPA 8310), Cyanides (EPA 335), and other inorganic, wet chemistry, and organic analytical tests on a wide variety of sample matrices. Responsible for the initial quality assurance review of work performed, supervised and managed personnel. Developed and implemented Laboratory Chemical Hygiene Plan. Directed personnel in regards to safety issues and hazardous waste management. Served as consultant and teacher regarding analytical methodology, environmental compliance, and industrial hygiene.

Education

40-Hour Hazmat Certification, *PBS Environmental,* 1996.
Industrial Emergency Response, *SFSP Seminar,* 1991
BS, Zoology, *Oregon State University, Corvallis, Oregon,* 1988.
BS, General Science, *Oregon State University, Corvallis, Oregon,* 1988.
COURSEWORK, General Studies, *Linfield College, McMinnville, Oregon,* 1981-1982.

Publications/ Presentations

Biochemical and Physical Factors Involved in the Application and Measurement of a Soil Bioremediation System. Biogeochemistry, Portland State University, 1996

Affiliations

American Chemical Society, Member since 1988

CHRISTINA KERKSIECK

2008 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**SCIENTIST II** 2011 to Present
Responsibilities	Experience in pharmaceuticals, food microbiology and environmental samples. Experience in validation/qualification of all laboratory equipment (IQ/OQ/PQ). Development of new methods, SOP, validation protocols and report writing. Experience in design and operation of custom Microbiology testing (e.g. MIC Test (Minimum Inhibitory Concentration Test). Subject Matter Expert (SME for CAS environmental and pharmaceutical microbiology laboratories. Technical Manager Environmental-Review and approval of method development and investigations. Final approval of SOP. Review and final approval of analyst training records. Assist in PT corrective actions.
Experience	**Scientist I,** *Columbia Analytical Services, Kelso, WA.,* 2008-2011. Microbiologist performing routine and non-routine microbiology testing of pharmaceutical raw materials, excipients and drug products in accordance with applicable methods (USP, BAM, AOAC). Method development and validation as required. Subject Matter Expert (SME) for CAS environmental and pharmaceutical microbiology laboratories. **Analyst III,** *Columbia Analytical Services, Kelso, WA.,* 2008-2011. Responsible with analysis of BOD, CBOD, Total Coliform, Fecal Coliform, E. Coli, Heterotrophic Plate Count/Total Plate Count, Colilert/Quantitray, Bacteria Swab, Enterococcus, Enterolert, Dissolved Oxygen, Yeast and mold, Aerobic Plate, Sheen Screen, IRB, SRB, SCYM-Bart. **Scientist,** *Roche Molecular Systems, Alameda, California,* 2000-2007. Produced master cell banks for new controls. Test and certify controls for manufacturing. Prepared DNA Panels for projects. Extensive mammalian cell culture experience with excellent sterile technique. Lyophlization, RNA transcription, and Bacteriophage Production, DNA extraction/purification from all cell types. Responsible for equipment calibration, validation, and preventative maintenance. cGMP experience. Experience in writing IR s (Investigation Reports), SOP s (Standard Operating Procedures), and satisfying CAPA s (Corrective Action Preventative Action). Responsible for cryostorage inventory/management. Maintained documentation updated database and produced Certificates of Analysis . Responsible for lab purchasing, lab and instrument maintenance. Point person for cell repository ordering. Prepared and participated in internal/external audits. **Lab Assistant,** *Center for Biomedical Laboratory Science, San Francisco University, San Francisco, Californian,* 1999-2000. Performed research in Dr. Lily Chen s lab using the following techniques: transformation of bacteria and yeast, plasmid isolation from bacteria and yeast, agarose gel electrophoresis, restriction digestion and PCR. **Internship Assistant,** *Center For Biomedical Laboratory Science, San Francisco University, San Francisco, California,* 1998-1999. Assisted with various laboratory preparations and organized Med-Tech Administrative Program.
Education	**BS, Microbiology,** *San Francisco State University, San Francisco, CA,* 2000.

AQILLA KAMAWAL

2002 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**TECHNICAL MANAGER II, Semivolatile Organics Department Manager**– 2009 to Present
Responsibilities	Oversee the operation of the Semivolatile Organics Department. Responsible for the quality and timeliness of analytical reports, departmental budgets, workload coordination, method development efforts, cost-effectiveness, and resource allocation. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**TECHNICAL MANAGER I, GC SEMI-VOA,** *Columbia Analytical Services, Inc., Kelso, Washington,* 2007to 2009. Responsible for supervision of GC Semi-VOA staff, interfacing with Project Management Team, working with Extractions group, method development, training, data review, tracking department workload, scheduling analyses, and operation, maintenance and troubleshooting GC instrumentation. Also responsible for department adherence to strict QA/QC policies of the organization. **SCIENTIST III, GC SEMI-VOA,** *Columbia Analytical Services, Inc., Kelso, Washington,* 2002 to 2007. Responsible for operation, maintenance, and troubleshooting of GC/ECD and GC/FPD instrumentation. Performed instrumental analysis and all stages of data review for tests performed in SVG. Also involved in problem-solving with Extractions group, training, and workload coordination. **Chemist II, Pesticide Laboratory,** *Oregon Department of Agriculture, Portland, Oregon,* 2000-2002. Responsible for non-routine sample extraction and analysis of phenoxy herbicides, chlorinated acids, organochlorines, organophosphates, organonitrogens, sulfonyl ureas, carbamates, and other unclassified pesticides using a wide array of GC and LC instrumentation, including ECD, ELCD, FPD, AED, MS, and fluorescence detection. Also responsible for instrument maintenance, method development, data review, training, and assisting in workload coordination. **Chemist I, Pesticide Laboratory,** *Oregon Department of Agriculture, Portland, Oregon,* 1999-2000. Performed sample extraction and analysis by GC and LC using FDA and EPA methodologies. **Research Technologist,** *Shriners Hospital, Portland, Oregon,* 1995-1999. Worked with extracellular matrix proteins independently and under the supervision of/as assistant to post-doctoral associates. Protein isolation, purification, and characterization using the following techniques: cell culture, liquid chromatography (reverse-phase, ion-exchange, affinity), differential centrifugation, immunopreciptation, SDS-PAGE, immunoblotting, and ELISA assay. **Research Assistant/Thesis Student,** *Reed College, Portland, Oregon,* 1994-1995. Reviewed literature, devised and conducted synthetic organic experiments, and analyzed results using NMR and IR instrumentation.
Education	**BA, Chemistry,** *Reed College, Portland, Oregon,* 1996.

JONATHAN H. "JON" JAMES

1991 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position

Technical Manager II, VOA/MS, Semivolatile GC/MS and HPLC Department Manager, 2009-Present

Responsibilities

Oversee the operation of the Volatiles GC/MS, Semivolatile GC/MS and HPLC laboratories. Responsibilities include organizing and prioritizing workload, training and development of staff, working with PCs on client specific project requirements, departmental budgets, workload coordination, method development efforts and resource allocation. Responsible for the quality and timeliness of analytical reports. Other responsibilities include ensuring compliance with CAS QA protocols and assisting staff with troubleshooting equipment and procedural problems.

Documentation of Demonstration of Capabilities is available for review.

Experience

Technical Manager I, VOA and PHC/HPLC Laboratories, *Columbia Analytical Services, Inc., Kelso, Washington.* 2004-2009. Oversee daily operation of the Volatiles GC/MS and PHC/HPLC laboratories. Responsibilities include organizing and prioritizing workload, initiating process improvements, training and development of staff and working wit PCs on client specific project requirements. Responsible for analytical duties as listed below for Scientist IV. Other responsibilities include ensuring compliance with CAS QA protocols and assisting staff with troubleshooting equipment and procedural problems.

Scientist IV, VOA Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington*, 1999-2004. Perform sample analysis and data review for EPA methods 524.2, 624 and 8260. Duties also include Project Management.

Scientist III/Project Chemist, Supervisor Pesticides GC Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington*, 1998-1999. Primary responsibilities included workload scheduling, data review, instrument maintenance and troubleshooting, and personnel training and evaluation. Also responsible for supervision of extraction personnel and instrument analysts.

Scientist III, Semi-volatile Gas Chromatography Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington*, 1996-1998. Primary responsibilities included analysis of samples using GC and HPLC techniques, report generation, data review, preparation of analytical standards, maintenance of instrumentation, Client Services and some Project Management. Routine duties included analysis of soil and water samples for pesticides, PCBs, CLP Pesticides, Explosives and PAHs using EPA methods.

Scientist II, Semi-volatile Gas Chromatography Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington*, 1994-1996. Primary responsibilities included analysis of samples using GC and HPLC techniques, report generation, data review, preparation of analytical standards, maintenance of instrumentation and client service/project management duties.

Laboratory Analyst III, Semi-volatile Gas Chromatography Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington*, 1992-1994. Primary responsibilities included analysis of samples by GC/ECD, GC/FID, GC/FPD, GC/NPD and HPLC techniques. Standard analytical methods performed were EPA method 515.1, 504, 8150, 8011, 8150M (for chlorinated phenols), 8310, and 8015.

Laboratory Analyst II, Organic Extractions Laboratory, *Columbia Analytical Services, Inc., Kelso, Washington*, 1991-1992. Responsibilities included extraction of soil and water samples for various SVOCs, and TCLP extraction of SVOC and VOC compounds using TCLP equipment. Other duties included performing cleanup procedures, validation studies, MDL studies, and the training of employees in advanced extraction procedures and techniques.

Education

Introduction to LC Methods Development & Troubleshooting, *Hewlett-Packard, Tacoma, Washington,* 1995.
HPLC Maintenance Seminar, *Waters, Portland, Oregon,* 1994.
GC/HPLC Maintenance Seminar, *Hewlett-Packard, Olympia, Washington,* 1993.
Gas Chromatography Seminar, *Curtis Matheson Scientific, Kelso, Washington,* 1992.
HPLC Seminar, *Hewlett-Packard, Kelso, Washington,* 1991.
BA, Chemistry/Biology, *The Evergreen State College, Olympia, Washington,* 1991.
AA, Arts and Sciences, *Lower Columbia College, Longview, Washington,* 1990.

EILEEN M. ARNOLD

1987 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**SCIENTIST IV, METALS LABORATORY, KELSO HEALTH AND SAFTEY OFFICER –** 1994 to Present
Responsibilities	Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Project Chemist, Client Services Group, Kelso Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1994. Duties included technical project management and customer service. Responsible for meeting the clients' needs of timely and appropriate analyses, and to act as liaison for all client-related activities within Columbia Analytical Services, Inc. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Scientist IV, Metals Laboratory, Health and Safety Officer,** *Columbia Analytical Services, Inc., Kelso, Washington, 1987-1992.* Duties include the operation and maintenance of the Inductively Coupled Argon Plasma (ICAP) Emission Spectrometer. This involves digestion, instrumental analysis, and report generation for environmental samples using approved EPA techniques. Health and Safety Officer responsibilities included development and implementation of the Kelso Health and Safety program, including accident investigation and incident review, maintenance of all safety related equipment and documents, and performance of monthly safety audits. **Chemist,** *Dow Corning Corporation, Springfield, Oregon,* 1986-1987. Responsibilities included ICP and atomic absorption work in silicon manufacturing. Methods development for ICP analysis of minor impurities found in silicon. **Chemist,** *Ametek, Inc., Harleysville, Pennsylvania,* 1982-1985. Responsibilities included product research and development chemist involved in production of thin-film semiconductors for use as solar cells. Work involved AA and SEM techniques. **Chemist,** *Janbridge, Inc., Philadelphia, Pennsylvania*, 1978-1982. Responsibilities included maintaining electroplating process lines through wet chemical analysis techniques, and performed Quality Assurance testing on printed circuit boards.
Education	**BA, Chemistry,** *Immaculata College, Immaculata, Pennsylvania,* 1977.
Affiliations	American Chemical Society, Member since 1987.

LYNDA A. HUCKESTEIN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**CLIENT SERVICES MANAGER IV** 1998 to Present
Responsibilities	Management of the Client Services Departments: Project Management, Electronic Data Deliverables and Report Generation, and Sample Management. Personally responsible for approximately 1.5 million dollars of client work annually performing technical project management and client service. Provides technical and regulatory interpretation assistance, as well as project organization of work received by the laboratory. **Documentation of Demonstration of Capabilities is available for review.**
Experience	**Project Chemist,** *Columbia Analytical Service, Inc., Kelso, Washington,* 1992-1998. Primary responsibilities included technical project management and client service in areas of pulp paper, marine services, mining, and DOD. Also responsible for providing technical and regulatory interpretation assistance as-well-as project organization to work received by the laboratory **Project Chemist and Department Manager, General Chemistry Laboratory,** *Columbia Analytical Services, Inc.,* 1989-1992. Responsible for management of the General Chemistry laboratory for routine wastewater, bioassay, and microbiological analyses. Also responsible for supervision of staff, data review, and reporting. **Analyst III,** *Columbia Analytical Services, Inc., Kelso, Washington*, 1989. Primary responsibilities included coliform testing, total recoverable petroleum hydrocarbon extractions and analysis, BODs, ammonias, and TKN, in addition to miscellaneous wet chemistry analyses. **Microbiologist/Chemist**, *Coffey Laboratories, Portland, Oregon,* 1983. Coliform analysis; water chemistry. **Laboratory Assistant**, *Oregon State University, Corvallis, Oregon,* 1983. Wheat spike dissection and tissue culture.
Education	**BS, Microbiology,** *Oregon State University, Corvallis, Oregon,* 1983.

JEFFREY D. CHRISTIAN

1989 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**DIRECTOR OF OPERATIONS –** 2010 to Present
Responsibilities	Responsible for oversight of operating units of Columbia Analytical Services, inc. with all Laboratory Directors reporting to the COO. Primary responsibilities include establishment of consistent quality, technical, and client service enhancements across the company, as well as the financial performance of the individual operating units. In addition, a significant role is to represent operations as a member of the Senior Management Team (SMT) consisting of the Chief Executive Officer, Chief Financial Officer, Chief Quality Officer, and the Director of Information Technology.
Experience	**Vice President/Laboratory Director, Kelso Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1993-2010. Responsible for all phases of laboratory operations, including project planning, budgeting, and quality assurance. **Operations Manager, Kelso Laboratory,** *Columbia Analytical Services, Inc., Kelso, Washington,* 1992-1993. Responsibilities included directing the daily operation of the Kelso laboratory. Other responsibilities and duties included functioning as a technical consultant to clients, providing assistance in developing and planning analytical schemes to match client objectives, and writing and developing analytical procedures/methods. Also, served as Project Manager for State of Alaska Department of Environmental Conservation contract and Coordinator for EPA Special Analytical Services (SAS) contracts. **Project Chemist and Manager, Metals Analysis Laboratory,** *Columbia Analytical Services, Kelso, Washington,* 1989-1992. Responsible for directing the daily operation of the Metals Laboratory, including the sample preparation, AAS, ICP-OES, and ICP-MS Laboratories. **Scientist,** *Weyerhaeuser Technology Center, Federal Way, Washington,* 1986-1989. Responsibilities included supervising atomic spectroscopy laboratory which included flame and furnace AAS, ICP-OES, and sample preparation capabilities to handle a wide variety of sample types. Interfaced with internal and external clients to provide technical support. Wrote and developed analytical procedures/methods. **Lead Technician, Metals Lab,** *Weyerhaeuser Technology Center, Federal Way, Washington*, 1981-1986. Responsibilities included primary ICP and AAS analyst for EPA-CLP contract work. Extensive experience in wide variety of environmental and product-related testing. **Research Assistant,** *ITT Rayonier, Olympic Research Division, Shelton, Washington,* 1978-1981. Responsibilities included performing water quality tests, product-related analytical tests, corrosion tests (i.e., potentiometric polarization techniques), and operated pilot equipment specific to the pulp and paper industry.
Education	**B.S., Chemistry,** *Evergreen State College, Olympia, Washington,* 1993. **Coursework,** *Pacific Lutheran University, Tacoma, Washington.* 1988-1989. **Coursework,** *Tacoma Community College, Tacoma, Washington.* 1970-1971, 1988-1989. **CERTIFICATION, Chemistry,** *L.H. Bates Technical, Tacoma, Washington,* 1976-1978. **Coursework,** *Central Washington University, Ellensburg, Washington.* 1969-1970. **Numerous Training/Educational Activities via Conferences, Professional Seminars, and Factory Training,** 1989-2010.
Publications/ Presentations	*Mr. Christian has a number of publications and presentations. For a list of these publications and presentations, please contact CAS.*

LEE E. WOLF

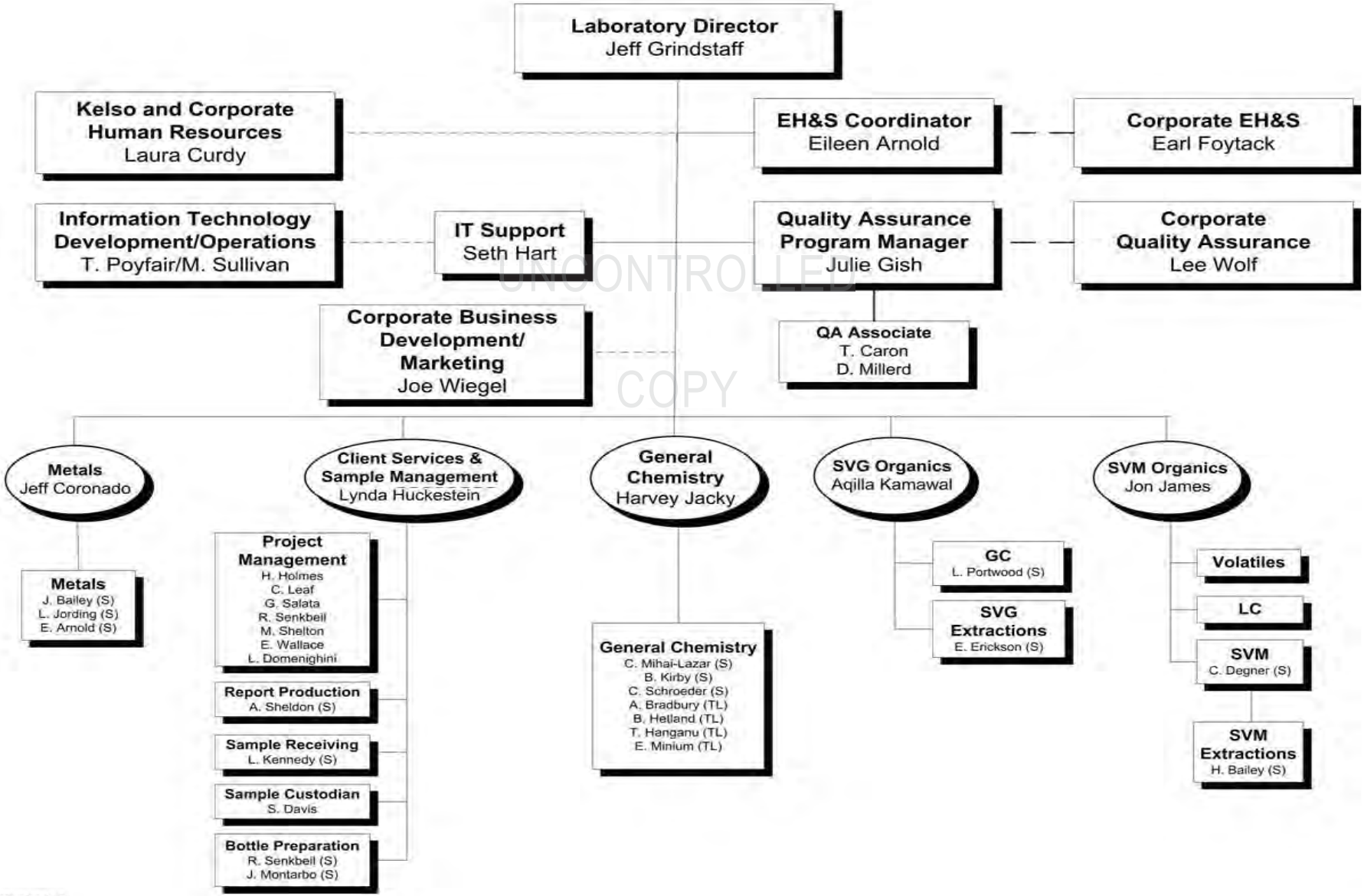
1988 TO PRESENT

Columbia Analytical Services, Inc., 1317 South 13th Ave., Kelso, WA 98626 360.577.7222

Current Position	**DIRECTOR OF QUALITY ASSURANCE** 2008 to Present
Responsibilities	Directing the overall corporate-wide quality systems and ethics programs for all CAS facilities. Responsible for ensuring that CAS quality systems and data integrity standards are implemented at all facilities. Act as liaison with government entities involving quality, technical and operational issues. Provide QA input and policy as needed for operations, development initiatives, special projects, planning, and information technology implementation. Provide assistance to QA Program Managers.
Experience	**Technical Manager IV, Quality Assurance Program Manager,** *Columbia Analytical Services, Inc., Kelso, Washington* **–** 2002 to 2008. As part of the management team, responsibilities included the overall management and implementation of the laboratory QA program. This included maintaining accreditations and certifications, and maintaining all necessary documents (QA Manual, SOPs, and QA records). Acted as primary point of contact during laboratory audits and provided audit responses and corrective actions. Coordinated performance audits (PE/PT testing) and conducted internal audits.
	Scientist IV, Quality Assurance Program Manager, *Columbia Analytical Services, Inc., Kelso, Washington,* 1996-2002. Duties primarily as listed above.
	Project Chemist/Principal Organic Scientist, *Columbia Analytical Services, Inc., Kelso, Washington,* 1994-1996. Responsibilities included GC and GC/MS method development and special projects coordination. Acts as technical advisor to the GC and GC/MS laboratories and GC/MS interpretation specialist and CLP organics specialist. Also responsible for Project Chemist functions, including management of projects for clients, identifying client needs, and preparation of data reports.
	Semivolatile Organics Department Manager, *Columbia Analytical Services*, 1988-1994. Responsibilities included overall management of the department. Supervised GC/MS analyses, data review, reporting and related QA/QC functions. Responsible for supervision of staff, training, and scheduling. Beginning in 1992, responsibilities included being a Project Chemist for organics EPA-SAS and other clients. This involved scheduling projects for clients, identifying client requirements, and preparing data reports.
	GC/MS Chemist, *U.S. Testing Co., Richland, Washington*, 1985-1988. Responsibilities included GC and GC/MS analysis of water and soil samples for volatiles and semivolatiles by EPA protocol, including Methods 8240, 8270 and CLP. Coordinated extraction and GC-GC/MS areas to manage sample/data flow through the lab. Also performed HPLC analysis and pesticide analysis by GC using EPA Methods.
	Laboratory Assistant, *Eastern Washington University, Cheney, Washington,* 1985. Responsibilities included supervision and instruction of organic chemistry labs. Experience with GC and IR operation. Responsible for lab safety.
Education	**Pharmaceutical Laboratory Control Systems,** *Univ. of Wisconsin Short Course, Las Vegas,* 2004 **Test Method Validation in Pharmaceutical Development and Production,** *Univ. of Wisconsin Short Course, Las Vegas,* 2004 **Documenting Your Quality System**, *A2LA Short Course, Las Vegas, Nevada*, 1998. **Internal Laboratory Audits,** *A2LA Short Course, Las Vegas, Nevada,* 1998. **Mass Spectra Interpretation**, *ACS Short Course, Denver, Colorado*, 1992. **BS, Chemistry,** *Eastern Washington University, Cheney, Washington,* 1985.
Publications/ Presentations	*Selected Ion Monitoring: Issues for Method Development*, Panel Discussion, Association of Official Analytical Chemists, (AOAC) Pacific Northwest Regional Meeting, 1995.
	Method Enhancement Techniques for Achieving Low level Detection of Butyl Tin in Marine Sediments and Tissues, Association of Official Analytical Chemists (AOAC) Pacific Northwest Regional Meeting, 1994.
	The Determination of Low-Level Concentrations of Polynuclear Aromatic Hydrocarbons (PAHs) in Soil and Water Using Gas Chromatography/Mass Spectroscopy Selected Ion Monitoring (GC/MS SIM), HazMat West, Long Beach, California, 1992.
Affiliations	American Chemical Society. American Society for Quality.

Columbia Analytical Services™

Environmental and General Testing Division
Kelso, Washington
Laboratory Organization



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APPENDIX C

MAJOR ANALYTICAL EQUIPMENT

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balances (16):			
Precisa,Mettler,OHOUS, Adams models	1990-2011	LM	13
Autoclave - Market Forge Sterilmatic	1988	LM	5
Autotitrator Thermo Orion 500	2007	LM	3
Calorimeters (2):			
Parr 1241 EA Adiabatic	1987	LM	4
Parr 6300 Isoparabolic	2005	LM	4
Centrifuge - Damon/IEC Model K	1992	LM	13
Colony Counter - Quebec Darkfield	1988	LM	2
Conductivity Meters (2):			
YSI Model 3200	2004	LM	4
VWR	2001	LM	4
Digestion Systems (5):			
COD (4)	1987, 1989	LM	4
Kjeldahl, Lachat 46-place (1)	1999	LM	3
Dissolved Oxygen Meter - YSI Model 58 (3)	1987, 1988, 1991	LM	4
Distillation apparatus (Midi) - Easy Still (2)	1996, 2000	LM	5
Drying Ovens (12):	1990-2010		
Shel-Lab and VWR models		LM	13
Air Drying Cabinets	2011	LM	NA
Flash Point Testers (2):			
ERDCO Setaflash Tester	1991	LM	3
Petroleum Systems Services	2005	LM	3
Flow-Injection Analyzers (2):			
Bran-Leubbe	2002	LM	2
Lachat 8500	2007	LM	2
Ion Chromatographs (4)			
Dionex DX-120 with Peaknet Data System	1998	LM	3
Dionex ICS-2500 with Chromchem Data System	2002	LM	3
Dionex ICS-2000 with Chromchem Data System	2006	LM	3
Dionex ICS-1600 with Chromchem Data System	2009	LM	
Ion Selective Electrode Meters (5)			
Fisher Scientific Accument Model 50	1997	LM	4
Fisher Scientific Accument Model 25	1993	LM	4
Fisher Scientific Accument Model 20	2000	LM	4
Orion Model 920A	1990	LM	4
Corning pH/ion Meter Model 135	1992	LM	4
Microscope - Olympus	1988	LM	1
Muffle Furnace- Sybron Thermolyne Model F-A1730	1991	LM	13

GENERAL CHEMISTRY/WATER CHEMISTRY LABORATORY (continued)			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
pH Meters (2):			
Fisher Scientific Accument Model 20	1993	LM	5
Fisher Scientific Accument Model AR25	2005	LM	5
Shatter Box (2):			
GP 1000	1989	LM	5
SPEX 8530	2011		
Sieve Shakers (2):			
CE Tyler - Portable RX 24	1990	LM	5
WS Tyler - RX 86	1991	LM	5
Thomas-Wiley Laboratory Mill, Model 4	1989	LM	5
Total Organic Carbon (TOC) Analyzers (2)			
Coulemetrics Model 5012	1997	LM	3
Teledyne Tekmar Fusion 1	2009	LM	3
Total Organic Halogen (TOX) Analyzers (2):			
Mitsubishi TOX-100	2001	LM	2
Turbidimeter - Hach Model 2100N	1996	LM	5
UV-Visible Spectrophotometers (3):			
Hitachi 100-40 Single Beam	1986	LM	4
Beckman-Coulter DU520	2005	LM	4
Perkin Elmer Lambda 25	2008	LM	4
Abrazix	2011	LM	2
Discrete Autoanlayzer Westco SmartChem AD20-1	2011	LM	2
Vacuum Pumps (3):			
Welch Duo-Seal Model 1376	1990	LM	13
Busch R-5 Series Single Stage	1991		
Chem Star 1402N-01	2011		
Water Baths/Incubators (6):	1986 - 2009	LM	13
Various Fisher Scientific and VWR Models			

METALS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (8)			
Mettler AE 200 analytical balance	1988-2010	MM	12
Various Mettler, Sartorius, and Ohaus models			
Atomic Absorption Spectrophotometers (5):			
Varian SpectrAA Zeeman/220 AA (2)	2000	LM	2
CETAC Mercury Analyzer M-6000A	2000	LM	2
Perkin Elmer AAnalyst 200 Flame AA	2005	MM	2
CETAC Mercury Analyzer M-6100	2010	MM	2
Buck AA Spectrophotometer Model 205	2008	LM	2
Atomic Fluorescence Spectrophotometer			
Brooks-Rand Model III (2)	1996, 2005	LM	3
Leeman Mercury Analyzer (1)	2006	LM	2
Centrifuge - IEC Model Clinical Centrifuge	1990	LM	12
Drying Oven - VWR Model 1370F	1990	LM	12
Freeze Dryers (1) - Labconco	2006	LM	5
Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) (2)			
Thermo Jarrell Ash, Model IRIS	2000	MM	3
Thermo Scientific Model iCAP 6500	2007	MM	3
Inductively Coupled Plasma Mass Spectrometers (ICP-MS):			
VG Excell	2001	MM	3
Thermo X-Series	2006	MM	2
Nexion Model 300D	2011	MM	2
Muffle Furnace (2) - Thermolyne Furnatrol - 53600	1991, 2005	LM	5
Shaker - Burrell Wrist Action Model 75	1990	LM	12
TCLP Extractors (3)	1989, 2002	LM	5

SEMIVOLATILE ORGANICS SAMPLE PREPARATION LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance (4) Mettler PM480, BB300 ,AG204 OHaus EP613	1999 - 2011	MM	12
Centrifuge Beckman J-6B	1988	LM	12
Drying Ovens (2) Fisher Model 655G VWR Model 1305U	 1991 1999	 LM LM	 12 12
Evaporators/concentrators Organomation N-Evap (8) Organomation S-Evap (8) Zymark Turbovap (2)	 1990-2010 1990-2010 1998-2000	 LM LM LM	 12 12 12
Extractor Heaters: Lab-Line Multi-Unit Models for Continuous Liquid-Liquid and Soxhlet Extractions (102)	1987-2007	LM	8
Solids Extractors: Sonic Bath VWR (2) Sonic Horn (5) Soxhtherm Gerhardt (2) OI Analytical (6)	 1991 -1994 1994 2000 2008	 LM LM LM	 6 6 6
Extractors, TCLP (10): Millipore TCLP Zero Headspace Extractors (5) TCLP Extractor - Tumbler (12 position)	 1987-1992 1989	 LM LM	 2 2
Gel Permeation Chromatography (GPC) (6) ABC single column (4) J2 Scientific AccuPrep (2)	 1998, 1999, 2007 2005, 2010	 LM LM	 4 4
Muffle Furnace - 4	1994-2006	LM	4
Solid Phase Extractors (18) Horizon SPE-Dex 4790	2003, 2006,2008	LM	4

GC SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Chromatography Data Systems (12)			
HP Enviroquant (8)	1994-2003	LM	7
Thruput Target (4)	1998-2000	LM	4
Varian Saturn (1)	2003	LM	2
Gas Chromatographs (17):			
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual ECD Detectors (2)	1990 1995	LM	6
Hewlett-Packard 5890 GC with HP 7673 Autosampler and Dual FPD Detectors	1991	LM	3
Agilent 6890 GC with Agilent 7683 Autosampler and Dual ECD Detectors (6)	2001, 2005, 2007,2011	LM	6
Agilent 6890 GC with Agilent 7683 Autosampler and Dual FPD Detectors	2003	LM	3
Agilent 7890A Dual ECD Detectors Agilent 7683B autosampler (2)	2010	LM	6
Hewlett-Packard 5890 GC with HP 7673 Autosampler and FID Detector	1995	LM	3
Agilent 6890 with Dual FID Detectors and Agilent 7873 Autosampler (4)	2001, 2005	LM	6
Varian Ion trap GC/MS:	2003	LM	2
Varian 3800 GC w/CP8400 autosampler	2006	LM	2
Varian Saturn 2100T mass spectrometer	2003	LM	2
Thermo Ion Trap ITQ-90C GC/MS w/TriPlus autosampler	2008	LM	2

GC/MS SEMIVOLATILE ORGANICS INSTRUMENT LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler AB 104-S	2000	MM	6
Enviroquant Chromatography Data Systems (9)	1994-2003	LM	6
Gas Chromatograph: Hewlett-Packard 5890 with HP 7673 autosampler and FID Detector	1994	LM	6
Semivolatile GC/MS Systems (11):			
Agilent 6890/5973 with ATAS Optic2 LVI and HP 7673 Autosampler (2)	1997, 2001	LM	6
Agilent 5890/5970 and HP 7673 Autosampler	1990	LM	6
Agilent 5890/5970 with ATAS Optic2 LVI and HP 7673 Autosampler	1994	LM	6
Agilent 5890/5972 with ATAS Optic2 LVI and HP 7673 Autosampler (3)	1993, 1994, 1998	LM	6
Agilent 6890/5973 with ATAS PTV and 7683 Autosampler	2004	LM	6
Agilent 6890/5973 with Agilent PTV Injector and 7683 Autosampler	2007	LM	6
Agilent7890A/5975C with Agilent 7693 Autosampler (2)	2010	LM	6
Semivolatile GC/MS/MS			
Waters Quattro Micro GC Micromass with Agilent 6890, Agilent PTV Injector, 7683B Autosampler	2008	MM	2

HPLC LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler BB240	1994	MM	4
Drying Oven - Fisher Model 630F	1991	LM	4
Evaporator Turbo Vap	2009	LM	4
Centrifuge Marathon 21K	1996	LM	4
HP Enviroquant Chromatography Data Systems 4	1994-2002	LM	3
High-Performance Liquid Chromatographs (3):			
HP 1090M Series II with Diode Array UV Detector	1999	LM	2
HP 1050/1100 Series with Fluorescence Diode Array UV Detectors	2004	LM	2
Agilent 1260 Infinity with Diode Array UV Detector	2011	LM	2
High-Performance LC/MS (3)			
Spectrometer - Thermo Electron TSQ Quantum LC/MS/MS and Autosampler	2005	MM	2
API 5000 LC/MS/MS and SIL-20AC Autosampler	2008	MM	2
AB Sciex 5500 and Schimadzo DGU 20A5	2011	MM	2
Agilent 1100 HPLC -UV/Fluoescence detectors- Pickering PCX-5200 Post-column derivitization unit	2003	LM	2

VOLATILE ORGANICS LABORATORY			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
Analytical Balance - Mettler PE 160	1989	MM	5
Fisher Vortex Mixer	1989	LM	5
HP Enviroquant Chromatography Data Systems (10)	1994-2002	LM	5
Drying Ovens (2):			
Boekel 107801	1989	LM	5
VWR 1305 U	1991	LM	5
Sonic Water Bath - Branson Model 2200	1989	LM	5
Volatile GC/MS Systems (9):			
Agilent 5890/5970	1989	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 5890/5971	1991	LM	5
Tekmar 3000 Purge and Trap Concentrator	2001	LM	5
Dynatech ARCHON 5100 Autosampler	1995	LM	5
Agilent 5890/5972A	1993	LM	5
Tekmar 3000 Purge and Trap Concentrator	1995	LM	5
Dynatech ARCHON 5100 Autosampler	1996	LM	5
Agilent 6890/5973	2001	LM	5
Tekmar 3100 Purge and Trap Concentrator	2001	LM	5
Varian Archon Autosampler	2001	LM	5
Agilent 6890/5973	2005	LM	5
Tekmar Velocity Purge and Trap Concentrator	2005	LM	5
Tekmar Aquatech Autosampler	2005	LM	5
Agilent 6890/5973 (2)	2007	LM	5
Tekmar 3000 Purge and Trap Concentrator	2007	LM	5
Varian Archon 5100 Autosampler	2007	LM	5
Agilent 7980A/5975C (2)	2010, 2011	LM	5
Teledyne Tekmar-Automx	2010,2011	LM	5
Hewlett-Packard 5890 Series II with PID/PID/FID	1991	LM	2
EST-ENCON Purge and Trap Concentrator	1991		
Dynatech Archon 5100 Autosampler	1992		

AUTOMATED DATA PROCESSING EQUIPMENT			
Equipment Description	**Year Acquired**	**Manufacturer or Laboratory Maintained (MM/LM)**	**# of Trained Operators**
1-WAN: LIMS Sample Manager using Oracle 10g 11g DBMS running on Redhat Advanced Server 4.0 (Linux) platform connected/linked via both fiber and MPLS circuits.	1994-2007	LM	NA
1 - Network Server Pentium 4 class, 1 for Reporting and Data Acquisition running Windows 2003 SP2 Advanced Server, 1 for Applications running Windows 2003 Advanced Server SP2. Data acquisition capacity at 195 GB with redundant tape and disk arrays.	2004-2008	LM	NA
Approximately 80+ HP, Dell, Kyocera Laserjet printers (various types including models III, 4, 5, 8150, 4000, 4041, 4050, 4200 4250, 8150, 1720dn, W5300, 1300D, M4000)	1991 - 2010	LM	NA
Approximately 280 + Gateway/Dell PC/Workstations running Windows 2000/XP on LAN connected via 10BT/100BT and TCP/IP for LIMs Terminal Emulation	1993 - 2010	LM	NA
Microsoft Office 2003 Professional as the base application for all PC/Workstations. Some systems using Office 2000/97, Office 2007.	1996 - 2010	LM	NA
E-Mail with link to SMTP for internal/external messaging. Web mail via Outlook Web Access interface. Microsoft Outlook 2003.	1994 - 2006	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Standard Excel (R) reporting platform application linked to LAN/WAN for data connectivity and EDD generation.	1996 - 2004	LM	NA
Facsimile Machines - Brother 4750e; Brother 2920; Brother 1860	1991 - 2010	LM	NA
Copiers/Scanners: Konica BizHub 420 (1), BizHub 600 (1), BizHub 920 (2), BizHub Pro 1050 (3), BizHub Pro 1051 (1). All are accessible via LAN for network scanning.	2000 - 2010	LM	NA
Dot Matrix Panasonic KX-P1150	1991 - 2004	LM	NA
Thruput, MARRS, Stealth, Harold, Blackbird, EDDGE, CASLIMS reporting software systems.	1998 - 2004	LM	NA

NA: Not applicable. This equipment administered by IT staff but may be used by all staff.

APPENDIX D

DATA QUALIFIERS AND ACRONYMS

Inorganic Data Qualifiers

*	The result is an outlier. See case narrative.
#	The control limit criteria are not applicable. See case narrative.
B	The analyte was found in the associated method blank at a level that is significant relative to the sample result as defined by the DOD or NELAC standards.
E	The result is an estimate amount because the value exceeded the instrument calibration range.
J	The result is an estimated value that was detected outside the quantitation range.
U	The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM definition*: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
i	The MRL/MDL or LOQ/LOD is elevated due to matrix interference.
X	See case narrative.
Q	See case narrative. One or more quality control criteria was outside the limits.

Metals Data Qualifiers

#	The control limit criteria are not applicable. See case narrative.
J	The result is an estimated value that was detected outside the quantitation range.
E	The percent difference for the serial dilution was greater than 10%, indicating a possible matrix interference in the sample.
M	The duplicate injection precision was not met.
N	The Matrix Spike sample recovery is not within control limits. See case narrative.
S	The reported value was determined by the Method of Standard Additions (MSA).
U	The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM 4.1 definition*: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
W	The post-digestion spike for furnace AA analysis is out of control limits, while sample absorbance is less than 50% of spike absorbance.
i	The MRL/MDL or LOQ/LOD is elevated due to matrix interference.
X	See case narrative.
+	The correlation coefficient for the MSA is less than 0.995.
Q	See case narrative. One or more quality control criteria were outside the limits.

Organic Data Qualifiers

*	The result is an outlier. See case narrative.
#	The control limit criterion is not applicable. See case narrative.
A	A tentatively identified compound, a suspected aldol-condensation product.
B	The analyte was found in the associated method blank at a level that is significant relative to the sample result as defined by the DOD or NELAC standards.
C	The analyte was qualitatively confirmed using GC/MS techniques, pattern recognition, or by comparing to historical data.
D	The reported result is from a dilution.
E	The result is an estimate amount because the value exceeded the instrument calibration range.
J	The result is an estimated value that was detected outside the quantitation range.
N	The result is presumptive. The analyte was tentatively identified, but a confirmation analysis was not performed.
P	The GC or HPLC confirmation criteria were exceeded. The relative percent difference is greater than 40% between the two analytical results.
U	The analyte was analyzed for, but was not detected ("Non-detect") at or above the MRL/MDL. *DOD-QSM 4.1 definition*: Analyte was not detected and is reported as less than the LOD or as defined by the project. The detection limit is adjusted for dilution.
i	The MRL/MDL or LOQ/LOD is elevated due to a chromatographic interference.
X	See case narrative.
Q	See case narrative. One or more quality control criteria was outside the limits.

