

# DATA VALIDATION REPORT

POPLAR POINT  
WASHINGTON, DC

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## 1.0 INTRODUCTION

This report summarizes the validation of analytical results generated from field sampling in November of 2002, in support of the site characterization for Poplar Point. Sampling and analyses were performed according to the *Soil Boring and Monitoring Well Installation Work Plan (Revision 1)* (Work Plan), (Ridolfi, October 2002). The criteria applied for this validation were consistent with U.S. EPA SW-846 analytical methods, laboratory established criteria, and the *U.S. EPA Contract Laboratory Program National Functional Guidelines (Functional Guidelines)*, (U.S. EPA, 1994). Qualified analytical results are presented in Appendix D. Data qualifiers applied to sample results are in accordance with the *Functional Guidelines*; qualifiers potentially applied are summarized below:

- U: The analyte was analyzed for, but was not detected above the reported sample quantitation limit.
- J: The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
- UJ: The analyte was not detected above the sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
- NJ: The analysis indicates the presence of an analyte that has been 'tentatively identified' and the associated numerical value represents its approximate concentration.
- R: The sample results are rejected due to serious deficiencies in the ability to analyze the sample and meet quality control criteria. The presence or absence of the analyte can not be verified.

This report represents review of all samples analyzed by the laboratory, including re-extractions (re) and dilutions (dil). Results for multiple analyses of the same sample material were selectively reported in Appendix D based on their usability. Consequently, certain results discussed in this report may not appear in Appendix D.

## 2.0 PETROLEUM HYDROCARBON ANALYSES - Diesel and Motor Oil – NWTPH-Dx

Petroleum hydrocarbon analyses were performed by Analytical Resources, Inc. (ARI) of Seattle, Washington, in accordance with the requirements of the Work Plan. The method used to determine diesel and motor oil in the samples is referenced as Northwest TPH-Dx (NWTPH-Dx [diesel range = C<sub>12</sub> - C<sub>24</sub>, lube or motor oil range = C<sub>24</sub> - C<sub>38</sub>]) - Semivolatile Petroleum Products Method for Soil and Water Analyses, as established by the Washington State Department of Ecology.

Forty-three samples were analyzed for NWTPH-Dx, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	22	0	0	22
Soil	19	2	0	21
Totals	41	2	0	43

The laboratory provided U.S. EPA CLP style deliverables for all sample delivery groups. Sample results are presented with associated data qualifiers in Appendix D.

**Sample Documentation, Custody and Holding Conditions/Times:** All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables were complete. Maximum holding times for extractables are specified as 14/40 days (sample/extract maximum holding times) and 7/40 days for solids and waters, respectively, at 4° C. ( $\pm 2^\circ$  C.). All holding times were met.

Upon receipt at the laboratory, the transport cooler temperature for SDG FA32 was 14.5 °C. The data were not qualified due to the elevated cooler temperatures since the elevated temperatures were not expected to significantly affect data quality and the sample integrity was considered acceptable since the samples were originally placed in coolers containing ice.

**Initial Calibration:** The laboratory performed initial multipoint calibration (linearity verification) for #2 diesel fuel at 25, 100, 250, 1000 and 2500 µg/mL (9/12/02 & 9/18/02); and motor oil at 50, 100, 250, 500, 2500 and 5000 µg/mL. The USEPA CLP *Functional Guidelines* specify that Relative Standard Deviations (RSDs) must be  $\leq 30\%$  for target analytes. Initial calibration levels, frequencies, and linearities were within pertinent guidance limits.

**Calibration Checks:** The laboratory performed calibration verifications for diesel and motor oil at concentrations of 250 µg/mL and 500 µg/mL, respectively. The acceptance window for the calibration check is  $\pm 15\%$  of the true value. Calibration check frequencies for all analyses were after every ten sample analyses. These criteria were well within the USEPA CLP *Functional Guidelines* specifications for similar parameters (Percent Differences (%Ds) must be  $\leq 25\%$ ). No results required qualification based on out-of-compliance procedures and performance criteria.

**Blanks:** Analytical method blanks were analyzed at the required frequency (once per analytical delivery group of 20 samples, or less). A total of six method blanks were analyzed, and no analyte responses were reported.

**Surrogate Compound Performance:** A surrogate compound was added to each sample prior to analysis to assess analytical performance for the sample. The surrogate compound was identified as o-terphenyl for both diesel and motor oil analyses. Surrogate performance was evaluated against the laboratory-established limits for NWTPH-D (water: 32-116%, soil 30-106%). All recoveries were within specification.