Additional Petroleum Hydrocarbon Specific Qualifiers

F	The chromatographic fingerprint of the sample matches the elution pattern of the calibration standard.
L	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of lighter molecular weight constituents than the calibration standard.
H	The chromatographic fingerprint of the sample resembles a petroleum product, but the elution pattern indicates the presence of a greater amount of heavier molecular weight constituents than the calibration standard.
O	The chromatographic fingerprint of the sample resembles an oil, but does not match the calibration standard.
Y	The chromatographic fingerprint of the sample resembles a petroleum product eluting in approximately the correct carbon range, but the elution pattern does not match the calibration standard.
Z	The chromatographic fingerprint does not resemble a petroleum product.

Acronyms

ASTM	American Society for Testing and Materials
A2LA	American Association for Laboratory Accreditation
CARB	California Air Resources Board
CAS Number	Chemical Abstract Service registry Number
CFC	Chlorofluorocarbon
CFU	Colony-Forming Unit
DEC	Department of Environmental Conservation
DEQ	Department of Environmental Quality
DHS	Department of Health Services
DOE	Department of Ecology
DOH	Department of Health
EPA	U. S. Environmental Protection Agency
ELAP	Environmental Laboratory Accreditation Program
GC	Gas Chromatography
GC/MS	Gas Chromatography/Mass Spectrometry
LUFT	Leaking Underground Fuel Tank
LOD	Limit of Detection
LOQ	Limit of Quantitation
M	Modified
MCL	Maximum Contaminant Level is the highest permissible concentration of a substance allowed in drinking water as established by the USEPA.
MDL	Method Detection Limit
MPN	Most Probable Number
MRL	Method Reporting Limit
NA	Not Applicable
NC	Not Calculated
NCASI	National Council of the Paper Industry for Air and Stream Improvement
ND	Not Detected
NIOSH	National Institute for Occupational Safety and Health
PQL	Practical Quantitation Limit
RCRA	Resource Conservation and Recovery Act
SIM	Selected Ion Monitoring
TPH	Total Petroleum Hydrocarbons

APPENDIX E

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Maint[a]	Frequency
Refrigerators and Coolers	Record temperatures	LM	Daily
	Clean coils	LM	Annually
	Check coolant	LM	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	LM	Every month or as needed
Fume Hoods	Face velocity measured	LM	Quarterly
	Sash operation	LM	As needed
	Change filters	LM	Annually
	Inspect fan belts	LM	Annually
Ovens	Clean	LM	As needed or if temperature outside lim.
	Record temperatures	LM	Daily, when in use
Incubators	Record temperatures	LM	Daily, morning and evening
Water Baths	Record temperatures	LM	Daily, morning and evening
	Wash with disinfectant solution	LM	When water is murky, dirty, or when growth appears
Autoclave	Check sterility	LM	Every month
	Check temperature	LM	Every month
	Clean	LM	When mold or growth appears
	Calibrate thermometer	VM	Once a year
Analytical Balances	Check alignment	LM	Before every use
	Verify calibration	LM	Daily
	Clean pans and compartment	LM	After every use
	Certified calibration	VM	Once a year
Dissolved Oxygen Meter	Change membrane	LM	When fluctuations occur
pH probes	Condition probe	LM	When fluctuations occur
Fluoride ISE	Store in storage solution	LM	Between uses
Ammonia ISE	Store in storage solution	LM	Between uses
UV-visible Spectrophotometer	Wavelength check	VM	Twice a year

Instrument	Activity	Maint[a]	Frequency
Total Organic Carbon Analyzers	Check IR zero	LM	Weekly
	Check digestion/condensation vessels	LM	Each use
	Clean digestion chamber	LM	Every 2000 hours, or as needed
	Clean permeation tube	LM	Every 2000 hours, or as needed
	Clean six-port valves	LM	Every 200 - 2000 hours, or as needed
	Clean sample pump	LM	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	LM	Every 200 - 2000 hours, or as needed
	Clean IR cell	LM	Every 2000 - 4000 hours, or as needed
Total Organic Carbon Analyzers	Change cell electrolyte	LM	Daily
	Change electrode fluids	LM	Daily
	Change pyrolysis tube	LM	As needed
	Change inlet and outlet tubes	LM	As needed
	Change electrodes	LM	As needed
Flow Injection Analyzer	Check valve flares	LM	Each use
	Check valve ports	LM	Each use
	Check pump tubing	LM	Each use
	Check light counts	LM	Each use
	Check flow cell flares	LM	Quarterly
	Change bulb	LM	As needed
	Check manifold tubing	LM	Each use
	Check T's and connectors	LM	Each use
Discrete Auto Analyzer	Clean probe, wash reservoirs	LM	Every 2 weeks
	Replace peristaltic pump tubing	LM	Every 3 months
	Replace hydraulic circuit tubing	LM	Once/year

Instrument	Activity	Maint[a]	Frequency
Ion Chromatographs	Change column	LM	Every six months or as needed
	Change valve port face hex nut	LM	Every six months or as needed
	Clean valve slider	LM	Every six months or as needed
	Change tubing	LM	Annually or as needed
	Eluent pump	LM	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	LM	Daily
	Clean burner head	LM	Daily
	Check aspiration tubing	LM	Daily
Atomic Absorption Spectro-photometers - GFAA	Clean optics	LM	Every three months
	Empty waste container	LM	Weekly
	Check gases	LM	Daily
	Check argon dewar	LM	Daily
	Change graphite tube	LM	Daily, as needed
	Clean furnace windows	LM	Monthly
ICP - AES	Check argon dewar	LM	Daily
	Replace peristaltic pump tubing	LM	Daily
	Empty waste container	LM	Weekly
	Clean nebulizer, spray chamber, and torch	LM	Every two weeks
	Replace water filter	LM	Quarterly
	Replace vacuum air filters	LM	Monthly
ICP - MS	Check argon dewar	LM	Daily
	Check water level in chiller	LM	Daily
	Complete instrument log	LM	Daily
	Replace peristaltic pump tubing	LM	Daily
	Clean sample and skimmer cones	LM	As needed
	Clean RF contact strip	LM	As needed
	Inspect nebulizer, spray chamber, and torch	LM	Clean as needed
	Clean lens stack/extraction lens	LM	As needed
	Check rotary pump oil	LM	Monthly
	Change rotary pump oil	LM	Every six months

Instrument	Activity	Maint[a]	Frequency
Gel-Permeation Chromatographs	Clean and repack column	LM	As needed
	Backflush valves	LM	As needed
HPCL Chromatographs	Backflush guard column	LM	As needed
	Backflush column	LM	As needed
	Change guard column	LM	As needed when back pressure too high
	Change column	LM	Annually or as needed
	Change in-line filters	LM	As needed
	Leak check	LM	After column maintenance
	Change pump seals	LM	As needed
	Change pump diaphragm	LM	Annually
	Clean flow cell	LM	As needed
	Fluorescence detector check	LM	Daily
	Diode array absorbance check	LM	Daily
HPLC MS/MS	Clean ion transfer tube	LM	Daily or noticeable decrease in signal
	Clean inlet assembly	LM	Monthly or as needed
	Forepump	LM	Blast weekly; change oil every 3 months
Gas Chromatographs, Semivolatiles	Check gas supplies	LM	Daily, replace if pressure reaches 50psi
	Change in-line filters	LM	Quarterly or after 30 tanks of gas
	Change septum	LM	Daily
	Change injection port liner	LM	Weekly or as needed
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Check system for gas leaks	LM	After changing columns and after any power failure
	Clean FID	LM	Weekly or as needed
	Clean ECD	LM	Quarterly or as needed
	Leak test ECD	LM	Annually

Instrument	Activity	Maint[a]	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	LM	Daily, replace if pressure reaches 50psi
	Change in-line filters	LM	Annually or as needed
	Change septum	LM	Daily, when in use
	Change injection port liner	LM	Weekly or as needed
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Clean source	LM	As needed when tuning problems
	Change pump oil	LM	As specified by service specifications
Purge and Trap Concentrators	Change trap	LM	Every four months or as needed
	Change transfer lines	LM	Every six months or as needed
	Clean purge vessel	LM	Daily
Gas Chromatographs, Volatiles	Check gas supplies	LM	Daily, replace when pressure reaches 50 psi
	Change in-line filters	LM	Quarterly or after 30 tanks of gas
	Change septum	LM	Daily
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Check system for gas leaks	LM	After changing columns and after any power failure
	Clean PID lamp	LM	As needed
	Clean FID	LM	As needed
	Change ion exchange resin	LM	Every 60 days
	Replace nickel tubing	LM	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	LM	Daily, replace when pressure reaches 50 psi
	Change in-line filters	LM	Annually or as needed
	Change septum	LM	Daily
	Clip first foot of capillary column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Clean source	LM	As needed when tuning problems
	Change pump oil		As specified by service specifications

APPENDIX E

PREVENTIVE MAINTENANCE PROCEDURES

Instrument	Activity	Maint[a]	Frequency
Refrigerators and Coolers	Record temperatures	LM	Daily
	Clean coils	LM	Annually
	Check coolant	LM	Annually or if temperature outside limits
Vacuum Pumps	Clean and change pump oil	LM	Every month or as needed
Fume Hoods	Face velocity measured	LM	Quarterly
	Sash operation	LM	As needed
	Change filters	LM	Annually
	Inspect fan belts	LM	Annually
Ovens	Clean	LM	As needed or if temperature outside lim.
	Record temperatures	LM	Daily, when in use
Incubators	Record temperatures	LM	Daily, morning and evening
Water Baths	Record temperatures	LM	Daily, morning and evening
	Wash with disinfectant solution	LM	When water is murky, dirty, or when growth appears
Autoclave	Check sterility	LM	Every month
	Check temperature	LM	Every month
	Clean	LM	When mold or growth appears
	Calibrate thermometer	VM	Once a year
Analytical Balances	Check alignment	LM	Before every use
	Verify calibration	LM	Daily
	Clean pans and compartment	LM	After every use
	Certified calibration	VM	Once a year
Dissolved Oxygen Meter	Change membrane	LM	When fluctuations occur
pH probes	Condition probe	LM	When fluctuations occur
Fluoride ISE	Store in storage solution	LM	Between uses
Ammonia ISE	Store in storage solution	LM	Between uses
UV-visible Spectrophotometer	Wavelength check	VM	Twice a year

Instrument	Activity	Maint[a]	Frequency
Total Organic Carbon Analyzers	Check IR zero	LM	Weekly
	Check digestion/condensation vessels	LM	Each use
	Clean digestion chamber	LM	Every 2000 hours, or as needed
	Clean permeation tube	LM	Every 2000 hours, or as needed
	Clean six-port valves	LM	Every 200 - 2000 hours, or as needed
	Clean sample pump	LM	Every 200 - 2000 hours, or as needed
	Clean carbon scrubber	LM	Every 200 - 2000 hours, or as needed
	Clean IR cell	LM	Every 2000 - 4000 hours, or as needed
Total Organic Carbon Analyzers	Change cell electrolyte	LM	Daily
	Change electrode fluids	LM	Daily
	Change pyrolysis tube	LM	As needed
	Change inlet and outlet tubes	LM	As needed
	Change electrodes	LM	As needed
Flow Injection Analyzer	Check valve flares	LM	Each use
	Check valve ports	LM	Each use
	Check pump tubing	LM	Each use
	Check light counts	LM	Each use
	Check flow cell flares	LM	Quarterly
	Change bulb	LM	As needed
	Check manifold tubing	LM	Each use
	Check T's and connectors	LM	Each use
Discrete Auto Analyzer	Clean probe, wash reservoirs	LM	Every 2 weeks
	Replace peristaltic pump tubing	LM	Every 3 months
	Replace hydraulic circuit tubing	LM	Once/year

Instrument	Activity	Maint[a]	Frequency
Ion Chromatographs	Change column	LM	Every six months or as needed
	Change valve port face hex nut	LM	Every six months or as needed
	Clean valve slider	LM	Every six months or as needed
	Change tubing	LM	Annually or as needed
	Eluent pump	LM	Annually
Atomic Absorption Spectro-photometers - FAA and CVAA	Check gases	LM	Daily
	Clean burner head	LM	Daily
	Check aspiration tubing	LM	Daily
Atomic Absorption Spectro-photometers - GFAA	Clean optics	LM	Every three months
	Empty waste container	LM	Weekly
	Check gases	LM	Daily
	Check argon dewar	LM	Daily
	Change graphite tube	LM	Daily, as needed
	Clean furnace windows	LM	Monthly
ICP - AES	Check argon dewar	LM	Daily
	Replace peristaltic pump tubing	LM	Daily
	Empty waste container	LM	Weekly
	Clean nebulizer, spray chamber, and torch	LM	Every two weeks
	Replace water filter	LM	Quarterly
	Replace vacuum air filters	LM	Monthly
ICP - MS	Check argon dewar	LM	Daily
	Check water level in chiller	LM	Daily
	Complete instrument log	LM	Daily
	Replace peristaltic pump tubing	LM	Daily
	Clean sample and skimmer cones	LM	As needed
	Clean RF contact strip	LM	As needed
	Inspect nebulizer, spray chamber, and torch	LM	Clean as needed
	Clean lens stack/extraction lens	LM	As needed
	Check rotary pump oil	LM	Monthly
	Change rotary pump oil	LM	Every six months

Instrument	Activity	Maint[a]	Frequency
Gel-Permeation Chromatographs	Clean and repack column	LM	As needed
	Backflush valves	LM	As needed
HPCL Chromatographs	Backflush guard column	LM	As needed
	Backflush column	LM	As needed
	Change guard column	LM	As needed when back pressure too high
	Change column	LM	Annually or as needed
	Change in-line filters	LM	As needed
	Leak check	LM	After column maintenance
	Change pump seals	LM	As needed
	Change pump diaphragm	LM	Annually
	Clean flow cell	LM	As needed
	Fluorescence detector check	LM	Daily
	Diode array absorbance check	LM	Daily
HPLC MS/MS	Clean ion transfer tube	LM	Daily or noticeable decrease in signal
	Clean inlet assembly	LM	Monthly or as needed
	Forepump	LM	Blast weekly; change oil every 3 months
Gas Chromatographs, Semivolatiles	Check gas supplies	LM	Daily, replace if pressure reaches 50psi
	Change in-line filters	LM	Quarterly or after 30 tanks of gas
	Change septum	LM	Daily
	Change injection port liner	LM	Weekly or as needed
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Check system for gas leaks	LM	After changing columns and after any power failure
	Clean FID	LM	Weekly or as needed
	Clean ECD	LM	Quarterly or as needed
	Leak test ECD	LM	Annually

Instrument	Activity	Maint[a]	Frequency
Gas Chromatograph/Mass Spectrometers, Semivolatiles	Check gas supplies	LM	Daily, replace if pressure reaches 50psi
	Change in-line filters	LM	Annually or as needed
	Change septum	LM	Daily, when in use
	Change injection port liner	LM	Weekly or as needed
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Clean source	LM	As needed when tuning problems
	Change pump oil	LM	As specified by service specifications
Purge and Trap Concentrators	Change trap	LM	Every four months or as needed
	Change transfer lines	LM	Every six months or as needed
	Clean purge vessel	LM	Daily
Gas Chromatographs, Volatiles	Check gas supplies	LM	Daily, replace when pressure reaches 50 psi
	Change in-line filters	LM	Quarterly or after 30 tanks of gas
	Change septum	LM	Daily
	Clip first 6-12" of capillary column	LM	As needed
	Change guard column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Check system for gas leaks	LM	After changing columns and after any power failure
	Clean PID lamp	LM	As needed
	Clean FID	LM	As needed
	Change ion exchange resin	LM	Every 60 days
	Replace nickel tubing	LM	Quarterly or as needed
Gas Chromatograph/Mass Spectrometers, Volatiles	Check gas supplies	LM	Daily, replace when pressure reaches 50 psi
	Change in-line filters	LM	Annually or as needed
	Change septum	LM	Daily
	Clip first foot of capillary column	LM	As needed
	Replace analytical column	LM	As needed when peak resolution fails
	Clean source	LM	As needed when tuning problems
	Change pump oil		As specified by service specifications

APPENDIX F

LABORATORY
STANDARD OPERATING PROCEDURES

Administrative SOP Kelso

SOP Title	FILE NAME
CHECKING VOLUMETRIC LABWARE	ADM-VOLWARE
CONTINGENCY PLAN FOR LABORATORY EQUIPMENT FAILURE	ADM-ECP
CONTROL CHARTING QUALITY CONTROL DATA	ADM-CHRT
DATA ARCHIVING	ADM-ARCH
DATA REPORTING AND REPORT GENERATION	ADM-RG
DEPARTMENT OF DEFENSE PROJECTS LABORATORY PRACTICES AND PROJECT MANAGEMENT	ADM-DOD
ELECTRONIC DATA BACKUP AND ARCHIVING	ADM-EBACKUP
INTERNAL QUALITY ASSURANCE AUDITS	ADM-IAUD
LABORATORY BALANCE MONITORING AND CALIBRATION	ADM-BAL
LABORATORY DATA REVIEW PROCESS	ADM-DREV
PROJECT MANAGEMENT	ADM-PCM
REAGENT LOGIN AND TRACKING	ADM-RLT
SUPPORT EQUIPMENT MONITORING AND CALIBRATION	ADM-SEMC
SAMPLE BATCHES	ADM-BATCH
SAMPLE MANAGEMENT SOPS	**FILE NAME**
BOTTLE ORDER PREPARATION AND SHIPPING	SMO-BORD
FOREIGN SOILS HANDLING TREATMENT	SMO-FSHT
SAMPLE DISPOSAL	SMO-SDIS
SAMPLE RECEIVING	SMO-GEN
SAMPLE TRACKING AND LABORATORY CHAIN OF CUSTODY	SMO-SCOC

Technical SOP - Kelso

COLIFORM, FECAL	BIO-9221FC
COLIFORM, TOTAL	BIO-9221TC
COLIFORM, FECAL (MEMBRANE FILTER PROCEDURE)	BIO-9222D
COLILERT , COLILERT-18 , COLISURE	BIO-9223
FECAL STREPTOCOCCUS/ENTEROCOCCUS	BIO-9230B
ENTEROLERT	BIO-ENT
HEPTEROTROPHIC PLATE COUNT	BIO-HPC
MICROBIOLOGY QUALITY ASSURANCE AND QUALITY CONTROL	BIO-QAQC
SHEEN SCREEN/OIL DEGRADING MICROORGANISMS	BIO-SHEEN
SEPARATORY FUNNEL LIQUID-LIQUID EXTRACTION	EXT-3510
CONTINUOUS LIQUID - LIQUID EXTRACTION	EXT-3520
SOLID PHASE EXTRACTION	EXT-3535
SOXHLET EXTRACTION	EXT-3540
AUTOMATED SOXHLET EXTRACTION	EXT-3541
ULTRASONIC EXTRACTION	EXT-3550
WASTE DILUTION EXTRACTION	EXT-3580
SILICA GEL CLEANUP	EXT-3630
GEL PERMEATION CHROMATOGRAPHY	EXT-3640A
REMOVAL OF SULFUR USING COPPER	EXT-3660
REMOVAL OF SULFUR USING MERCURY	EXT-3660M
SULFURIC ACID CLEANUP	EXT-3665
CARBON CLEANUP	EXT-CARCU
DIAZOMETHANE PREPARATION	EXT-DIAZ
DMD SYNTHESIS	EXT-DMD

Technical SOP - Kelso

FACILITY AND LABORATORY CLEANING	FAC-CLEAN
OPERATION AND MAINTENANCE OF LABORATORY REAGENT WATER SYSTEMS	FAC-WATER
FLASHPOINT DETERMINATION - SETAFLASH	GEN-1020
COLOR	GEN-110.2
TOTAL SOLIDS	GEN-160.3
SOLIDS, TOTAL VOLATILE AND PERCENT ASH IN SOIL AND SOLID SAMPLES	GEN-160.4
SETTEABLE SOLIDS	GEN-160.5
HALIDES, ADSORBABLE ORGANIC (AOX)	GEN-1650
GRAVIMETRIC DETERMINATION OF HEAXANE EXTRACTABLE MATERIAL (1664)	GEN-1664
ALKALINITY TOTAL	GEN-2320
HARDNESS, TOTAL	GEN-2340
DETERMINATION OF INORGANIC ANIONS IN DRINKING WATER BY ION CHROMATOGRAPHY	GEN-300.1
ACIDITY	GEN-305.2
PERCHLORATE BY ION CHROMATOGRAPHY	GEN-314.0
CHLORIDE (TITRIMETRIC, MERCURIC NITRATE)	GEN-325.3
CHLORINE, TOTAL/FREE RESIDUAL	GEN-330.4
TOTAL RESIDUAL CHLORINE - METHOD 330.5	GEN-330.5
AMMONIA BY FLOW INJECTION ANALYSIS	GEN-350.1
AMMONIA AS NITROGEN BY ION SPECIFIC ELECTRODE	GEN-350.3
NITRATE/NITRITE, NITRITE BY FLOW INJECTION ANALYSIS	GEN-353.2
PHOSPHORUS DETERMINATION USING COLORMETRIC PROCEDURE	GEN-365.3
PHENOLICS, TOTAL	GEN-420.1
ORTHOPHOSPHATE DETERMINATION USING COLORIMETRIC PROCEDURE	GEN-4500-PE

Technical SOP - Kelso (CONT.)

DISSOLVED SILICA	GEN-4500 SIO_2C
GRAVIMETRIC SULFATE	GEN-4500 SO4 C
NITRITE BY COLORIMETRIC PROCEDURE	GEN-4500NO2 B
SULFIDE, METHYLENE BLUE	GEN-4500S2D
SULFIDE, TITRIMETRIC (IODINE)	GEN-4500S2F
TRIAZINES AS ATRAZINE by QUANTITATIVE IMMUNOASSAY	GEN-4670
HALOGENS TOTAL AS CHLORIDE BY BOMB COMBUSTION	GEN-5050
BIOCHEMICAL OXYGEN DEMAND	GEN-5210B
HALIDES, ADSORBABLE ORGANIC (AOX) - SM 5320B	GEN-5320B
DETERMINATION OF METHYLENE BLUE ACTIVE SUBSTANCES (MBAS)	GEN-5540C
TANNIN AND LIGNIN	GEN-5550
HALIDES, TOTAL ORGANIC (TOX)	GEN-9020
HALIDES, EXTRACTABLE ORGANIC (EOX)	GEN-9020M
TOTAL SULFIDES BY METHYLENE BLUE DETERMINATION	GEN-9030
TOTAL HALIDES BY OXIDATIVE COMBUSTION AND MICROCOULOMETRY	GEN-9076
CARBON, TOTAL ORGANIC IN SOIL	GEN-ASTM
AUTOFLUFF	GEN-AUTOFLU
SULFIDES, ACIDS VOLATILE	GEN-AVS
HEAT OF COMBUSTION	GEN-BTU
CHLOROPHYLL-a BY COLORIMETRY	GEN-CHLOR
TOTAL CYANIDES AND CYANIDES AMENABLE TO CHLORINATION	GEN-CN

Technical SOP - Kelso (CONT.)

CYANIDE, WEAK ACID DISSOCIABLE	GEN-CNWAD
CHEMICAL OXYGEN DEMAND	GEN-COD
CONDUCTIVITY IN WATER AND WASTES	GEN-COND
CORROSIVITY TOWARDS STEEL	GEN-CORR
HEXAVALENT CHROMIUM - COLORIMETRIC	GEN-CR6
STANDARD TEST METHODS FOR DETERMINING SEDIMENT CONCENTRATION IN WATER SAMPLES	GEN-D3977
CARBONATE (CO3) BY EVOLUTION AND COLUMETRIC TITRATION	GEN-D513-82M
SULFIDE, SOLUBLE DETERMINATION OF SOLUBLE SULFIDE IN SEDIMENT	GEN-DIS.S2
BULK DENSITY OF SOLID WASTE FRACTIONS	GEN-E1109
FDA EXTRACTABLES	GEN-FDAEX
FERROUS IRON IN WATER	GEN-FeII
FLUORIDE BY ION SELECTIVE ELECTRODE	GEN-FISE
FORMALDEHYDE COLORIMETRIC DETERMINATION	GEN-FORM
HYDROGEN HALIDES BY ION CHROMATOGTRAPHY (METHOD 26)	GEN-HA26
HYDAZINE IN WATER USING COLORIMETRIC PROCEDURE	GEN-HYD
TOTAL SULFUR FOR ION CHROMATOGRAPHY	GEN-ICS
ION CHROMATOGRAPHY	GEN-IONC
COLOR, NCASI	GEN-NCAS
NITROCELLULOSE IN SOIL	GEN-NCEL
OXYGEN CONSUMPTION RATE	GEN-O2RATE
CARBON, TOTAL ORGANIC DETERMINATION (WALKELY BLACK METHOD)	GEN-OSU
Ph IN SOIL AND SOLIDS	GEN-Phs
Ph IN WATER	GEN-Phw

Technical SOP - Kelso (CONT.)

PARTICLE SIZE DETERMINATION - ASTM PROCEDURE	GEN-PSASTM
PARTICLE SIZE DETERMINATION	GEN-PSP
SULFIDES, REACTIVE	GEN-RS
TOTAL SULFIDE BY PSEP	GEN-S2PS
SULFITE	GEN-SO3
SPECIFIC GRAVITY	GEN-SPGRAV
SUBSAMPLING AND COMPOSITING OF SAMPLES	GEN-SUBS
SOLIDS, TOTAL DISSOLVED (TDS)	GEN-TDS
THIOCYANATE	GEN-THIOCN
NITROGEN, TOTAL AND SOLUBLE KJELDAHL	GEN-TKN
TOTAL NITROGEN AND TOTAL PHOSPHORUS BY ALKALINE PERSULFATE DIGESTION NCASI METHOD TNTP-W10900	GEN-TNTP
TOTAL ORGANIC CARBON IN WATER	GEN-TOC
SOLIDS, TOTAL SUSPENDED (TSS)	GEN-TSS
TURBIDITY MEASUREMENT	GEN-TURB
ULTIMATE BOD	GEN-UBOD
GLASSWASHING FOR INORGANIC ANALYSES	GEN-WASH
PHARMACEUTICALS, PERSONAL CARE PRODUCTS AND ENDOCRINE DISRUPTING COMPOUNDS HPLC/TANDEM MASS SPECTROMETRY (HPLC/MS/MS)	LCP-1694
DETERMINATION OF TRIAZINE PESTICIDES AND THIER DEGRADATES IN WATER BY LIQUID CHROMATOGRAPHY ELECTROSPRAY IONIZATION TANDEM MASS SPECTROMETRY	LCP-536
ALDEHYDES BY HPLC	LCP-8315
Quantitative Determination of Carbamate Pesticides by High Performance Liquid Chromatography/Tandam Mass Spectrometry (HPLC/MS/MS)	LCP-8321
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY(HPLC)	LCP-8330B
Acrylamide by High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/ms/ms)..	LCP-ACRYL
QUANTITATIVE DETERMINATION OF AFLATOXINS By High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/ms/ms)	LCP-AFLA

Technical SOP - Kelso (CONT.)

Dioctyl sulfosuccinate by High Performance Liquid Chromatography/tandem mass spectrometry (HPLC/ms/ms)..	LCP-DOS
QUANTITATION OF NITROAROMATICS AND NITRAMINES IN WATER, SOIL, AND TISSUE BY LIQUID CHROMATOGRAPHY AND TANDEM MASS SPECTROMETRY (LC-MS/MS)	LCP-LCMS4
NITROGUANIDINE BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	LCP-NITG
QUANTITATION OF NITROPHENOLS IN SOILS BY LIQUID CHROMATOGRAPHYAND TANDEM MASS SPECTORMETRY (LC-MS/MS)	LCP-NITRO
ORGANIC ACIDS IN AQUEOUS MATRICES BY HPLC	LCP-OALC
QUANTITATIVE DETERMINATION OF OPTICAL BRIGHTENER 220 By High Performance Liquid Chromatography (HPLC)	LCP-OPBr
PERFLUORINATED COMPOUNDS BY HPLC/MS/MS	LCP-PFC
METHYL MERCURY IN SOIL AND SEDIMENT BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630S
METHYL MERCURY IN TISSUE BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630T
METHYL MERCURY IN WATER BY ATOMIC FLUORESCENCE SPECTROMETRY	MET-1630W
MERCURY IN WATER BY OXIDATION, PURGE TRAP, AND COLD VAPOR ATOMIC FLUORES. SPECTROMETRY	MET-1631
DETERMINATION OF ARSENIC SPECIES BY HYDRIDE GENERATION CRYOGENIC TRAPPING GAS CHROMATOGRAPY ATOMIC ABSORPTION SPECTROPOTOMETRY	MET-1632
MERCURY IN WATER	MET-245.1
METALS DIGESTION	MET-3010A
METALS DIGESTION	MET-3020A
METALS DIGESTION	MET-3050
CLOSED VESSEL OIL DIGESTION	MET-3051M
CLOSED VESSEL DIGESTION OF SILICEOUS AND ORGANICALLY BASED MATRICIES	MET-3052M
DETERMINATION OF METALS TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 6020)	MET-6020
ARSENIC BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7062
METALS DIGESTION FOR HEXAVALENT CHROMIUM	MET-7195
MERCURY IN LIQUID WASTE	MET-7470A
MERCURY IN SOLID OR SEMISOLID WASTE	MET-7471

Technical SOP - Kelso (CONT.)

SELENIUM BY BOROHYDRIDE REDUCTION ATOMIC ABSORPTION	MET-7742
SAMPLE PREPARATION OF AQUEOUS SAMPLES BY "CLEAN" TECHNIQUES	MET-ACT
BIOACCESSIBILITY OF METALS IN SOIL AND SOLID WASTE	MET-BIOACC
METALS DIGESTION	MET-DIG
SAMPLE FILTRATION FOR METALS ANALYSIS	MET-FILT
METALS LABORATORY GLASSWARE CLEANING	MET-GC
DETERMINATION OF TRACE METALS BY GRAPHITE FURNACE ATOMIC ABSORPTION SPECTROMETRY (GFAA)	MET-GFAA
DETERMINATION OF METALS AND TRACE ELEMENTS BY ICP/AES	MET-ICP
DETERMINATION OF METALS - TRACE ELEMENTS BY INDUCTIVELY COUPLED PLASMA-MS (METHOD 200.8)	MET-ICP.MS
TRACE METALS IN WATER BY PRECONCENTRATION USING REDUCTIVE PRECIPITATION FOLLOWED BY ICP-MS	MET-RPMS
METALS AND SEMIVOLATILES SPLP EXTRACTION (EPA METHOD 1312)	MET-SPLP
WASTE EXTRACTION TEST (WET) PROCEDURE (STLC) for NONVOLATILE and SEMIVOLATILE PARAMETERS	MET-STLC
METALS AND SEMIVOLATILES TCLP EXTRACTION (EPA METHOD 1311)	MET-TCLP
SAMPLE PREPARATION OF BIOLOGICAL TISSUES FOR METALS ANALYSIS BY GFAA, ICP-OES, AND ICP-MS	MET-TDIG
TISSUE SAMPLE PREPARATION	MET-TISP
ANALYSIS OF WATER AND SOLID SAMPLES FOR ALIPHATIC HYDROCARBONS	PET-ALIPHAT
GASOLINE RANGE ORGANICS BY GAS CHROMATOGRAPHY	PET-GRO
ANALYSIS OF WATER, SOLIDS AND SOLUBLE WASTE SAMPLES FOR SEMI-VOLATILE FUEL HYDROCARBONS	PET-SVF
ANALYSIS OF WATER AND SOLIDS SAMPLES FOR TOTAL PETROLEUM HYDROCARBONS	PET-TPH
ANALYSIS OF SOLID AND AQUEOUS SAMPLES FOR STATE OF WISCONSIN DIESEL RANGE ORGANICS	PHC-WIDRO

Technical SOP - Kelso (CONT.)

ORGANOCHLORINE PESTICIDES AND PCBs (METHOD 608)	SOC-608
GLYCOLS	SOC-8015M
ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY: CAPILLARY COLUMN TECHNIQUE	SOC-8081
PCBS AS AROCLORS	SOC-8082Ar
CONGENER-SPECIFIC DETERMINATION OF PCBS BY GC/ECD	SOC-8082Co
DETERMINATION OF NITROGEN OR PHOSPHORUS CONTAINING PESTICIDES	SOC-8141
CHLORINATED HERBICIDES	SOC-8151
CHLORINATED PHENOLS METHOD 8151 MODIFIED	SOC-8151M
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SOC-8270C
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - METHOD 8270D	SOC-8270D
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS - LOW LEVEL PROCEDURE	SOC-8270L
POLYNUCLEAR AROMATIC HYDROCARBONS BY HPLC	SOC-8310
NITROAROMATICS AND NITRAMINES BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY	SOC-8330
RESIN AND FATTY ACIDS BY GC/MS - NCASI METHOD 85.02 MODIFIED	SOC-85.02
METHANOL IN PROCESS LIQUIDS AND STATIONARY SOURCE EMISSIONS	SOC-9403
HAZARDOUS AIR POLLUTANTS (HAPS) IN PULP AND PAPER INDUSTRY CONDENSATES	SOC-9901
HAPS AND OTHER COMPOUNDS IN IMPINGER/CANISTER SAMPLES FROM WOOD PRODUCTS FACILITIES	SOC-9902
ALCOHOLS	SOC-ALC
BUTYLTINS	SOC-BUTYL
CALIBRATION OF INSTRUMENTS FOR ORGANICS CHROMATOGRAPHIC ANALYSES	SOC-CAL
CONFIRMATION PROCEDURE FOR GC AND HPLC ANALYSES	SOC-CONF
CPSC PHTHALATES BY GC/MS SELECTIVE ION MONITORING	SOC-CPSC

Technical SOP - Kelso (CONT.)

DIMP	SOC-DIMP
TOTAL OLEANOLIC ACID SAPONINS IN WATER BY ACID HYDROLYSIS AND HPLC/MS/MS	SOC-LCMS3
MONOCHLOROACETIC ACID BY GC-ECD	SOC-MCA
NONYLPHENOLS ISOMERS AND NONYLPHENOL ETHOXYLATES	SOC-NONYL
ORGANOPHOSPHOROUS PESTICIDES BY GC/MS/MS	SOC-OPPMS2
DETERMINATION OF OTTO FUEL II IN WATER	SOC-OTTO
PICRIC ACID AND PICRAMIC ACID BY HPLC	SOC-PICRIC
POLYBROMINATED DIPHENYL ETHERS (PBDEs) AND POLYBROMINATED BIPHENYLS (PBBs) BY GC/MS	SOC-ROHS
SEMI-VOLATILE ORGANICS SCREENING	SOC-SCR
1,2-DIBROMOETHANE, 1,2-DIBROMO-3-CHLOROPROPANE, AND 1,2,3-TCP BY GC	SVD-504
ORGANOCHLORINE PESTICIDES AND PCBS IN DRINKING WATER	SVD-508_1
CHLORINATED HEBICIDES IN DRINKING WATER	SVD-515.4
N-NITROSAMINES BY GC/MS/MS	SVD-521
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS (METHOD 525.2)	SVD-525
SELECTED PESTICIDES AND FLAME RETARDANTS IN DRINKING WATER BY GC/MS (EPA METHOD 527)	SVD-527
CARBAMATES AND CARBAMOYLOXIMES IN WATER BY POST-COLUMN DERIVITIZATION HPLC	SVD-531 -1
GLYPHOSATE IN DRINKING WATER BY HPLC	SVD-547
ENDOTHALL IN DRINKING WATER BY GC/MS	SVD-548
DIQUAT AND PARAQUAT BY HPLC	SVD-549
HALOACETIC ACIDS IN DRINKING WATER	SVD-552

Technical SOP - Kelso (CONT.)