**Matrix Spike/Matrix Spike Duplicate Analyses:** Sample 470E-PZ5-01, 470E-SS-29-01 and 470E-SB41-01 were analyzed as matrix spike and spike duplicate pairs. Diesel fuel was evaluated at 178 mg/kg and 3 mg/L. Laboratory-established control limits applied were 26-132% for soil and 57-151% for water. All recoveries were acceptable.

**Laboratory Duplicate Analysis:** Samples 470E-SS-29-02 and 470E-SB-41-02 were analyzed in duplicate by the laboratory. The results of the laboratory duplicate analysis were below the precision limit of 35% RPD and were acceptable.

**Laboratory Control Samples:** Spiked blanks (LCS) were analyzed in conjunction with the matrix spikes and method blanks. Six LCS samples were analyzed for diesel. The LCS spiking levels were 150 mg/kg and 3 mg/L. Performance was considered acceptable and the analytical systems were determined to be in control.

**Compound Quantitation and Reported Detection/Quantitation Limits:** Compound quantitation and detection limits were acceptable as reported. No anomalies were identified.

**System Performance:** Raw data show no indication of degradation of system performance during or between analytical runs. Chromatograms show no abrupt shifts in baseline, high background levels, excessive baseline rise with increased temperature, or other indications of system performance degradation.

**Field Replicates:** One field replicate sample pair was submitted and analyzed for petroleum hydrocarbons for determination of analytical variability. Sample results for replicate pair (470E-SB32-01, 470E-SB32-03) are presented in Appendix D. RPDs for the pair are greater than 100%, indicating poor comparability. No results were qualified based on field replicate performance.

**Overall Assessment:** All deliverables required by the project were present and data packages were complete. Sample holding times and shipping requirements were met with the exception noted above. Initial calibration and calibration check requirements were met. Method blank performances were within specification. Surrogate compound recoveries were within specification. MS/MSD results were acceptable. Compound identification and quantitation were acceptable. Raw data show no indications of system performance degradation. Reporting limits were sufficient. Field replicate analyses showed poor agreement, suggesting sample heterogeneity. Overall analytical performance was considered acceptable, and data quality sufficient for project use.

### 3.0 SEMIVOLATILE ORGANICS ANALYSES - U.S. EPA SW-846, Method 8270C and Selective Ion Monitoring

Semivolatile organics analyses were performed by Analytical Resources, Inc. (ARI) of Seattle, Washington, in accordance with the requirements of the Work Plan. Soil and water samples were analyzed for semivolatile organic compounds and several soils were also analyzed for polyaromatic hydrocarbons (PAHs) using Method 8270C modified for selective ion monitoring (SIM) to enhance sensitivities to 0.25 parts per billion.

Fourteen samples were analyzed for semivolatile organics and/or selected PAHs, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	2	0	0	2
Soil	12	0	0	12
Totals	14	0	0	14

The laboratory provided U.S. EPA CLP style deliverables for all sample delivery groups. Sample results are presented with associated data qualifiers in Appendix D.

**Sample Documentation, Custody and Holding Conditions/Times:** All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables were complete. Maximum holding times for extractables are specified as 14/40 days (sample/extract maximum holding times) and 7/40 days for solids and waters, respectively, at 4° C. ( $\pm 2^\circ$  C.). All extraction and analytical holding times were met.

Upon receipt at the laboratory, the transport cooler temperature for SDG FA32 was 14.5 °C. The data were not qualified due to the elevated cooler temperatures since the elevated temperatures were not expected to significantly affect data quality and the sample integrity was considered acceptable since the samples were originally placed in coolers containing ice.

**GC/MS Tuning:** GC/MS tune performance was checked using decafluorotriphenylphosphine (DFTPP) prior to all initial calibrations runs and all subsequent sample analytical runs. All sample analyses were performed within 12 hours of DFTPP analyses. All ion abundances and relative ion abundances met *Functional Guidelines* acceptance criteria. Review of mass spectral plots and associated mass listings supplied with the raw data, and transcription of mass data to Form V (GC/MS Tuning and Mass Calibration) showed no inconsistencies. No errors were identified during spot checks of calculations.

**Initial Calibration:** Initial multipoint calibrations were performed at 5, 10, 25, 40 and 60 µg/mL for SVOC 8270C analysis; 0.25, 0.5, 1.0, 5.0 and 10 µg/mL for SIM analysis, for all target compounds and surrogate compounds. The USEPA CLP *Functional Guidelines* specify that Average Relative Response Factors (Average RRFs) must be  $\geq 0.05$ , and selected analytes must meet additional minimum RRF and maximum %RSD criteria ( $>30\%$ ). Average RRFs for all Target Compound List (TCL) compounds in all initial calibrations showed compliance with technical requirements. All requirements were met.

**Continuing Calibration:** Continuing calibrations were performed for all TCL compounds at 2.5 µg/mL for SIM analysis, and 25 µg/mL for SVOC 8270C analysis. The USEPA CLP *Functional Guidelines* specify that RRFs must be  $\geq 0.05$ , and compounds must meet a maximum percent difference (%D) criteria ( $\leq 25\%$ ). All RRFs were in compliance while deviations from the %D criteria were as follows:

Calibration Date	Compound	Samples Affected	Qualifier
12/12/02	Benzoic acid, Hexachlorocyclopentadiene, 2,4-dinitrophenol	470E-SS-29-03dil	UJ
12/12/02	2,4-dinitrophenol, 4-nitroaniline	470E-SS-30, 470E-SS-31, 470E-SS-32	UJ

**Blanks:** Analytical method blanks were analyzed at least once for each analytical group and matrix, as specified. Method blank results were acceptable.

**Surrogate Compound Performance:** Surrogate compounds were added to each sample prior to analysis to assess analytical performance on each sample. Surrogate compounds and associated performance criteria for the analysis of TCL analytes in soil and water matrices were those specified in U.S. EPA Method SW-846 8270. Laboratory-established surrogate compound recovery specifications were as follows:

Compound	Acceptance Range (%)	Acceptance Range (%)
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	Water	Soil
d <sub>5</sub> -Nitrobenzene	49-118	20-102
2-Fluorobiphenyl	53-106	34-99
d <sub>14</sub> -p-Terphenyl	45-126	43-128
d <sub>5</sub> -Phenol	43-112	29-93
2-Fluorophenol	47-109	25-92
2,4,6-Tribromophenol	34-142	23 - 131
d <sub>4</sub> -2-Chlorophenol	51-110	32 - 91
d <sub>4</sub> -1,2-Dichlorobenzene	39-92	26 - 85
<b>SIM</b>		
d10-2-methylnaphthalene	36-122	24-114
d14-dibenzo(a,h)anthracene	18-139	10-130

According to the U.S. EPA *Functional Guidelines*, if two or more surrogates of a compound class (neutrals vs. acids) are outside specification, the associated results require qualification. The following is a summary of the samples in which two or more surrogate recoveries for a fraction are outside the control limits:

Surrogate	Affected Sample	Compounds Qualified	Qualifier
d <sub>5</sub> -Phenol 2-Fluorophenol d <sub>4</sub> -2-Chlorophenol	470E-SS-29-02	acid	J, UJ
2-Fluorobiphenyl d <sub>4</sub> -1,2-Dichlorobenzene 2-Fluorophenol d <sub>4</sub> -2-Chlorophenol	470E-SS-29-01	all	J, UJ
d <sub>5</sub> -Phenol 2-Fluorophenol d <sub>4</sub> -2-Chlorophenol	470E-SS-28d	acid	J, UJ

**Matrix Spike/Matrix Spike Duplicate Analyses:** Matrix spike (MS) and matrix spike duplicate (MSD) analyses were performed on soil samples 470E-SW-01 and 470E-SS-28s. Analyte spike concentrations were 387 - 4830 µg/kg for low level soils (8270C) and 2.5 - 3.7 µg/L for SIM water analysis. MS/MSD compounds and associated performance criteria for the analysis of TCL analytes in soil and water matrices were those specified in U.S. EPA Method SW-846 8270. Laboratory-established spike compound recovery specifications were:

Compound	Recovery Acceptance Range (%)	Recovery Acceptance Range (%)
	Water	Soil
Phenol	47-129	11-118
2-Chlorophenol	49-131	11-115
1,4-Dichlorobenzene	20-88	10-91
N-Nitroso-di-n-propylamine	38-112	10-96
1,2,4-Trichlorobenzene	20-85	10-94
4-Chloro-3-methylphenol	48-113	15-111
Acenaphthene	42-110	13-113
4-Nitrophenol	42-155	10-148
2,4-Dinitrotoluene	39-127	10-126
Pentachlorophenol	26-175	14-153
Pyrene	43-110	12-120
<b>SIM</b>		
Phenanthrene	49-118	10-131
Benzo(k)fluoranthene	38-124	10-132
Chrysene	42-117	10-128

The recoveries (%R) for the matrix spike analyses were acceptable.