CHLORINATED PHENOLICS BY IN-SITU ACETYLATION AND GC/MS	SVM-1653A
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS	SVM-625
POLYNUCLEAR AROMATIC HYDROCARBONS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY SIM	SVM-8270P
SEMIVOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTED ION MONITORING	SVM-8270S
CHLORINATED PESTICIDES BY GC/MS/MS, EPA METHOD 1699 MODIFIED	SVM-PESTMS2
PURGE AND TRAP FOR AQUEOUS SAMPLES	VOC-5030
PURGE AND TRAP/EXTRACTION FOR VOC IN SOIL AND WASTE SAMPLES , CLOSED SYSTEM	VOC-5035
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-524.2
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-624
AROMATIC VOLATILE ORGANICS (BTEX) BY GC - METHOD 8021	VOC-8021BTEX
VOLATILE ORGANIC COMPOUNDS BY GC/MS	VOC-8260
VOLATILE ORGANIC COMPOUNDS BY GC/MS SELECTIVE ION MONITORING	VOC-8260S
VOA STORAGE BLANKS	VOC-BLAN
SAMPLE SCREENING FOR VOLATILE ORGANIC COMPOUNDS IN SOIL, WATER AND MISC. MATRICES	VOC-BVOC
ZERO HEADSPACE EXTRACTION (EPA METHOD 1311)	VOC-ZHE

APPENDIX G

List of Laboratory Certifications and Accreditations

Federal and National Programs

- The TNI (The NELAC Institute) National Environmental Laboratory Accreditation Program (NELAP) Accredited Drinking Water, Non-Potable Water, Solid Hazardous Waste, and Biological Tissue Laboratory
- ANSI-ASQ National Accreditation Board/ACLASS ISO 17025:2005
- DoD- ELAP Environmental Laboratory Accreditation Program
- U.S. EPA Region 8
  Approved Drinking Water Laboratory

State and Local Programs

- State of Alaska, Department of Environmental Conservation
  UST Laboratory, Lab I.D. UST040
- State of Arizona, Department of Health Services
  License No. AZ0339
- State of Arkansas, Department of Environmental Quality
  Certified Environmental Laboratory, Lab I.D. 88-0637
- State of California, Department of Health Services, Environmental Laboratory Accreditation Program
  Certification No. 2286
- State of Florida, Department of Health
  Accredited Environmental Laboratory No. E87412
- State of Georgia, Department of Natural Resources
  Certified Drinking Water Laboratory
- State of Hawaii, Department of Health
  Certified Drinking Water Laboratory
- State of Idaho, Department of Health and Welfare
  Certified Drinking Water Laboratory
- State of Indiana, Department of Health
  Certified Drinking Water Laboratory, Lab I.D. C-WA-01
- State of Louisiana, Department of Environmental Quality
  Accredited Environmental Laboratory, Lab I.D. 3016
- State of Louisiana, Department of Health and Hospitals
  Accredited Drinking Water Laboratory, Lab I.D. LA080001
- State of Maine, Department of Human Services
  Certified Environmental Laboratory, Lab I.D. WA0035
- State of Michigan, Department of Environmental Quality
  Certified Drinking Water Laboratory, Lab I.D. 9949

State and Local Programs (continued)

- State of Minnesota, Department of Health
  Certified Environmental Laboratory, Lab I.D. 053-999-368
- State of Montana, Department of Health and Environmental Sciences
  Certified Drinking Water Laboratory, Lab I.D. 0047
- State of Nevada, Division of Environmental Protection
  Certified Drinking Water Laboratory, Lab I.D. WA35
- State of New Jersey, Department of Environmental Protection
  Accredited Environmental Laboratory, Lab I.D. WA005
- State of New Mexico, Environment Department
  Certified Drinking Water Laboratory
- State of North Carolina, Department of Environment and Natural Resources
  Certified Environmental Laboratory, Lab I.D. 605
- State of Oklahoma, Department of Environmental Quality
  General Water Quality/Sludge Testing, Lab I.D. 9801
- State of Oregon, ORELAP Laboratory Accreditation Program
  Accredited Environmental Laboratory, Lab I.D. WA200001
- State of South Carolina, Department of Health and Environmental Control
  Certified Environmental Laboratory, Lab I.D. 61002
- State of Washington, Department of Ecology
  Accreditation Program Lab I.D. C1203
- State of Wisconsin, Department of Natural Resources
  Accredited Environmental Laboratory, Lab I.D. 998386840

A complete listing of and certifications and accreditations can be found at:

http://www.caslab.com/Certifications/

ALS Environmental – SIMI VALLEY, CALIFORNIA

Summary of Calibration and Internal Quality Control Procedures for U.S. EPA Method TO-15			
Quality Control Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**
BFB Tuning Verification	Once every 24-hours or analytical batch	Ion abundance criteria as described in Table 3 of Method TO-15	1) Repeat BFB analysis 2) Retune instrument
Initial Calibration (ICAL) – minimum of five levels	Initially or if continuing calibration no longer meets criteria	1) <30% RSD with 2 exceptions up to 40% [AFCEE: only $\leq$ 30% RSD] 2) Area response at each calibration level within 40% of IS mean area response over the ICAL range. 3) Retention time for each IS within 20s of the mean retention time over the ICAL range.	1) May repeat 1 point (if 5 levels) or 2 points (if 6 levels) 2) Inspect the system for problems and perform required maintenance 3) Repeat initial calibration Problem must be corrected. Samples may not be analyzed until there is a valid ICAL.
Initial Calibration Verification (ICV)	Following every ICAL	Percent difference of +/-30%	Correct problem and verify second source standard. Rerun second source verification. If that fails, correct problem and repeat initial calibration. Problem must be corrected. Samples may not be analyzed until there is a valid ICV.
Continuing Calibration Verification (CCV)	Once every 24 hours, if an ICAL has not been performed (within the last 24 hours).	Percent difference of +/-30% Note: If CCV is biased high and analyte is ND (not detected) results are acceptable. It will be noted in case narrative	1) Reanalyze CCV 2) Identify and correct problem; re-analyze or if necessary qualify the data. 3) Repeat initial calibration if CCV corrective action is unsuccessful.
Internal Standards (IS)	All samples, duplicates, blanks and standards	1) RT must be <20 sec from most recent valid calibration (ICAL midpoint or CCV) 2) Area response +/-40% of IS area response of most recent valid calibration (ICAL midpoint or CCV)	1) Identify and correct the problem 2) Reanalyze the sample unless obvious matrix interference exists. 3) Problem persists, qualify data.
Surrogate Standards	All samples, duplicates, blanks and standards	70-130% recovery [AFCEE: 60-140%]	1) Identify and correct the problem 2) Reanalyze the sample unless obvious matrix interference exists 3) Problem persists, qualify data.
Laboratory Method Blank (MB)	Once every analytical batch of 20 or fewer samples	No analyte detected equal to or above the method reporting limit (MRL) [DoD: No analytes > ½ MRL; common lab contaminants none detected > MRL]	1) Reanalyze blank 2) Identify and correct problem 3) Reanalyze blank and affected samples 4) Qualify data

ALS Environmental – SIMI VALLEY, CALIFORNIA

Summary of Calibration and Internal Quality Control Procedures for U.S. EPA Method TO-15			
Quality Control Check	**Minimum Frequency**	**Acceptance Criteria**	**Corrective Action**
Laboratory Control Sample (LCS)	Once every analytical batch of 20 or fewer samples	Percent recovery (%R) within laboratory generated limits [AFCEE, AZ: 70-130% recovery]	1) Reanalyze 2) Identify and correct the problem 3) Qualify data *DoD projects require corrective action for all exceedances.
Laboratory Duplicate (LD)	Once every analytical batch of 20 or fewer samples	Relative percent difference (RPD) within +/-25% for positive hits	1) Analyze third aliquot 2) Flag data if third aliquot unacceptable
Holding Time (HT)	N/A	SUMMA Canisters - 30 days [EPA Region 9 - 14 days] Tedlar Bags - 72 hours [not included in AFCEE Manual]	Contact client and qualify data
Method Reporting Limit (MRL)	DoD: Quarterly LOQ/MRL Verification Required	At or above the low standard of the current initial calibration AFCEE: Minimum 2x method detection limit	N/A
Method Detection Limit (MDL) with Limit of Detection Verification	Initially and once per 12 month period DoD: Quarterly LOD Verification Required	Limit of Detection Verification - Response with a minimum signal to noise ratio of 3:1	N/A
Report results between MDL and MRL	N/A	Upon request [Required for AFCEE and DoD projects; verify with Client QAPP]	Qualify results as estimated

AFCEE – Air Force Center for Environmental Excellence, Appendix C: QAPP, Final Version 4.0.02, May 2006.
DoD – Department of Defense Quality Systems Manual for Environmental Laboratories, Final Version 4.1, April 2009.
AZ – Requirements for State of Arizona compliance samples.
Summary complies with Method TO-15 and 2003 NELAC Standard.

Appendix B-2
Method Detection, Quantitation and Reporting Limits

Attachment B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edibles Oils
Portland, Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

									Accuracy	Matrix Spike	Precision
METHOD	**ANALYTE**	**CAS No.**	**MATRIX**	**MDLa**	**MRL**	**LODb**	**LOQc**	**UNITS**	**(LCS %Rec.)**	**(%Rec.)**	**(% RPD)**
8260C	1,1,1,2-Tetrachloroethane	630-20-6	Water	0.047	0.5	0.2	0.5	ug/L	66-124	67-127	30
8260C	1,1,1-Trichloroethane (TCA)	71-55-6	Water	0.050	0.5	0.1	0.5	ug/L	59-136	57-151	30
8260C	1,1,2,2-Tetrachloroethane	79-34-5	Water	0.064	0.5	0.2	0.5	ug/L	70-127	72-129	30
8260C	1,1,2-Trichloroethane	79-00-5	Water	0.061	0.5	0.3	0.5	ug/L	74-118	74-124	30
8260C	1,1-Dichloroethane	75-34-3	Water	0.042	0.5	0.15	0.5	ug/L	66-129	69-141	30
8260C	1,1-Dichloroethene	75-35-4	Water	0.10	0.5	0.2	0.5	ug/L	66-129	59-171	30
8260C	1,1-Dichloropropene	563-58-6	Water	0.051	0.5	0.1	0.5	ug/L	59-134	61-148	30
8260C	1,2,3-Trichlorobenzene	87-61-6	Water	0.10	2	0.2	2	ug/L	68-120	57-137	30
8260C	1,2,3-Trichloropropane	96-18-4	Water	0.14	0.5	0.4	0.5	ug/L	69-123	71-127	30
8260C	1,2,4-Trichlorobenzene	120-82-1	Water	0.13	2	0.2	2	ug/L	58-126	57-133	30
8260C	1,2,4-Trimethylbenzene	95-63-6	Water	0.037	2	0.2	2	ug/L	63-122	61-132	30
8260C	1,2-Dibromo-3-chloropropane	96-12-8	Water	0.22	2	0.5	2	ug/L	55-132	59-133	30
8260C	1,2-Dibromoethane (EDB)	106-93-4	Water	0.084	2	0.2	2	ug/L	74-118	73-122	30
8260C	1,2-Dichlorobenzene	95-50-1	Water	0.044	0.5	0.2	0.5	ug/L	72-115	72-119	30
8260C	1,2-Dichloroethane (EDC)	107-06-2	Water	0.073	0.5	0.2	0.5	ug/L	56-142	56-141	30
8260C	1,2-Dichloropropane	78-87-5	Water	0.042	0.5	0.2	0.5	ug/L	67-126	63-131	30
8260C	1,3,5-Trichlorobenzene	108-70-3	Water	0.10	5	0.2	5	ug/L	63-118	58-118	30
8260C	1,3,5-Trimethylbenzene	108-67-8	Water	0.042	2	0.2	2	ug/L	62-126	60-136	30
8260C	1,3-Dichlorobenzene	541-73-1	Water	0.041	0.5	0.2	0.5	ug/L	70-116	70-121	30
8260C	1,3-Dichloropropane	142-28-9	Water	0.032	0.5	0.2	0.5	ug/L	75-116	74-121	30
8260C	1,4-Dichlorobenzene	106-46-7	Water	0.054	0.5	0.2	0.5	ug/L	73-115	72-121	30
8260C	1,4-Dioxane	123-91-1	Water	13	100	40	100	ug/L	67-160	64-145	30
8260C	1-Chlorohexane	544-10-5	Water	0.057	0.5	0.1	0.5	ug/L	50-118	50-118	30
8260C	2,2-Dichloropropane	594-20-7	Water	0.050	0.5	0.2	0.5	ug/L	37-145	39-161	30
8260C	2-Butanone (MEK)	78-93-3	Water	3.8	20	4	20	ug/L	71-149	65-147	30
8260C	2-Chloroethyl Vinyl Ether	110-75-8	Water	0.19	5	0.2	5	ug/L	61-126	10-150	30
8260C	2-Chlorotoluene	95-49-8	Water	0.035	2	2.7	2	ug/L	55-131	55-139	30
8260C	2-Hexanone	591-78-6	Water	2.9	20	10	20	ug/L	59-131	53-132	30
8260C	2-Nitropropane	79-46-9	Water	0.91	5	10	5	ug/L	10-160	10-160	30
8260C	3-Chloro-1-propene	107-05-1	Water	0.19	5	0.2	5	ug/L	42-147	70-151	30
8260C	4-Chlorotoluene	106-43-4	Water	0.025	2	0.13	2	ug/L	66-121	57-138	30
8260C	4-Isopropyltoluene	99-87-6	Water	0.044	2	0.51	2	ug/L	61-128	57-141	30
8260C	4-Methyl-2-pentanone (MIBK)	108-10-1	Water	3.0	20	2.6	20	ug/L	64-134	64-139	30
8260C	Acetone	67-64-1	Water	2.5	20	10	20	ug/L	68-135	68-134	30
8260C	Acetonitrile	75-05-8	Water	7.3	50	20	50	ug/L	69-132	77-127	30
8260C	Acrolein	107-02-8	Water	2.0	20	2	20	ug/L	42-118	14-180	30
8260C	Acrylonitrile	107-13-1	Water	0.31	5	4	5	ug/L	65-129	73-131	30
8260C	Benzene	71-43-2	Water	0.045	0.5	0.1	0.5	ug/L	69-124	63-144	30
8260C	Bromobenzene	108-86-1	Water	0.027	2	0.2	2	ug/L	72-116	72-122	30
8260C	Bromochloromethane	74-97-5	Water	0.091	0.5	0.2	0.5	ug/L	75-131	73-135	30
8260C	Bromodichloromethane	75-27-4	Water	0.036	0.5	0.2	0.5	ug/L	63-129	61-134	30
8260C	Bromoform	75-25-2	Water	0.080	0.5	0.2	0.5	ug/L	52-144	54-140	30
8260C	Bromomethane	74-83-9	Water	0.072	0.5	0.2	0.5	ug/L	35-113	36-127	30
8260C	Carbon Disulfide	75-15-0	Water	0.045	0.5	0.2	0.5	ug/L	46-144	52-156	30
8260C	Carbon Tetrachloride	56-23-5	Water	0.068	0.5	0.2	0.5	ug/L	55-140	53-161	30
8260C	Chlorobenzene	108-90-7	Water	0.045	0.5	0.2	0.5	ug/L	72-116	69-126	30
8260C	Chloroethane	75-00-3	Water	0.13	0.5	0.2	0.5	ug/L	58-134	56-147	30
8260C	Chloroform	67-66-3	Water	0.042	0.5	0.1	0.5	ug/L	70-129	64-133	30
8260C	Chloromethane	74-87-3	Water	0.053	0.5	0.1	0.5	ug/L	34-130	49-127	30
8260C	Chloroprene	126-99-8	Water	0.15	10	4	10	ug/L	43-146	67-126	30
8260C	cis-1,2-Dichloroethene	156-59-2	Water	0.045	0.5	0.2	0.5	ug/L	71-118	61-139	30
8260C	cis-1,3-Dichloropropene	10061-01-5	Water	0.038	0.5	0.2	0.5	ug/L	62-132	66-134	30
8260C	cis-1,4-Dichloro-2-butene	1476-11-5	Water	0.84	10	4	10	ug/L	26-171	49-172	30
8260C	Cyclohexane	110-82-7	Water	0.36	1			ug/L	70-130	70-130	30
8260C	Dibromochloromethane	124-48-1	Water	0.057	0.5	0.5	0.5	ug/L	67-126	68-125	30
8260C	Dibromomethane	74-95-3	Water	0.089	0.5	0.5	0.5	ug/L	69-128	68-132	30
8260C	Dichlorodifluoromethane	75-71-8	Water	0.083	0.5	0.2	0.5	ug/L	32-124	29-133	30
8260C	Dichlorofluoromethane (CFC 21)	75-43-4	Water	0.053	0.5	0.2	0.5	ug/L	54-140	79-135	30
8260C	Diisopropyl Ether	108-20-3	Water	0.046	2	0.1	2	ug/L	62-123	50-134	30
8260C	Ethyl Acetate	141-78-6	Water	0.81	5			ug/L	59-117	70-130	30
8260C	Ethyl Ether	60-29-7	Water	0.069	1	0.1	1	ug/L	54-137	31-141	30
8260C	Ethyl Methacrylate	97-63-2	Water	0.11	5	0.2	5	ug/L	48-143	63-134	30
8260C	Ethylbenzene	100-41-4	Water	0.042	0.5	0.1	0.5	ug/L	67-121	66-136	30
8260C	Ethylene Oxide	75-21-8	Water	1.1	10			ug/L	70-130	70-130	30
8260C	Hexachlorobutadiene	87-68-3	Water	0.19	2	0.2	2	ug/L	57-119	60-132	30
8260C	Iodomethane	74-88-4	Water	0.27	5	0.4	5	ug/L	51-164	65-155	30
8260C	Isobutyl Alcohol	78-83-1	Water	12	100	20	100	ug/L	36-142	27-182	30
8260C	Isopropylbenzene	98-82-8	Water	0.031	2	0.2	2	ug/L	67-129	58-144	30
8260C	m,p-Xylenes	179601-23-1	Water	0.078	0.5	0.1	0.5	ug/L	69-121	67-135	30
8260C	Methacrylonitrile	126-98-7	Water	0.25	5	0.8	5	ug/L	47-136	68-129	30
8260C	Methyl Acetate	79-20-9	Water	0.38	1			ug/L	70-130	70-130	30
8260C	Methyl Methacrylate	80-62-6	Water	0.18	5	0.5	5	ug/L	46-138	61-143	30
8260C	Methyl tert-Butyl Ether	1634-04-4	Water	0.070	0.5	0.2	0.5	ug/L	54-126	54-126	30

Attachment B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edibles Oils
Portland, Oregon

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	(LCS %Rec.)	(%Rec.)	(% RPD)
8260C	Methylcyclohexane	108-87-2	Water	0.33	1	0.2	1	ug/L	70-130	65-154	30
8260C	Methylcyclopentane	96-37-7	Water					ug/L		65-154	30
8260C	Methylene Chloride	75-09-2	Water	0.23	2	0.2	2	ug/L	71-122	70-133	30
8260C	Naphthalene	91-20-3	Water	0.10	2	0.2	2	ug/L	64-126	52-147	30
8260C	n-Butylbenzene	104-51-8	Water	0.056	2	0.5	2	ug/L	55-130	52-144	30
8260C	n-Heptane	142-82-5	Water	0.28	2			ug/L		99-162	
8260C	n-Hexane	110-54-3	Water	0.29	1	0.5	1	ug/L	54-160	54-160	30
8260C	n-Octane	111-65-9	Water	0.33	5	0.5	5	ug/L	64-138	43-157	30
8260C	n-Propylbenzene	103-65-1	Water	0.037	2	1	2	ug/L	61-124	55-144	30
8260C	o-Xylene	95-47-6	Water	0.037	0.5	0.1	0.5	ug/L	71-119	67-127	30
8260C	Propionitrile	107-12-0	Water	1.1	5	4	5	ug/L	46-137	72-122	30
8260C	sec-Butylbenzene	135-98-8	Water	0.036	2	0.1	2	ug/L	59-128	56-142	30
8260C	Styrene	100-42-5	Water	0.039	0.5	0.2	0.5	ug/L	74-121	66-131	30
8260C	tert-Amyl Methyl Ether	994-05-8	Water	0.078	2	0.2	2	ug/L	58-138	47-162	30
8260C	tert-Butyl Alcohol	75-65-0	Water	4	20	10	20	ug/L	38-138	49-142	30
8260C	tert-Butyl Ethyl Ether	637-92-3	Water	0.027	2	0.1	2	ug/L	60-123	52-130	30
8260C	tert-Butylbenzene	98-06-6	Water	0.038	2	0.1	2	ug/L	61-127	59-139	30
8260C	Tetrachloroethene (PCE)	127-18-4	Water	0.077	0.5	0.2	0.5	ug/L	62-126	61-131	30
8260C	Tetrahydrofuran	109-99-9	Water	0.89	5	2.5	5	ug/L	31-157	71-153	30
8260C	Toluene	108-88-3	Water	0.048	0.5	0.1	0.5	ug/L	69-124	71-136	30
8260C	trans-1,2-Dichloroethene	156-60-5	Water	0.048	0.5	0.2	0.5	ug/L	67-125	65-143	30
8260C	trans-1,3-Dichloropropene	10061-02-6	Water	0.041	0.5	0.2	0.5	ug/L	59-125	56-127	30
8260C	trans-1,4-Dichloro-2-butene	110-57-6	Water	0.20	10	1	10	ug/L	46-170	63-157	30
8260C	Trichloroethene (TCE)	79-01-6	Water	0.061	0.5	0.2	0.5	ug/L	67-128	53-139	30
8260C	Trichlorofluoromethane	75-69-4	Water	0.086	0.5	0.2	0.5	ug/L	52-141	45-124	30
8260C	Trichlorotrifluoroethane	76-13-1	Water	0.079	0.5	0.5	0.5	ug/L	56-141	56-161	30
8260C	Vinyl Acetate	108-05-4	Water	0.91	5	1	5	ug/L	44-156	69-148	30
8260C	Vinyl Chloride	75-01-4	Water	0.071	0.5	0.2	0.5	ug/L	55-123	49-136	30
8260C	1,2-Dichloroethane-D4 (Surr.)	17060-07-0	Water	NA	NA	NA	NA	%	59-127	NA	NA
8260C	4-Bromofluorobenzene (Surr.)	460-00-4	Water	NA	NA	NA	NA	%	68-117	NA	NA
8260C	Dibromofluoromethane (Surr.)	1868-53-7	Water	NA	NA	NA	NA	%	73-122	NA	NA
8260C	Toluene-D8 (Surr.)	2037-26-5	Water	NA	NA	NA	NA	%	78-129	NA	NA

CAS - Columbia Analytical Services
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Verification is acceptable if the response is > 3x instrument noise & ion abundance
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CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
8270D SIM PAH LL	1-Methylnaphthalene	90-12-0	Water	0.37	3.4	0.6	3.4	ng/L	50-169	48-134	30
8270D SIM PAH LL	1-Methylphenanthrene	832-69-9	Water	0.72	3.4	0.72	3.4	ng/L	61-128	61-128	30
8270D SIM PAH LL	2,3,5-Trimethylnaphthalene	2245-38-7	Water	0.3	3.4	0.6	3.4	ng/L	45-174	45-174	30
8270D SIM PAH LL	2,6-Dimethylnaphthalene	581-42-0	Water	0.24	3.4	0.6	3.4	ng/L	45-174	45-174	30
8270D SIM PAH LL	2-Methylnaphthalene	91-57-6	Water	0.4	3.4	0.6	3.4	ng/L	34-176	34-112	30
8270D SIM PAH LL	Acenaphthene	83-32-9	Water	0.36	3.4	0.6	3.4	ng/L	50-159	40-124	30
8270D SIM PAH LL	Acenaphthylene	208-96-8	Water	0.37	3.4	0.6	3.4	ng/L	51-159	46-120	30
8270D SIM PAH LL	Anthracene	120-12-7	Water	0.29	3.4	0.6	3.4	ng/L	53-151	55-119	30
8270D SIM PAH LL	Benz(a)anthracene	56-55-3	Water	0.34	3.4	0.6	3.4	ng/L	53-140	58-119	30
8270D SIM PAH LL	Benzo(a)pyrene	50-32-8	Water	0.41	3.4	0.6	3.4	ng/L	52-146	48-129	30
8270D SIM PAH LL	Benzo(b)fluoranthene	205-99-2	Water	0.25	3.4	0.6	3.4	ng/L	57-142	56-125	30
8270D SIM PAH LL	Benzo(e)pyrene	192-97-2	Water	0.49	3.4	0.6	3.4	ng/L	60-145	42-153	30
8270D SIM PAH LL	Benzo(g,h,i)perylene	191-24-2	Water	0.36	3.4	0.6	3.4	ng/L	50-144	58-118	30
8270D SIM PAH LL	Benzo(k)fluoranthene	207-08-9	Water	0.41	3.4	0.6	3.4	ng/L	55-144	60-129	30
8270D SIM PAH LL	Benzo[j,k]fluoranthene	CASID30895	Water	0.41	3.4	0.6	3.4	ng/L	55-144	60-129	30
8270D SIM PAH LL	Biphenyl	92-52-4	Water	0.39	3	0.6	3	ng/L	51-168	45-129	30
8270D SIM PAH LL	Carbazole	86-74-8	Water	0.39	3.4	0.6	3.4	ng/L	57-155	60-140	30
8270D SIM PAH LL	Chrysene	218-01-9	Water	0.65	3.4	0.65	3.4	ng/L	54-143	61-121	30
8270D SIM PAH LL	Dibenz(a,h)anthracene	53-70-3	Water	0.45	3.4	0.6	3.4	ng/L	46-148	49-127	30
8270D SIM PAH LL	Dibenzofuran	132-64-9	Water	0.42	3.4	0.6	3.4	ng/L	52-159	51-128	30
8270D SIM PAH LL	Dibenzothiophene	132-65-0	Water	0.52	3.4	0.6	3.4	ng/L	56-147	56-147	30
8270D SIM PAH LL	Fluoranthene	206-44-0	Water	0.46	3.4	0.6	3.4	ng/L	58-158	56-130	30
8270D SIM PAH LL	Fluorene	86-73-7	Water	0.42	3.4	0.6	3.4	ng/L	53-162	51-124	30
8270D SIM PAH LL	Indeno(1,2,3-cd)pyrene	193-39-5	Water	0.44	3.4	0.6	3.4	ng/L	45-149	44-126	30
8270D SIM PAH LL	Naphthalene	91-20-3	Water	0.71	3.4	0.71	3.4	ng/L	43-169	33-125	30
8270D SIM PAH LL	Perylene	198-55-0	Water	0.36	3.4	0.6	3.4	ng/L	54-144	46-141	30
8270D SIM PAH LL	Phenanthrene	85-01-8	Water	0.72	3.4	0.72	3.4	ng/L	56-144	59-119	30
8270D SIM PAH LL	Pyrene	129-00-0	Water	0.78	3.4	0.78	3.4	ng/L	57-157	54-129	30

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CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	(LCS %Rec.)	(%Rec.)	(% RPD)
NWTPH-Dx	Diesel Range Organics	68334-30-5	Water	8.2	250	20	250	ug/L	44-143	44-143	30
NWTPH-Dx	Residual Range Organics	NA	Water	19	500	50	500	ug/L	55-139	45-140	30
NWTPH-Dx	o-Terphenyl (Surr.)	84-15-1	Water	NA	NA	NA	NA	%	50-150	NA	NA
NWTPH-Dx	n-Triacontane (Surr.)	638-68-6	Water	NA	NA			%	54-136		

CAS/KELSO DATA QUALITY OBJECTIVES

									Accuracy	Matrix Spike	Precision
METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	(LCS %Rec.)	(%Rec.)	(% RPD)
NWTPH-Gx	Gasoline Range Organics	8006-61-9	Water	13	250	25	50	ug/L	77-122	71-128	30
NWTPH-Gx	4-Bromofluorobenzene (Surr.)	460-00-4	Water	NA	NA	NA	NA	%	50-150	NA	NA

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
SM2320 B	Alkalinity as CaCO3	NA	Water	3	9	6	9	mg/L	90-110	NA	20
300.0	Chloride	16887-00-6	Water	0.03	0.2	0.03	0.2	mg/L	90-110	90-110	20
SM4500 CN-E	Cyanide, Total	57-12-5	Water	0.003	0.01	0.009	0.01	mg/L	84-115	23-148	20
7196A	Hexavalent Chromium	18540-29-9	Water	0.004	0.05	0.012	0.05	mg/L	80-120	85-115	20
300.0	Nitrate as Nitrogen	14797-55-8	Water	0.004	0.1	0.02	0.1	mg/L	90-110	90-110	20
314	Perchlorate	14797-73-0	Water	0.4	1	1	3	ug/L	85-115	80-120	20
300.0	Sulfate	14808-79-8	Water	0.01	0.2	0.02	0.2	mg/L	90-110	90-110	20

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Attachment B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edible Oils
Portland, Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
6010C LL	Aluminum	7429-90-5	Water	0.5	2	6	18	ug/L	92-112	75-125	20

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
6020A	Antimony	7440-36-0	Water	0.02	0.05	0.013	0.05	ug/L	91-112	75-125	20
6020A	Arsenic	7440-38-2	Water	0.1	0.5	0.13	0.5	ug/L	89-112	75-125	20
6020A	Cadmium	7440-43-9	Water	0.005	0.02	0.01	0.03	ug/L	92-111	75-125	20
6020A	Chromium	7440-47-3	Water	0.04	0.2	0.05	0.2	ug/L	88-113	75-125	20
6020A	Cobalt	7440-48-4	Water	0.006	0.02	0.01	0.03	ug/L	87-114	75-125	20
6020A	Copper	7440-50-8	Water	0.02	0.1	0.025	0.1	ug/L	89-113	75-125	20
6020A	Manganese	7439-96-5	Water	0.006	0.05	0.013	0.05	ug/L	89-115	75-125	20
6020A	Nickel	7440-02-0	Water	0.03	0.2	0.05	0.2	ug/L	89-113	75-125	20
6020A	Selenium	7782-49-2	Water	0.3	1	0.5	1.5	ug/L	87-115	75-125	20
6020A	Zinc	7440-66-6	Water	0.2	0.5	0.25	0.75	ug/L	86-119	75-125	20

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
7470A	Mercury	7439-97-6	Water	0.02	0.2	0.05	0.2	ug/L	83-117	75-125	20

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	LODb	LOQc	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
1632	Total Inorganic Arsenic [TIA]	7440-38-2	Water	0.003	0.02	0.01	0.02	ug/L	80-120	50-150	35
1632	Monomethylarsonic acid [MMA]	7440-38-2	Water	0.002	0.02	0.01	0.02	ug/L	80-120	60-140	25
1632	Dimethylarsinic acid [DMA]	7440-38-2	Water	0.006	0.05	0.01	0.05	ug/L	70-130	60-140	40
1632	Arsenite [As(III)]	7440-38-2	Water	0.003	0.02	0.005	0.02	ug/L	70-130	30-170	35

Notes:
* (pH 6.5–9.0)

Attachment B-2
Method Detection , Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edible Oils
Portland, Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

				Accuracy	Matrix Spike	Precision
METHOD	**ANALYTE**	**MATRIX**	**UNITS**	**(LCS %Rec.)**	**(%Rec.)**	**(% RPD)**
8260C	1,1,1,2-Tetrachloroethane	Soil	ug/kg	71-119	16-131	40
8260C	1,1,1-Trichloroethane (TCA)	Soil	ug/kg	59-146	26-144	40
8260C	1,1,2,2-Tetrachloroethane	Soil	ug/kg	60-128	10-153	40
8260C	1,1,2-Trichloroethane	Soil	ug/kg	72-118	35-130	40
8260C	1,1-Dichloroethane	Soil	ug/kg	59-137	31-135	40
8260C	1,1-Dichloroethene	Soil	ug/kg	64-152	31-153	40
8260C	1,1-Dichloropropene	Soil	ug/kg	62-142	25-143	40
8260C	1,2,3-Trichlorobenzene	Soil	ug/kg	52-138	10-118	40
8260C	1,2,3-Trichloropropane	Soil	ug/kg	53-134	23-149	40
8260C	1,2,4-Trichlorobenzene	Soil	ug/kg	65-132	10-121	40
8260C	1,2,4-Trimethylbenzene	Soil	ug/kg	55-140	10-142	40
8260C	1,2-Dibromo-3-chloropropane	Soil	ug/kg	55-127	10-146	40
8260C	1,2-Dibromoethane (EDB)	Soil	ug/kg	71-116	26-131	40
8260C	1,2-Dichlorobenzene	Soil	ug/kg	67-124	10-124	40
8260C	1,2-Dichloroethane (EDC)	Soil	ug/kg	65-121	32-134	40
8260C	1,2-Dichloropropane	Soil	ug/kg	71-121	31-132	40
8260C	1,3,5-Trimethylbenzene	Soil	ug/kg	66-132	10-160	40
8260C	1,3-Dichlorobenzene	Soil	ug/kg	69-128	10-126	40
8260C	1,3-Dichloropropane	Soil	ug/kg	72-118	32-133	40
8260C	1,4-Dichlorobenzene	Soil	ug/kg	69-125	10-123	40
8260C	1,4-Dioxane	Soil	ug/kg	10-172	10-172	40
8260C	1-Chlorohexane	Soil	ug/kg	62-136	62-136	40
8260C	2,2-Dichloropropane	Soil	ug/kg	50-138	34-140	40
8260C	2-Butanone (MEK)	Soil	ug/kg	54-116	27-113	40
8260C	2-Chloroethyl Vinyl Ether	Soil	ug/kg	63-130	10-139	40
8260C	2-Chlorotoluene	Soil	ug/kg	66-129	10-140	40
8260C	2-Hexanone	Soil	ug/kg	67-121	15-162	40
8260C	2-Nitropropane	Soil	ug/kg	10-142	39-128	40
8260C	3-chloro-1-propene	Soil	ug/kg			
8260C	4-Chlorotoluene	Soil	ug/kg	65-129	10-134	40
8260C	4-Isopropyltoluene	Soil	ug/kg	61-132	10-126	40
8260C	4-Methyl-2-pentanone (MIBK)	Soil	ug/kg	69-126	30-129	40
8260C	Acetone	Soil	ug/kg	32-135	18-117	40
8260C	Acetonitrile	Soil	ug/kg	38-128	38-128	40
8260C	Acrolein	Soil	ug/kg	10-218	10-140	40
8260C	Acrylonitrile	Soil	ug/kg	18-179	42-163	40
8260C	Benzene	Soil	ug/kg	68-122	30-137	40

Attachment B-2
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Phase II RI Work Plan
Premier Edible Oils
Portland, Oregon

				Accuracy	Matrix Spike	Precision
METHOD	**ANALYTE**	**MATRIX**	**UNITS**	**(LCS %Rec.)**	**(%Rec.)**	**(% RPD)**
8260C	Bromobenzene	Soil	ug/kg	71-124	13-134	40
8260C	Bromochloromethane	Soil	ug/kg	65-131	34-132	40
8260C	Bromodichloromethane	Soil	ug/kg	61-143	14-146	40
8260C	Bromoform	Soil	ug/kg	62-134	10-139	40
8260C	Bromomethane	Soil	ug/kg	22-180	10-160	40
8260C	Carbon Disulfide	Soil	ug/kg	55-141	18-140	40
8260C	Carbon Tetrachloride	Soil	ug/kg	51-135	10-144	40
8260C	Chlorobenzene	Soil	ug/kg	70-116	15-124	40
8260C	Chloroethane	Soil	ug/kg	51-122	15-149	40
8260C	Chloroform	Soil	ug/kg	61-137	43-133	40
8260C	Chloromethane	Soil	ug/kg	37-146	30-133	40
8260C	Chloroprene	Soil	ug/kg	10-167	55-142	40
8260C	cis-1,2-Dichloroethene	Soil	ug/kg	62-138	32-137	40
8260C	cis-1,3-Dichloropropene	Soil	ug/kg	58-138	20-132	40
8260C	cis-1,4-Dichloro-2-butene	Soil	ug/kg	10-175	54-132	40
8260C	Cyclohexane	Soil	ug/kg	70-130	70-130	40
8260C	Dibromochloromethane	Soil	ug/kg	69-120	21-132	40
8260C	Dibromomethane	Soil	ug/kg	68-125	41-127	40
8260C	Dichlorodifluoromethane	Soil	ug/kg	38-160	14-158	40
8260C	Dichlorofluoromethane	Soil				
8260C	Diethylether	Soil				
8260C	Diisopropyl Ether	Soil	ug/kg	55-158	55-158	40
8260C	Ethyl Acetate	Soil	ug/kg	61-117	10-180	40
8260C	Ethyl Methacrylate	Soil	ug/kg	10-149	51-129	40
8260C	Ethylbenzene	Soil	ug/kg	70-118	13-128	40
8260C	Ethylene Oxide	Soil	ug/kg	29-158	29-158	40
8260C	Hexachlorobutadiene	Soil	ug/kg	54-140	10-114	40
8260C	Iodomethane (Methyl Iodide)	Soil	ug/kg	33-160	21-122	40
8260C	Isobutanol	Soil	ug/kg	21-128	47-118	40
8260C	Isopropylbenzene	Soil	ug/kg	67-133	10-153	40
8260C	m,p-Xylenes	Soil	ug/kg	69-127	10-139	40
8260C	Methacrylonitrile	Soil	ug/kg	15-140	47-120	40
8260C	Methyl Acetate	Soil	ug/kg	42-121	10-172	40
8260C	Methyl Methacrylate	Soil	ug/kg	10-147	46-125	40
8260C	Methyl tert-Butyl Ether	Soil	ug/kg	66-118	44-116	40
8260C	Methylcyclohexane	Soil	ug/kg	70-130	70-130	40
8260C	Methylene Chloride	Soil	ug/kg	65-122	36-123	40
8260C	Naphthalene	Soil	ug/kg	54-134	10-127	40
8260C	n-Butylbenzene	Soil	ug/kg	53-139	10-125	40
8260C	n-Hexane	Soil	ug/kg	38-173	27-186	40

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Premier Edible Oils
Portland, Oregon

				Accuracy	Matrix Spike	Precision
METHOD	**ANALYTE**	**MATRIX**	**UNITS**	**(LCS %Rec.)**	**(%Rec.)**	**(% RPD)**
8260C	n-Octane	Soil	ug/kg	49-138		
8260C	n-Propylbenzene	Soil	ug/kg	57-143	10-145	40
8260C	o-Xylene	Soil	ug/kg	69-124	10-146	40
8260C	Propionitrile	Soil	ug/kg	16-138	47-120	40
8260C	Propylene Oxide	Soil	ug/kg			
8260C	sec-Butylbenzene	Soil	ug/kg	55-146	10-141	40
8260C	Styrene	Soil	ug/kg	62-135	10-130	40
8260C	tert-Amyl Methyl Ether	Soil	ug/kg	64-148	64-148	40
8260C	tert-Butyl Alcohol	Soil	ug/kg	35-141	64-148	40
8260C	tert-Butyl Ethyl Ether	Soil	ug/kg	58-152	58-152	40
8260C	tert-Butylbenzene	Soil	ug/kg	58-152	10-152	40
8260C	Tetrachloroethene (PCE)	Soil	ug/kg	66-126	10-132	40
8260C	Toluene	Soil	ug/kg	75-117	24-142	40
8260C	trans-1,2-Dichloroethene	Soil	ug/kg	63-127	29-139	40
8260C	trans-1,3-Dichloropropene	Soil	ug/kg	63-121	19-125	40
8260C	trans-1,4-Dichloro-2-butene	Soil	ug/kg	26-204	10-179	40
8260C	Trichloroethene (TCE)	Soil	ug/kg	67-126	18-145	40
8260C	Trichlorofluoromethane	Soil	ug/kg	51-140	20-137	40
8260C	Trichlorotrifluoroethane	Soil	ug/kg	53-135	24-144	40
8260C	Vinyl Acetate	Soil	ug/kg	45-158	31686	40
8260C	Vinyl Chloride	Soil	ug/kg	54-127	31-140	40
8260C	1,2-Dichloroethane-D4 (Surr.)	Soil	%	71-119	NA	NA
8260C	4-Bromofluorobenzene (Surr.)	Soil	%	77-124	NA	NA
8260C	Dibromofluoromethane (Surr.)	Soil	%	83-128	NA	NA
8260C	Toluene-D8 (Surr.)	Soil	%	83-135	NA	NA

NA - not available

Attachment B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edible Oils
Portland, Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
7471B	Mercury	7439-97-6	Soil	0.002	0.02	mg/kg	71-128	75-125	20

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
6020A	Aluminum	7429-90-5	Soil	0.4	2	mg/kg	41-158	75-125	20
6020A	Antimony	7440-36-0	Soil	0.02	0.05	mg/kg	50-150	75-125	20
6020A	Arsenic	7440-38-2	Soil	0.06	0.5	mg/kg	78-122	75-125	20
6020A	Cadmium	7440-43-9	Soil	0.004	0.02	mg/kg	81-119	75-125	20
6020A	Chromium	7440-47-3	Soil	0.03	0.2	mg/kg	80-119	75-125	20
6020A	Copper	7440-50-8	Soil	0.08	0.1	mg/kg	83-116	75-125	20
6020A	Lead	7439-92-1	Soil	0.009	0.05	mg/kg	79-121	75-125	20
6020A	Manganese	7439-96-5	Soil	0.03	0.05	mg/kg	81-119	75-125	20
6020A	Nickel	7440-02-0	Soil	0.03	0.2	mg/kg	81-118	75-125	20
6020A	Selenium	7782-49-2	Soil	0.2	1	mg/kg	80-120	75-125	20
6020A	Silver	7440-22-4	Soil	0.008	0.02	mg/kg	66-134	75-125	20
6020A	Zinc	7440-66-6	Soil	0.2	0.5	mg/kg	73-121	75-125	20

Attachment B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edible Oils
Portland, Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

							Accuracy	Matrix Spike	Precision
METHOD	**ANALYTE**	**CAS No.**	**MATRIX**	**MDLa**	**MRL**	**UNITS**	**(LCS %Rec.)**	**(%Rec.)**	**(% RPD)**
314	Perchlorate	14797-73-0	Soil	7	20	ug/Kg	72-133	30-160	20
7196A	Hexavalent Chromium	18540-29-9	Soil	0.08	0.5	mg/Kg	80-120	85-115	30
9013/9012B	Cyanide, Total and Amenable	57-12-5	Soil	0.06	0.2	mg/Kg	62-128	10-171	20

Attachment B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edible Oils
Portland, Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

							Accuracy	Matrix Spike	Precision
METHOD	**ANALYTE**	**CAS No.**	**MATRIX**	**MDLa**	**MRL**	**UNITS**	**(LCS %Rec.)**	**(%Rec.)**	**(% RPD)**
NWTPH-Dx	Diesel Range Organics	68334-30-5	Soil	0.79	25	mg/kg	42-134	23-144	40
NWTPH-Dx	Residual Range Organics	NA	Soil	2.9	100	mg/kg	48-141	29-167	40
NWTPH-Dx	o-Terphenyl (Surr.)	84-15-1	Soil	NA	NA	%	50-150	NA	NA

CAS/KELSO DATA QUALITY OBJECTIVES

							Accuracy	Matrix Spike	Precision
METHOD	**ANALYTE**	**CAS No.**	**MATRIX**	**MDLa**	**MRL**	**UNITS**	**(LCS %Rec.)**	**(%Rec.)**	**(% RPD)**
NWTPH-Gx	Gasoline Range Organics	8006-61-9	Soil	1.5	5	mg/kg	81-111	32-154	40
NWTPH-Gx	4-Bromofluorobenzene (Surr.)	460-00-4	Soil	NA	NA	%	50-150	NA	NA

NA- not available

Attachment B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edible Oils
Portland Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

							Accuracy	Matrix Spike	Precision
METHOD	**ANALYTE**	**CAS No.**	**MATRIX**	**MDLa**	**MRL**	**UNITS**	**(LCS %Rec.)**	**(%Rec.)**	**(% RPD)**
8270D SIM PAH	1-Methylnaphthalene	90-12-0	Soil	0.51	5	ug/kg	37-129	26-133	40
8270D SIM PAH	1-Methylphenanthrene	832-69-9	Soil	0.28	5	ug/kg	53-109	52-102	40
8270D SIM PAH	2,3,5-Trimethylnaphthalene	2245-38-7	Soil	0.21	5	ug/kg	34-137	28-162	40
8270D SIM PAH	2,6-Dimethylnaphthalene	581-42-0	Soil	0.36	5	ug/kg	37-135	36-151	40
8270D SIM PAH	2-Methylnaphthalene	91-57-6	Soil	0.39	5	ug/kg	27-126	24-115	40
8270D SIM PAH	Acenaphthene	83-32-9	Soil	0.5	5	ug/kg	39-124	33-118	40
8270D SIM PAH	Acenaphthylene	208-96-8	Soil	0.56	5	ug/kg	38-126	32-117	40
8270D SIM PAH	Anthracene	120-12-7	Soil	0.55	5	ug/kg	38-130	30-127	40
8270D SIM PAH	Benz(a)anthracene	56-55-3	Soil	0.72	5	ug/kg	46-120	35-122	40
8270D SIM PAH	Benzo(a)fluoranthene	203-33-8	Soil			ug/kg	57-146	40-162	40
8270D SIM PAH	Benzo(a)pyrene	50-32-8	Soil	0.76	5	ug/kg	49-122	37-123	40
8270D SIM PAH	Benzo(b)fluoranthene	205-99-2	Soil	0.92	5	ug/kg	51-121	35-124	40
8270D SIM PAH	Benzo(b)fluorene	243-17-4	Soil			ug/kg	50-150	50-150	40
8270D SIM PAH	Benzo(b)thiophene	95-15-8	Soil	0.55	5	ug/kg	47-150	20-154	40
8270D SIM PAH	Benzo(e)pyrene	192-97-2	Soil	0.61	5	ug/kg	56-122	37-133	40
8270D SIM PAH	Benzo(g,h,i)perylene	191-24-2	Soil	0.85	5	ug/kg	49-122	33-128	40
8270D SIM PAH	Benzo(k)fluoranthene	207-08-9	Soil	0.87	5	ug/kg	55-120	38-124	40
8270D SIM PAH	Benzo[j,k]fluoranthene	CASID3089	Soil	0.87	5	ug/kg	55-120	38-124	40
8270D SIM PAH	Chrysene	218-01-9	Soil	0.8	5	ug/kg	49-120	36-126	40
8270D SIM PAH	Dibenz(a,h)anthracene	53-70-3	Soil	0.8	5	ug/kg	43-125	32-125	40
8270D SIM PAH	Dibenzofuran	132-64-9	Soil	0.63	5	ug/kg	41-130	34-131	40
8270D SIM PAH	Fluoranthene	206-44-0	Soil	0.98	5	ug/kg	39-135	35-139	40
8270D SIM PAH	Fluorene	86-73-7	Soil	0.61	5	ug/kg	39-129	33-125	40
8270D SIM PAH	Indeno(1,2,3-cd)pyrene	193-39-5	Soil	0.87	5	ug/kg	40-128	28-133	40
8270D SIM PAH	Naphthalene	91-20-3	Soil	0.6	5	ug/kg	32-124	23-114	40
8270D SIM PAH	Perylene	198-55-0	Soil	0.72	5	ug/kg	41-119	30-128	40
8270D SIM PAH	Phenanthrene	85-01-8	Soil	1.4	5	ug/kg	39-123	29-125	40
8270D SIM PAH	Pyrene	129-00-0	Soil	0.76	5	ug/kg	39-134	27-134	40
8270D SIM PAH	2,4,6-Tribromophenol (Surr.)	118-79-6	Soil	NA	NA	%	35-109	NA	NA
8270D SIM PAH	Fluoranthene-d10 (Surr.)	93951-69-0	Soil	NA	NA	%	26-118	NA	NA
8270D SIM PAH	Fluorene-d10 (Surr.)	81103-79-9	Soil	NA	NA	%	22-113	NA	NA
8270D SIM PAH	Terphenyl-d14 (Surr.)	1718-51-0	Soil	NA	NA	%	32-130	NA	NA

NA - not available

Table B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edible Oils Site
Portland, Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
7471B	Mercury	7439-97-6	Soil	0.002	0.02	mg/kg	71-128	75-125	20

DATA QUALITY OBJECTIVES

METHOD	ANALYTE	CAS No.	MATRIX	MDLa	MRL	UNITS	Accuracy (LCS %Rec.)	Matrix Spike (%Rec.)	Precision (% RPD)
8082	Aroclor-1016	12674-11-2	Soil	2.1	10	ug/kg	37-121	27-128	40
8082	Aroclor-1221	11104-28-2	Soil	2.1	20	ug/kg	-	-	-
8082	Aroclor-1232	11141-16-5	Soil	2.1	10	ug/kg	-	-	-
8082	Aroclor-1242	53469-21-9	Soil	2.1	10	ug/kg	-	-	-
8082	Aroclor-1248	12672-29-6	Soil	2.1	10	ug/kg	-	-	-
8082	Aroclor-1254	11097-69-1	Soil	2.1	10	ug/kg	-	-	-
8082	Aroclor-1260	11096-82-5	Soil	2.1	10	ug/kg	42-123	29-131	40
8082	Aroclor-1262	37324-23-5	Soil	2.1	10	ug/kg	-	-	-
8082	Aroclor-1268	11100-14-4	Soil	2.1	10	ug/kg	-	-	-

Attachment B-2
Method Detection, Quanitation and Reporting Limits
Phase II RI Work Plan
Premier Edible Oils
Portland, Oregon

CAS/KELSO DATA QUALITY OBJECTIVES

				Accuracy	Matrix Spike
METHOD	**ANALYTE**	**Matrix**	**UNITS**	**(LCL %R)**	**(UCL %R)**
TO-15	Ethylbenzene	Soil Gas	ug/m^3	60	131
TO-15	Trichloroethene (TCE)	Soil Gas	ug/m^3	61	135

Fixed Limits; LOQ analyzed at 5ng for these compounds
Precision (RPD) criteria for each compound shall be as stated in the method and specific SOP as +/- 25%
The base reporting limit from a 1 L cansiter is 1.3 ug/m^3
The base reporting limit from a 6 L cansiter is 0.50 ug/m^3
RL adjusted for canister dilution factor and volume analyzed (if a dilution is required)

Appendix C
- Analytical Methods for Petroleum Hydrocarbons, Washington State Department of Ecology, June 1997

ANALYTICAL METHODS FOR PETROLEUM HYDROCARBONS

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June 1997

Printed on Recycled Paper

ANALYTICAL METHODS
FOR
PETROLEUM HYDROCARBONS

Prepared by:

Washington State Department of Ecology
Toxics Cleanup Program
And
The Ecology Environmental Laboratory

Publication No. ECY 97-602
June 1997

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PREFACE LETTER

ANALYTICAL METHODS FOR PETROLEUM HYDROCARBONS

This document contains analytical methods for analyzing "Total Petroleum Hydrocarbons" (TPH) for compliance with the Model Toxics Control Act (MTCA) Cleanup Regulation (chapter 173-340 WAC). The Washington Department of Ecology (Ecology) adopted changes to the MTCA Cleanup Regulation on February 12, 2001. These changes became effective on August 15, 2001. Some of the analytical methods provided in this document may also be used for analyzing TPH in Oregon (check with the Oregon Department of Environmental Quality for specific requirements).

The MTCA Cleanup Regulation sets forth the procedures and requirements for conducting remedial investigations, establishing cleanup standards, and selecting cleanup actions for the cleanup of hazardous waste sites. Part VII of the regulation describes how to establish cleanup levels for hazardous substances. MTCA provides three approaches for establishing TPH cleanup levels: Method A (ARARs and Tables), Method B (Universal method), and Method C (Conditional method). Each of these approaches require specific analytical testing methods based on soil and/or groundwater contamination and the hazardous substance found on the site.

Under Method A, the “total” approach may be used to collect and analyze the presence, location, and concentration of TPH. This “total” approach includes two analytical methods: NWTPH-Gx for gasoline range organics (GRO) and NWTPH-Dx for diesel range organics (DRO) and heavy oils. See Table 830-1 in WAC 173-340-900.

Under Method B and Method C, the “fractionated” approach must be used to collect and analyze the presence, location, and concentration of TPH, except as provided in WAC 173-340-700(8)(b)(ii)(D). This “fractionated” approach includes two analytical methods: one for “volatile” aliphatic and aromatic petroleum hydrocarbons (VPH) for gasoline range organics (GRO), and another for “extractable” aliphatic and aromatic petroleum hydrocarbons (EPH) for diesel range organics (DRO) and heavy fuel oils. See Table 830-1 in WAC 173-340-900. Ecology is in the process of accrediting laboratories for the VPH and EPH analytical methods. Pending the evaluation of information from the laboratory accreditation process, Ecology will be updating this document.

In conjunction with the Evironmental Protection Agency (EPA), Ecology is establishing a laboratory accreditation process. The accreditation of Washington laboratories will provide some assurance of the technical proficiency and competence of a laboratory to assess conformance to a set of prescribed standards. The laboratory accreditation will be a formal recognition that a testing laboratory is competent to carry out tests specifically related to TPH analytical testing methods.

Ecology is currently revising and expanding the TPH guidance document, "Guidance for Remediation of Petroleum Contaminated Soils," Pub. No. 91-30. The revised document will provide updated and expanded guidance regarding the conduction of remedial investigations, the establishment of cleanup levels, and the selection of cleanup actions for petroleum contaminated sites.

Questions about analytical methods for petroleum hydrocarbons or the laboratory accreditation process should be directed to Charles San Juan at the Department of Ecology (360-407-7191); e-mail: csan461@ecy.wa.gov. Access to a variety of technical guidance documents is also available at the Toxics Cleanup Program's Internet address: http://www.ecy.wa.gov/programs/tcp/cleanup.html.

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TOTAL PETROLEUM HYDROCARBONS (TPH)

ANALYTICAL DECISION TREE FOR NWTPH

* The chromatograms are not available in the "electronic" version. They are included in the printed publication.

TOTAL PETROLEUM HYDROCARBONS (TPH)

The following compilation of analytical methods may be used in Oregon and Washington for Underground Storage Tank cleanups and other cleanups of TPH (check with applicable state rules). Each of these Total Petroleum Hydrocarbon (TPH) Methods has its own niche in the overall analytical scheme. The methods are:

NWTPH-HCID.....Hydrocarbon Identification

NWTPH-Gx .....Volatile Petroleum Products (Extended)

NWTPH-Dx .....Semi-volatile Petroleum Products (Extended)

NWTPH-HCID is a qualitative and semi-quantitative screen to determine the presence and type of petroleum products that may exist in water or soil. This method should be used if the type of petroleum contamination is unknown. It should be performed on contaminated soil or water that is representative of the contamination at the site. The results of this method will determine what fully quantitative method/methods, if any, are to be used in determining compliance with the matrix criteria. Should the value of the analysis for gasoline, diesel or heavy oils (or any other identified petroleum product) exceed the reporting limits, then the specific analytical method for that product must be employed.

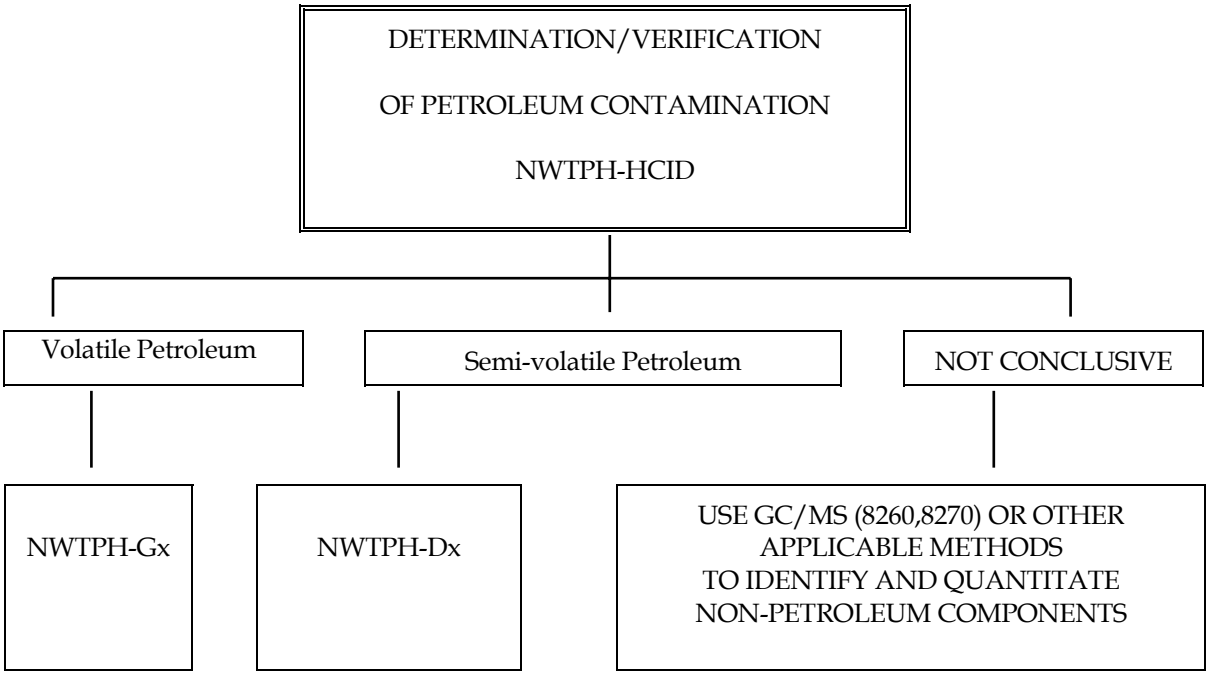
NWTPH-Gx is the qualitative and quantitative method (extended) for volatile ("gasoline") petroleum products in soil and water. Petroleum products applicable for this method include aviation and automotive gasolines, mineral spirits, stoddard solvent and naphtha.

NWTPH-Dx is the qualitative and quantitative method (extended) for semi-volatile ("diesel") petroleum products in soil and water. Petroleum products applicable for this include jet fuels, kerosene, diesel oils, hydaulic fluids, mineral oils, lubricating oils and fuel oils.

NOTE: These "NWTPH" methods result in single TPH values that can be used when compliance with a single cleanup level is desired. When TPH "fractions" are needed, then the VPH and EPH methods must be used.

ANALYTICAL DECISION TREE FOR NWTPH

The following flow chart depicts the laboratory analytical scheme to be used when analyzing samples for single TPH levels. The first step is the qualitative determination of the existence and nature of petroleum contamination and this should be used when the site contamination is unknown. It is required that this first step will be performed on a representative sample from the area that is suspected to be the most contaminated at the site. For those samples containing analytes which, due to their retention times, ratios to each other or their non-hydrocarbon pattern (e.g., creosote), do not suggest petroleum hydrocarbons, then GC/MS methods should be employed to ascertain the components. At those sites where the petroleum contaminants are known or have been identified using the NWTPH-HCID method, the specific product method is to be used.



NWTPH-HCID

Hydrocarbon Identification Method for Soil and Water

Summary

This method is a qualitative and semi-quantitative procedure. It is used for groundwater or surface water, and soil/sediment from sites where the petroleum products are unknown and/or when multiple types of petroleum products are suspected to be present. This method is used to identify petroleum products containing components from C7 to C30 range, as well as heavy oils, with specific product confirmation by pattern matching ("fingerprinting") employing capillary gas chromatography with flame ionization detection (GC/FID). EPA method 3510 has been adapted as the extraction procedure for the water portion of this method.

While this method is intended to be primarily qualitative, it can be used to eliminate the need for further analyses for those samples which demonstrate TPH levels significantly below the regulatory limits. If the sample contains toluene to dodecane (gasoline range), dodecane through tetradecane (diesel range) and/or an unresolved chromatographic envelope greater than tetradecane (e.g. motor oils) above the reporting limits of this method, then the final quantitation must be performed by methods specific for these mixtures. Since the water extraction procedure in this method is identical to that found in the water portion of NWTPH-Dx (semi-volatile petroleum products, i.e. from kerosene through heavy fuel oils), these products may be quantitated using this extract. Because of the possible loss of volatile compounds in the extraction and concentrations steps, gasoline, mineral spirits and other volatile petroleum products that exceed the reporting limits of this method must be quantitated using the NWTPH-Gx method.

The reporting limits for water are 0.25 mg/L for gasoline, 0.63 mg/L for #2 diesel and motor oils. The reporting limits for soil/sediment are 20 mg/Kg for gasoline, 50 mg/Kg for #2 diesel, and 100 mg/Kg for motor oil, all reported on a dry weight basis. These values for soil/sediment assumes 100% solids and will be higher depending on the actual moisture content.

The method relies heavily upon the experience of the analyst for the identification of the specific petroleum product(s) that may be present. Therefore, this method must be run by, or under the direct supervision of, analysts experienced in the use of GC and in the interpretation of gas chromatograms of both fresh and weathered petroleum products.

Equipment and Reagents

Gas Chromatograph, w/wo autosampler
Capillary split/splitless injector
Flame ionization detector
Suggested Capillary Column:
J&W DB-1 or DB-5, 30 M x 0.25 mm or 0.32 mm with 0.25 um film thickness capillary column or equivalent
Chromatography Data System
VOA Vial: 40 mL glass vial with Teflon coated cap septum, Eagle Picher or equivalent
Syringe: Hamilton #701, 10 uL or equivalent
Ultrasonic Bath
Glass Wool: Pyrex or equivalent
Centrifuge tubes: 5 or 15 mL, calibrated in 0.1 mL increments
Analytical Balance: accurate to at least 0.0001 g
Volumetric Flasks: 10 mL, ground glass with ground glass stopper
Separatory funnels: 500 mL, Teflon stopcocks
Kuderna-Danish (KD) Flasks: 250 mL or equivalent
Snyder Columns: 3-ball, 24/40 ground glass joint
Concentrator Tubes: 10 mL
Methylene Chloride: Burdick and Jackson Brand or equivalent
N-Evap Concentrator or equivalent

Standards

Retention Time Standards. Prepare a composite standard, using methylene chloride as the solvent, consisting of toluene, dodecane and tetracosane at 25 ug/mL each. Additional compounds may be added at the discretion of the analyst. The use of this standard is to establish the retention time windows for the quantitation of gasoline #2 diesel and motor oils.

Reference Standards. Prepare individual petroleum product reference standards (i.e. gasoline, mineral spirits, kerosene and #2 diesel oil), using methylene chloride as the solvent, at approximately 50 ug/mL. Prepare a non-synthetic motor oil (pennzoil SAE 30 or equivalent) reference standard at 200 ug/mL. The preparation of reference standards for other types of petroleum products is recommended. The use of these reference standards is to insure the accurate identification of petroleum product contamination by chromatographic pattern matching ("fingerprinting") and establish retention time windows for those petroleum products not determined with the individual compound retention time standard.

Gasoline Stock Standard. A stock standard is prepared by placing approximately 9 mL of methylene chloride in a 10 mL volumetric flask. Tare the flask/methylene chloride and add about five drops of non-oxygenated regular unleaded gasoline, assuring that the liquid falls directly into the methylene chloride without contacting the neck of the flask. Reweigh the flask and dilute to volume with methylene chloride, stopper and mix by inverting the flask several times. It is important that the analyst minimize the amount of time that the flask is left unstoppered, to reduce

the loss of gasoline through volatilization. The use of a commercially prepared gasoline standards is acceptable if it is certified as non-oxygenated gasoline or if the gasoline concentration has been adjusted to reflect the contribution of the oxygenate. Calculate the gasoline concentration as follows:

$$Stock,\ ug / mL = \frac{(final\ wt,\ mg) - (tare\ wt,\ mg)}{10\ mL} \times \frac{1000\ ug}{mg}$$

Note: The use of oxygenated regular unleaded gasoline for the gasoline stock standard is allowed if the weight (mass) of the gasoline used is adjusted for the weight (mass) contribution of the oxygenate to the gasoline. This will necessitate the analysis of the gasoline for the specific oxygenate(s) present to determine the concentration. The analysis for the oxygenates will be conducted by either of the methods published in the Federal Register - Appendix B and C - Testing Procedures - Vol. 57, No. 24, Wednesday, February 5, 1992, Notices.Alternate methods for the analysis of gasoline oxygenates must be approved by the Oregon's Department of Environmental Quality and/or Washington's Department of Ecology prior to use.

Diesel Stock Standard. A stock standard is prepared by adding about five drops of #2 diesel oil stock to tared 10 mL volumetric flask. Reweigh the flask and bring it to volume with methylene chloride, stopper and mix by inverting the flask several times. Calculate the concentration of this standard in the same manner as the gasoline stock standard. The use of a commercially prepared #2 diesel standard is an acceptable alternative to the above procedure.

Motor Oil Stock Standard. A stock standard is prepared by adding about ten drops of a non-synthetic SAE 30 weight motor oil (Pennzoil or equivalent) to a tared 10 mL volumetric flask. Reweigh the flask, bring it to volume with methylene chloride, stopper and mix by inverting the flask several times. Calculate the concentration of this standard in the same manner as the gasoline stock standard. The use of commercially prepared motor oil standards is an acceptable alternative to the above procedure.

Note: The Diesel and Motor Oil Stock Standards required in this method are identical to those required for NWTPH-Dx (extended diesel method including all semi-volatile petroleum products eluting after gasoline, e.g. kerosene, diesels, mineral oils, lubricating oils, heavy fuel oils, etc.).

Surrogate Stock Standard. Suggested surrogates for use in this method are bromofluorobenzene and pentacosane. The use of different or additional surrogates is optional. Prepare the surrogate stock standard by weighing 50 mg of each surrogate compound into a 10 mL volumetric flask, then bring to volume with methylene chloride for a final concentration of 5000 ug/mL for each surrogate compound. The use of a commercially prepared surrogate solution(s) is an acceptable alternative to the above procedure.

Composite Calibration Working Standard. Using serial dilutions of the stock standards, prepare a mixture for water analyses that contains 10 ug/mL of gasoline, 25 ug/mL of #2 diesel oil and the

surrogate standard. For soil/sediment analyses this standard should be prepared to contain 20 ug/mL of gasoline, 50 ug/mL of #2 diesel and the surrogate standard. Add the appropriate volumes, using the equations listed below, and adjusting for the concentration change created by any serial dilutions, of gasoline stock standard, #2 diesel stock standard and the surrogate stock standard to a 10 mL volumetric flask, then dilute to volume with methylene chloride. Stopper and mix by inverting the flask several times. The surrogate standard should be added to a level sufficient to produce a surrogate concentration of between 5 and 50 ug/mL.

$$\textit{Volume Gasoline Stock, uL} = \frac{\textit{20 (soil) / 10 (water) ug / mL x 10 mL}}{\textit{Gasoline Stock Conc, ug / mL}} \; x \; \frac{\textit{1000 uL}}{\textit{mL}}$$

$$\textit{Volume Diesel Stock, uL} = \frac{\textit{50 (soil) / 25 (water) ug / mL x 10 mL}}{\textit{Diesel Stock Conc, ug / mL}} \; x \; \frac{\textit{1000 uL}}{\textit{mL}}$$

This mixture corresponds to 0.25 mg/L gasoline and 0.63 mg/L #2 diesel oil for water and 20 mg/Kg gasoline and 50 mg/Kg diesel in soil following the extraction and analytical procedures of this method.

The motor oil calibration working standard should be made at a concentration of 250 ug/mL for water and 100 ug/mL for soil following the procedure outlined above. This will correspond to a reporting value of 0.63 mg/L for water and 100 mg/kg for soil. If, in the opinion of the analyst, the GC sensitivity to 100 ug/mL of motor oil is insufficient, the analyst is allowed to increase the concentration of this standard and to concentrate a portion of the extract to achieve the reporting limit for soil.

Surrogate Working Standard. Prepare a surrogate working (spiking) standard, using the procedure outlined above, that will yield between 5 ug and 50 ug/mL of the surrogate compounds in the 10 mL sample extract produced in this method.

Note: All samples must be collected in glass jars with Teflon lined lids (Eagle Picher or equivalent) and held at 4 degrees C until extracted. A volume of 400 mL is the minimum sample size to achieve the reporting limits as stated, however, larger volumes are allowed as long as the solvent/sample ratio is maintained. Samples must be extracted within 7 days (for water) or 14 days (for soil/sediment) of the date of collection. The preservation of water samples in the field, to a pH of less than 2 with 1+1 HCL, is recommended.

Extraction Procedures

Water Samples

Mark the water meniscus on the sample jar for later volume determination. Pour the sample into a 500 mL separatory funnel, add the surrogate solution (to achieve the desired concentration in a 10 mL extract). Add 30 mL of methylene chloride to the sample jar, cap and shake the jar vigorously

for a few seconds to wash off any hydrocarbons adhering to the side of the jar. Add this solvent to the separatory funnel, stopper and shake vigorously, venting frequently, for one minute. Allow the two phases to separate, then drain the solvent into a 250 mL K-D flask fitted with a 10 mL concentrator tube. Repeat the extraction twice more using 30 mL of methylene chloride, each time, adding the solvent to the K-D.

Attach a 3-ball Snyder column to the K-D and concentrate the sample extract to 5 - 10 mL on a stream bath. Remove the K-D apparatus and allow it to cool prior to disassembly. During disassembly, rinse the Snyder/K-D joint and K-D/concentrator joint with approximately 1 mL of methylene chloride and add these rinsings to the extract. Adjust the volume of the extract to 10 mL. For those samples which exceed 10 mL, place the concentrator tube into an N-Evap and, under a gentle stream of nitrogen, reduce the volume to 10 mL. Transfer 1 mL of the extract (to be used for the gasoline or gasoline/diesel determination) to an autosampler vial fitted with a screw top and a Teflon coated septum. Concentrate the remainder of the extract (to be used for heavy oils determination) to 0.9 mL, transfer it to an 2 mL autosampler vial equipped with a screw top and a Teflon coated septum. Store both vials in a refrigerator until analysis.

Each extraction set must include one method blank (organic-free water of similar volume to the samples) per 20 samples. The method blank is to receive the surrogate solution and to be extracted and analyzed in the same manner as the samples.

EPA method 3520, Continuous Liquid-Liquid Extraction, may be substituted as an alternate extraction procedure to that outlined above.

Soil Samples

Weigh approximately 10 grams of soil into a 40 mL VOA vial and record the weight to the nearest 0.001 grams. Add 5 grams of anhydrous sodium sulfate, surrogate working solution and 10 mL of methylene chloride to the VOA vial. Cap the vial and place it (no more than 5 at a time) in a sonic bath for 5 minutes. Shake the vials well and return them to the sonic bath for 5 more minutes. A minimum of one method blank per extraction set or 20 samples, whichever is more frequent, must be prepared along with the samples. One sample duplicate must also be extracted for samples set from one to ten samples and two duplicates for sets from eleven to twenty samples.

Determine the moisture content of the samples, for use in the final calculations, by the following procedure. Immediately after weighing the sample for extraction, weigh 5-10 grams of the sample into a tared crucible. Dry the sample/crucible overnight at 105 degrees C. Reweigh the sample/crucible after allowing it to cool to room temperature. Calculate the % solids as follows: [(grams of dry sample/grams of wet sample) x 100].

If the extract contains significant moisture, elute the solvent phase through an anhydrous sodium sulfate micro-column. Place a portion of the extract in a 2 mL autosampler vial (screw cap, Teflon coated septum), taking care to minimize the volume of headspace, and store the vial in a refrigerator until analyzed. Concentration of the extract, when necessary, will be conducted using an N-Evap

with a gentle stream of nitrogen. This concentrate should be stored in the same manner as noted above.

Note: Anhydrous sodium sulfate micro-columns are prepared by plugging a 5 3/4" length disposable Pasteur pipette (pre-rinsed with methylene chloride) with glass wool (precleaned with methylene chloride) and adding approximately 3 cm of anhydrous sodium sulfate (previously muffled at 430 degrees C overnight in a shallow pan and stored in a glass jar with a Teflon lid liner).

For samples containing petroleum products other than gasoline, diesel, or motor oil, the analyst must either prepare calibration standards of them by the methods listed above or analyze the sample by the fully quantitative method, e.g. NWTPH-Gx. Other calibration standards produced for use in this method must be at an equivalent concentration to the previously established standards, e.g. mineral spirits at the gasoline concentration, kerosene at the diesel concentration.

As more information becomes available on new extraction techniques, the Washington State Dept. of Ecology's Manchester Laboratory and Oregon's Department of Environmental Quality will publish descriptions of acceptable alternate extraction methods.

Suggested GC Parameters

Sample injection Volume = 2 uL
Injector Temperature = 290°C
Detector Temperature = 300°C
Hydrogen Flow = 25-35 mL/min
Air Flow = 300-400 mL/min
Make-up Gas Flow = 30 mL/min
GC Temperature Program = Initial temperature of 50°C and hold for 2 minutes; ramp the temperature 10°C/min to 320°C and hold for 5 minutes.

Product Identification

Petroleum products are to be identified by pattern matching with reference product chromatograms generated the same day as the sample analysis. The term "gasoline range" or "diesel range" hydrocarbons, or derivations of them, should only be used when the analyst is unable to identify the petroleum product present. When these terms are used, it is to indicate the presence of compounds eluting from toluene to dodecane, for the former term, and from dodecane through tetracosane, for the latter term. Motor oils, hydraulic fluids and similar petroleum products which present an unresolved chromatographic envelope of compounds, originating or extending beyond tetracosane, may be reported using the collective term, lube oil, unless specific identification is possible. Heavy fuel oils, e.g. fuel oil #6 or Bunker C, which contain a diesel range component as well as a lube oil (and higher) range may be reported using the collective term, heavy fuel oil, unless specific identification is possible. These products should not, however, be confused with mixtures of #2 diesel and motor oils.

Note: The actual identification of the grade or type of lube oil and heavy fuel oil may require equipment and techniques beyond the scope of this method.

Analysis Procedure

The analysis is accomplished by injecting 2 uL of the 10 mL extract, either manually or by autosampler, into the GC using the splitless injection mode. The results from this injection are, for quantitation purposes, compared directly against the single point calibration standard for the product(s) identified. The injection of 2 uL of the concentrated extract (1 mL equivalent) is used primarily for heavy oil determination, because of the reduced sensitivity of the FID to late eluting motor oil-like products. It may be used for the determination of #2 diesel oil and other diesel range petroleum products but it may not be used for the determination of gasoline or gasoline range petroleum products because of the potential for loss of the more volatile components during the concentration step.

Calculations

Gasoline. The area of the components, toluene to dodecane of the calibration standard, is integrated to the baseline as a group. The samples and method blanks are integrated in the same manner and the group areas are compared. If the sample area exceeds the calibration standard area, proceed with method NWTPH-Gx for accurate quantitation using a fresh aliquot of the sample. If the sample area does not exceed the calibration standard area, then report the gasoline concentration as less than 0.25 mg/L for water or 20 mg/kg for soil. This soil value for gasoline, and the subsequent petroleum products, assumes 100% solids and will be higher depending on the actual moisture content.

Other volatile petroleum products, identified as being present (e.g. mineral spirits), must be compared to the appropriate standard produced at the same concentration as gasoline. The requirement for further analyses is determined following the criteria for gasoline as noted above.

Note: For samples containing only #2 diesel oil, kerosene, etc., the portion of these products, that elute within the retention time range of gasoline, should not be identified and/or quantitated as gasoline or gasoline range petroleum.

Diesel. The area of the components, dodecane through tetracosane of the calibration standard, is integrated to the baseline as a group. This integration must include the unresolved envelope of compounds as well as the discrete component peaks. The sample is integrated in the same manner and the group areas are compared. If the sample area exceeds the calibration standard area, then proceed to method NWTPH-Dx. If the sample does not exceed the calibration standards area, then report the #2 diesel concentration as less than 0.63 mg/L for water or 50 mg/Kg for soil.

Other semi-volatile petroleum products, identified as being present (e.g. kerosene), must be compared to the appropriate standard produced at the same concentration as #2 diesel. The requirement for further analyses is determined following the criteria for #2 diesel as noted above.

Lube Oil. For those samples which consist primarily of only an unresolved chromatographic envelope of components eluting after tetracosane, compare their area to the area of the motor oil standard by integrating the unresolved envelope to baseline. If the sample exceeds the standard area, then proceed to method NWTPH-Dx. If the sample area is less than the standard, then report the lube oil concentration as less than 0.63 mg/L for water or 100 mg/Kg for soil. Samples identified as containing heavy fuel oil must be quantitated to the same value as lube oil by utilizing a heavy fuel oil standard and following the procedure outlined for lube oil.

Note: The analyst is expected to adjust the retention time windows used for quantitation of petroleum products, other than gasoline, #2 diesel, or motor oil/lube oils, to incorporate the majority of the chromatographable components associated with those identified products.

If, in the judgement of the analyst, the area of an identified petroleum product other than gasoline, diesel, and motor oil would exceed the reporting limit, the analyst is allowed to report a "greater than" value for that product without performing the quantitative analysis. The analyst must then proceed to the appropriate fully quantitative analytical procedure, e.g. NWTPH-Dx, for the identified petroleum product.

Quality Assurance

The addition of an appropriate extraction surrogate to samples and method blank(s) is required. The surrogate recovery for all samples and blanks should be between 50% and 150% and must be reported with the petroleum results unless the quantity of the petroleum product(s) preclude its determination. The laboratory should analyze one sample from each site in duplicate for sample sets of 10 or less and two samples in duplicate for sets of 11 to 20 samples (i.e. 10% QA). If either of the duplicate results are positive, the sample is to be considered positive. Since this method precludes the preparation of analytical duplicates, the laboratory should recommend that the project manager collect and submit field duplicates for analysis. The laboratory must analyze method blanks prepared identically to the samples. Organic free water must be used in the preparation of water method blank. No "sample" is necessary for use with soil/sediment method blanks.

Author - Bob Carrell, Manchester Environmental Laboratory, Dept. of Ecology, State of Washington. Reviewed and edited by Steve Robb, Toxics Cleanup Program, Department of Ecology, State of Washington. This method is based on Oregon’s Department of Environmental Quality TPH methods and Washington’s Department of Ecology WTPH methods.

NWTPH-Gx Chromatograms

Gasoline
Weathered Gasoline
Naptha
Mineral Spirits #1
Mineral Spirits #2
Mineral Spirits #3

NWTPH-Dx Chromatograms

#2 Diesel
#2 Diesel/Motor Oil
#2 Fuel Oil
Kerosene (Deodorized)
Jet A Fuel
Bunker C #1
Bunker C #2
Motor Oil 30 Wt.
Mineral Oil (USP)
Hydraulic Fluid
Transformer Oil
Gas Oil

See Appendix 6

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NWTPH-Gx

Volatile Petroleum Products Method for Soil and Water

Summary

The NWTPH-Gx method is intended to replace the Oregon's TPH-G and Washington's WTPH-G methods and to present a more comprehensive approach to volatile petroleum product analyses. NWTPH-Gx adapts Oregon's TPH, Washington's WTPH and EPA SW846 Methods 5030 and/or 8020 and covers the quantitative and qualitative analyses of volatile petroleum products, e.g. gasolines, naphtha, mineral spirits, stoddard solvent, and other volatile petroleum products in soil and water. Soil samples are extracted with methanol and analyzed by gas chromatograph with a flame ionization detector (GC/FID). The methanol extracts may be injected directly into the GC or they may be diluted with organic free reagent water and introduced by a purge/trap concentrator. Water samples are introduced directly to the purge/trap concentrator. This method specifies the criteria for the identification and quantitation of volatile petroleum products. When the type of petroleum product is unknown, regular unleaded gasoline will initially be used as the default petroleum standard. The use of GC/PID, GC/MS or GC/AED (Atomic Emission Detector) for the analysis of gasoline may be substituted for GC/FID as long as all other method parameters are met.

The reporting limits for soil are 5 mg/kg by the purge/trap procedure and 20 mg/kg for direct injection. All soil results are reported on a dry weight basis. Since this value assumes 100% solids, the analyst may wish to adjust the amount of soil extracted and/or purge a larger quantity of extract to achieve the reporting limits. For water, the reporting limits are 0.25 mg/L.

The method is applicable for the identification, by pattern matching ("fingerprinting") and quantitation of volatile petroleum products, i.e. those petroleum products for which the majority of the components elute within the gasoline range.

Note: Benzene, Toluene, Ethylbenzene and Xylenes (BTEX) may be determined simultaneously with gasoline if the requirements of EPA Method 8021 or EPA Method 8260 are met (i.e. the use of a PID (Photoionization Detector) for method 8020 or a MS (Mass Spectrometry) for method 8260 and the QA/QC associated with these methods).

This method must be performed by, or under the direct supervision of, analysts experienced in the use of GC and in the interpretation of gas chromatographs of both fresh and weathered volatile petroleum products.

Equipment and Reagents

Gas Chromatograph
Purge/Trap Liquid Concentrator - Tekmar or Equivalent - Autosampler (Optional)
Flame Ionization (FID) or Photo Ionization/Flame Ionization Detectors (PID/FID)
Suggested Capillary Column: Restex RTX-502.2, 60M x 0.53 mm x 3.0 um film thickness or equivalent
Chromatography Data System
Gas tight syringes, various volumes, Hamilton brand or equivalent
Volumetric flasks: 10 mL, ground glass joint with stopper
Methanol: Burdick and Jackson Purge/Trap grade or equivalent
Petroleum Product Standards: Available from commercial sources
Refer to methods 5030 and/or 8020 for the remaining equipment and reagent requirement

Sample Containers/Holding Times

All soil samples for NWTPH-Gx analyses must be collected in soil VOA bottles with Teflon coated septum lined tops. They should be filled to the top to minimize headspace above the soil and stored at 4 degrees C until analyzed. The maximum holding time (the time from the date of collection to the date of analysis) for gasoline and any other volatile petroleum product is 14 days.

All water samples for NWTPH-Gx analyses must be collected in glass VOA vials with Teflon coated septum lined screw tops. They must be filled so that there is no air space (headspace) above the water. Samples should be preserved with 1+1 HCl to a pH <2 and refrigerated at 4 degrees C until analyzed. The maximum holding time (the time from the date of collection to the date of analysis) is 7 days for unpreserved samples and 14 days for preserved samples. The results of any samples which exceed these limits must be qualified as an estimated value.

Standards

Stock Standards. A stock standard is prepared by placing approximately 9 mL of methanol in a 10 mL volumetric flask. Allow the flask to stand, unstoppered, until the methanol wetted surfaces have dried (about ten minutes), then tare the methanol/flask. Add about 5 drops of the petroleum product to the flask while assuring that the liquid falls directly into the methanol without contacting the neck of the flask. Weigh the flask again and dilute to volume with methanol, stopper and mix by inverting the flask several times. The use of commercially prepared standards is acceptable to the above procedure. Commercially prepared gasoline standards must be certified as non-oxygenated gasoline or the gasoline concentration has been adjusted to reflect the contribution of the oxygenate. Calculate the concentration as follows:

$$\textit{Gasoline Stock Standard, ug / mL} = \frac{\textit{(final wt, mg) - (tare wt, mg)}}{\textit{10 mL}} \times \frac{\textit{1000 ug}}{\textit{mg}}$$

The standard for gasoline will be regular unleaded gasoline and this standard is to be used as the default petroleum product for reporting purposes.

Note: The use of oxygenated regular unleaded gasoline for this standard is allowed if the weight (mass) of the gasoline used is adjusted for the weight contribution of the oxygenate to the gasoline. This will necessitate the analysis of the gasoline for the specific oxygenate(s) present to determine their concentration. This analysis must be conducted by either of the methods published in the Federal Register - Appendix B and C - Testing procedures - Vol. 57, No. 24, Wednesday, February 5, 1992, Notices. Alternate methods for the analysis of gasoline oxygenates must be approved by the Oregon's Department of Environmental Quality and/or Washington's Department of Ecology prior to use.

Stock Surrogate Standard

Note: The suggested surrogates are 1,4-difluorobenzene and bromofluorobenzene. The use of additional surrogates is optional. Selected surrogate compounds should be non-polar, purgeable from water and must not coelute with any significant component of gasoline.