**Laboratory Control Samples:** Laboratory control samples (LCSs) were analyzed per batch for the samples. Laboratory-established LCS recovery specifications were as follows:

Compound 8270C	Recovery Acceptance Range (%)	Recovery Acceptance Range (%)
	Water	Soil
Phenol	59-119	43-108
2-Chlorophenol	63-120	46-104
1,4-Dichlorobenzene	27-85	35-83
N-Nitroso-di-n-propylamine	44-111	25-94
1,2,4-Trichlorobenzene	26-83	29-85
4-Chloro-3-methylphenol	50-101	42-98
Acenaphthene	51-108	40-99
4-Nitrophenol	39-130	27-133
2,4-Dinitrotoluene	44-118	34-108
Pentachlorophenol	34-144	51-128
Pyrene	37-126	32-122
<b>SIM</b>		
Phenanthrene	58-114	48-112
Benzo(k)fluoranthene	50-115	44-123
Chrysene	58-108	42-118

The LCS results were compared to the above criteria. The LCS concentrations were 200 - 2500 µg/kg for low level soils and 25 - 37 µg/L for low level waters for (8270C), and 3 µg/L for SIM analysis. LCS performance indicates the analytical systems were in control. All LCS recoveries were acceptable.

**Internal Standards Performance:** Six internal standards were added at a concentration of 20 ng/µL to all sample extracts prior to instrumental analyses of the full list of ABNs, including quality control samples. Retention times (RT) for all internal standards were within method specified acceptance limits

in all samples ( $\pm 30$  seconds). Internal standard areas were within method specified acceptance limits (0.5 to 2 times the 12-hour calibration standard area) for all samples.

**TCL Compound Identification:** Relative retention times (RRTs) for all reported TCL compounds are within the method specified acceptance limits ( $\pm 0.06$  RRT units). Mass spectra generally show good comparison with library reference spectra.

**Compound Quantitation and Reported Detection/Quantitation Limits:** The detection limits were acceptable as reported. No problems were noted.

**System Performance:** Raw data showed no indication of degradation of system performance during or between analytical runs. Reconstructed ion chromatograms (RICs) showed no abrupt shifts in baseline, high background levels, excessive baseline rise with increased temperature, or other indications of system performance degradation.

**Overall Assessment:** All deliverables required by the project were present and data packages were complete. With the exception of the elevated cooler temperatures, the holding time criteria were met. GC/MS tuning requirements were met. Initial and continuing calibration requirements were acceptable. Internal standard (IS) performance was acceptable. Surrogate performance was acceptable with exceptions noted. Compound identification and quantitation were acceptable. Raw data showed no indications of system performance degradation. Overall analytical performance was considered acceptable, and data quality sufficient for project use.

#### 4.0 PESTICIDES - U.S. EPA SW-846, Method 8081A

Pesticide analyses were performed by Analytical Resources, Inc. (ARI) of Seattle, Washington, in accordance with the requirements of the Work Plan. The samples were analyzed using EPA SW-846 method 8081A.

Forty-seven samples were analyzed for pesticides, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	12	0	0	12
Soil	35	0	0	35
Totals	47	0	0	47

The laboratory provided U.S. EPA CLP style deliverables for all sample delivery groups. Sample results are presented with associated data qualifiers in Appendix D.

**Sample Documentation, Custody and Holding Conditions/Times:** All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables were complete. Maximum holding times for extractables were specified as 14/40 days (sample/extract maximum holding times) for solids and 7/40 for waters, at 4° C. ( $\pm 2^\circ$  C). All extraction and analysis holding times were met.

Upon receipt at the laboratory, the transport cooler temperature for SDG FA32 was 14.5 °C. The data were not qualified due to the elevated cooler temperatures since the elevated temperatures were not



expected to significantly affect data quality and the sample integrity was considered acceptable since the samples were originally placed in coolers containing ice.