Make up a stock standard by accurately weighing the surrogate compound(s) into a 10 mL volumetric flask. Utilize the same procedure as the stock gasoline standard preparation if neat material is used or by adding the appropriate volumes of dilute surrogate if solutions are used. Bring it to volume with methanol. The use of commercially prepared surrogate solutions is an acceptable alternative to the above procedure.

Working Surrogate Spike. Add the appropriate volume of stock surrogate standard to methanol in a partially filled 10 mL volumetric flask and dilute to volume with methanol. The final concentration of the working surrogate solution is left to the discretion of the analyst, however, this solution should produce between 5 ng and 50 ng of surrogate introduced to the GC.

Secondary Dilution Standard. Using serial dilutions of the stock standard, prepare a 50 ug/mL standard by adding the appropriate volumes, as calculated below, to a 10 mL volumetric flask. The appropriate volume of the stock surrogate standard may also be added to this volumetric flask. Dilute to volume with methanol to yield a final working standard concentration of 50 ug/mL for the standard. Surrogate compound(s) may be added automatically during the sampling process by an autosampler. Gasoline is the default petroleum product for reporting purposes.

$$\textit{Stock Std (Gasoline), uL} = \frac{\textit{50 ug / mL x 10 mL}}{\textit{Stock Std Conc, ug / mL}} \; x \; \frac{\textit{1000 uL}}{\textit{mL}}$$

Store all standards in a refrigerator until needed. Allow them to come to room temperature prior to use.

Calibration Standard

The aqueous purge standards are each prepared by adding 5 uL, 10 uL, 20 uL and 50 uL and 100 uL of the secondary dilution standard per 5 mL of organic free water. The five point calibration standard quantities in the purged water are then 250 ng, 500 ng, 1000 ng, 2500 ng and 5000 ng for the volatile petroleum products. At the discretion of the analyst, the concentration of the surrogate can increase with increasing standard concentration or remain at a fixed value for all calibration standards and samples. Extending the calibration range, either up or down, is allowed as long as the standards remain within the linear range of the instrument and a minimum of a five point calibration is produced. In order to be acceptable, the calibration curve must have a linear correlation coefficient of at least 0.990 and none of the standards may vary from their true (known) value by more than plus/minus 15%.

The use of an autosampler may be substituted for this manual method of standards and sample introduction.

Direction Injection. Prepare calibration standards from the stock gasoline standard and surrogate standards at concentrations of 10 ug/mL, 25 ug/mL, 50 ug/mL, 100 ug/mL and 250 ug/mL for gasoline by adding the appropriate volumes to volumetric flasks and diluting to volume with methanol. Increasing the calibration range with higher standards is acceptable as long as the linear range of the instrument is not exceeded.

Purge/Trap and GC Parameters

Follow the procedure outlined in Method 8021 for the set up of the purge and trap operating parameters and for the GC. Adjust the hydrogen/air flow rates to optimize the FID sensitivity.

Soil Extraction Procedure

Weigh approximately 5 grams of soil into a 40 mL VOA vial and record the weight to 0.001 grams. Add 50 uL of the surrogate working standard and 10 mL of methanol. Quickly cap the vial and shake for 1 minute or use an ultrasonic bath for 2 minutes shaking well after 1 minute. Allow the soil methanol mixture to separate, centrifuging if necessary to clarify the methanol extract. For storage, transfer a portion of the extract into a 2 mL glass autosampler vial, with a Teflon-lined cap, minimizing the headspace and store in a freezer for no longer than one week prior to analysis. Along with the samples, prepare at least one method blank and one sample duplicate per ten samples.

Determine the moisture content of the sample, for use in the final calculations, by the following method. Immediately after weighing the sample for extraction, weigh 5-10 grams of the sample into a tared crucible. Dry the sample/crucible overnight at 105 degrees C. Reweigh the sample/crucible after allowing it to cool to room temperature. Calculate the % solids as follows: [(grams of dry sample/grams of wet sample) x 100].

Analysis Procedure

Prior to the analysis of any samples or method blanks, prepare and analyze a mid-range calibration check standard to insure that the instrument is functioning correctly and that the calibration is valid. This standard should be produced daily using the secondary gasoline standard. The value obtained for this analysis must not vary from the true (known) value by more than plus/minus 20%. If the value falls outside this range then a second mid-range calibration standard should be produced and the analysis repeated. If the reanalysis of the fresh standard fails to meet the acceptance criteria, then the instrument must be recalibrated prior to the analysis of any samples. Once the instrument is shown to be in calibration, the analyses of samples may proceed.

After the last sample has been analyzed, a mid-range calibration check sample must be run to demonstrate that the instrument is still operating within the required parameters. Should this standard fail to meet those parameters, then all samples analyzed after the last successful calibration check must either be reanalyzed or the results obtained must be qualified as an estimated value. An increase in the frequency of mid-range calibration check standard analyses beyond the minimum required is recommended.

Significant interferences may be encountered due to the presence of other petroleum products (or non-petroleum products) eluting within the retention time range of the volatile petroleum product being analyzed. If this occurs, the analyst is allowed to adjust the retention time range used for quantitation to exclude the interferences or to subtract the area of the interfering components from the total area prior to the quantity determination. With the former method, the calibration curve must be adjusted in the same manner to reflect the change in retention time range and integration area.

For volatile petroleum products other than gasoline that have a more narrow boiling point range, e.g. mineral spirits, the retention time range used for quantitation should be adjusted to encompass the expected range of the product. Petroleum products which cannot be identified should be quantitated with the gasoline calibration curve. The term "gasoline range" hydrocarbons, or derivations of it, should not be used when reporting the petroleum values unless the analyst is unable to identify the petroleum product present.

Purge/Trap - Soil. A 100 uL aliquot of the methanol extract is transferred via a 100 uL gas tight syringe to 5 mL of organic free water in a 5 mL gas tight syringe and immediately injected into the purging vessel of the purge and trap device. For samples expected to contain concentrations of gasoline range volatiles outside the calibration linear range, or if dilution is required, a smaller aliquot of the methanol extract, or sample, should be used. The analysis then proceeds as in Method 8020. Autosampler techniques may be substituted for this manual method of sample introduction.

Purge/Trap - Water. Sample water (5 mL) is transferred to a 10 mL gas tight syringe and the working surrogate standard spike is added, via a gas tight syringe, into the 5 mL water sample. Immediately inject this water into the purge vessel of the purge/trap device. If necessary, a smaller sample aliquot may be used in order to remain within the linear calibration range of the instrument.

Larger sample volumes may be analyzed, at the discretion of the analyst, if lower quantitation limits are required. Autosampler techniques may be substituted for this manual method of sample introduction.

Direct Injection - For Soil. Allow the extract to come to room temperature, then inject, either manually or by autosampler, 2 uL of the extract into the GC using the splitless injection mode.

Quantitation

The retention time range (window) for gasoline integration must, at a minimum, include toluene through naphthalene. For other volatile petroleum products, the retention time range for integration must be adjusted to incorporate the majority of the components of the petroleum product(s) identified as present in the samples. If specific product identification can not be made, the analyst must quantitate the samples with the calibration curve of the petroleum product that most closely resembles that of the sample.

For those surrogates which elute within the retention time range used for integration, the analyst must subtract the area of the surrogate(s) from the total area to yield the appropriate area of the petroleum product.

The analyst shall use regular unleaded gasoline as the default petroleum product for reporting purposes when no petroleum products were identified in any initial screening or when the type(s) of petroleum products are unknown prior to analysis.

Sample chromatograms of various volatile petroleum products are included at the end of this method to assist the analyst in determining the appropriate integration ranges.

Result Calculation

The area of the components is integrated, as a group, to the baseline and compared to concentrations of the standards which are integrated in the same manner.

For Soil

$$\textit{Soil Sample Conc, mg / kg} = \frac{(A \times R) \times D}{E \times W \times S}$$

where

A = group area of sample

R = Response factor from std curve (ng injected/area count)

D = Extract Volume, mL

W = Weight of sample, grams

E = Volume of extract purged or injected, uL

S = Decimal percent solids of sample

For Water

$$\textit{Water Sample Conc, ug / L} = \frac{(A \times R)}{V}$$

where

A = group area of sample

R = Response factor from std curve (ng purged/area count)

V = Volume of sample purged, mL

The recovery of the surrogate should be between 50% and 150% and must be reported with the results. Report any surrogate recoveries that can not be calculated due to a high level of gasoline contamination.

Author: Bob Carrell, Manchester Environmental Laboratory, Dept. of Ecology, State of Washington. Reviewed and edited by Steve Robb, Toxics Cleanup Program, Department of Ecology, State of Washington. This method is based on Oregon's Department of Environmental Quality TPH-G and Washington's Department of Ecology WTPH-G methods.

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NWTPH-Dx

Semi-Volatile Petroleum Products Method for Soil and Water

Summary

The NWTPH-Dx method is intended to replace the Oregon's Department of Environmental Quality TPH-D and Washington's Department of Ecology WTPH-D methods and to present a more comprehensive approach to semi-volatile petroleum product analyses. NWTPH-Dx adapts Oregon's TPH, Washington's WTPH and EPA SW-846 Methods 3510, 3540/3550 and 8000 and covers the quantitative and qualitative analysis of semi-volatile petroleum products, i.e. jet fuels through heavy fuel oils, in soil and water. The method involves extracting the samples with methylene chloride and injecting a portion of the extract into a gas chromatograph (GC) equipped with a flame ionization detector (FID). This method specifies criteria for the identification and quantitation of semi-volatile petroleum products. A clean-up procedure, which may be used to aid in the removal of non-petroleum based organic interferences, i.e. biogenic interferences, has been included. When the type of petroleum product is unknown, #2 diesel will initially be used as the default petroleum standard.

The reporting limits are 25 mg/kg (soil) and 0.25 mg/L (water) for the petroleum products in the elution range of jet fuels through #2 diesel. For petroleum products eluting after #2 diesel oil, e.g. motor oils, hydraulic fluids, and heavy fuel oils, the reporting limits are 100 mg/kg (soil) and 0.50 mg/L (water). All soil results are reported on a dry weight basis. Since this value assumes 100% solids and therefore will be higher depending on the actual moisture content, the analyst is permitted to concentrate the extract to obtain these reporting limits. When doubt exists as to which reporting limit is applicable for the petroleum product present, the analyst should use the lower value.

The method is applicable for the identification, by pattern matching ("fingerprinting"), and quantitation of semi-volatile petroleum products. These include kerosenes, jet fuels, diesel oils, fuel oils, lubricating oils, hydraulic fluids, mineral oils and insulating oils, e.g. transformer oils. In general, those petroleum products which do not contain a substantial volatile fraction, i.e. the majority of the components eluting outside of the gasoline range, should be analyzed by this method.

Note: The use of GC/MS (Mass Spectrometry) or GC/AED (Atomic Emission Detector) may be substituted for GC/FID as long as all other method parameters are met.

This method is to be used by, or under the direct supervision of, analysts experienced in the use of GC and in the interpretation of gas chromatograms of both fresh and weathered petroleum products.

Equipment and Reagents

Gas Chromatograph, w/wo Autosampler
Flame Ionization Detector
Capillary Split/Splitless Injector
Suggested Column:
J & W Scientific: DB-1 or DB-5, 30 M x 0.25 mm or 0.32 mm I.D. with 0.25 um film thickness capillary column or equivalent
Chromatographic Data System: Capable of group integrations
Analytical Balance, accurate to a least 0.0001 grams
Volumetric Flasks, 10 mL, ground glass stoppered
N-Evap Concentrator or equivalent
Centrifuge tubes, 10 or 15 mL, glass, calibrated in 0.1 mL increments
Centrifuge tubes, 10 or 15 mL, glass, disposable
Kaderna-Danish (K-D) Flasks, 250 mL
Concentrator Tubes, 10 mL
Snyder Columns, 3-ball, 300 mm length
Sodium Sulfate, anhydrous
Methylene Chloride, Burdick and Jackson brand, gas chromatography/pesticide residue grade or equivalent
Sulfuric acid, concentrated
Silica gel, 100/200 mesh, Baker Analyzed Reagent grade or equivalent - Before use, activate for at least 16 hours at 130 degrees C in a shallow tray
Petroleum Product Standards: Available from commercial sources

Note: All samples shall be collected in Eagle Picher, or equivalent, glass jars and held at 4 degrees C until extracted. The holding time, from the date of collection to extraction, is 14 days for soil and preserved water. For unpreserved water, the holding the holding time is 7 days. Preservation is accomplished by adjusting the pH of the water sample to approximately 2 with the addition of 1+1 HCl.

Suggested GC Parameters

Sample Extract Injection Volume = 2 uL
Injector Temperature = 290 degrees C
Detector Temperature = 300 degrees C
Hydrogen Flow = 25-35 cc/min
Air Flow = 300-400 cc/min
Helium Make-up Gas Flow = 30 cc/min
Helium Carrier Gas Head Pressure = 15 psi
GC Temperature Program:
Initial temperature = 50 degrees C, hold 2 minutes
Temperature Ramp Rate = 20 degrees C per minute
Final Temperature = 320 degrees C, hold for 10 minutes

Standards

Reference/Stock Standards. Prepare individual petroleum product reference/stock standards, e.g. kerosene, #2 diesel oil, transformer oil (mineral oil based) and Bunker-C fuel oil.

Add 5 to 10 drops of the pure petroleum product to a zero tared 10 mL flask. Record the weight and bring the flask to volume with methylene chloride, stopper and mix by inverting the flask several times. Calculate the concentration of these standards using the equation shown below. The use of commercially prepared standards is an acceptable alternative to the above procedure. Analysts may not use artificial standards, e.g. diesel range organics mixtures, etc., for quantitation purposes in place of authentic petroleum products.

These standards are to be used to produce calibration working standards which should be used to insure the proper identification of petroleum products by chromatographic pattern matching ("fingerprinting") as well as accurate quantitation.

$$\textit{Stock Conc, ug / mL} = \frac{\textit{(final wt, mg) - (tare wt, mg)}}{\textit{10 mL}} \times \frac{\textit{1000 ug}}{\textit{mg}}$$

Calibration Working Standards. Using the stock standards, prepare calibration working standards for the identified petroleum product(s) to be quantitated. Add the appropriate volume(s), using the equation shown below and adjusting for the concentration change created by any serial dilutions, to a 10 mL volumetric flask(s). Dilute to volume with methylene chloride. Calibration standards must, at a minimum, (1) provide a five point calibration curve, (2) include a sufficiently low standard to provide the necessary reporting limits, and (3) define the linear working range of the instrument.

In order to be acceptable, the calibration curve must have a linear correlation coefficient of at least 0.990 and none of the standards may vary from their true (known) value by more than plus/minus 15%. #2 diesel oil is the default petroleum product for reporting purposes.

Stock Surrogate Standard. Prepare the stock surrogate standard by weighing 50 mg of the surrogate compound(s) into a 10 mL volumetric flask. Bring the flask to volume with methylene chloride for a final concentration of 5000 ug/mL for the surrogate compound. The use of commercially prepared surrogate solutions is an acceptable alternative to the above procedure.

Note: The suggested surrogates are 2-fluorobiphenyl, o, or p-terphenyl or pentacosane. The use of other surrogates is optional. Selected surrogate compounds must be non-polar, unaffected by the cleanup procedure, i.e. the concentrated sulfuric acid/silica gel treatment, and lacking in significant interferences in most standard petroleum products.

Working Surrogate Spike. Using serial dilutions of the stock standard, prepare a surrogate working standard. Add the appropriate volume of the stock surrogate standard, using the equation listed

below, and adjusting for any serial dilutions, to a 10 mL volumetric flask and dilute to volume with methylene chloride. Stopper and mix by inverting the flask several times. The surrogate working standard should be added to a level sufficient to produce a surrogate concentration between 5 and 50 ug/mL.

$$Volume\ Stock,\ uL\ =\ \frac{(Cal\ Std\ Conc,\ ug\ /\ mL)\ x\ 10\ mL}{Stock\ Conc,\ ug\ /\ mL}\ x\ \frac{1000\ uL}{mL}$$

Store all standards in a refrigerator until needed. Allow them to come to room temperature prior to use.

Sample Extraction

Soil Matrix

Weigh approximately 20 grams of soil, recording the weight to the nearest 0.01 grams, and approximately 20 grams of anhydrous sodium sulfate into a 150 mL beaker. Mix completely with a spatula. The mixture should have a grainy texture. If it forms a large clump, add more anhydrous sodium sulfate and grind to grainy texture. Add the appropriate volume of working surrogate standard, 50 mL of methylene chloride and sonicate for 3 minutes utilizing the horn sonicator and power settings in SW-846 Method 3550. Allow the mixture to settle then collect the extract in a 250 mL Kuderna-Danish (KD) flask to which is connected a 10 mL concentrator tube.

Repeat the extraction twice more and add these extracts to the KD. Attach a 3 ball Snyder column and concentrate the extract on a steam bath to a volume of 5-10 mL. Allow the K-D to cool to room temperature. Disassemble the K-D, rinsing the Snyder/K-D and K-D/concentrator tube joints with 1-2 mL of methylene chloride. Add these rinsings to the extract. If necessary, place the concentrator tube in an N-Evap and reduce the volume to 10 mL under a gentle stream of nitrogen. At this point, proceed to the sample cleanup procedure if applicable or transfer a portion of the extract to a 2 mL autosampler vial fitted with a screw top and a Teflon lined septum. Store the extract in a refrigerator until analyzed. If the extract is highly colored or forms a precipitate, a dilution may be necessary to stay within the calibration range. The use of the EPA method 3540 (soxhlet) in place of Method 3550 is optional.

Determine the moisture content of the samples by the following method. Immediately after weighing the sample for extraction, weigh approximately 10 grams of the sample into a tared crucible and record the weight. Dry the sample/crucible overnight at 105 degrees C. Reweigh the sample/crucible after allowing it to cool to room temperature and record the weight. Calculate the % solids as follows: [(grams of dry sample/grams of wet sample) x 100].

Along with each sample set, run at least one duplicate sample per set of 10 or fewer samples (10%) and, for each extraction day, at least one method blank (5%). Spiking of surrogates, extraction and

analyses of the QC samples will be conducted identically to the regular samples with the exception that no soil is added to the method blank.

Water Matrix

Allow the sample to come to room temperature and mark the meniscus for later use in volume determination. Pour the sample into a separartory funnel and adjust the pH to approximately 2 with 1+1 HCl and add the appropriate volume of surrogate working solution. Add 30 mL of methylene chloride to the sample jar and rotate the jar at a sufficient angle to wash the walls. Pour the solvent into the separatory funnel, stopper, and shake it vigorously for one minute, venting frequently. After the two phases have separated, drain the solvent into a 250 mL K-D flask to which is attached a 10 mL concentrator tube.

Note: Due to possible loss of analytes from the water to the sample jar walls, the entire sample must be consumed in the extraction and no aliquots may be used. Since the reporting limits are calculated on a 400 mL sample volume, sample jar size should be appropriate for this volume. For larger sample volume extractions, the analyst must increase the quantity of solvent used to maintain the original solvent/sample ratio.

Repeat the extraction twice more and add these extracts to the K-D. Attach a 3-ball Snyder column to the K-D and concentrate the extract on a steam bath to 5-10 mL. Allow the K-D to cool to room temperature and disassemble it, rinsing the Snyder/K-D and K-D/ concentrator joints with 1-2 mL of methylene chloride. Add these rinsings to the extract. Place the concentrator tube into an N-Evap and reduce the volume to 2 mL under a gentle stream of nitrogen. Transfer the extract to a 2 mL autosampler vial fitted with a screw top and a Teflon lined septum. Store the extract in a refrigerator until analyzed.

Along with each sample set, run at least one duplicate sample per set of 10 or fewer samples (10%) and, for each extraction day, at least one method blank (5%). Spiking of surrogates, extraction and analyses of the QC samples will be conducted identically to the regular samples with the exception that organic free water will be used for the method blank.

As more information becomes available on new extraction techniques, Washington State Dept. of Ecology's Manchester Laboratory and/or Oregon’s Department of Environmental Quality will publish descriptions of acceptable alternative extraction methods.

Sample Cleanup. In those cases where samples contain a significant amount of naturally occurring non-petroleum organics, e.g. leaf litter, bark, etc., which may contribute biogenic interferences, the following cleanup technique may be employed to assist in their reduction or elimination.

Transfer the 10 mL sample extract to a 10 to 15 mL centrifuge tube, add 1 mL of concentrated sulfuric acid to the extract and stopper the tube. Mix thoroughly for 1 minute by either shaking the tube or with the use of a vortex-genie adjusted to the highest setting.

Caution: Since sulfuric acid produces a highly exothermic reaction with water and other polar materials, extreme care should be exercised with its use.

Allow the two phases to separate. Centrifugation can be used to facilitate this process. Using a disposable glass pipet, transfer the methylene chloride (top) phase to another centrifuge tube and add approximately 0.4 grams (roughly equivalent to 1 mL of volume) of silica gel to the tube, stopper and mix as before. Allow the silica gel to settle or centrifuge. Repeat the sulfuric acid/silica gel treatment once more. Transfer a portion of the extract to a 2 mL autosampler vial equipped with a Teflon-lined cap and store the extract in a refrigerator until analyzed. A smaller aliquot of the extract may be used for this cleanup procedure as long as the ratio of extract to acid/silica gel is maintained.

It has been noted that some petroleum products, i.e. heavy fuel oils such as #6 fuel oil or Bunker-C, may experience a concentration loss of between 10 and 20 percent when subjected to this cleanup technique. This loss appears to be primarily associated with the removal of petroleum compounds which contain sulfur. To account for this loss when analyzing samples that have been subjected to the cleanup procedure in preparation for heavy fuel oil determination, the analyst must use utilize standards which have undergone the cleanup technique to calibrate the GC.

Note: The use of EPA method 3611 (Alumina column cleanup) may be substituted for the above cleanup technique if it is demonstrated to provide equivalent results.

Analysis Procedure. Prior to the analysis of any samples or method blanks, the analyst must prepare and analyze a mid-range calibration check standard to insure that the instrument is functioning correctly and that the calibration is still valid. The value obtained for this analysis must not vary from the true (known) value by more than plus/minus 15%. If the value falls outside this range then a second mid-range calibration check standard should be analyzed. If the analysis of the second check standard fails to meet the acceptance criteria, then the instrument must be recalibrated prior to the analysis of any samples. Once the instrument has been shown to be in calibration, the analyses of samples may proceed.

The analyst shall use #2 diesel as the default petroleum product for reporting purposes when no petroleum products were identified in any initial screening or when the type(s) of petroleum products are unknown prior to analysis.

After the last sample has been analyzed, a mid-range calibration check sample must be run to demonstrate that the instrument is still operating within the required parameters. Should this standard fail to meet those parameters, then all samples analyzed after the last successful calibration check standard must be reanalyzed. An increase in the frequency of mid-range calibration check standard analyses beyond the minimum required is recommended.

Qualitative Analysis - Identification

If NWTPH-HCID has not been previously performed on the samples and/or the type of petroleum present is unknown, the analyst should pre-screen the samples to determine the petroleum product.

The observed petroleum product shall be determined by pattern matching with the standard(s) analyzed the same day. Chromatograms used for this "fingerprinting" should be normalized to approximately 90% of full scale for the largest component of the particular petroleum product observed.

When reporting the results, the terms such as "diesel range" or "motor oil range", or derivations of them, should only be used when the analyst is unable to identify the petroleum product(s) present. Motor oils, hydraulic fluids and similar petroleum products which consist primarily of an unresolved chromatographic envelope of compounds originating at, or extending beyond tetracosane, may be reported using the collective term "lube oil" unless specific identification is possible. Heavy fuel oils, e.g. #6 fuel oil or Bunker-C, which contain a diesel range component as well as a lube oil range, may be reported using the collective term "heavy fuel oil" unless specific identification is possible. Heavy fuel oils should not, however, be confused with mixtures of #2 diesel and lube oils.

Note: The actual identification of the grade or type of lube oil and/or heavy fuel oil may require equipment and techniques beyond the scope of this method.

Quantitative Analysis - Integration

The retention time range (window) for integration must be adjusted to incorporate the majority of the components of petroleum product(s) identified as present in the samples. If specific product identification can not be made, the analyst must quantitate the samples with the calibration curve for the petroleum product that most closely resembles that of the sample. In all cases, the selected retention time range (windows) used for quantitation must, at a minimum, include any unresolved envelope of compounds as well as all discrete component peaks with an area greater than or equal to 10% of the largest peak. These components must be integrated to the baseline as a group.

For those surrogates which elute within the retention time range used for integration of a petroleum product, the analyst must subtract the area of the surrogate from the total area to yield the appropriate area of the petroleum product. In this case, the analyst may wish to generate separate calibrations for the petroleum standards and the surrogate(s) to facilitate integration and quantitation.

At the discretion of the analyst, the range of components included in the integration may be adjusted in order to minimize the potential contribution of any co-eluting fractions arising from the presence of multiple petroleum products. Any change in the integration range must be reflected in a concomitant change to the calibration standards integration.

Sample chromatograms of various petroleum products are included at the end of this method to assist the analyst in determining the appropriate integration ranges.

Result Calculation

For Soil

$$Soil\ Sample\ Conc,\ mg / kg = \frac{(A \times R) \times V \times Dilution\ Factor}{E \times W \times S}$$

where

A = Area Count from Sample

R = Response Factor (ng injected/area count)

V = Extract Volume (mL)

W = Weight of Sample (g)

E = Volume injected, (uL)

S = Decimal percent solids of sample

For Water

$$Water\ Sample\ Conc,\ mg / L = \frac{(A \times R) \times V}{E \times S}$$

where

A = Area Count from Sample

R = Response Factor (ng injected/area count)

V = Extract Volume (mL)

S = Volume of Sample (mL)

E = Volume Injected (uL)

The recovery of the surrogate should be between 50% and 150% and must be reported with the results. If the recovery of the surrogate is not able to be obtained due to a high levels of petroleum contamination, then this fact needs to be reported.

Author: Bob Carrell, Manchester Environmental Laboratory, Dept. of Ecology, State of Washington. Reviewed and edited by Steve Robb, Toxics Cleanup Program, Department of Ecology, State of Washington. This method is based on Orgeon's Department of Environmental Quality TPH-D and Washington's Department of Ecology WTPH-D methods.

METHOD FOR THE DETERMINATION

OF

VOLATILE PETROLEUM HYDROCARBONS (VPH) FRACTIONS

Washington State Department of Ecology

June 1997

METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS (VPH)

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DISCLAIMER

Mention of trade names or commercial products does not constitute endorsement by the Washington State Department of Ecology Equipment and materials cited in this method may be replaced by similar products, as long as adequate data exists or has been produced documenting equivalent or superior performance.

METHOD FOR THE DETERMINATION OF VOLATILE PETROLEUM HYDROCARBONS

1.0 SCOPE AND APPLICATION

1.1 This method is designed to measure the collective concentrations of volatile aliphatic and aromatic petroleum hydrocarbons in water and soil. **The carbon ranges used through out this document are given in equivalent carbon (EC) numbers which are related to the boiling point of a chemical normalized to the boiling point of the n-alkanes, and its retention time in a boiling point gas chromatographic (GC) column.** Volatile aliphatic hydrocarbons are collectively quantitated within four ranges: C5 through C6, >C6 through C8, >C8 through C10 and > C10 through C12. Volatile aromatic hydrocarbons are collectively quantitated within the C8 through C10, >C10 through C12 and >C12 through C13 ranges. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 36°C and 220°C.

1.2 This method is also designed to measure the individual concentrations of benzene, toluene, ethylbenzene, xylenes, and methyl tert butylether (MTBE) in water and soil.

1.3 Petroleum products suitable for evaluation by this method include gasoline, mineral spirits, and certain petroleum naphthas. This method, in and of itself, is not suitable for the evaluation of samples contaminated with kerosene, jet fuel, heating oils, lubricating oils, or other petroleum products which contain a significant percentage of hydrocarbons larger than C10. When samples are known or suspected to contain petroleum products containing significant concentration of hydrocarbons >C10, the Extractable Petroleum Hydrocarbon (EPH) method should also be employed to fully evaluate the hydrocarbons present.

1.4 For reporting purposes, the practical quantitation limits (PQL), given the sample volume purged, mass and/or methanol extract volume purged are: 50.0 ug/L for water and 5.0 mg/kg for soil/sediments for the aliphatic and aromatic carbon ranges and 5.0 ug/L and 0.5 mg/kg respectively for the individually targeted compounds. The procedure for Method Detection Limits (MDL) determination in this method and the

Single Laboratory Precision, Accuracy and MDL data (generated by the State of Massachusetts) is added for informational purposes only.

1.5 This method is based on a purge-and-trap, gas chromatography (GC) procedure. This method should be used by, or under the supervision of, analysts experienced in the use of purge-and-trap systems and gas chromatographs. The analysts should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

1.6 Like all GC procedures, this method is subject to a "false positive" bias in the reporting of targeted analytes, in that non-petroleum compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated with the respective carbon ranges. Confirmatory analysis by a GC/MS, EPA Method 8260, or other suitable procedures are recommended in cases where significant concentrations of non-hydrocarbon compounds are known or suspected. If the results of these analyses lead to identification and quantitation of non-petroleum compounds, the analyst may subtract those values from the affected carbon ranges as long as the identities and quantities of subtracted compounds are provided in the analytical report.

2.0 SUMMARY OF METHOD

2.1 Samples are analyzed using purge-and-trap sample concentration. The gas chromatograph is temperature programmed to facilitate separation of organic compounds. Detection is achieved by a photo-ionization detector (PID) and flame ionization detector (FID) in series. Quantitation is based on comparing the PID and FID detector response of a sample to a standard comprised of aromatic and aliphatic hydrocarbons. The PID chromatogram is used to determine the individual concentrations of targeted analytes (BTEX/MTBE) and collective concentration of aromatic hydrocarbons within the C8 through C10, C10 through C12 and C12 through C13 ranges. The FID chromatogram is used to determine the collective concentration of aliphatic hydrocarbons within the C5 through C6, C6 through C8, C8 through C10 and C10 through C12 ranges. To avoid double counting of the aromatic contribution to the aliphatic ranges, the PID concentrations are subtracted from the FID concentrations to yield the aliphatic ranges values.

2.2 This method is suitable for the analysis of waters, soils, and sediments. Water samples may be analyzed directly for volatile petroleum hydrocarbons by purge-and-trap concentration and gas chromatography. Soil samples are either dispersed in methanol to dissolve the volatile organic constituents and a portion of the methanol is analyzed by purge-and-trap GC or combined with water for purging directly from a soil purge vessel (EPA method 5035).

2.3 This method is based on, and constitutes a significant modification of, the “Method for the Determination of Volatile Petroleum Hydrocarbons (VPH)”, Public Comment Draft 1.0, developed by the Massachusetts Department of Environmental Protection and on EPA method 5035. They in turn based their method on (1) USEPA Methods

5030, 8000, 8020, and 8015, SW-846, "Test Methods for Evaluating Solid Wastes", 3rd Edition, 1986; (2) Draft "Method for Determination of Gasoline Range Organics", EPA UST Workgroup, November, 1990; and (3) "Method for Determining Gasoline Range Organics", Wisconsin Department of Natural Resources, PUBL-SW-140, 1992.

3.0 DEFINITIONS

3.1 **Volatile Petroleum Hydrocarbons (VPH)** are defined as all hydrocarbon compounds eluting just prior to n-pentane through 1-methylnaphthalene. VPH is comprised of C5 through C6, >C6 through C8, >C8 through C10 and >C10 through C12 Aliphatic Hydrocarbons, as well as >C8 through C10, >C10 through C12 and >C12 through C13 Aromatic Hydrocarbons as well as benzene and toluene. VPH concentration data are reported as the aggregate concentrations of the aliphatic and aromatic hydrocarbon ranges and as selected targeted analytes.

3.2 **Equivalent Total Petroleum Hydrocarbons (E-TPH)** For samples contaminated ONLY with gasoline or other low molecular weight petroleum products, the E-TPH value is equivalent to the VPH value. For samples contaminated with BOTH light and heavy molecular weight petroleum products (e.g., gasoline and diesel fuel), the E-TPH value is a summation of the VPH value and the Extractable Petroleum Hydrocarbon (EPH) values. In order to avoid double counting of analytes due to the overlap in the carbon ranges existing between the two methods, the analyst will report the highest of the two values determined for the overlapping ranges. In those cases where both the VPH and EPH methods are employed on samples, the analyst may select to quantitate the >C10 through C12 Aliphatic and the >C10 through C12 Aromatic Hydrocarbon ranges only using the EPH method.

3.3 **C5 through C6 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds which elute on the FID chromatogram from (and including) n-pentane through n-hexane.

3.4 **C6 through C8 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbons which elute on the FID chromatogram after n-hexane through n-octane.

3.5 **C8 through C10 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds which elute on the FID chromatogram after n-octane through n-decane.

3.6 **C10 through C12 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbons which elute on the FID chromatogram after n-decane through n-dodecane.

3.7 **C8 through C10 Aromatic Hydrocarbons** are defined as all hydrocarbon compounds which elute on the PID chromatogram after toluene through 1,2,3-trimethylbenzene.

3.8 **C10 through C12 Aromatic Hydrocarbons** are defined as all hydrocarbons which elute on the PID chromatogram after 1,2,3- trimethylbenzene through naphthalene.

3.9 **C12 through C13 Aromatic Hydrocarbons** are defined as all hydrocarbons which elute on the PID chromatogram after naphthalene through 1-methylnaphthalene. This is a hybrid range which is designed to acquire the methylnaphthalenes associated with petroleum products like gasoline and is only used when VPH is run without an accompanying EPH method request.

3.10 **Targeted VPH Analytes** are defined as benzene, toluene, ethylbenzene, p,m,o-xylenes, and MTBE.

3.11 **Volatile Petroleum Hydrocarbon (VPH) Component Standard** is defined as a 15 component mixture of the aliphatic and aromatic compounds, plus surrogate, listed in Table 1. The compounds comprising the VPH Component Standard are used to (a) define the individual retention times and chromatographic response factors for each of the Targeted VPH Analytes, (b) define and establish the windows for the collective aliphatic and aromatic hydrocarbon ranges of interest, and (c) determine average chromatographic response factors that can in turn be used to calculate the collective concentration of hydrocarbons within these ranges.

3.12 **Analytical Batch** is defined as a group of 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.

3.13 **Laboratory Duplicates** are defined as split samples taken from the same sampling container and analyzed separately with identical procedures. The analysis of laboratory duplicates give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.14 **Field Duplicates** are defined as two separate samples collected at the same time and place under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation, and storage, as well as laboratory procedures.

3.15 **E-TPH Duplicates** are defined as two separate samples collected at the same time and location, for analysis by both EPH and VPH methods. E-TPH duplicates are taken at locations where significant concentrations of petroleum hydrocarbons lighter and heavier than C9 are likely to be present (e.g., locations contaminated by releases of both gasoline and diesel fuel). The resultant EPH and VPH concentrations, adjusted to prevent double counting of overlapping hydrocarbon ranges between the methods, are then summed to determine the Equivalent TPH (E-TPH) concentration.

3.16 **Calibration Standards** are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compound of interest.

3.17 **Calibration Check Standard** 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 solution as calibration standards, and is generally one of the mid-level range calibration standard dilutions.

Table 1. Volatile Petroleum Hydrocarbon (VPH) Component Standard

Compound	Equivalent Carbon Number
n-Pentane	5.0
n-Hexane	6.0
Methyl tert butylether	N/A
Benzene	6.5
Toluene	7.6
n-Octane	8.0
Ethylbenzene	8.5
m- & p- Xylene	8.6
o-Xylene	8.8
1,2,3,-Trimethylbenzene	10.1
n-Decane	10.0
Naphthalene	11.7
n-Dodecane	12.0
1-Methylnaphthalene	13.0
2,5-Dibromotoluene (surrogate)	N/A

3.18 **Matrix Spiking Solution** is defined as a solution which is generally prepared independently from the calibration standards, containing known concentrations of method analytes.

3.19 **Laboratory Method Blank** is defined as, depending on the matrix of the samples, either reagent water or clean sand spiked with a surrogate standard. The laboratory method blank is treated identically as with samples, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is analyzed with every batch of samples, to determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

3.20 **Laboratory Fortified Blank (LFB)** is defined as, depending on the matrix of the samples, either reagent water or a clean sand blank fortified with a matrix spiking solution.

The LFB is treated and analyzed identically as with samples and blanks, and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required practical quantitation limits..

3.21 **Laboratory Fortified Matrix (LFM) Sample** is defined as an environmental sample which has been spiked with a matrix spiking solution containing known concentrations of method analytes. The LFM sample is treated and analyzed exactly as a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined through the separate analyses of a laboratory or field duplicate, and the measured values in the LFM sample corrected for background concentrations.

3.22 All other terms are as defined in SW-846, "Test Method for Evaluating Solid Waste", USEPA, September, 1986, and as amended.

4.0 INTERFERENCES

4.1 Samples can become contaminated by diffusion of volatile organics through the sample container septum during shipment and storage. Trip blanks prepared from reagent water should be carried through sampling and subsequent storage and handling to serve as a check on such contamination.

4.2 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. To reduce carryover, the sample syringe and/or purging device must be rinsed between samples with reagent water or solvent. The trap and other parts of the system are also subject to contamination, therefore, frequent bake-out and purging of the entire system may be required. A screening step is recommended to protect analytical instrumentation. Whenever an unusually concentrated sample is encountered, it must be followed by the analysis of a solvent blank to check for cross-contamination.

4.3 Certain organic compounds not associated with releases of petroleum products, including chlorinated solvents, ketones, and ethers, will be quantitated as Volatile Petroleum Hydrocarbons. Some samples may require additional analytical procedures to be employed , e.g. GC/MS, to document the presence and quantity of such compounds.

4.4 The response selectivity of a photo-ionization detector (PID) is used in this method to differentiate aromatic hydrocarbons from aliphatic hydrocarbons. All compounds eluting on the PID chromatogram after toluene are identified by the method as aromatic hydrocarbons. This will lead to an overestimation of aromatic hydrocarbons within samples, as certain aliphatic compounds will elicit a response on the PID, particularly unsaturated compounds such as alkenes. The significance and implications of this overestimation will vary from sample to sample and, where less conservative data are desired, additional actions should be considered to

minimize the detection of non-aromatic compounds, such as the use of a lower energy PID lamp or GC/MS.

5.0 HEALTH AND SAFETY ISSUES

The toxicity and carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets should also be made available to all personnel involved in the chemical analysis.

6.0 APPARATUS AND MATERIALS

6.1 The following glassware is used in this method:

6.1.1 VOC Vials: Wide mouth 60-mL VOC vials or 40-mL VOC vials with teflon/ silicone septa for soils; 40-mL VOC vials with teflon/silicone septa for waters.

6.1.2 Volumetric flasks: 10-mL, 50-mL, 100-mL, and 1,000-mL with a ground-glass stopper.

6.1.3 Disposal pipets: Pasteur.

6.2 Analytical balance: An analytical balance capable of accurately weighing 0.0001 g must be used for weighing standards. A top-loading balance capable of weighing to the nearest 0.1 g must be used for weighing soil samples.

6.3 **Gas Chromatography**

6.3.1 Gas Chromatograph: An analytical system complete with temperature programmable gas chromatograph and purge-and-trap concentrator. The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection.

6.3.2 Columns

6.3.2.1 Recommended column: 105M x 0.53 mm I.D. Restek RTX 502.2, 3.0 micron film thickness, or equivalent.

6.3.2.2 Other columns, such as a 60 M x 0.53 mm J&W DB-5, may be used. Capillary columns are required to achieve necessary resolution. The column must be capable of resolving typical gasoline components. It must also resolve ethylbenzene from m/p-xylene. Some columns may require subambient cooling to achieve these criteria.

6.3.3 Detectors: The method utilizes a Photo-ionization Detector (PID) in series with a Flame Ionization Detector (FID); the PID first in the series. The method is based upon the use of a 10.0 eV PID lamp, although lower energy lamps are permissible in order to minimize PID response to aliphatic compounds.

6.3.4 Purge-and-trap device: The purge-and-trap device consists of a sample purger, a trap, and a desorber. Several complete devices are commercially available.