**Instrument Performance:** The breakdown of the 4,4'-DDT and Endrin were evaluated at the beginning of every 12-hour shift using a standard that contained only 4,4'-DDT and Endrin. Degradation of these compounds occurs in the injection port. The percent breakdown of 4,4'-DDT and Endrin were acceptable (<20%) with the following exceptions, requiring qualification of the associated results:

Breakdown Check Date	Sample	Compound	Qualifier
12/4/02	470E-SS-29-01, 470E-SS-28s, 470E-SS-29-02	4,4'-DDT	J
12/4/02	470E-SS-29-01, 470E-SS-28s, 470E-SS-29-02	4,4'-DDE 4,4'-DDD	NJ
12/4/02	470E-SS-28d	4,4'-DDD 4,4'-DDT	NJ J
12/4/02	470E-SS-28dMS 470E-SS-28dMSD	4,4'-DDD	NJ

**Initial Calibration:** Initial five point calibrations were performed for all pesticides, with varying concentrations depending on the compound. Retention windows were defined along with calibration factors for each compound at each calibration concentration. The relative standard deviations for the calibration factors in the initial calibrations were <20% per method 8081A, demonstrating acceptable linearity.

**Continuing Calibration:** Per method 8081A, calibration verification was performed every 12 hours. Calibration standards were injected after the analysis of ten samples and at the end of each analytical sequence. The average percent differences of the calibration verification solution were less than 15%. The percent difference for several individual compounds exceeded 25%, which were qualified as estimated as follows:

Calibration Date	Compounds	Samples Affected	Qualifier
12/4/02	4,4'-DDT	470E-SS-28s, 470E-SS-29-01, 470E--SS-29-02	J

**Blanks:** Method blanks were analyzed for each analytical group. Method blanks showed no detections of target analytes above reporting limits.

**Surrogate Compound Performance:** Surrogate compounds tetrachloro-m-xylene and decachlorobiphenyl were added to each sample prior to analysis to assess analytical performance on each sample. Laboratory-established acceptance limits were 24-111% for tetrachloro-m-xylene and 20-119% for decachlorobiphenyl. All surrogate recoveries were acceptable.

**Matrix Spike/Matrix Spike Duplicate Analyses:** Matrix spike and matrix spike duplicate analyses were performed on 470E-SB33-01, 470E-SS-28d and 470E-SB41-01. Analyte spike concentrations ranged from 21 to 60 µg/kg. The following is a summary of the compounds and laboratory-established acceptance limits for the spiked samples:

Compound	Soil Acceptance Limits
Lindane	10-130
Heptachlor	10-127
Aldrin	10-112
Dieldrin	10-144
Endrin	10-147
DDT	10-161

MS/MSD performances were acceptable for all samples.

**Laboratory Control Samples:** Five spiked blanks (LCS) were analyzed with acceptable results. The laboratory-established acceptance windows for the analysis were as follows:

Compound	Water Acceptance Limits (%)	Soil Acceptance Limits (%)
Lindane	47-121	34-120
Heptachlor	54-103	49-108
Aldrin	48-94	43-102
Dieldrin	63-116	54-116
Endrin	64-122	56-123
DDT	48-127	48-127

LCS performance was considered acceptable for all samples.

**Target Compound Identification and Reporting Limits:** The compounds reported were within established retention time windows. The laboratory reported several compounds with a 'Y' qualifier, indicating an elevated detection limit. The 'Y' qualifier was considered a 'U' for data qualification purposes. The laboratory reported a few compounds with an 'S' qualifier, which indicated saturation of the detector. Since there was no compound concentration associated with an 'S' qualifier, no qualification was made when an 'S' was reported. Several compounds were qualified by the laboratory with an 'E' to indicate the compound concentration exceeded the calibration range. In all cases the laboratory diluted and reanalyzed the associated sample to bring the result within range. These latter results are reported in Appendix D.

**System Performance:** System and analytical performance was evaluated by the breakdown of 4,4'-DDT in addition to a review of chromatograms and quantitation reports. Sample 470E-SS-32 was qualified as estimated for 4,4'-DDT due to the difference between the primary and secondary analytical columns exceeding 40%.

**Overall Assessment:** All deliverables required by the project were present and data packages were complete. Recommended sample holding times and conditions were met with the exceptions noted above. Calibration requirements were met and acceptable with the exception of data that were qualified due to the percent difference exceedance. Method blanks showed no target analytes to be present. Compound identification and quantitation were acceptable as reported. Overall analytical performance was considered acceptable, and data quality sufficient for project use.

## 5.0 Polychlorinated Biphenyls - U.S. EPA SW-846, Method 8082

Pesticide analyses were performed by Analytical Resources, Inc. (ARI) of Seattle, Washington, in accordance with the requirements of the Work Plan. The samples were analyzed for polychlorinated biphenyls (PCBs) using EPA SW-846 method 8082.

Forty-one samples were analyzed for polychlorinated biphenyls, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	12	0	0	12
Soil	29	0	0	29
Totals	41	0	0	41

The laboratory provided U.S. EPA CLP style deliverables for all sample delivery groups. Sample results are presented with associated data qualifiers in Appendix D.

**Sample Documentation, Custody and Holding Conditions/Times:** All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are complete. Maximum holding times for extractables are specified as 14/40 days (sample/extract maximum holding times) for solids and 7/14 for waters, at 4° C. ( $\pm 2^\circ$  C.). All extraction and analysis holding times were met

Upon receipt at the laboratory, the transport cooler temperature for SDG FA32 was 14.5 °C. The data were not qualified due to the elevated cooler temperatures since the elevated temperatures were not expected to significantly affect data quality and the sample integrity was considered acceptable since the samples were originally placed in coolers containing ice.

**Initial Calibration:** Initial five point calibrations were performed for all Aroclors at 0.1, 0.25, 0.5, 1.0, and 2.0 ppm. Retention windows were defined along with calibration factors for each aroclor. The percent relative standard deviations for the calibration factors in the initial calibrations were <20%, with the following exceptions:

Calibration Date	Compounds	Samples Affected	Qualifier
11/27/02	Aroclor 1016, Aroclor 1260	all	J, UJ

**Continuing Calibration:** Calibration verification was performed every 12 hours. Aroclor standards were injected at the required frequency and at the end of each analytical sequence. The average percent differences of the calibration verification solution were less than 15%.

**Blanks:** Method blanks were analyzed for each analytical group. Method blanks showed no detections of target analytes above reporting limits.

**Surrogate Compound Performance:** Two surrogate compounds were added to each sample prior to analysis to assess analytical performance on each sample. The surrogates were identified as tetrachloro-m-xylene and decachlorobiphenyl. Surrogate performance was evaluated against the laboratory-established limits 20-118% (soil) and 36-104% (water) for tetrachloro-m-xylene and 16-146% (soil) and 26-131% (water) for decachlorobiphenyl. All recoveries were within specification.

**Matrix Spike/Matrix Spike Duplicate Analyses:** Matrix spike and matrix spike duplicate analyses were performed on 470E-SS-28d, 470E-SB41-01, 470E-PZ6-01 and 470E-SB33-01. Analyte spike concentrations for Aroclor 1242 were 419 - 598 µg/kg and 20.6 µg/L. The laboratory-established acceptance windows for the analysis were as follows:

Compound	Water Acceptance Limits (%)	Soil Acceptance Limits (%)
Aroclor 1242	47-115	15-143

The MS/MSD performances were acceptable for all samples.

**Laboratory Control Samples:** The LCS samples were analyzed for Aroclor 1242 at 333 µg/kg and 0.5 µg/L. LCS samples were analyzed with acceptable results. The laboratory-established acceptance windows for the analysis were as follows:

Compound	Water Acceptance Limits (%)	Soil Acceptance Limits (%)
Aroclor 1242	58-114	54-125

**Target Compound Identification and Reporting Limits:** The compounds reported were within established retention time windows. The laboratory reported several compounds with a 'Y' qualifier, indicating an elevated detection limit. The 'Y' qualifier was considered a 'U' for data qualification purposes.

**System Performance:** The chromatograms were reviewed for baseline shifts, aroclor patterns and general instrument response. No problems were identified during review of the raw data.

**Overall Assessment:** All deliverables required by the project were present and data packages were complete. Recommended sample holding times and conditions were met with the exceptions noted above. Initial and continuing calibration requirements were acceptable with the exceptions noted for Aroclor 1260 and Aroclor 1016. Method blanks showed no target analytes were present. Compound identification and quantitation were acceptable. Raw data show no indications of system performance degradation. Overall analytical performance was considered acceptable, and data quality sufficient for project use.