6.3.4.1 The purging chamber must be designed to accept 5 mL samples with a water column at least 3 cm deep. Purging devices larger than 5 mL have a reduced purging efficiency and should not be used. The gaseous headspace between the water column and the top of the vessel should be at least 3 cm deep. The gaseous headspace between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. Fritted glass or needle sparge cells may be used. If needle sparge cells are used, the purge gas must be introduced no more than 5 mm from the base of the water column. Alternate sample purge devices may be used, provided equivalent performance is demonstrated.

6.3.4.2 The trap should be at least 25 cm long and have an inside diameter of at least 0.105 inches. The trap should be packed with 400 mg of Carbopack B (Supelco Cat. No. 209273). Alternative trap packing materials include: Tenax GC (or equivalent); 7.6 cm Carbopack B and 1.3 cm Carbosieve S-III (Supelco Cat No. 2-0321); 7 cm Carbopack C and 1.2 cm Carbopack B (Supelco Cat No. 2-1064); or equal volumes of Tenax, silica gel, and charcoal as described in EPA SW-846 Method 5030. In general, Carbopack trap packing materials are recommended because they have less of a tendency to retain methanol, which could interfere with the elution of pentane and quench the FID flame. The trap length and packing materials may be varied as long as equivalent performance has been verified.

6.3.4.3 Prior to initial use, the Carbopack B trap should be conditioned overnight at 270°C by backflushing with an inert gas flow of at least 20 mL/min. Vent the trap effluent to a hood, not to the analytical column. Prior to daily use, the trap should be conditioned for 10 min. at 260°C with backflushing. The trap may be vented to the analytical column during daily conditioning, however, the column must be run through the temperature program prior to analysis of samples. Devices other than the traps recommended in Section 6.3.4.2 should be conditioned and desorbed according to the manufacturer's guidelines.

6.3.4.4 The desorber should be capable of rapidly heating the trap to 240°C for desorption.

6.4 Ultrasonic bath.

6.5 Syringes: 5-mL Luerlock glass hypodermic and 5-mL gas-tight syringe with shutoff valve.

6.6 Syringe valve: Two-way, with luer ends.

6.7 Microsyringes: 1-µL, 5-µL, 10-µL, 25-µL, 100-µL, 250-µL, 500-µL, and 1,000-µL.

6.8 Spatula: Stainless steel.

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**

7.1.1 Reagent Water: organic free water (ASTM Type I reagent grade water).

7.1.2 Solvent: methanol; purge and trap grade or equivalent. Store away from other solvents.

7.1.3 Ottawa and/or masonry sand: free of volatile petroleum hydrocarbons.

7.2 **Stock Standard Solution**

Prepare a stock standard solution in methanol at approximately 20 µg/µL, or purchase certified solutions. Preparation of stock standards and component standards should be done using volumetric glassware.

7.2.1 The stock standard solution consists of the 15 VPH component standards listed in Table 1 and a surrogate standard. Prepare the stock standard solution by accurately weighing approximately 0.2000 g of each standard component. Dissolve the component in methanol and dilute to volume in a 10-mL volumetric flask. At the discretion of the analyst, the surrogate standard may be made up as a separate solution from the VPH standards.

7.2.1.1 Place about 8 mL of methanol in a 10-mL tared ground-glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min. or until all alcohol-wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

7.2.1.2 Using a 500-µL syringe, immediately add 100 to 200 µL of each VPH Component Standard to the flask; then reweigh. The liquid must fall directly into the alcohol without contacting the neck of the flask.

7.2.1.3 Dilute to volume, stopper, and then mix by inverting the flask three times. Calculate the concentration in micrograms per microliter (µg/µL) from the net gain in weight. When compound purity is assayed to be 98% or greater, the weight may be used without correction to calculate the

concentration of the stock standard, provided such purities are certified by the manufacturer or by an independent source.

7.2.1.4 Transfer the stock standard solution into a Teflon-lined screw-cap/crimp cap bottle. Store, with minimal headspace, at -10°C to -20°C and protect from light.

7.2.2 Stock standard solutions must be replaced after 6 months, or sooner if comparison with check standards indicates a problem. The use of commercially prepared certified standard solutions is an acceptable alternative to the use of neat compounds in the preparation of standards.

7.3 **Primary Dilution Standard**

Using the stock standard solution, prepare primary dilution standards in methanol, as needed. The primary dilution standard should be prepared at the concentrations shown in Table 2. These standards should be stored with minimal headspace, at -10°C to -20°, and should be checked frequently for signs of degradation or evaporation. The analyst is allowed to prepare separate surrogate and VPH analyte standards.

The primary dilution standards should be replaced at least monthly.

7.4 **VPH Calibration Standards**

Prepare VPH Calibration standards in reagent water from the primary dilution standard at a minimum of five concentration levels. One of the concentration levels must be near, but above, the method detection limit and must be at a sufficiently low level to allow the PQL reporting of at least 5.0 ug/L for water and 0.5 mg/kg for soil/sediments for the targeted analytes (BTEX and MTBE) and 50.0 ug/L and 5.0 mg/kg respectively for the aliphatic and aromatic carbon ranges. The other standard concentrations must correspond to the expected range of concentrations found in real samples and/or should define the working range of the detector.

7.4.1 Rapidly inject the methanolic standard into the water in the expanded area of a filled 100-mL volumetric flask. Remove the needle quickly after injection.

7.4.2 Mix aqueous standards by inverting the flask three times.

7.4.3 Discard the solution contained in the neck of the flask, and fill the sample syringe from the standard solution contained in the expanded area of the flask.

7.4.4 Do not use pipets to dilute or transfer samples or aqueous standards.

7.4.5 Do not inject more than 20 µL of methanolic standards into 100 mL of reagent water. Aqueous standards are not stable and should be discarded after one hour.

7.5 **Surrogate Control Standard (SCS)**

The analyst must monitor both the performance of the analytical system and the effectiveness of the method in dealing with sample matrices by spiking each sample, blank, and matrix spike with a surrogate standard. The surrogate standard is also added to the VPH calibration standard solutions. The recommended surrogate standard is 2,5-dibromotoluene, which elutes after all aliphatic and aromatic compounds of interest. The use of additional surrogates or surrogates other than those listed above may be used, at the discretion of the analyst, as long as their performance in the method is demonstrated as acceptable and does not compromise the quantitation of the various target analytes or carbon ranges.

7.5.1 Surrogate Spiking Solution: From a stock standard solution prepared as in Section 7.2.1, prepare a surrogate spiking solution at 50 µg/mL in methanol. Add 4.0 µL of this surrogate spiking solution directly into the 5-mL syringe with every aqueous sample, blank, and matrix spike. 1.0 mL of the surrogate spiking solution is added to soil samples during the extraction step (See 9.1.2.2).

7.6 **Matrix Spiking Solution**

The recommended matrix spiking solution, consisting of the Targeted VPH Analytes, is prepared in methanol at concentrations of 50 µg/mL.

7.7 **Petroleum Reference Standard**

The use of a Petroleum Reference Standard is recommended for quality control purposes. The Petroleum Reference Standard consists of an API or commercial gasoline standard. Prepare a Petroleum Reference Standard Spiking Solution by accurately weighing approximately 0.0100 g of neat product. Dissolve the neat product in methanol and dilute to volume in a 10-mL volumetric flask.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 **Aqueous Samples**

8.1.1 Aqueous samples should be collected in duplicate (or the number of vials directed by the laboratory) without agitation and without headspace in contaminant-free amber glass VOC vials with Teflon-lined septa screw caps. Samples must be acidified to a pH of 2.0 or less at the time of collection and this can generally be accomplished by adding 3 or 4 drops (0.1 to 0.2 mL) of 1:1 HCl (1 part reagent water and 1 part concentrated HCl) to a 40-mL VOC vial. Samples must be cooled to 4°C immediately after collection.

8.1.2 A chain of custody form should accompany all sampling vials and should document the date and time of sample collection and acid preservation. The pH of all water samples must be determined by the laboratory unless sample vials containing acid for field preservation were supplied by the laboratory (this must be noted on the chain of custody). The pH measurement may be performed on left over sample. Any sample found to contain a pH above 2.0 should be so noted on the laboratory/data report sheet.

8.1.3 Any sample received by the laboratory that is not packed in ice or cooled to 4°C must be so noted on the laboratory/data report sheet.

8.1.4 Acid preserved aqueous samples must be analyzed within 14 days of collection. Aqueous samples which, for whatever reason, have not received acid preservation must be analyzed within 7 days of collection or the data must be reported as an “estimate quantity”.

8.2 **Soil Samples**

8.2.1 Soil samples must be collected in a manner that minimizes sample handling and agitation. All sediment must be removed from the glass threads of the vial to ensure an adequate seal. Samples must be cooled to 4°C immediately after collection.

8.2.3 Samples for VPH analysis should be collected in duplicate 2 or 4 ounce VOC jars with Teflon coated septum lined lids.

8.2.4 Sampling must be accomplished in a manner that ensures a minimum of headspace in the sample vial.

8.2.5 A chain of custody form should accompany all sampling vials and should document the date and time of sample collection.

8.2.6 Soil samples must be analyzed within 14 days of collection.

8.3 A summary of sample collection, preservation and holding times is provided in Table 2 .

Table 2 . Holding Times and Preservatives for VPH Samples

Matrix	Container	Preservation	Holding Time
Aqueous Samples	40-mL VOC vials w/ Teflon-lined septa screw caps	Add 3 to 4 drops of 1:1 HCl; cool to 4°C	14 days 7 days, if not preserved
Soil/Sediments Samples	VOC vials w/ Teflon-lined septa screw caps. 40-mL vials	cool to 4°C	14 days

9.0 PROCEDURE

9.1 **Sample Preparation and Purging**

9.1.1 It is recommended that samples known or suspected to have extremely high levels of volatile petroleum hydrocarbons be screened prior to analysis in order to establish the appropriate volume/mass to be used for analysis. This screening step may be analysis of a soil sample's methanol extract (diluted), the headspace method (SW-846 method 3810), the hexadecane extraction and screening method (SW-846 Method 3820) or other applicable method as determined by the analyst.

9.1.2 Water Samples

Introduce volatile compounds into the gas chromatograph using a purge-and-trap concentrator. The use of autosampling devices for sample introduction is recommended or the analyst may use the manual method outlined below.

9.1.2.1 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample or standard bottle, which has been allowed to come to ambient temperature, and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and

compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. This process of taking an aliquot destroys the validity of the liquid sample for future analysis; therefore, if there is only one 40-mL vial, the analyst should fill a second syringe at this time to protect against possible loss of sample integrity. This second sample is maintained only until such time when the analyst has determined that the first sample has been analyzed properly. Filling one 20-mL syringe would allow the use of only one syringe. If a second analysis is needed from a syringe, it must be analyzed within 24 hours. Care must be taken to prevent air from leaking into the syringe.

9.1.2.2 If necessary, samples should be diluted prior to injection into the purge chamber. In such cases, all steps must be performed without delay until the diluted sample is in a gas-tight syringe.

9.1.2.2.1 Dilutions may be made in volumetric flasks (10 mL to 100 mL). Select the volumetric flask that will allow for the necessary dilution. Intermediate dilutions may be necessary for highly concentrated samples.

9.1.2.2.2 Calculate the approximate volume of reagent water to be added to the volumetric flask selected and add slightly less than this volume of reagent water to the flask.

9.1.2.2.3 Inject the proper aliquot of sample from the syringe prepared in Paragraph 9.1.2.1 into the flask. Aliquots of less than 1 mL are not recommended. Dilute the sample to the mark with reagent water. Cap the flask, invert, and shake three times. Repeat the above procedure for additional dilutions. Alternatively the dilutions can be made directly in the glass syringe to avoid further loss of volatiles.

9.1.2.2.4 Fill a 5-mL syringe with diluted sample as in Paragraph 9.1.2.1.

9.1.2.3 Add 4.0 µL of the surrogate spiking solution through the valve bore of the syringe. Close the valves.

9.1.2.4 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

9.1.2.5 Close both valves and purge the sample for 11 min. Recommended purge and trap operating parameters are provided in Table 3.

Table 3. Suggested Purge and Trap Operating Parameters

Purge gas	Helium
Purge gas flow rate (mL/min)	40
Purge time (min)	11.0
Purge temperature	Ambient
Desorb temperature °C	250
Backflush inert gas flow (mL/min)	15-20

9.1.2.6 At the conclusion of the purge time, attach the trap to the chromatograph (if necessary), adjust the device to the desorb mode, and begin the gas chromatographic temperature program (in Section 9.2.1) and GC data acquisition. Concurrently, introduce the trapped materials to the gas chromatographic column by rapidly heating the trap to 240°C and backflushing the trap with inert gas between 15 and 20 mL/min for 4 minutes.

9.1.2.7 While the trap is desorbing into the gas chromatograph, empty the purging chamber. Wash the chamber with a minimum of two 5 mL flushes of reagent water (or methanol followed by reagent water) to avoid carryover of pollutant compounds into subsequent analyses.

9.1.2.8 After desorbing the sample, recondition the trap by returning the purge-and-trap device to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 260°C. After approximately 7 to 15 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. After a highly concentrated sample, a longer baking time may be necessary. When cool, the trap is ready for the next sample.

9.1.2.9 If the concentration of an analyte in a sample exceeds the calibration range, a dilution of the sample is required. If a sample analysis results in a saturated detector response for a compound, the analysis must be followed by one or more blank reagent water analyses. If the final blank analysis is not free of significant interferences, the system must be decontaminated. Sample analysis may not resume until a blank can be analyzed that is free of significant interferences.

9.1.2.10 All dilutions should keep the detector response of the major constituents (previously saturated peaks) in the upper half of the linear range of the calibration curve.

9.1.3 Soil/Sediments

Soil and sediment samples are extracted with methanol. An aliquot of the extract is added to reagent water and introduced into the gas chromatograph using a purge and trap concentrator. Should lower values for the targeted analytes be desired, the analyst may employ direct purging of the soil sample using the method outlined in EPA methods 5030 and 5035. The use of autosampling devices for sample introduction is recommended or the analyst may use the manual method outlined below.

9.1.3.1 Weigh the sample vial to 0.1 g in a top loading balance and add 4 to 5 grams of the sample to the vial, reseal and determine the weight of the soil/sediment sample.

9.1.3.2 Quickly add 9.0 mL of methanol and 1.0 mL of the surrogate spiking solution. Cap and shake the sample vial for 2 minutes.

9.1.3.3 Allow soil/sediment to settle, or centrifuge the vial, until a layer of methanol is apparent.

9.1.3.4 Using a microliter syringe, withdraw an appropriate aliquot of the methanol extract for sparging. Sample screening data can be used to determine the volume of methanol extract to add to the 5 mL of reagent water for analysis. All dilutions must keep the response of the major constituents in the upper half of the linear range of the calibration curve.

9.1.3.5 Remove the plunger from one 5.0-mL Luerlock type syringe equipped with a syringe valve and fill until overflowing with reagent water. Replace the plunger and compress the water to vent trapped air. Adjust the volume to allow for addition of the extract (e.g., for 100 µL of extract adjust to 4.9 mL). Pull the plunger to 5.0 mL for addition of the sample extract. Add the volume of methanol extract determined from screening (100 µL maximum).

9.1.3.6 Attach the syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber. Complete operations as specified in Paragraphs 9.1.2.5 through 9.1.2.8.

9.1.4 Proceed with the analysis as described in Sections 9.2 through 9.5. Analyze all laboratory method blanks and QC samples under the same conditions as that used for samples.

9.1.5 If the responses exceed the calibration or linear range of the system, use a smaller aliquot of methanol or aqueous sample.

9.1.6 Determination of Percent Moisture

9.1.6.1 Soil and sediment results must be reported on a dry-weight basis.

9.1.6.2 Transfer 5 to 10 g of sample into a tared crucible. The amount of material used to determine the percent moisture will depend on the amount of moisture present, thus relatively dry samples require more sample addition in order to achieve a significant weight change than wet samples. Dry the sample overnight in an oven at 105°C. Remove the sample from the oven and allow it to cool in a desiccator before reweighing. Reweigh and calculate the percent moisture of the sample using the equations provided in Section 9.6.2.2.

9.2 **GC Conditions**

9.2.1 Oven Program: Oven temperature 45°C, hold for 1 min, then to 100°C at 3°C/min, to 160°C at 8°C/min, to 230°C at 20°C/min; hold for 7.5 min. Conditions may be altered to improve resolution of volatile petroleum hydrocarbons.

9.2.2 Gas Flows: The recommended carrier gas is helium.

9.2.2.1 Carrier gas flow: 12.5 mL/min.

9.2.2.2 Air: 350 mL/min

9.2.2.3 Hydrogen: 30 mL/min

9.2.2.4 Make up gas flow: 17.5 mL/min

9.2.3 Miscellaneous:

9.2.3.1 FID temperature: 230°C

9.2.3.2 PID temperature: 230°C

9.2.3.3 Injection port temperature: 250°C

9.2.3.4 Column head pressure: 15 psi

9.3 **Retention Time Windows**

9.3.1 Before establishing retention time windows, make sure the GC system is within optimum operating conditions. Make three injections of the VPH Component Standard throughout the course of a 72-hr period. Serial injections over less than a 72-hr period may result in retention time windows that are too tight.

9.3.2 Calculate the standard deviation of the three absolute retention times for each individual compound in the VPH Component Standard. The retention time window is defined as plus or minus three times the standard deviation of the absolute retention times for each standard. However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.3.3 In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window.

9.3.4 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed. This data must be retained by the laboratory.

9.3.5 VPH retention time (Rt) windows are defined as beginning 0.1 minutes after the Rt of the beginning marker compound and ending 0.1 minutes after the Rt of the ending marker compound. The exception to this is the C5 through C6 aliphatic hydrocarbon range, where its Rt window is defined a beginning 0.1 minutes prior to the beginning marker compound and ending 0.1 minutes after the ending marker compound. VPH marker compounds and windows are summarized in Table 4 .

Table 4 . VPH Marker Compounds

Hydrocarbon Range	Beginning Marker Compound	Ending Marker Compound
C5-C6 Aliphatic Hydrocarbons (FID)	just before n-Pentane	just after n-Hexane
C6-C8 Aliphatic Hydrocarbons (FID)	just after n-Hexane	just after n-Octane
C8-C10 Aliphatic Hydrocarbons (FID)	just after n-Octane	just after n-Decane
C10-C12 Aliphatic Hydrocarbons (FID)	just after n-Decane	just after n-Dodecane
C8-C10 Aromatic Hydrocarbons (PID)	just after Toluene	just after 1,2,3-Trimethylbenzene
C10-C12 Aromatic Hydrocarbons (PID)	just after 1,2,3-Trimethylbenzene	just after Naphthalene
C12-C13 Aromatic Hydrocarbons (PID)	just after Naphthalene	just after 1-Methylnaphthalene

9.4 **Calibration**

9.4.1 External Standard Calibration Procedure

9.4.1.1 Prepare VPH Calibration Standards at a minimum of five concentration levels in accordance with the procedures and specifications contained in Section 7.0.

9.4.1.2 Analyze each VPH Calibration Standard following the procedures outlined in Section 9.5. Tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each targeted analyte. Alternatively, the ratio of the response to the amount injected, defined as the calibration factor (CF), may be calculated for method analytes using Equation 1. If the percent relative standard deviation (%RSD) of the calibration factor is equal to or less than 20 % over the working range for all analytes of interest, as determined using Equation 2, linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve.

Equation 1: Calibration Factor

$$Calibration\ Factor\ (CF) = \frac{area\ of\ peak}{mass\ purged\ (ng)}$$

Equation 2: Relative Standard Deviation

$$\%RSD = \frac{Stand\ Dev\ of\ 5\ CFs}{Mean\ of\ 5\ CFs} \times 100$$

9.4.1.3 A collective calibration curve or factor must also be established for each hydrocarbon range of interest. Calculate the collective Calibration Factors (CF) for C5-C6, >C6-C8, >C8-C10 and >C10-C12 Aliphatic Hydrocarbons ranges using the FID chromatogram. Calculate the collective CF for the >C8-C10, >C10-C12 and C12-C13 Aromatic Hydrocarbons ranges using the PID chromatogram. Tabulate the summation of the peak areas of all components, the compounds which define the various ranges, in those fractions (e.g. C6-C8 Aliphatic Hydrocarbons, n-pentane and n-hexane or >C6 - C8, n-octane) against the total mass injected using Equation 3.

Note: Do not include the area of any surrogate standard or internal standard in calculating a Range CF.

Equation 3: Range Calibration Factor

$$Range\ CF = \frac{Total\ area\ of\ peaks}{Total\ mass\ purged\ (ng)}$$

9.4.1.4 At a minimum, the working calibration curve or calibration factor must be verified on each working day, and after every 20 samples, whichever is more frequent, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any targeted analyte response varies from the predicted response by more than ± 15 %, as determined using Equation 4, a new calibration curve must be prepared for that analyte. Similarly, if the percent difference of the carbon range compound response varies from the predicted response by more than ± 20 %, a new calibration curve must be prepared.

Equation 4: Percent Difference (%D)

$$\%D = \frac{\overline{CF} - CFv}{\overline{CF}} x\ 100$$

9.4.1.5 Targeted VPH Analytes and Aromatic Hydrocarbons ranges are quantitated on the PID chromatogram.

9.4.1.6 The Aliphatic Hydrocarbons ranges are quantitated on the FID chromatogram after subtraction of the collective concentrations of MTBE, and the BTEX compounds identified on the PID chromatogram from the collective concentration values of the C5 through C6 and C6 through C8 Aliphatic Hydrocarbon concentration values determined using the FID chromatogram. Similarly, the PID concentrations determined for the remaining aromatic ranges are subtracted from the FID concentrations for those ranges to yield the actual FID Aliphatic Hydrocarbon ranges values.

9.4.1.7 The concentration of specific analytes or hydrocarbon ranges in aqueous and non-aqueous samples may also be calculated from a calibration curve by use of linear regression analysis.

9.5 GC Analysis

9.5.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration followed by samples interspersed with blanks and QC samples. The analytical sequence ends must end with an acceptable calibration verification. If the final calibration verification is not acceptable, then all samples analyzed after the last acceptable calibration verification must either be reanalyzed or if this is not possible, then the data associated with those samples must be qualified as an "estimate quantity".

9.5.2 Establish daily retention time windows for each analyte. Use the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 9.3.

9.5.2.1 Tentative identification of an analyte occurs when a peak from a sample falls within the daily retention time window. In cases where interferences are suspected, confirmation on a second dissimilar GC column or by GC/MS analysis may be necessary.

9.5.2.3 Validation of GC system qualitative performance must be accomplished by the analysis of standards, generally mid-level, within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.

9.5.3 If the response for Targeted VPH Analytes exceeds the linear range of the system, dilute the sample and reanalyze. It is recommended that samples be diluted so that all peaks are on scale. Overlapping peaks are not always evident when peaks are off scale. Computer reproduction of chromatograms, manipulated to ensure that all peaks are on scale over a 100-fold range, are acceptable if linearity is demonstrated prior to the analysis of samples. Peak height measurements are recommended over peak area integration when overlapping peaks cause errors in area integration.

9.5.4 If detection of targeted analyte peaks are prevented by the presence of interferences, additional analytical techniques, e.g. GC/MS, are recommended.

9.5.5 Peak area and carbon range area quantification/integration must be from baseline (i.e. forced baseline projection which must include the unresolved complex mixture "hump" areas). The exception to this is the C5 through C6 Aliphatic Hydrocarbon range which, due to the use of capillary columns shorter than 100 meters and/or the purging of methanol volumes greater than 20 uL, may experience a lack of complete resolution between C5 (n-pentane) and methanol. In this event, the analyst is allowed to use tangential skim integration, or other suitable integration techniques, to integrate n-pentane, and subsequent hydrocarbons within the C5-C6 aliphatic range if it/they appear(s) on the trailing edge of the methanol solvent peak.

9.6 External Standard Calculations

The concentration of targeted analytes and hydrocarbon ranges in a sample may be determined by calculating the amount of analyte or hydrocarbon range purged, from the peak response, using the calibration curve or the calibration factor determined in Section 9.4.

9.6.1 Aqueous samples:

The general equation to determine the concentration of a specific analyte or hydrocarbon range in aqueous samples in provided in Equation 5.

Equation 5

$$Concentration\ (\mu g / L) = \frac{(A_x)(A)(D)}{(A_s)(V_s)}$$

where:

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

A = Amount of external standard purged, ng.

D = Dilution factor; if no dilution was made, D = 1, dimensionless.

A_s = Response for the external standard, units same as for A_x.

V_s = Volume of sample purged, mL.

If a Calibration Factor is used, the concentration of a specific analyte or hydrocarbon range in an aqueous sample may be calculated using equations 6 and 7, respectively.

Equation 6

$$Conc\ Analyte\ (\mu g / L) = \frac{(A_x)(D)}{(V_s)(CF)}$$

Equation 7

$$Conc\ HC\ Range\ (\mu g / L) = \frac{(A_x)(D)}{(V_s)(Range\ CF)}$$

where:

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

D = Dilution factor; if no dilution was made, D = 1, dimensionless.

V_s = Volume of sample purged, mL.

CF = Calibration Factor, area counts/ng.

Range CF = Calibration Factor for hydrocarbon range, (collective area count/collective mass), area counts/ng.

9.6.2 **Non-Aqueous Samples (Methanol Extraction):**

9.6.2.1 The general equation to determine the concentration of a specific analyte or hydrocarbon range in a soil or sediment sample is provided in Equation 8.

Equation 8

$$Concentration\ (\mu g / kg) = \frac{(A_x)(A)(V_t)(D)}{(A_s)(V_i)(W_d)}$$

where:

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

A = Amount of external standard purged, ng.

V_t = Volume of total extract, µL (Note: this value must include the 1.0 mL surrogate spiking solution added to soil samples)

D = Dilution factor; if no dilution was made, D = 1, dimensionless.

A_s = Response for the external standard, units same as for A_x.

V_i = Volume of methanol extract added to reagent water for purge and trap analysis, µL.

W_d = Dry weight of sample purged, g (see equations 11 through 13)

If a Calibration Factor is used, the concentration of a specific analyte or hydrocarbon range in a soil or sediment sample may be calculated using Equations 9 and 10, respectively.

Equation 9

$$Conc\ Analyte\ (\mu g / kg) = \frac{(A_x)(V_t)(D)}{(V_i)(W_d)(CF)}$$

Equation 10

$$Conc\ HC\ Range\ (\mu g / kg) = \frac{(A_x)(V_t)(D)}{(V_i)(W_d)(Range\ CF)}$$

where:

V_t = Volume of total extract, µL.

Vi = Volume of extract added for purging, µL

W_d = Dry Weight of sample purged, g (see Equations 11 through 13)

A_x, CF, Range CF and D have the same definition as for aqueous samples.

9.6.2.2 Calculation of Dry Weight of Sample

In order to calculate the dry weight of sample purged (W_d), it is necessary to determine the moisture content of the soil/sediment sample, using the procedure outlined in Section 9.1.6. Using the data obtained from Section 9.1.6, W_d is calculated using Equations 11 through 13.

Equation 11

$$\%\ Moisture = \frac{g\ sample - g\ dry\ sample}{g\ sample}\ X\ 100$$

Equation 12

$$\%\ Dry\ Solids = (1) - (\%\ Moisture)$$

Equation 13

$$W_d\ (g) = (\%\ Dry\ Solids)(g\ of\ extracted\ sample)$$

9.6.3 The concentration of specific analytes or hydrocarbon ranges in aqueous and non-aqueous samples may also be calculated from the calibration curve by linear regression, provided that the correlation coefficient (r) is at least 0.99 and the % D for any targeted analyte must not vary from the predicted response by more than +/- 15%, nor any hydrocarbon range standard from its predicted response by more than +/- 20%.

9.6.4 Peak areas measured from blanks may not be subtracted from sample peak areas.

9.6.5 All integration of collective hydrocarbon ranges must be to baseline.

9.6.6 Required Adjustment of Range Concentration Data: In order to minimize the "double counting" of the same hydrocarbon compounds on both the FID and PID chromatograms, the collective concentrations of MTBE, benzene, toluene, ethylbenzene, and m, p, o-xylene identified on the PID chromatogram must be subtracted from the collective Aliphatic Hydrocarbon concentration values, for the ranges in which they elute, determined using the FID chromatogram. Similarly, the collective concentrations of the remaining aromatic ranges values determined on the PID chromatogram must be subtracted from the corresponding aliphatic ranges values determined on the FID chromatogram.

10.0 QUALITY CONTROL

10.1 **General Requirements and Recommendations**

10.1.1 Each laboratory that uses this method should operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document the quality of data. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance standards for the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

10.1.2 A acidified reagent water blank should continually accompany each soil sample or water sample batch, respectively, over the course of sampling, storage, and analysis.

10.1.3 A Laboratory Method Blank should be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

10.1.4 At a minimum, for each analytical batch (up to 20 samples), a Laboratory Method Blank, Laboratory Fortified Blank, Laboratory Fortified Matrix Spike, and sample

duplicate must be analyzed. For analytical batches with more than 10 samples, the analysis of an additional mid-range calibration check standard is recommended. The blank and spiked samples must be carried through all stages of the sample preparation and measurement process.

10.1.5 The recommended sequence of analysis is as follows:

(1) Calibration Standards (initial) or mid-range Calibration Check Standard (daily check on initial calibration)
(2) Laboratory Method Blank
(3) Samples
(4) QC Samples
(5) Mid-range Calibration Check Standard (also recommended after each 10 samples)

10.1.6 It is recommended that a system of control charts be developed to plot surrogate standard recoveries as a function of time. When surrogate recovery from a sample, blank, or QC sample is less than 60% or more than 140%, check calculations to locate possible errors, the fortifying solution for degradation, and changes in instrument performance. If the cause cannot be determined, the analyst may reanalyze the sample or report the surrogate values as outside acceptance limits.

10.2 **Minimum Instrument QC**

10.2.1 While it is recommended that the n-pentane (C5) peak be adequately resolved from the methanol solvent on the FID chromatogram, the analyst is allowed to follow the guidance as outlined in 9.5.5. Coelution of the m- and p- xylene isomers is permissible. Any surrogates and/or internal standards used must be adequately resolved from individual compounds in the VPH Component Standard.

10.2.2 Retention time windows must be established for each analyte of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis. (See Section 9.3)

10.2.3 Calibration curves must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels. The linearity of calibration or response factors may be assumed if the percent relative standard deviation (%RSD) over the working range of the curve is less than or equal to 20%. Alternatively, if linear regression analysis is used for quantitation, the correlation coefficient (r) must be at least 0.99 and no targeted analyte or carbon range standard may vary from the true value by more than +/- 15% and +/- 20% respectively. (See Section 9.4.)

10.3 **Initial and Periodic Method QC Demonstrations**

The following must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

10.3.1 Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate reagent water and/or clean sand blanks spiked with each analyte of interest at approximately 20 to 60 µg/L and/or 20 to 60 mg/kg, respectively.

10.3.1.1 Add an appropriate aliquot of the stock or primary dilution standard solution(s) to each of the four replicate reagent water or clean sand blanks. Purge and analyze each replicate according to the procedures described in Section 9.0.

10.3.1.2 Calculate the measured concentrations of each analyte in all replicates, the mean accuracy (as a percentage of true value) for each analyte, and the precision (as %RSD) of the measurements for each analyte.

10.3.1.3 For each analyte, the mean accuracy, expressed as a percentage of the true value, must be between 80% and 120%. For each analyte, the %RSD must be less than or equal to 20%.

10.3.1. If desired, the Accuracy and Precision evaluation may be combined with the MDL evaluation specified in Paragraph 10.3.2.

10.3.2 Method Detection Limits (Optional)

Analyze a minimum of seven replicate reagent water and/or clean sand blanks which have been fortified with all analytes of interest at approximately 0.5 to 5 µg/L and/or 1 to 5 mg/kg, respectively. Calculate the Method Detection Limit (MDL) of each analyte using the procedure described in Section 12.0.

10.3.2.1 Water MDLs are determined by analyzing 7 to 10 replicates of reagent water samples in 100-mL flasks spiked with the VPH Component standard and with 40 µg/L of the surrogate compound 2,5-dibromotoluene.

10.3.2.2 Soil/sediment MDLs are determined by analyzing 7-10 replicates of 5 -g of VPH-free sand blanks spiked with the VPH Component standard and with 2 mg/kg of the surrogate 2,5-dibromotoluene.

10.3.3 Petroleum Reference Standard

As an optional demonstration of the validity and relevance of VPH calibration, analyze a reagent water and/or clean sand blank spiked with a known concentration of a neat gasoline product.

10.3.3.1 Fortify a reagent water and/or clean sand blank with 5 µL and/or 0.5 mL of the Petroleum Standard Spiking Solution, respectively. Purge and analyze in accordance with the procedures outlined in Section 9.0.

10.3.3.2 Calculate the total concentration of all petroleum hydrocarbons within the Aliphatic Hydrocarbon ranges using the FID chromatogram. Add these values together. Do not subtract the concentration of Targeted VPH analytes.

10.3.3.3 The concentration calculated in Paragraph 10.3.3.2 is expected to be within 30% +/- of the known concentration of Petroleum Standard in the reagent water or sand blank.

10.4 **Ongoing Method QC Demonstrations**

10.4.1 Each sample, blank, and matrix spike must be spiked with the surrogate spiking solution. Required surrogate recovery is 60% to 140%.

10.4.2 At a minimum, with every batch of 20 samples or less the laboratory must analyze the following:

10.4.2.1 **Calibration Check Standard** - A mid-range calibration standard, prepared from the same stock standard solution used to develop the calibration curve, must be analyzed prior to sample analysis to verify the calibration state of the instrument. For large analytical batches that contain more than 10 samples, the analysis of an additional mid-range calibration check standard is recommended after the analysis of the tenth sample. If the percent difference (% D) of any analyte within the calibration check standard varies from the predicted response by more than 20 %, a new calibration curve must be prepared for that analyte.

10.4.2.2 **Laboratory Method Blank** - A water or soil laboratory method blank is prepared by fortifying a 5 mL reagent water blank with 4 µL of the surrogate spiking solution, or by fortifying a 5 g sample of clean sand with 1.0 mL of the surrogate spiking solution. Peaks within the retention time windows of any hydrocarbon ranges of interest may not be present at or above the lowest calculated PQL for any sample within its batch. When determining the PQL for soil method blanks, incorporate the lowest percent solids value found for any sample within its batch in the calculation.

10.4.2.3 **Laboratory Fortified Blank (LFB)** - A water or soil component spike is prepared by fortifying a 5 mL reagent water blank with 4 µL of the matrix spiking solution, or by fortifying a 5 g sample of clean sand with 1.0 mL of the matrix spiking solution. The spike recovery must be between 70% and 130% and if these values can not be obtained the analyst must identify and correct the problem before analyses can continue.

10.4.2.4 **Sample duplicates** - Sample duplicates may be laboratory or field duplicates. The RPD of duplicate samples should not exceed +/- 25%. The lack of sample homogeneity may contribute to RPD's for duplicates which exceed this value. Should the values exceed 25%, the analyst must report that occurrence.

10.4.2.5 **Laboratory Fortified Matrix (LFM) Spike** - The water or soil LFM spike is prepared by fortifying an actual 5 mL water sample with 4 µL of the matrix spiking solution, or by fortifying an actual 5 g soil/sediment sample with 1.0 mL of the matrix spiking solution. The purpose

of the LFM is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations. The corrected concentrations of each analyte within the matrix spiking solution must be within 70 - 130% of the true value. Should the values determined exceed this range, the analyst must report that occurrence.

10.4.3 If any of the performance standards specified in Section 10.4 are not met, the problem must be corrected before further samples are analyzed. Exceptions to this are duplicate samples RPD's and high recoveries for LFM spikes which, due to sample non-homogeneity may exceed the allowable limits. Any samples run between the last calibration check standard that meets the criteria and one that does not must be rerun. If this is not possible, that data must be reported as an "estimated concentration".

11.0 DATA PRODUCTION AND REPORTING

11.1 **Sample Analysis**

11.11 PID Chromatogram

11.1.1.1 Determine the peak area count for the Targeted VPH Analytes.

11.1.1.2 Determine the peak area count for the surrogate 2,5-dibromotoluene.

11.1.1.3 Separately determine the total area count for all peaks eluting 0.1 minutes after the Rt's for toluene through 0.1 minutes after the Rt for 1,2,3-trimethylbenzene, for all peaks eluting 0.1 minutes after the retention time (Rt) for 1,2,3-trimethylbenzene and 0.1 minutes after the retention time for naphthalene and for all peaks eluting 0.1 minutes after the Rt for naphthalene through 0.1 minutes after the Rt of 1-methylnaphthalene.

11.1.1.4 Using the equations contained in Section 9.6 or linear regression analysis, calculate the concentrations of the Targeted VPH Analytes, the surrogate standard 2,5-dibromotoluene, and >C8 through C10, >C10 through C12 and >C12 through C13 Aromatic Hydrocarbons.

11.2.1 FID Chromatogram

11.2.1.1 Separately determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for n-pentane to 0.1 minutes after the Rt for n-hexane and for all peaks eluting 0.1 minutes after the Rt for n-hexane to 0.1 minutes after the Rt for n-octane. It is not necessary to identify or quantitate individual aliphatic compounds within these ranges.

11.2.1.2 Determine the total area count for all peaks eluting 0.1 minutes after the Rt for n-octane to 0.1 minutes after the Rt for n-decane and 0.1 minutes after n-decane to 0.1 minutes after n-dodecane. It is not necessary to identify or quantitate individual aliphatic compounds within this range.

11.2.1.3 Determine the peak area count for the surrogate standard 2,5-dibromotoluene.

11.2.1.4 Using the equations contained in Section 9.6 or linear regression analysis, calculate the concentrations of C5 through C6 Aliphatic Hydrocarbons, >C6 through C8 Aliphatic Hydrocarbons, >C8 through C10 Aliphatic Hydrocarbons, >C10 through C12 Aliphatic Hydrocarbons and the surrogate standard 2,5-dibromotoluene.

11.2.1.5 To avoid "double counting" of the same analytes, adjust the concentrations of Aliphatic Hydrocarbons calculated in Paragraph 11.2.1.4 by subtracting the collective concentrations of methyl tert butylether, benzene, toluene, >C8 through C10 and >C10 through C12 Aromatic Hydrocarbons, as determined from the PID chromatogram in Section 11.11, from the Aliphatic Hydrocarbon range values effected.

11.3 **Data Reporting Format**

11.3.1 The following information and data must be reported:

11.3.1.1 The sample matrix (aqueous, soil or sediment);

11.3.1.2 The date(s) the sample was collected, received by the laboratory, and analyzed;

11.3.1.3 A description of the sample(s) received by the laboratory, relative to the physical condition of the containers, the temperature of the samples, and use of appropriate preservatives;

11.3.1.4 Moisture content (for soil/sediment samples);

11.3.1.5 The calculated concentrations of C5 through C6, C6 through C8, C8 through C10 and C10 through C12 Aliphatic Hydrocarbons ranges and C8 through C10, C10 through C12 and C12 through C13 Aromatic Hydrocarbons ranges

11.3.1.6 Surrogate recovery (expressed as percent recovery);

11.3.1.7 The calculated concentrations of Targeted VPH Analytes determined

11.3.1.8 The concentration units for aqueous samples are expressed as ug/L or mg/L and for soil or sediment samples the units are expressed as ug/Kg or mg/Kg on a dry-weight basis.

12.0 METHOD PERFORMANCE

12.1 **Method Detection Limits (Optional)**

12.1.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.1.2 The MDL is determined according to the following equation:

Equation 14

$$MDL = (t) \times (SD)$$

where:

t = student t value at the 99% confidence level

SD = standard deviation of the replicate analysis

Student t values are as follows:

Number of replicates	t value
7	3.14
8	3.00
9	2.90
10	2.82

12.1.3 For the purposes of this method, the designated MDL value for a hydrocarbon range of interest shall be the highest value calculated for the individual analytes within that hydrocarbon range.

12.2 **Single Laboratory Accuracy, Precision, and MDL Data**

Single laboratory accuracy, precision and MDL data for method analytes are provided in Tables 1-1 through 1-4 in Appendix 1. Additional investigation will be conducted to further evaluate the low recoveries for naphthalene.

13.0 REFERENCES

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3. USEPA, "Measurements of Petroleum Hydrocarbons: Report on Activities to Develop a Manual," prepared by Midwest Research Institute, Falls Church, VA under EPA Contract No. 68-WO-0015, WA No. 4; Submitted to USEPA Office of Underground Storage Tanks; Washington, DC; November 20, 1990.

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5. Parr, J. L., G. Walters, and M. Hoffman, Sampling and Analysis of Soils for Gasoline Range Organics," presented at First Annual West Coast Conference Hydrocarbon contaminated Soils and Groundwater, 2/21/90.

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7. "Leaking Underground Fuel Tank (LUFT) Field Manual," State Water Resources Control Board, State of California, Sacramenta, CA, May 1988.

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9. Senn, R. B., and M. S. Johnson, "Interpretation of Gas Chromatographic Data in Subsurface Hydrocarbon Investigations," Ground Water Monitoring Review, 1987.

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METHOD FOR THE DETERMINATION

OF

EXTRACTABLE PETROLEUM HYDROCARBONS (EPH) FRACTIONS

Washington State Department of Ecology

June 1997

METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS (EPH)

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DISCLAIMER

Mention of trade names or commercial products does not constitute endorsement by the Washington State Department of Ecology. Equipment and materials cited in this method may be replaced by similar products, as long as adequate data exists or has been produced documenting equivalent or superior performance.

METHOD FOR THE DETERMINATION OF EXTRACTABLE PETROLEUM HYDROCARBONS

1.0 SCOPE AND APPLICATION

1.1 This method is designed to measure the collective concentrations of extractable aliphatic and aromatic petroleum hydrocarbons in water and soil. **The carbon ranges used through out this document are given in equivalent carbon numbers (EC) which are related to the boiling point of a chemical normalized to the boiling point of the n-alkanes and its retention time in a boiling point gas chromatographic column**. Extractable aliphatic hydrocarbons are collectively quantitated within five ranges: C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34. Extractable aromatic hydrocarbons are collectively quantitated within five ranges: C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34. These aliphatic and aromatic hydrocarbon ranges correspond to a boiling point range between approximately 150 °C and 500 °C.

1.2 Petroleum products suitable for evaluation by this method include, but are not limited to, kerosene and jet fuels, diesel and fuel oils and hydraulic, insulating and lubricating oils. This method, in and of itself, is not suitable for the evaluation of gasoline, mineral spirits, petroleum naphthas, and other petroleum products which contain a significant percentage of hydrocarbons lighter than C10. When samples are known or suspected to contain petroleum hydrocarbons of these or similar types, the Volatile Petroleum Hydrocarbon (VPH) method must also be employed to fully evaluate the hydrocarbons present.

1.3 For reporting purposes, the practical quantitation limits (PQL), given the sample volume/mass, final extract volume and assuming 100 percent solids for soil/sediments are: 50.0 ug/L for aliphatic and aromatic carbon ranges in water and 5.0 mg/Kg for these components in soil. If lower quantitation limits are desired, the analyst is allowed to extract larger volumes/masses and/or concentrate the extracts to smaller volumes prior to analysis. The procedure for Method Detection Limits

(MDL) determination in this method and the Single Laboratory Precision, Accuracy and MDL data (generated by State of Massachusetts) is added for informational purposes only.

1.4 This method is based on a solvent extraction, silica gel fractionation process and gas chromatography (GC) analysis using a flame ionization detector (FID). This procedure should be used by, or under the supervision of, analysts experienced in extractable organics analysis. Analysts using this method should be skilled in the interpretation of gas chromatograms and their use as a quantitative tool.

1.5 Like all GC procedures, this method is subject to a "false positive" bias, in that non-hydrocarbon compounds eluting or co-eluting within a specified retention time window may be falsely identified and/or quantitated with the respective carbon ranges. While the cleanup procedure specified in this method to segregate aliphatic and aromatic fractions will serve to mitigate this concern, confirmatory analyses by gas chromatography/mass spectrometry (GC/MS) analysis, EPA Method 8270, or other suitable techniques are recommended in cases where significant concentrations of non-hydrocarbon compounds are known or suspected. Non-petroleum compounds identified and quantitated by GC/MS may be subtracted from the carbon ranges affected as long as the quantity and identities of the compounds are reported along with the carbon range data.

2.0 SUMMARY OF METHOD

2.1 A sample submitted for EPH analysis is extracted with methylene chloride, dried over sodium sulfate, solvent exchanged into hexane, and concentrated. Sample cleanup and separation into aliphatic and aromatic fractions is conducted using a modification of EPA method 3630 (silica gel cleanup). The use of commercially available silica gel cartridges (Sep-Pak cartridges, Waters, Milford, MA) may be substituted for the silica gel column outlined in method 3630 if they can be shown to achieve comparable results. The two extracts produced are then concentrated to final volumes of 10 mL each (i.e., an aliphatic extract and an aromatic extract) and are then separately analyzed by a gas chromatograph equipped with a capillary column and a flame ionization detector. The resultant chromatogram of aliphatic compounds is collectively integrated within the C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 aliphatic hydrocarbon ranges. The resultant chromatogram of aromatic compounds is collectively integrated within the C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 ranges.

2.2 Average calibration factors or response factors determined using an aliphatic hydrocarbon standard mixture are used to calculate the collective concentrations of the different aliphatic hydrocarbons ranges. An average calibration factor or response factor determined using the aromatic hydrocarbon standard mixture is used to calculate a collective concentrations of the aromatic hydrocarbon ranges.

2.3 This method is suitable for the analysis of waters, soils, and sediments.

2.4 This method is based on, and constitutes a significant modification of, the “Method for the Determination of Extractable Petroleum Hydrocarbons (EPH)” Public Comment Draft 1.0 developed by the Massachusetts Department of Environmental Protection. They in turn based their method on (1) USEPA Methods 8000, 8100, and 3630, SW-846, "Test Methods for Evaluating Solid Waste", 3rd Edition, 1986; (2) Draft "Method for Determination of Diesel Range Organics", EPA UST Workgroup, November, 1990; and (3) "Method for Determining Diesel Range Organics", Wisconsin Department of Natural Resources, PUBL-SW-141, 1992.

Table 1. Aromatic Hydrocarbon Standard

Equivalent Carbon Number	Compound
7. 6	Toluene
10.1	1,2,3-Trimethylbenzene
11.7	Naphthalene
15.5	Acenaphthene
Surrogate	Ortho-Terphenyl
20.8	Pyrene
34.01	Benzo(g,h,i)Perylene

Table 2. Aliphatic Hydrocarbon Standard

Carbon Number	Compound
8	Octane
10	Decane
12	Dodecane
16	Hexadecane
Surrogate	1-Chloro-octadecane
21	Henicosane
34	Tetratriacontane

3.0 DEFINITIONS

3.1 **Extractable Petroleum Hydrocarbons (EPH)** are defined as all hydrocarbon compounds eluting from toluene through benzo(g,h,i)perylene. EPH is comprised of C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 Aliphatic Hydrocarbons and C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 Aromatic Hydrocarbons. EPH concentration data are reported as the aggregate concentration of the aliphatic and aromatic hydrocarbon ranges.

3.2 **Equivalent Total Petroleum Hydrocarbons (E-TPH)** For samples contaminated with petroleum products in the C10 to C34 range, the E-TPH value is equivalent to the EPH value. For samples contaminated with a petroleum product(s) containing significant concentrations of hydrocarbons lighter and heavier than C10, (e.g., contaminated with both gasoline and diesel fuel), the E-TPH value is a summation of the EPH value and the Volatile Petroleum Hydrocarbon (VPH) value. In order to avoid double counting of analytes due to the overlap in the carbon ranges existing between the two methods, the analyst will report the highest of the two values determined for the overlapping ranges. In those cases where both the VPH and EPH methods are employed on samples, the analyst may select to quantitate the >C10 through C12 Aliphatic and the >C10 through C12 Aromatic Hydrocarbon ranges by the EPH method only. Similarly, the analyst may select to quantitate the C8 through C10 Aromatic and C8 through C10 Aliphatic Hydrocarbon ranges by the VPH method only.

3.3 **C8 through C10 Aromatic Hydrocarbons** are defined as all of the aromatic hydrocarbon compounds eluting from (and including) toluene through 1,2,3-trimethylbenzene.

3.4 **>C10 through C12 Aromatic Hydrocarbons** are defined as all aromatic hydrocarbon compounds eluting after 1,2,3-trimethylbenzene through naphthalene.

3.5 **>C12 through C16 Aromatic Hydrocarbons** are defined as all aromatic hydrocarbon compounds eluting after naphthalene through acenaphthene.

3.6 >**C16 through C21 Aromatic Hydrocarbons** are defined as all aromatic hydrocarbons compounds eluting after acenaphthene through pyrene.

3.7 >**C21 through C34 Aromatic Hydrocarbons** are defined as all aromatic hydrocarbons compounds eluting after pyrene through benzo(g,h,i,)perylene.

3.8 **C8 through C10 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds eluting from (and including) n-octane (n-C8) through n-decane (n-C10).

3.9 **>C10 through C12 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds eluting after n-decane through n-dodecane (n-C12).

3.10 **>C12 through C16 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbon compounds eluting after n-dodecane through n-hexadecane (nC16)

3.11 >**C16 through C21 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbons eluting after n-hexadecane through n-henicosane (nC21).

3.12 **>C21 through C34 Aliphatic Hydrocarbons** are defined as all aliphatic hydrocarbons eluting after n-henicosane through tetratriacontane (nC34).

3.13 **Aromatic Hydrocarbon Standard** is defined as a 6 component mixture (plus surrogate) of the aromatic hydrocarbons listed in Table 1. The compounds comprising the Aromatic Hydrocarbon Standard are used to (a) define and establish the windows for the Aromatic Hydrocarbon ranges, and (b) determine chromatographic response factors that can in turn be used to calculate the collective concentration of aromatic hydrocarbons in environmental samples within those hydrocarbon ranges.

3.14 **Aliphatic Hydrocarbon Standard** is defined as a 6 component mixture (plus surrogate) of the normal alkanes listed in Table 2. The compounds comprising the Aliphatic Hydrocarbon Standard are used to (a) define and establish windows for the aliphatic hydrocarbons ranges, and (b) determine chromatographic response factors that can in turn be used to calculate the collective concentration of aliphatic hydrocarbons in environmental samples within those hydrocarbon ranges.

3.15 **Analytical Batch** is defined as a group of 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.

3.16 **Laboratory Duplicates** are defined as split samples taken from the same sampling container and analyzed separately with identical procedures. The analysis of laboratory duplicates give a measure of the precision associated with laboratory procedures, but not with sample collection, preservation, or storage procedures.

3.17 **Field Duplicates** are defined as two separate samples collected at the same time and location under identical circumstances and managed the same throughout field and laboratory procedures. Analyses of field duplicates give a measure of the precision associated with sample collection, preservation and storage, as well as laboratory procedures.

3.18 **E-TPH Duplicates** are defined as two separate samples collected at the same time and location, for analysis by both EPH and VPH methods. E-TPH duplicates are taken at locations where significant concentrations of petroleum hydrocarbons lighter and heavier than C10 are likely to be present (e.g., locations contaminated by releases of both gasoline and diesel fuel). The resultant EPH and VPH concentrations are then summed to determine the Equivalent TPH (E-TPH) concentration.

3.19 **Calibration Standards** are defined as a series of standard solutions prepared from dilutions of a stock standard solution, containing known concentrations of each analyte and surrogate compounds of interest.

3.20 **Calibration Check Standard** 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.

3.21 **Matrix Spiking Solution** is defined as a solution which is generally prepared independently from the calibration standards and which contain known concentrations of method analytes.

3.22 **Laboratory Method Blank** is defined as, depending on the matrix of the samples, either reagent water or clean sand spiked with a surrogate standard. The laboratory method blank is treated identically as with samples, exposed to all glassware, solvents, reagents, and equipment. A laboratory method blank is analyzed with every batch of samples, to determine if method analytes or other interferences are present in the laboratory environment, reagents, or equipment.

3.23 **Laboratory Fortified Blank (LFB)** is defined as, depending on the matrix of the samples, either reagent water or clean sand blank fortified with a matrix spiking solution. The LFB is treated and analyzed identically as with samples and blanks and its purpose is to determine whether the methodology is in control and whether the laboratory is capable of making accurate and precise measurements at the required practical quantitation limits.

3.24 **Laboratory Fortified Matrix (LFM) Sample** is defined as an environmental sample which has been spiked with a matrix spiking solution containing known concentrations of method analytes. The LFM sample is treated and analyzed exactly as with samples and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of analytes in the sample matrix must be determined through the separate analyses of a laboratory or field duplicate, and the measured values in the LFM sample corrected for background concentrations.

3.25 All other terms are as defined in SW-846, "Test Methods for Evaluating Solid Waste", USEPA, September, 1986, and as amended.

4.0 INTERFERENCES

4.1 Method interferences are reduced by washing all glassware with hot soapy water and then rinsing with warm tap water, acetone, and methylene chloride.

4.2 High purity reagents must be used to minimize interference problems.

4.3 Contamination by carryover can occur whenever high-level and low-level samples are sequentially analyzed. Whenever an unusually concentrated sample is analyzed, it should be followed by the analysis of a solvent blank to check for cross-contamination.

4.4 Matrix interferences may be caused by contaminants that are coextracted along with the analytes of interest from the sample. The type and extent of matrix interference will vary considerably from one source to another depending upon the nature and diversity of the site being sampled and may include certain solvents, halogenated hydrocarbons and phthalate esters. A silica gel cleanup procedure is used to overcome many of these interferences however, some samples may require additional cleanup procedures/approaches or analytical techniques, e.g. gel permeation chromatography and/or GC/MS, which are beyond the scope of this method.

5.0 HEALTH AND SAFETY ISSUES

The toxicity and carcinogenicity of each reagent used in this method has not been precisely defined. However, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of Occupational Safety and Health Administration (OSHA) regulations regarding the safe handling of the chemicals specified in this method. A reference file of material safety data sheets (MSDS) should also be made available to all personnel involved in the chemical analysis.

6.0 APPARATUS AND MATERIALS

6.1 The following glassware is used for this method:

6.1.1 1-L amber glass bottles

6.1.2 4 oz. (120 mL) amber glass wide-mouth jars

6.1.3 Vials:

6.1.3.1 autosampler: 2-mL glass vials with Teflon-lined rubber crimp caps

6.1.3.2 10-mL vials with Teflon-lined caps

6.1.4 Glass funnels

6.1.5 2-L Separatory funnels with Teflon stopcock

6.1.6 Kuderna-Danish apparatus including 10-mL concentrator tube, 500-mL Evaporative flask, & 3-ball Snyder column

6.1.7 Chromatographic column: 250 mm long x 10 mm I.D. with teflon stopcock

6.1.8 Disposable pipets: Pasteur

6.1.9 25-mL graduated cylinder

6.1.10 1-Liter graduated cylinder

6.1.11 100-mL beakers

6.1.12 25-mL volumetric flasks

6.2 Analytical balance: An analytical balance capable of accurately weighing 0.0001 g must be used for weighing standards. A top-loading balance capable of weighing to the nearest 0.1 g must be used for weighing soil samples.

6.3 A nitrogen blowdown apparatus for use in concentrating extracts.

6.4 **Gas Chromatography**

6.4.1 Gas Chromatograph: An analytical system complete with temperature programmable gas chromatograph for use with capillary columns is required. The data station must be capable of storing and reintegrating chromatographic data and must be capable of determining peak areas using a forced baseline projection.

6.4.2 Recommended Column: 30-m long x 0.32-mm I.D., 0.25-µm film DB-5 column (J&W Scientific) or equivalent. This column will allow for the adequate resolution of alkanes from n-C8 to n-C34.

6.4.3 Detector: A Flame Ionization Detector (FID).

6.4.4 Autosampler: An autosampler capable of making 1 to 2 µL injections is recommended.

6.5 Water bath: heated with a concentric ring covers, capable of temperature control (± 2°C). The bath should be used in a hood.

6.6 Microsyringes: 10-µL, 100-µL, 250-µL, 500-µL, 1000-µL

6.7 Boiling Chips: glass or teflon, precleaned prior to use

6.8 Soxhlet or Sonication extraction apparatus

6.9 Drying oven

7.0 REAGENTS AND STANDARDS

7.1 **Reagents**

7.1.1 Reagent Water: organic free water

7.1.2 Solvents: hexane, methylene chloride, and acetone; pesticide grade or better. Store away from other solvents.

7.1.3 Sodium sulfate: (ACS) granular, anhydrous. Purify by heating at 400°C for 4 hours in a shallow tray.

7.1.4 Ottawa and/or masonry sand: free of extractable petroleum hydrocarbons.

7.1.5 Silica gel: 100/200 mesh (Davison Chemical grade 923 or equivalent). Heat to 150-160 ^{o}C for several hours before use. Silica Gel Sep-Pak Cartridges: 5 g/20-mL (Waters, Milford, MA) , if it is demonstated to give equivalent separations.

7.2 **Stock Standard Solutions**

Prepare stock standard solutions at approximately 1000 ng/µL, or purchase as certified solutions.

7.2.1 Aromatic Hydrocarbon Standard: The Aromatic Hydrocarbon Standard consists of the 6 aromatic compounds listed in Table 1 and a surrogate compound. Prepare stock standard solutions by accurately weighing approximately 0.0100 g of pure material. Dissolve the material in methylene chloride and dilute to volume in a 10-mL volumetric flask. The use of commercially prepared stock standards solutions is an acceptable alternative to the use of neat material. The analyst is allowed to prepare a separate surrogate solution.

7.2.2 Aliphatic Hydrocarbon Standard: The Aliphatic Hydrocarbon Standard consists of the 6 normal alkanes listed in Table 2 and a surrogate compound. Prepare stock standard solutions by accurately weighing approximately 0.0100 g of pure material. Dissolve the material in hexane and dilute to volume in a 10-mL volumetric flask. The use of commercially prepared stock standard solutions is an acceptable alternative to the use of neat material. The analyst is allowed to prepare a separate surrogate solution.

7.2.3 Petroleum Reference Standard: The use of a Petroleum Reference Standard is recommended for quality control purposes. The Petroleum Reference Standard consists of an API or commercial diesel, fuel oil, kerosene, and/or lubricating oil. Prepare stock standard solutions by accurately weighing approximately 0.0100 g of neat product. Dissolve neat product in hexane and dilute to volume in a 10-mL volumetric flask.

7.3 **Surrogate Standards**

7.3.1 The recommended surrogate standards are chloro-octadecane (COD, available from Restek Corporation, Bellefonte, PA) and ortho-terphenyl (OTP, available from EM Sciences, Gibbstown, NJ). Additional surrogates or surrogates other than those listed above may be used at the discretion of the analyst as long as their performance in the method is demonstrated as acceptable. Preparation and use of those surrogates will be in the same manner as the listed surrogate.

7.3.2 The surrogate standard COD is prepared by accurately weighing approximately 0.0100 g of pure material in a 10-mL volumetric flask. Dissolve the material in hexane. This solution is added to the Aliphatic Hydrocarbon standard.

7.3.3 The surrogate standard OTP is prepared by accurately weighing approximately 0.0100 g of pure material in a 10-mL volumetric flask. Dissolve the material in methylene chloride. This solution is added to the Aromatic Hydrocarbon standard.

7.3.4 Surrogate Spiking Solution: The recommended surrogate spiking solution is comprised of a mixture of the COD and OTP surrogate standards. Prepare a surrogate spiking solution which contains the surrogate standards at a concentration of 20 ng/µL in acetone. Each sample, blank, and matrix spike is fortified with 1.0 mL of the surrogate spiking solution.

7.4 **Internal Standard**

If an internal standard method of calibration is to be used, the recommended internal standard is 5-alpha-androstane (EM Sciences, Gibbstown, NJ).

7.5 **Matrix Spike Standard**

7.5.1 Four or more analytes from each analyte group (i.e., aromatic and aliphatic hydrocarbons) are selected for use in a matrix spiking solution, which is prepared independently from the calibration standards.

7.5.2 The recommended spiking solution, consisting of C10, C12, C16 and C21 normal alkanes and naphthalene, acenaphthene, anthracene, pyrene, benzo(a)pyrene and benzo(g,h,i)perylene, is prepared in acetone at concentrations of 25 ng/µL.

7.5.3 The samples selected as the matrix spike are fortified with 1.0 mL of the matrix spiking solution.

7.6 **Fractionation Check Solution**

7.6.1 The Fractionation Check Solution is used to monitor the fractionation efficiency of different batches (lot numbers) of silica gel used to prepare the silica gel hydrocarbon fractionation columns (and Sep-Pak cartridges if used) as well as check for sample preparation errors (e.g., insufficient/excessive pentane use).

7.6.2 Prepare a Fractionation Check Solution in hexane containing 25 ng/µL each of the n-C8, n-C10, n-C12, n-C16, n-C21, and n-C34 alkanes) and 25 ng/µL each of naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, anthracene, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene and benzo(g,h,i)perylene. The final solution will contain 6 alkanes and 16 PAHs at concentrations of 25 ng/µL each. The use of commercially available check solutions containing these compounds is acceptable.

8.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

8.1 Aqueous samples are collected in 1 liter amber glass bottles with teflon-lined screw caps.

8.2 Soil and sediment samples are collected in 4 oz. (120 mL) amber wide-mouth glass jars with teflon-lined screw caps.

8.3 Aqueous samples must be preserved at the time of sampling by the addition of an acid to reduce the pH of the sample to less than 2.0. This is accomplished by the addition of approximately 5 mL of 1:1 HCl to a 1 liter sample. Following collection and the addition of acid, the sample must be cooled to 4°C.

8.4 Soil and sediment samples must be cooled to 4°C immediately after collection.

8.5 A chain of custody form must accompany all aqueous, soil and sediment samples, documenting the time and date of sampling and any preservative additions.

8.6 Aqueous, soil and/or sediment samples must be extracted within 14 days of collection. If for whatever reason aqueous samples were not preserved at the time of collection they must be extracted within 7 days of collection. Both aqueous and soil/sediment extracts must be analyzed within 40 days of extraction.

8.7 A summary of sample collection, preservation, and holding times is provided in Table 3.

Table 3. Holding Times and Preservatives for EPH Samples

Matrix	Container	Preservation	Holding Time
Aqueous Samples	1-Liter amber glass bottle with Teflon-lined screw cap	Add 5 mL of 1:1 HCl; cool to 4°C	Samples must be extracted within 14 days and extracts analyzed within 40 days
Soil/Sediments Samples	4-oz. (120 mL) wide mouth amber glass jar with Teflon-lined screw cap	Cool to 4°C	Samples must be extracted within 14 days and extracts analyzed within 40 days

9.0 PROCEDURE

9.1 **Sample Preparation**

9.1.1 Water Extraction

9.1.1.1 Mark the water meniscus on the side of the 1 liter of sample bottle (for later volume determination) and transfer the water to a 2-liter separatory funnel. For blanks and quality control samples, pour 1 liter of reagent water into the separatory funnel. Add 1.0 mL of the surrogate spiking solution to all samples, blanks and matrix spikes. For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking solution.

9.1.1.2 Check the pH of the sample with wide-range pH paper. Note the pH in the laboratory notebook. The pH of the sample need not be adjusted.

9.1.1.3 Add 60 mL methylene chloride to the sample bottle to rinse the inner walls of the container, then add this solvent to the separatory funnel.

9.1.1.4 Seal and shake the separatory funnel vigorously for 1 to 2 minutes with periodic venting to release excess pressure.

NOTE: Methylene chloride creates excessive pressure very rapidly; therefore, venting should be done immediately after the separatory funnel has been sealed and shaken once.

9.1.1.5 Allow the organic layer to separate from the water phase for a minimum of 5 minutes. If the emulsion interface between layers is more than one-third the size of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample and may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods.
Collect the solvent extract in an 500ml Kuderna-Danish (K-D) evaporation flask to which is attached a 10 mL concentrator tube..

9.1.1.6 Repeat the extraction two more times using fresh portions of solvent and adding the solvent extracts to the KD flask. (Steps 9.1.1.3 to 9.1.1.5) The use of continuous liquid-liquid extraction (EPA method 3520) is an acceptable alternative to the separatory funnel extraction method described above.

9.1.1.7 For sample volume determination add water to the sample bottle to the level of the meniscus previously marked then transfer this water to a graduated cylinder and record the volume.

9.1.1.8 Add one clean boiling chip to the K-D flask and attach a three ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 10 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 4-6 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

9.1.1.9 Solvent exchange the methylene chloride with hexane by adding at least 50 mL of hexane to the top of the Snyder column. and concentrate the extract to less than 10 mL, as described in Paragraph 9.1.1.8, raising the temperature of the water bath, if necessary, to maintain proper distillation.

Note: An alternative solvent exchange method is to use the nitrogen blowdown apparatus to exchange the methylene chloride with hexane. This is accomplished by placing a disposable pipet in the concentrator tube and adding the small amounts of hexane via the pipet to the bottom of the concentrator tube during the concentration process in the apparatus.

9.1.1.10 Remove the Snyder column and evaporation flask from the 10-mL concentrator tube. Place the concentrator tube containing the hexane extract onto a nitrogen blowdown apparatus. Adjust the extract volume to 1-2 mL under a gentle stream of nitrogen . If the extract is highly colored, forms a precipitate, or stops evaporating, the final volume will need to be larger.

9.1.1.11 The extract obtained is now ready to be cleaned and fractionated on a silica gel column. If cleanup will not be performed immediately, transfer the extract to a Teflon-lined screw-cap vial, label, and refrigerate.

9.1.1.12 Record the sample preparation information for the extraction and concentration steps. At a minimum, record the date, sample laboratory number, sample volume, volume and concentration of added surrogate and matrix spike solutions, and any deviations or problems associated with the extraction of the samples.

9.1.1.13 For cleanup and fractionation, refer to Section 9.1.4.

9.1.2 Soil/Sediment Extraction using Ultrasonic Extraction

9.1.2.1 The following steps should be performed rapidly to minimize the loss of the more volatile fractions. In a beaker blend 10 g of the soil/sediment sample with sufficient anhydrous sodium sulfate to form a free flowing powder using a spatula. Add 1.0 mL of the surrogate spiking solution to the samples and blanks. For the samples in each analytical batch selected for matrix spiking, add 1.0 mL of the matrix spiking standard. Immediately add approximately 50 mL of 1:1 methylene chloride:acetone to the beaker. If lower PQL's are desired, the analyst is allowed to increase the sample size extracted. This will necessitate a proportional increase in the volume of extraction solvent used.

9.1.2.2 Place the bottom surface of the tip of the ultrasonic horn about 1/2 inch below the surface of the solvent but above the soil/sediment layer.

9.1.2.3 Sonicate for 3 minutes with the control knob set to the maximum output, pulse mode and 50% duty cycle.

9.1.2.4 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.

9.1.2.5 Decant and filter the extract through Whatman No. 41 filter paper into the K-D concentrator. Repeat the extraction two more times with additional 50 mL portions of the solvent. Decant off and filter the extract into the K-D flask each time. On the last extraction, pour

the entire sample into the funnel and rinse the beaker and sample with additional portions of solvent and add these to the K-D flask. The analyst may find it useful to (1) either add anhydrous sodium sulfate to the filter for drying the extract or (2) to pass the extract through a drying column containing about 10 cm of anhydrous sodium sulfate prior to collecting the extract in the K-D flask.

9.1.2.6 Add one clean boiling chip to the K-D flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10 to 20 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1-2 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

9.1.2.7 Exchange the methylene chloride with hexane by adding 50 mL of hexane to the top of the Snyder column and concentrating the extract as described in Paragraph 9.1.2.6, raising the temperature of the water bath, if necessary, to maintain proper distillation. The alternate solvent exchange technique described in note in 9.1.1.9 may also be used.

9.1.2.8 Remove the Snyder column and evaporation flask from the 10-mL concentrator tube. Place the concentrator tube containing the hexane extract onto a nitrogen blowdown apparatus. Adjust the extract volume to 1-2 mL under a gentle stream of nitrogen . If the extract is highly colored, forms a precipitate, or stops evaporating, the final volume will need to be larger.

9.1.2.9 The extract obtained is now ready to be cleaned and fractionated on a silica gel column. If cleanup will not be performed immediately, transfer the extract to a Teflon-lined screw-cap vial, label, and refrigerate.

9.1.2.10 Record the sample preparation information for the extraction and concentration steps, as specified in paragraph 9.1.1.14.

9.1.2.11 For cleanup and fractionation, refer to Section 9.1.4.

9.1.3 <u>Soil/Sediment Extraction by Soxhlet Extraction</u>

9.1.3.1 Blend 10 g of the soil/sediment sample with sufficient anhydrous sodium sulfate to produce a free flowing powder and place this material in an extraction thimble. The extraction thimble must drain freely for the duration

of the extraction period. Add 1.0 mL of the surrogate spiking solution onto the sample. For the samples in each analytical batch selected for matrix spiking, add 1.0 mL of the matrix spiking solution. If lower PQL's are desired, the analyst may increase the quantity of sample extracted.

9.1.3.2 Place approximately 200 mL of methylene chloride into a 250-mL Erlenmeyer or round bottomed flask (depending on the heating source) containing several clean boiling chips. Attach the flask to the extractor and extract the sample for 8-16 hr.

9.1.3.3 Allow the extract to cool after the extraction is completed.

9.1.3.4 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporation flask.

9.1.3.5 Dry the extract by passing it through a glass powder funnel containing anhydrous sodium sulfate. Collect the dried extract in the K-D concentrator. Wash the extractor flask and sodium sulfate column with 50 to 75 mL of methylene chloride to complete the quantitative transfer.

9.1.3.6 Add one or two clean boiling chips to the flask and attach a three-ball Snyder column. Pre-wet the Snyder column by adding about 1 mL of methylene chloride to the top of the column. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10 to 20 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1-2 mL, remove the K-D apparatus from the water bath and allow it to drain and cool for at least 10 minutes.

9.1.3.7 Exchange the methylene chloride with hexane by adding 50 mL of hexane to the top of the Snyder column and concentrating the extract as described in Paragraph 9.1.3.6, raising the temperature of the water bath, if necessary, to maintain proper distillation. The alternate solvent exchange technique outlined in the note in 9.1.1.9 may also be used.

9.1.3.8 Remove the Snyder column and evaporation flask from the 10 mL concentrator tube. Place the concentrator tube containing the hexane extract onto a nitrogen blowdown apparatus. Adjust the extract volume to 1-2 mL under a gentle stream of nitrogen . If the extract is highly colored, forms a precipitate, or stops evaporating, the final volume will need to be larger.

9.1.3.9 The extract obtained is now ready to be cleaned and fractionated on a silica gel column. If cleanup will not be performed immediately, transfer the extract to a Teflon-lined screw-cap vial, label, and refrigerate.

9.1.3.10 Record the preparation information for the extraction and concentration steps, as specified in 9.1.1.14.

9.1.3.11 For cleanup and fractionation, refer to Section 9.1.4.

9.1.3.12 The use of the Accelerated Solvent Extraction (ASE), EPA Method 3545, is an acceptable alternative to either ultrasonic or soxhlet extraction techniques as long as it is demonstrated to achieve comparable results.

9.1.4 Silica Gel Cleanup and Separation

9.1.4.1 Silica gel is a regenerative adsorbent of amorphous silica with weakly acidic properties. It is produced from sodium silicate and sulfuric acid. Silica gel can be used for column chromatography and is used for separating analytes from interfering compounds of a different chemical polarity. Silica gel is also used to separate petroleum distillates into aliphatic and aromatic fractions. Before the silica gel technique can be utilized, the extract solvent must be exchanged to hexane. This procedure may be performed immediately before the extract concentration step is complete by adding hexane to the K-D (when the volume of remaining methylene chloride is approximately 10 to 15 mL) and reducing the extract volume to 1 to 2 mL. After cooling, disassemble the K-D apparatus, rinsing the joints into the concentrator tube with a minimum of hexane. With a nitrogen blowdown concentrator, further reduce the extract to a final volume of 2 mL.

9.1.4.2 Prepare a slurry of 10g of activated silica gel in methylene chloride and place this into a 10 mm I.D.x 250 mm long chromatographic column. Tap the column to settle the silica gel and elute the methylene chloride. Add 1 to 2 cm of anhydrous sodium sulfate to the top of the silica gel. Note: The activity of each new batch (lot number) of silica gel must be evaluated prior to its use in this procedure. This requires the use of the fractionation check solution to insure that there is an efficient separation of the aromatic fraction from the aliphatic fraction. If necessary, the analyst is allowed to adjust the volumes of the eluants used to facilitate this process.

9.1.4.3 Pre-elute the column with 40 mL of pentane. Let the solvent flow through the column, at a rate of about 2 mL/minute, until the head of the liquid in the

column is just above the sodium sulfate layer. Close the stopcock to stop solvent flow. Discard this pentane.

9.1.4.4 Open the stopcock and immediately transfer the hexane sample extract onto the silica gel column. Rinse the concentrator tube with an additional 1-2 mL of hexane and add this to the column as well.

9.1.4.5 Just prior to exposure of the sodium sulfate layer to the air, add 25 mL of pentane to the column. Collect the eluant in a 250 mL K-D flask to which is attached a 10 mL concentrator tube and label this fraction "aliphatics". Concentrate and solvent exchange this extract into hexane.

9.1.4.6 Following recovery of the aliphatic fraction and just prior to exposure of the sodium sulfate layer, elute the column with 50 mL of methylene chloride/pentane (40:60) (v/v) and collect the eluant in a 250 mL K-D flask equipped with a 10 mL concentrator tube.. Label this fraction "aromatics". Concentrate and solvent exchange this extract into methylene chloride.

9.1.4.7 Concentrate each fraction to a final volume of 10 mL under a gentle stream of nitrogen from a nitrogen blowdown apparatus.

9.1.4.8 Transfer 1 mL of the fractions to a labeled two-mL glass autosampler vials with teflon-rubber crimp caps for analysis and save the remaining extract in a teflon-rubber septum screw topped vial. If lower PQL's are required, the analyst may concentrate the extract to as low as 1 mL prior to analysis

9.1.5 Proceed with the analysis in accordance with Sections 9.2 through 9.5. Analyze all laboratory method blanks and QC samples under the same conditions as that used for samples.

9.1.6 If chromatographic responses exceed the linear range of the system, dilute the extract(s) and re-analyze

9.1.7 <u>Determination of Percent Moisture</u>

9.1.7.1 Soil and sediment sample results must be reported on a dry-weight basis. A portion of sample for moisture determination should be weighed out at the same time as the portion used for hydrocarbon determination.

9.1.7.2 Immediately after weighing a sample for extraction, transfer 5 to 10 g of the sample into a tared crucible. Dry the sample overnight at 105°C in an oven. After drying remove it from the oven allow it to cool in a desiccator before reweighing. Calculate the percent moisture of the sample using the equations provided in Section 9.6.3.

9.2 **GC Conditions**

9.2.1 Suggested Gas Chromatographic Conditions

9.2.1.1 Oven Program: Set oven temperature to 50 °C for 2 minute, then 8°C/min to 320 °C and hold for 10 minutes.

9.2.1.2 Sample/autosampler injection volume is 1 to 2 µL.

9.2.1.3 Gas Flows: The recommended carrier gas is helium.

9.2.1.3.1 Helium carrier gas flow: 1 to 2 mL/min.

9.2.1.3.2 Air: 400 mL/min.

9.2.1.3.3 Hydrogen: 35 mL/min.

9.2.1.3.4 Make up gas flow: 30 mL/min.

9.2.1.4 Miscellaneous:

9.2.1.4.1 FID temperature, 310 to 320 °C

9.2.1.4.2 Injection port temperature, 290 to 300 °C

9.2.1.4.3 GC operated in splitless mode

9.2.1.4.4 Column head pressure 15.0 psi at 50°C

9.2.1.4.5 Linear velocity approximately 50 cm/sec

9.3 **Retention Time Windows**

9.3.1 Before establishing windows, make sure the GC system is within optimum operating conditions. Make three injections of the Aromatic and Aliphatic Hydrocarbon standard mixtures throughout the course of a 72-hr period. Serial injections over less than a 72-hr period may result in retention time windows that are too tight.

9.3.2 Calculate the standard deviation of the three absolute retention times for each standard.

9.3.3 Plus or minus three times the standard deviation of the absolute retention times for each standard should be used to define the retention time window. However, the

experience of the analyst should weigh heavily in the interpretation of chromatograms.

9.3.4 In those cases where the standard deviation for a particular standard is zero, the laboratory should substitute the standard deviation of a closely eluting structurally similar compound to develop a valid retention time window.9.3.5 The laboratory must calculate retention time windows for each standard on each GC column and whenever a new GC column is installed or a portion removed as part of GC maintenance. This data must be retained by the laboratory.

9.3.6 EPH retention time (Rt) windows for all fractions (carbon ranges and hydrocarbon types) except the C8 through C10 are defined as beginning 0.1 minutes after the Rt of the beginning marker compound and ending 0.1 minutes after the Rt of the ending marker compound. Since the first fraction for both the aliphatics and aromatics includes the beginning marker compounds, the Rt windows for them are defined as beginning 0.1 minutes before the Rt of the beginning marker compound and ends 0.1 minutes after the ending marker compound.

EPH marker compounds and windows are summarized in Table 4.

Table 4. EPH Marker Compounds

Range/ Hydrocarbon Standard	Beginning Marker Compound	Ending Marker Compound
C8-C10 Aliphatic Hydrocarbons	just before n-octane	just after n-decane
C10-C12 Aliphatic Hydrocarbons	just after n-decane	just after n-dodecane
C12-C16 Aliphatic Hydrocarbons	just after n-dodecane	just after n-hexadecane
C16-C21 Aliphatic Hydrocarbons	just after n-hexadecane	just after n-henicosane
C21-C34 Aliphatic Hydrocarbons	just after n-henicosane	just after n-tetratriacontane
C8-C10 Aromatic Hydrocarbons	just before toluene	just after 1,2,3-trimethylbenzene
C10-C12 Aromatic Hydrocarbons	just after 1,2,3-trimethylbenzene	just after naphthalene
C12-C16 Aromatic Hydrocarbons	just after naphthalene	just after acenaphthene
C16-C21 Aromatic Hydrocarbons	just after acenaphthene	just after pyrene
C21-C34 Aromatic Hydrocarbons	just after pyrene	just after benzo(g,h,i)perylene

9.4 **Calibration**

Calibrate the GC system using either the external standard procedure (Section 9.4.1) or the internal standard procedure (Section 9.4.2).

9.4.1 External standard calibration procedure

9.4.1.1 Prepare Aromatic and Aliphatic Hydrocarbon calibration standards at a minimum of five concentration levels by adding volumes of one or more stock standard solutions to volumetric flasks and diluting to volume with methylene chloride and hexane, respectively. The surrogate OTP is added to the Aromatic Hydrocarbon Standard; the surrogate COD is added to the Aliphatic Hydrocarbon Standard. One of the calibration

standards must be at a concentration near, but above, the method detection limit and is used to determine the reporting PQL. In order to report the PQL's listed in 1.4, given the volume/mass water or soil extracted of 1 liter or 10 grams and assuming 100% solids on the soil, this standard must be at a 5 ng/uL level. The other concentrations must correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

9.4.1.2 A collective calibration curve or factor must be established for each hydrocarbon range of interest. Inject each calibration standard using the technique that will be used to introduce the actual samples into the gas chromatograph (e.g., 1 to 2 µL injections). To calculate the collective CF for C8-C10, >C12-C12, >C12-C16, >C16-C21 and C21-C34 Aromatic Hydrocarbons, tabulate the summation of all peak areas of the calibration compounds in that range (e.g. C8-C10, toluene and 1,2,4-trimethylbenzene or >C10-C12, naphthalene) against the total mass injected. Although a forced baseline projection is required for samples and blanks, the analyst is allowed to integrate only the area of the calibration compound(s) to generate the calibration factors. The ratio of the response to the amount injected, defined as the calibration factor (CF), or range CF, may be calculated for hydrocarbon ranges at each standard concentration using Equation 1. If the percent relative standard deviation (%RSD) of the calibration factor is equal to or less than 20 % over the working range for the ranges of interest, as determined using Equation 2, linearity through the origin can be assumed, and the average calibration factor can be used in place of a calibration curve. The Aliphatic Hydrocarbon collective CF is calculated in the same manner using the aliphatic standards.

Equation 1: Range Calibration Factor

$$Range\ CF = \frac{total\ area\ of\ peaks}{mass\ injected\ (ng)}$$

Equation 2: Percent Relative Standard Deviation

$$\%RSD = \frac{Stand\ Dev\ of\ 5\ CFs}{Mean\ of\ 5\ CFs} \times 100$$

Note: The area for the surrogates COD and OTP must be subtracted from the area of the range in which they elute (e.g., COD is subtracted from the C16-C21 Aliphatic Hydrocarbon range.

9.4.1.4 At a minimum, the working calibration curve or calibration factor must be verified at the beginning of each working day and after the final analysis of that day, or after every 20 samples, whichever is more frequent, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any range varies from the predicted response by more than ± 20 %, as calculated using Equation 4, a new calibration curve must be prepared.

Equation 4: Percent Difference (%D)

$$\%D = \frac{\overline{CF} - CFv}{\overline{CF}} \times 100$$

where:

$\overline{CF}$ = Average Calibration Factor from calibration curve.

CFv = Calibration Factor from verification calibration check.

9.4.1.5 The concentrations of hydrocarbon ranges may also be calculated from a calibration curve by use of linear regression analysis.

9.4.2 Internal standard calibration procedure

9.4.2.1 The suggested internal standard for this method is 5-alpha-androstane (EM Science, Gibbstown, NJ).

9.4.2.2 Prepare Aromatic and Aliphatic Hydrocarbon calibration standards at a minimum of five concentration levels by adding volumes of stock standard solutions to volumetric flasks. The surrogate OTP is added to the Aromatic Hydrocarbon Standard; the surrogate COD is added to the Aliphatic Hydrocarbon Standard. To each calibration standard, add a known constant amount of an internal standard. One of the calibration standards must be at a concentration near, but above, the method detection limit and is used to determine the reporting PQL. The other concentrations must correspond to the expected range of concentration found in real world samples or should define the working range of the detector.

9.4.2.3 Inject each calibration standard using the same technique that will be applied to the samples (e.g., 1 to 2 µL injection). Tabulate the peak area responses against the concentration of each hydrocarbon range and internal standard. Calculate the collective response factors (RF) for each hydrocarbon range using Equation 5.

Note: The area for the surrogates COD and OTP must be subtracted from the area of the range in which they elute (e.g., COD is subtracted from the appropriate Aliphatic Hydrocarbon range).

uation 5: Range Response Factor

$$Range\ RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

A_s = Summation of peak areas of the fraction (e.g., C10-C12 Aliphatic Hydrocarbons).

C_{is} = Concentration of internal standard, ng/µL.

A_{is} = Response for the internal standard.

C_s = Total mass concentration of injected standards, ng/µL.

9.4.2.4 At a minimum, the working calibration curve or RF must be verified at the beginning of each working day and after the final analysis of that day, or after every 20 samples, whichever is more frequent, by the injection of a mid-level calibration standard to verify instrument performance and linearity. If the percent difference (%D) for any hydrocarbon range varies from the predicted response by more than ± 20 %, as calculated using Equation 4, a new calibration curve must be prepared for that range.

9.4.2.5 The concentrations of hydrocarbon ranges may also be calculated from a calibration curve by the use of linear regression analysis.

9.5 **GC Analysis**

9.5.1 Samples are analyzed in a set referred to as an analysis sequence. The sequence begins with instrument calibration, or verification of calibration, followed by sample extracts interspersed with blanks and QC samples. The analytical sequence must end with an acceptable verification calibration standard. If the ending calibration standard is not acceptable, all samples analyzed after the last acceptable calibration verification must be reanalyzed. If, for whatever reason, reanalysis is not possible, then the data for those samples must be qualified as an "estimate".

9.5.2 Aliphatic and aromatic extracts are introduced into the gas chromatograph by slitless injection.

9.5.3 Inject 1 to 2 µL of the sample extract. Record the volume injected and the resulting peak size in area units.

9.5.4 Confirm the established retention time windows for each hydrocarbon range on a daily basis. Use the absolute retention time for each analyte as the midpoint of the window for that day. The daily retention time window equals the midpoint ± three times the standard deviation determined in Section 9.3.

9.5.4.1 Validation of GC system qualitative performance must be accomplished by the analysis of standards within the analysis sequence. If any of the standards fall outside their daily retention time window, the system is out of control. In such cases, the cause of the problem must be determined and corrected.

9.5.5 When quantifying on a peak area basis by internal or external calibration, peak area integration for the aliphatic and aromatic carbon ranges must be <u>from baseline</u> (i.e. must include the unresolved complex mixture "hump" areas).

9.6 **Calculations**

9.6.1 <u>External Standard Calibration</u>

The concentration of each hydrocarbon range in a sample may be determined by calculating the amount of hydrocarbon range compounds injected, from the peak response, using the calibration curve or the calibration factor determined in Section 9.4.

9.6.1.1 **Aqueous samples:**

The general equation to determine the concentration of a hydrocarbon range in aqueous samples in provided in Equation 6.

Equation 6

$$Concentration\ (ug / L) = \frac{(A_x)(A)(V_t)(D)}{(A_s)(V_i)(V_s)}$$

w

here:

A_x = Response for a hydrocarbon range in the sample, units may be in area counts or peak height.

A = Amount of standard injected, ng.

A_s = Response for the external standard, units same as for A_x.

V_i = volume of extract injected, µL.

D = Dilution factor: if no dilution was made, D = 1, dimensionless.

V_t = Volume of total extract, µL.

V_s = Volume of sample extracted, mL.

If a Calibration Factor is used, the concentration of a hydrocarbon range may be calculated using Equation 7.

Equation 7

$$Conc\ HC\ Range\ (ug / L) = \frac{(A_X)(V_t)(D)}{(V_i)(V_s)(Range\ CF)}$$

where:

Range CF = Calibration Factor for hydrocarbon ranges (collective area count/collective mass).

9.6.1.2 **Nonaqueous samples:**

The general equation to determine the concentration of a hydrocarbon range in soil or sediment samples is provided in Equation 8.

Equation 8

$$Concentration\ (ug / kg) = \frac{(A_x)(A)(V_t)(D)}{(A_s)(V_i)(W_d)}$$

where:

W_d = Dry weight of sample extracted, g. (See Equations 12 through 14)

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

If a Calibration Factor is used, the concentration of specific hydrocarbon ranges in a soil or sediment sample may be calculated using Equation 9.

Equation 9

$$Conc\ HC\ Range\ (ug / kg) = \frac{(A_x)(V_t)(D)}{(V_i)(W_d)(Range\ CF)}$$

where:

Range CF = Calibration Factor for a hydrocarbon range (collective area count/collective mass).

9.6.2 Internal Standard Calibration

The concentration of each hydrocarbon range in a sample may be determined by calculating the amount of hydrocarbon range compound(s) injected, from the peak response, based upon the compound/internal-standard response ratio.

9.6.2.1 **Aqueous samples:**

The general equation to determine the concentration of a specific hydrocarbon range in aqueous samples is provided in Equation 10.

Equation 10

$$Concentration\ (ug / L) = \frac{(A_x)(C_{is})(D)}{(A_{is})(RF)(V_s)}$$

where:

A_x = Response of the hydrocarbon range being measured, units may be in area counts or in peak height.

C_{is} = Amount of internal standard added to extract, ng.

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

A_{is} = Response of the internal standard, units same as A_x.

RF = Response factor for the hydrocarbon range, dimensionless.

V_s = Volume of aqueous sample extracted, mL.

9.6.2.2 **Nonaqueous samples:**

The general equation to determine the concentration of a specific or hydrocarbon range in soil or sediment samples is provided in Equation 11.

Equation 11

$$Concentration\ (ug / kg) = \frac{(A_x)(C_{is})(D)}{(A_{is})(RF)(W_d)}$$

where:

W_d = Dry weight of sample extracted, g. (See Equations 12 through 14).

A_x, C_{is}, D, A_{is}, and RF have the same definition as for aqueous samples.

9.6.3 Calculation of Dry Weight of Sample

In order to calculate the dry weight of sample extracted (W_d), it is necessary to determine the moisture content of the soil/sediment sample, using the procedure outlined in Section 9.1.7. Using the data obtained from Section 9.1.7, W_d is calculated using Equations 12 through 14.

Equation 12

$$\% \text{ Moisture} = \frac{g \text{ sample} - g \text{ dry sample}}{g \text{ sample}} X\ 100$$

Equation 13

$$\% \text{ Dry Solids} = (1) - (\% \text{ Moisture})$$

Equation 14

$$W_d\ (g) = (\% \text{ Dry Solids})(g \text{ of extracted sample})$$

10.0 QUALITY CONTROL

10.1 **General Requirements and Recommendations**

10.1.1 Each laboratory that uses this method should operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of the data generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance standards for the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

10.1.2 Hexane or methylene chloride solvent blanks should be run after samples suspected of being highly contaminated to determine if sample carryover has occurred.

10.1.3 At a minimum, for each analytical batch (up to 20 samples), a Laboratory Method Blank, Laboratory Fortified Blank Spike (Water or Soil), Laboratory Fortified

Matrix Spike, and sample duplicate must be analyzed. For analytical batches with more than 10 samples, the analysis of an additional mid-range calibration check standard is recommended. The blank and spiked samples must be carried through all stages of the sample preparation and measurement process.

10.1.4 The recommended sequence of analysis is as follows:

(1) Calibration Standards (initial) or mid-range Calibration Check (Verification) Standard (i.e. daily check of initial calibration)
(2) Laboratory Method Blank
(3) Samples
(4) QC Samples
(5) Mid-range Calibration Check (Verification) Standard (also recommended after each 10 samples)

10.1.5 It is recommended that a system of control charts be developed to plot surrogate standard recoveries as a function of time. When surrogate recovery from a sample, blank, or QC sample is less than 50% or more than 150%, check calculations to locate possible errors, the fortifying solution for degradation, and changes in instrument performance. If the cause cannot be determined, the analyst may reanalyze the sample extract, report the surrogate values as outside acceptance limits or, if additional sample is available, re-extract and reanalyze the sample.

10.2 **Minimum Instrument QC**

10.2.1 The instrument must be able to achieve adequate separation and resolution of peaks of interest.

10.2.1.1 The n-octane (n-C8) and toluene peaks must be adequately resolved from the solvent front of the chromatographic run.

10.2.1.2 The surrogates COD and OTP and any internal standards used must be adequately resolved from any individual components in the Aliphatic Hydrocarbon and Aromatic Hydrocarbon standards.

10.2.2 Retention time windows must be established for each hydrocarbon range of interest each time a new GC column is installed, and must be verified and/or adjusted on a daily basis. (See Section 9.3)

10.2.3 Calibration curves must be developed based upon the analysis of calibration standards prepared at a minimum of 5 concentration levels. The linearity of calibration or response factors may be assumed if the percent relative standard deviation (%RSD) over the working range of the curve is less than or equal to 20 %. Alternatively, if linear regression analysis is used for

quantitation, the correlation coefficient (r) must be at least 0.99. and the percent difference (% D) of the response for any hydrocarbon range from the predicted response may not vary by more than +/- 20%. (See Section 9.4.)

10.3 **Initial and Periodic Method QC Demonstrations**

The following must be conducted as an initial demonstration of laboratory capability, prior to the analysis of any samples. Subsequent to this initial demonstration, additional evaluations of this nature should be conducted on a periodic basis, in response to changes in instrumentation or operations, and/or in response to confirmed or suspected systems, method, or operational problems.

10.3.1 Accuracy and Precision

To demonstrate initial laboratory capability, analyze a minimum of four replicate reagent water and/or clean sand blanks spiked with the calibration compounds for each range of interest at approximately 50 µg/L and/or 5 mg/kg, respectively.

10.3.1.1 Extract and analyze each replicate according to the procedures described in Section 9.0.

10.3.1.2 Calculate the measured concentrations of each range in all replicates, the mean accuracy (as a percentage of true value) for each compound, and the precision (as %RSD) of the measurements for each compound.

10.3.1.3 For each compound, the mean accuracy, expressed as a percentage of the true value, should be between 70 % and 130 %. For each compound, the %RSD must be less than or equal to 20%.

10.3.2 Method Detection Limits (Optional)

Analyze a minimum of seven replicate reagent water and/or clean sand blanks which have been fortified with all analytes of interest at approximately 0.5 to 2 µg/L and/or 1.0 to 5.0 mg/kg, respectively. Extract and analyze each replicate according to the procedures described in Section 9.0. Calculate the Method Detection Limit (MDL) of each analyte using the procedure described in Section 12.0.

10.3.2.1 Water MDLs are determined by extracting 7 to 10 replicates of 1-L reagent water blanks spiked with OTP, COD and each analyte of interest.

10.3.2.2 Soil/sediment MDLs are determined by extracting 7-10 replicates of 10-g of EPH-free sand blanks spiked with OTP, COD, and each analyte of interest.

10.3.3 Fractionation

To demonstrate the capability of properly fractionating aliphatic and aromatic hydrocarbons in a sample, the analyst must first prepare and analyze the Fractionation Check Solution specified in Section 7.6.

10.3.3.1 Prepare a silica gel column as outlined in 9.1.4

10.3.3.2 Load 1.0 mL of the Fractionation Check Solution onto column and proceed with the elution and collection of the aliphatic and aromatic fractions as outline in 9.1.4.

NOTE: The amount of pentane used during the elution/fractionation is critical. Excessive pentane will cause elution of aromatics into the aliphatic fraction. Insufficient pentane will cause low recoveries of the aliphatic fraction. The volume of pentane and/or methylene chloride/pentane recommended may need to be adjusted to meet QC limits.

10.3.3.3 Solvent exchange the aliphatic fraction to hexane and the aromatic fraction to methylene chloride. Concentrate each solution to a final volume of 1.0 mL under a gentle stream of nitrogen from an nitrogen blowdown apparatus.

10.3.3.4 Transfer the final 1.0 mL extracts to two labeled glass auto sampler vials with teflon-lined rubber crimp caps. Analyze by GC/FID (see Section 9.0).

10.3.3.5 For each analyte within the Fractionation Check Solution, the mean accuracy, expressed as a percentage of the true value, must be between 70 % and 130 %.

10.3.4 Petroleum Reference Standard

As an optional demonstration of the validity and relevance of EPH calibration, analyze a reagent water and/or clean sand blank spiked with a known concentration of a neat petroleum product.

10.3.4.1 Dilute a Petroleum Reference Standard stock standard solution with acetone to a concentration of approximately 250 ng/µL. Spike a 1 Liter reagent water blank or 10 g clean sand blank with 1.0 mL of this spiking solution. Extract and analyze in accordance with the procedures outlined in Section 9.0.

10.3.4.2 Calculate the total concentration of all petroleum hydrocarbons within each range. Add these values together.

10.3.4.3 The concentration calculated in Paragraph 10.3.4.2 is expected to be within +/- 30 % of the known concentration of Petroleum Standard in the reagent water or sand blank.

10.4 **Ongoing Method QC Demonstrations**

10.4.1 Each sample, blank, and matrix spike sample must be spiked with the surrogate spiking solution. Required surrogate recovery is 60% to 140%.

10.4.2 At a minimum, with every batch of 20 samples or less the lab must analyze the following:

10.4.2.1 **Calibration Check Standard** - A mid-range calibration standard, prepared from the same stock standard solution used to develop the calibration curve, must be analyzed prior to sample analysis to verify the calibration state of the instrument. For large analytical batches that contain more than 10 samples, the analysis of an additional mid-range calibration check standard is recommended after the analysis of the tenth sample. If the percent difference (% D) of any compound within a calibration check standard varies from the predicted response by more than 20 %, a new calibration curve must be prepared. (See Section 9.4)

10.4.2.2 **Laboratory Method Blank** - A water or soil laboratory method blank is prepared by fortifying a reagent water or clean sand blank with 1.0 mL of the surrogate spiking solution. Peaks within the retention time windows of any hydrocarbon ranges of interest may not be present at or above the lowest calculated PQL for any sample within its batch. When determining the PQL of soil method blanks, incorporate the lowest percent solids value found for any sample within its batch in the calculation.

10.4.2.3 **Laboratory Fortified Blank Spike** - A water or soil component spike is prepared by fortifying a reagent water or clean sand blank with 1.0 mL of the matrix spiking solution. The spike recovery must be between 70 % and 130 % and, if these values can not be obtained, the analyst must identify and correct the problem before analyses can continue.

10.4.2.4 **Sample duplicates** - Sample duplicates may be laboratory duplicates or field duplicates and the RPD of the duplicate samples should not exceed +/- 25%. The lack of sample homogeneity may contribute to the RPD's values for duplicates which exceed this value. Should the values exceed 25% the analyst must report that occurrence.

10.4.2.5 **Laboratory Fortified Matrix (LFM) Spike** - The water or soil LFM spike is prepared by fortifying an actual water or soil sample with 1.0 mL of the matrix spiking solution. The purpose of the LFM spike is to

determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations. The corrected concentrations of each analyte within the LFM spike sample must be within 70 to 130 % of the true value. Should the values determined exceed this range, the analyst must report that occurrence.

10.4.3 If any of the performance standards specified in Section 10.4 are not met, the problem should be corrected before further samples are analyzed. Exceptions to this are duplicate samples RPD's and high recoveries for LFM spikes which, due to sample non-homogeneity, may exceed the allowable limits. Any samples run between the last calibration check standard that meet the criteria and one that does not must be reanalyzed. If this is not possible, then the data must be reported as an "estimated concentration."

11.0 DATA PRODUCTION AND REPORTING

11.1 **Sample Analysis**

11.1.1 Aliphatic Fraction

11.1.1.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for C8 to 0.1 minutes after the Rt for C10.

11.1.1.2 Determine the total area count for all peaks eluting 0.1 minutes after the Rt for C10 to 0.1 minutes after the Rt for C12. Determine the total area count of all peaks eluting 0.1 minutes after the Rt for C12 to 0.1 minutes after the Rt for C16, for all peaks eluting 0.1 minutes after the Rt for C16 to 0.1 minutes after the Rt for C21and for all peaks eluting 0.1 minutes after the Rt for C21 to 0.1 minutes after the Rt for C34. It is not necessary to identify or quantitate individual aliphatic compounds within these ranges.

11.1.1.3 Determine the peak area count for the surrogate COD and any internal standard used. Subtract these values from the collective area count value within the appropriate hydrocarbon range(s).

11.1.1.4 Using the equations contained in Section 9.6 or linear regression analysis, calculate the concentrations of C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 Alipiphatic Hydrocarbon ranges and the surrogate standard COD.

11.1.1.5 The term "all peak areas" must include any unresolved envelope of peaks which elute within the Rt windows listed above and below for the aliphatic and aromatic fractions.

11.2.1 Aromatic Fraction

11.2.1.1 Determine the total area count for all peaks eluting 0.1 minutes before the retention time (Rt) for toluene to 0.1 minutes after the Rt for 1,2,3 trimethylbenzene. In the same manner, determine the total area count for all peaks eluting 0.1 minutes after the Rt for 1,2,3-trimethylbenzene to 0.1 minutes after the Rt for naphthalene, from 0.1 minutes after the Rt for naphthalene to 0.1 minutes after the Rt for acenaphthene, from 0.1 minutes after the Rt for acenaphthene to 0.1 minutes after the Rt for pyrene and 0.1 minutes after the Rt for pyrene to 0.1 minutes after the Rt for benzo(g.h.i)perylene.

11.2.1.2 Determine the peak area count for the surrogate OTP and any internal standard used.

11.2.1.3 Subtract the peak area value from 11.2.1.2 from the collective area count value for the range effected determined in 11.2.1.1 to calculate the area count for Aromatic Hydrocarbons ranges.

11.2.1.4 Using the equations contained in Section 9.6 or linear regression analysis, calculate the concentrations of C8 through C10, >C10 through C12, >C12 through C16 , >C16 through C21 and >C21 through C34 Aromatic Hydrocarbons and the surrogate standard COD.

11.3 Data Reporting Format

11.3.1 The following information and data must be reported:

11.3.1.1 The sample matrix (aqueous, soil or sediment);

11.3.1.2 The date(s) the sample was collected, received by the laboratory, extracted, and analyzed;

11.3.1.3 A description of the sample(s) received by the laboratory, relative to the physical condition of the containers, the temperature of the samples, and use of appropriate preservatives;

11.3.1.4 Moisture content (for soil/sediment samples);

11.3.1.5 The calculated concentrations of C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 Aliphatic Hydrocarbons, and C8 through C10, >C10 through C12, >C12 through C16, >C16 through C21 and >C21 through C34 Aromatic Hydrocarbons.

11.3.1.6 Surrogate recovery (expressed as percent recovery);

11.3.1.7 The concentration units for aqueous samples are expressed as ug/L or mg/L and soil or sediment samples the units are expressed as µg/Kg or mg/Kg on a dry-weight basis.

12.0 METHOD PERFORMANCE

12.1 **Method Detection Limits (Optional)**

12.1.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.1.2 The MDL is determined according to Equation 15.

Equation 15

$$MDL = (t) \times (SD)$$

where:

t = student t value at the 99% confidence level.

SD = standard deviation of the replicate analysis.

Student t values are as follows:

Number of replicates	t value
7	3.14
8	3.00
9	2.90
10	2.82

12.2 **Single Laboratory Accuracy, Precision and MDL Data**

Single laboratory accuracy, precision and MDL data for method analytes are provided in Tables 1-1 through 1-4 in Appendix 1. Tables 1-1 and 1-2 present data collected from spiking reagent water at 5.0 µg/L; Tables 1-3 and 1-4 by spiking sand at 1.0 mg/kg.

13.0 REFERENCES

1. Massachusetts Department of Environmental Protection and ABB Environmental Services, Inc., Wakefield, MA "Interim Petroleum Policy: Development of Health-based Alternative to the Total Petroleum Hydrocarbon (TPH) Parameter," August 1994.

2. USEPA, "Measurement of Petroleum Hydrocarbons: Report on Activities to Develop a Manual." Prepared by Midwest Research Institute, Falls Church, VA, under EPA Contract #68-WO-0015, WA No. 4; submitted to USEPA Office of Underground Storage Tanks, Washington, DC; November 20, 1990.

3. USEPA Federal Register 40 CFR Part 136, Appendix B, "Guidelines Establishing Test procedures for the Analysis of Pollutants," July 1992.

4. USEPA Test Methods for Evaluating Solid Waste (SW-846); Method 3510: Separatory Funnel Liquid-Liquid Extraction; September 1986.

5. USEPA Test Methods for Evaluating Solid Waste (SW-846); Method 3540: Soxhlet Extraction; September 1986.

6. USEPA Test Methods for Evaluating Solid Waste (SW-846); Method 3630: Silica Gel Cleanup; September 1986.

7. USEPA Test Methods for Evaluating Solid Waste (SW-846): Method 8000: Gas Chromatography; September 1986.

8. USEPA Test Methods for Evaluating Solid Waste (SW-846); Method 8100: Polynuclear Aromatic Hydrocarbons; September 1986.

9. Wisconsin Department of Natural Resources, "Modified DRO - Method for Determining Diesel Range Organics," PUBL-SW-141, 1992.

APPENDIX 1

SINGLE LABORATORY ACCURACY, PRECISION, AND METHOD DETECTION LIMITS (MDL) DATA FOR VPH

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Table 1-1. Single Laboratory Accuracy, Precision, and Method Detection Limits (MDLs) for Compounds in Component Standard Spiked Into Reagent Water and Analyzed by the VPH Method

Compound	Spiked Conc. (µg/L)	Method Accuracy[a] (Mean % Recovery[b])		Method Precision[a] (RSD[c] - %)		MDL[a] (µg/L)	
		PID[d]	FID[e]	PID	FID	PID	FID
n-Pentane	6.0		98		7.8		1.4
Methyl tert butylether	1.8	77	79	4.8	15	0.21	0.69
Benzene	0.6	82	97	4.9	10	0.08	0.19
Toluene	0.6	79	104	7.3	6.5	0.11	0.13
n-Nonane	2.0		83		8.0		0.42
Ethylbenzene	0.6	77	94	4.2	9.1	0.06	0.16
m- & p-Xylene	1.2	68	85	4.3	6.4	0.11	0.20
o-Xylene	0.6	79	88	16	6.4	0.23	0.11
1,2,3-Trimethylbenzene	0.6	69	89	8.2	6.4	0.11	0.11
Naphthalene	3.6	58	56	16	18	1.0	1.2
2,5-Dibromotoluene (surrogate)	40	88	92	4.9	13		

[a] Based on analysis of seven samples spiked with component standard.
[b] Recovery (%) of spiked concentration.
[c] RSD = relative standard deviation (%) of mean concentration measured.
[d] PID = photoionization detector.
[e] FID = flame ionization detector.

Table 1-2. Single Laboratory Accuracy, Precision, and Method Detection Limits (MDLs) for Compounds in Component Standard Spiked Into VPH- Free Sand and Analyzed by the VPH Method

Compound	Spiked Conc. (µg/Kg)	Method Accuracy[a] (Mean % Recovery[b])		Method Precision[a] (RSD[c] - %)		MDL[a] (µg/Kg)	
		PID[d]	FID[e]	PID	FID	PID	FID
n-Pentane	120		124		6.0		28
Methyl tert butylether	120	70	96	3.1	27	8.1	99
Benzene	60	122	128	3.3	5.4	7.6	13
Toluene	60	139	137	4.8	8.6	13	22
n-Nonane	120		119		4.6		21
Ethylbenzene	60	107	103	4.3	5.0	8.6	9.7
m- & p-Xylene	120	109	104	4.1	5.2	17	20
o-Xylene	60	103	111	3.8	6.6	7.4	14
1,2,3-Trimethylbenzene	60	93	102	5.5	5.3	9.6	10
Naphthalene	ND[f]	ND	ND	ND	ND	ND	ND
2,5-Dibromotoluene (surrogate)	2,000	119	117	8.6	9.7		

[a] Based on analysis of seven samples spiked with component standard.
[b] Recovery (%) of spiked concentration.
[c] RSD = relative standard deviation (%) of mean concentration measured.
[d] PID = photoionization detector.
[e] FID = flame ionization detector.
[f] ND = not determined.

Table 1-3. Single Laboratory Accuracy and Precision for Compounds in Component Standard Spiked Into Reagent Water at Concentrations in the Low to Middle End of the Analytical Range and Then Analyzed by the VPH Method

Compound	Spiked Conc. (µg/L)	Method Accuracy[a] (Mean % Recovery[b])		Method Precision[a] (RSD[c] - %)	
		PID[d]	FID[e]	PID	FID
n-Pentane	40		96		7.7
Methyl tert butylether	60	87	88	4.0	3.9
Benzene	20	99	99	4.4	3.7
Toluene	60	99	99	4.1	4.0
n-Nonane	40		104		10
Ethylbenzene	20	99	99	4.2	4.2
m- & p-Xylene	80	99	99	4.3	4.2
o-Xylene	40	99	99	3.7	3.6
1,23-Trimethylbenzene	40	99	99	4.1	4.0
Naphthalene	40	70	81	4.3	3.2
2,5-Dibromotoluene (surrogate)	40	92	94	5.2	4.6

[a] Based on analysis of five samples spiked with component standard.
[b] Recovery (%) of spiked concentration.
[c] RSD = relative standard deviation (%) of mean concentration measured.
[d] PID = photoionization detector.
[e] FID = flame ionization detector.

Table 1-4. Single Laboratory Accuracy and Precision for Compounds in Component Standard Spiked Into VPH-Free Sand at Concentrations in the Low to Middle End of the Analytical Range and Then Analyzed by the VPH Method

Compound	Spiked Conc. (mg/Kg)	Method Accuracy[a] (Mean % Recovery[b])		Method Precision[a] (RSD[c] - %)	
		PID[d]	FID[e]	PID	FID
n-Pentane	2.0		118		1.9
Methyl tert butylether	3.0	100	94	2.4	2.8
Benzene	2.0	119	109	3.0	0.7
Toluene	2.0	118	109	1.7	1.3
n-Nonane	ND	ND	ND	ND	ND
Ethylbenzene	1.0	121	110	1.4	1.2
m- & p-Xylene	3.0	122	110	1.4	1.1
o-Xylene	2.0	118	107	1.3	1.1
1,2,3-Trimethylbenzene	2.0	115	109	1.4	1.3
Naphthalene	2.0	61	57	11	1.8
2,5-Dibromotoluene (surrogate)	2.0	75	64	9.7	3.3

[a] Based on analysis of five samples spiked with component standard.
[b] Recovery (%) of spiked concentration.
[c] RSD = relative standard deviation (%) of mean concentration measured.
[d] PID = photoionization detector.
[e] FID = flame ionization detector.
[f] ND = not determined.

APPENDIX 2

SUGGESTED VPH DATA REPORTING FORMAT

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VOLATILE PETROLEUM HYDROCARBON (VPH) ANALYSIS

Client:

Client Sample ID: Date Received:

Laboratory Sample ID: Date Analyzed:

Matrix: Date Reported:

Percent Moisture:

VPH RESULTS

Parameter	Results	PQL				Units
C5-C6 Aliphatics (FID) *						
C6-C8 Aliphatics (FID) *						
C8-C10 Aliphatics (FID) *						
C10-C12 Aliphatics (FID) *						
C8-C10 Aromatics (PID)						
C10-C12 Aromatics (PID)						
C12-C13 Aromatics (PID)						

*** Excludes any MTBE or BTEX compounds**

SURROGATE RECOVERY

Surrogate	%Recovery	Acceptance Range
2,5-dibromotoluene		80% - 120%

TARGETED VPH ANALYTES

Analyte	**Results**	**PQL**	**Units**
Methyl tert butylether			
Benzene			
Toluene			
Ethylbenzene			
p-Xylene *			
m-Xylene *			
o-Xylene			
Naphthalene			

* p and m-xylenes may not be able to be resolved and may be reported as the total of p and m-xylenes.

APPENDIX 3

SINGLE LABORATORY ACCURACY, PRECISION, AND METHOD DETECTION LIMIT (MDL) DATA FOR EPH

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Table 1-1. Single Laboratory Accuracy, Precision, and Method Detection Limits (MDLs) for Alkanes Spiked Into Reagent Water and Analyzed by the EPH Method

Compound[a]	Compound Conc. Measured (µg/L)		Mean Accuracy (Mean % Recovery[b])	Method Precision (RSD[c] - %)	MDL (µg/L)
	Mean	Std. Dev.			
C_{10}	4.1	0.040	82	0.98	0.12
C_{12}	4.1	0.021	82	0.51	0.064
C_{16}	4.0	0.062	80	1.6	0.19
COD[d]	44	0.93	88	2.1	-
C21	3.7	0.047	74	1.3	0.14

[a] Compounds were spiked into 7 or 8 samples at a concentration of 5.0 µg/L.
[b] Recovery (%) of spiked concentration.
[c] RSD = relative standard deviation (%) of mean concentration measured.
[d] Surrogate (COD = 1-Chloro-octadecane) was spiked into three samples at a concentration of 50 µg/L.

Table 1-2. Single Laboratory Accuracy, Precision, and Method Detection Limits (MDLs) for Polynuclear Aromatic Hydrocarbons (PAHs) Spiked Into Reagent Water and Analyzed by the EPH Method

Compound[a]	Compound Conc. Measured (µg/L)		Mean Accuracy (Mean % Recovery[b])	Method Precision (RSD[c] - %)	MDL (µg/L)
	Mean	Std. Dev.			
Naphthalene	5.6	0.088	112	1.6	0.26
2-Methylnaphthalene	6.4	0.096	128	1.5	0.29
Acenaphthylene	5.6	0.085	112	1.5	0.26
Acenaphthene	5.7	0.092	114	1.6	0.28
Fluorene	5.6	0.094	112	1.7	0.28
Phenanthrene	5.5	0.097	110	1.8	0.29
Anthracene	7.0	0.10	140	1.4	0.31
OTP[d]	44	1.6	88	3.6	-
Fluoranthene	5.7	0.090	114	1.6	0.27
Pyrene	6.1	0.098	122	1.6	0.29
Benzo(a)Anthracene	5.9	0.089	118	1.5	0.27
Chrysene	5.8	0.098	116	1.7	0.29
Benzo(b)Fluoranthene	6.3	0.082	126	1.3	0.25
Benzo(k)Fluoranthene	5.6	0.065	112	1.2	0.20
Benzo(a)Pyrene	6.0	0.075	120	1.2	0.22
Indeno(123 cd)Pyrene	5.1	0.17	102	3.3	0.50
Dibenzo(ah)Anthracene	2.8	0.21	56	7.5	0.62
Benzo(ghi)Perylene	3.8	0.18	76	4.7	0.54

[a] Compounds were spiked into 7 or 8 samples at a concentration of 5.0 µg/L.
[b] Recovery (%) of spiked concentration.
[c] RSD = relative standard deviation (%) of mean concentration measured.
[d] Surrogate (OTP = ortho-Terphenyl) was spiked into three samples at a concentration of 50 µg/L.

Table 1-3. Single Laboratory Accuracy, Precision, and Method Detection Limits (MDLs) for Alkanes Spiked Into EPH-Free Sand and Analyzed by the EPH Method

Compound[a]	Compound Conc. Measured (mg/Kg)		Mean Accuracy (Mean % Recovery[b])	Method Precision (RSD[c] - %)	MDL (mg/Kg)
	Mean	Std. Dev.			
C_{10}	0.72	0.074	72	10	0.22
C_{12}	0.83	0.079	83	9.5	0.24
C_{16}	1.2	0.14	120	12	0.42
COD[d]	4.3	0.48	86	11	-
C21	0.81	0.044	81	5.4	0.13

[a] Compounds were spiked into 8 samples at a concentration of 1.0 mg/Kg.
[b] Recovery (%) of spiked concentration.
[c] RSD = relative standard deviation (%) of mean concentration measured.
[d] Surrogate (COD = 1-Chloro-octadecane) was spiked into three samples at a concentration of 5.0 mg/kg.

Table 1-4. Single Laboratory Accuracy, Precision, and Method Detection Limits (MDLs) for Polynuclear Aromatic Hydrocarbons (PAHs) Spiked Into EPH-Free Sand and Analyzed by the EPH Method

Compound[a]	Compound Conc. Measured (mg/Kg)		Mean Accuracy (Mean % Recovery[b])	Method Precision (RSD[c] - %)	MDL (mg/Kg)
	Mean	Std. Dev.			
Naphthalene	0.64	0.063	64	9.8	0.19
2-Methylnaphthalene	0.64	0.058	64	9.1	0.17
Acenaphthylene	0.68	0.060	68	8.8	0.18
Acenaphthene	0.70	0.064	70	9.1	0.19
Fluorene	0.79	0.060	79	7.6	0.18
Phenanthrene	0.83	0.041	83	4.9	0.12
Anthracene	1.2	0.082	120	6.8	0.25
OTP[d]	4.8	0.45	96	9.4	-
Fluoranthene	0.92	0.038	92	4.1	0.12
Pyrene	0.91	0.037	91	4.1	0.11
Benzo(a)Anthracene	0.96	0.042	96	4.4	0.12
Chrysene	0.90	0.050	90	5.6	0.15
Benzo(b)Fluoranthene	0.96	0.038	96	4.0	0.11
Benzo(k)Fluoranthene	0.87	0.037	87	4.3	0.11
Benzo(a)Pyrene	0.96	0.036	96	3.8	0.11
Indeno(123 cd)Pyrene	0.90	0.033	90	3.7	0.099
Dibenzo(ah)Anthracene	0.82	0.033	82	4.0	0.099
Benzo(ghi)Perylene	0.82	0.032	82	3.9	0.096

[a] Compounds were spiked into 7 or 8 samples at a concentration of 1.0 mg/Kg.
[b] Recovery (%) of spiked concentration.
[c] RSD = relative standard deviation (%) of mean concentration measured.
[d] Surrogate (OTP = ortho-Terphenyl) was spiked into three samples at a concentration of 5.0 mg/kg.

APPENDIX 4

SUGGESTED EPH DATA REPORTING FORMAT

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EXTRACTABLE PETROLEUM HYDROCARBON (EPH) ANALYSIS

Client:	
Client Sample ID:	Date Received:
Laboratory Sample ID:	Date Extracted:
Matrix:	Date Analyzed:
Percent Moisture:	Date Reported:

EXTRACTABLE PETROLEUM HYDROCARBON (EPH)

Parameter	Results	PQL				Units
C8-C10 Aliphatics						
C10-C12 Aliphatics						
C12-C16 Aliphatics						
C16-C21 Aliphatics						
C21-C34 Aliphatics						
C8-C10 Aromatics						
C10-C12 Aromatics						
C12-C16 Aromatics						
C16-C21 Aromatics						
C21-C34 Aromatics						

SURROGATE RECOVERIES

Surrogate	%Recovery	Acceptance Range
Chloro-octadecane (COD)		50% - 150%
Ortho-terphenyl (OTP)		50% - 150%

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APPENDIX 5

REPORT OF ANALYSIS

SUMMARY SHEET

Client: Client Sample ID Number(s): Laboratory ID Number(s):

SAMPLE INFORMATION

Sample Matrix	¨ Aqueous ¨ Soil ¨ Sediment ¨ Other:	
Analysis Performed	¨ VPH ¨ EPH ¨ VPH and EPH (E-TPH Duplicate samples)	
Condition of Containers	¨ Satisfactory ¨ Broken ¨ Leaking ¨ Other:	
Sample Preservatives	AQUEOUS	¨ N/A ¨ pH $\leq$ 2 ¨ pH > 2
	SOIL/SEDIMENT	¨ N/A
Sample Temperature	¨ Received on Ice ¨ Received at 4 °C ¨ Other	

ANALYTICAL RESULTS

Parameter	Results	PQL	Units
Volatile Petroleum Hydrocarbons (VPH)			
Extractable Petroleum Hydrocarbons (EPH)			
Equivalent TPH (E-TPH) ** [IF BOTH VPH AND EPH ANALYSES PERFORMED]			

**** Due to overlapping carbon ranges between the VPH and EPH methods and in order to avoid double counting, the analyst must select the higher of the two values to be included for reporting purposes for those ranges effected.**

COMMENTS

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APPENDIX 6*

Petroleum Product Chromatograms

Carburator Cleaner
Automotive Gasoline
Mineral Spirits #1
Mineral Spirits #2
JP-4 Jet Fuel (old)
JP-4 Jet Fuel (new)
JP-5 Jet Fuel
Kerosene
Kerosene (ICP grade)
Gasoline and #2 Diesel Oil
Natural Gas Condensate
#2 Fuel Oil
#2 Fuel Oil (38% aromatic) (See chromatogram in NWTPH-Dx)
#2 Diesel Oil (See chromatogram in NWTPH-Dx)
#2 Diesel Oil and Motor Oil (30w) (See chromatogram in NWTPH-Dx)
2-Cycle Engine Oil #1
2-Cycle Engine Oil #2
2-Cycle Engine Oil #3
Motor Oil (30w) (See chromatogram in NWTPH-Dx)
Automatic Transmission Fluid
Power Steering Fluid
Transformer Oil (used)
Transformer Oil (new)
Hydraulic Fluid #1
Hydraulic Fluid #2
Hydraulic Fluid #3
Mineral Oil #1
Mineral Oil #2
Cutting Oil #1
Cutting Oil #2
Bunker-C #1
Bunker-C #2
CSS-1 (emulsion asphalt)
AR-4000 (asphalt cement)

Non-petroleum Product Chromatograms

Turpentine
Creosote
Synthetic Motor Oil

*** The chromatograms are not available in the "electronic" version. They are included in the printed publication.**

Chromatograms: NWTPH-Gx

Gasoline
Weathered Gasoline
Naphtha
Mineral Spirits #1
Mineral Spirits #2
Mineral Spirits #3

Chromatograms: NWTPH-Dx

#2 Diesel Oil
#2 Diesel Oil/Motor Oil
#2 Fuel Oil (38% Aromatic)
Kerosene (Deodorized)
Jet Fuel A
Bunker C #1
Bunker C #2
Motor Oil 30 Wgt.
Mineral Oil (USP)
Hydraulic Fluid
Transformer Oil
Gas Oil