## 6.0 PETROLEUM HYDROCARBON ANALYSES - Gasoline – NWTPH-G

Petroleum hydrocarbon analyses were performed by Analytical Resources, Inc. (ARI) of Seattle, Washington, in accordance with the requirements of the Work Plan. The method used to determine gasoline in the samples is referenced as Northwest TPH-G (NWTPH-G [gasoline range = toluene - C<sub>12</sub>]) - Volatile Petroleum Products Method for Soil and Water Analyses, as established by the Washington State Department of Ecology.

Thirty samples were analyzed for NWTPH-G, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Trip	Totals
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Blanks					
Water	9	0	0	4	13
Soil	18	0	0	0	18
Totals	27	0	0	3	31

The laboratory provided U.S. EPA CLP style deliverables for all sample delivery groups. Sample results are presented with associated data qualifiers in Appendix D.

**Sample Documentation, Custody and Holding Conditions/Times:** All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are complete. Maximum holding times were specified as 14 days for waters and solids, at 4° C. ( $\pm 2^\circ$  C.).

Upon receipt at the laboratory, the transport cooler temperature for SDG FA32 was 14.5 °C. Associated sample results for 470E-SS-30, 470E-SS-31, 470E-SS-32 and 470E-TB-021126 were qualified as estimated due to the elevated cooler temperatures. The elevated temperatures were not expected to significantly affect data quality and the sample integrity was considered acceptable since the samples were originally placed in coolers containing ice.

**Initial Calibration:** The laboratory performed initial multipoint calibration (linearity verification) for NWTPH-G (toluene - C12) at 0.1, 0.25, 1.00, 2.5, 5.0 and 20 µg/mL. All %RSDs were  $\leq 30\%$  for all initial calibrations.

**Calibration Checks:** The laboratory performed calibration verifications for NWTPH-G at 2.5 ppm and BTEX at 25 ppb. The acceptance window for the calibration check is  $\pm 15\%$  of the true value. Calibration check frequencies for all analyses were conducted after every ten sample analyses. No results required qualification based on out-of-compliance procedures and performance criteria.

**Blanks:** Analytical method blanks were analyzed at the required frequency (once per analytical delivery group of 20 samples, or less) and no analyte responses were reported. Four trip blank were analyzed for petroleum hydrocarbons to evaluate potential contributions of analytes and introduction of contaminants during transport. No positive results were reported for the trip blanks.

**Surrogate Compound Performance:** Two surrogate compounds were added to each sample prior to analysis to assess analytical performance on each sample. The surrogates were identified as trifluorotoluene and bromobenzene. Surrogate performance was evaluated against the laboratory-established limits for NWTPH-G as follows:

Compound	Water Acceptance Limits (%)	Soil Acceptance Limits (%)
Trifluorotoluene	66-129	61-132
Bromobenzene	69-124	52-141

All surrogate recoveries were within specification.

**Matrix Spike/Matrix Spike Duplicate Analyses:** Sample 470E-SB34-02 was analyzed as the matrix spike/spike duplicate pair. Gasoline range hydrocarbons were evaluated at 333 mg/kg. Laboratory-established control limits applied were 41-148% recovery. The results were acceptable.

**Laboratory Control Samples:** Spiked blanks (LCS) were analyzed in conjunction with the matrix spikes and method blanks. Three soil and two liquid LCS analyses were performed for gasoline range hydrocarbons with acceptable recoveries. The LCS spiking level 250 mg/kg for soils and was 25 mg/L for waters. The laboratory-established acceptance limits for the LCS were 61 - 122% for solids and 64 - 118% for liquids. Performance was considered acceptable and the analytical systems were determined to be in control.

**Compound Quantitation and Reported Detection/Quantitation Limits:** No problems were noted during review of the raw data and chromatograms.

**System Performance:** Raw data show no indication of degradation of system performance during or between analytical runs. Chromatograms show no abrupt shifts in baseline, high background levels, excessive baseline rise with increased temperature, or other indications of system performance degradation.

**Overall Assessment:** All deliverables required by the project are present and data packages are complete. Sample holding times were met with the exception noted above. Initial calibration and calibration check requirements were met. Method and field blank performances were within specification. Surrogate compound recoveries were within specification. LCS performances were acceptable. Compound identification and quantitation were acceptable. Raw data show no indications of system performance degradation. Reporting limits were sufficient. Overall analytical performance was considered acceptable, and data quality sufficient for project use.

## 7.0 VOLATILE ORGANICS ANALYSES - U.S. EPA SW-846, Method 8260B

Volatile organics analyses were performed by Analytical Resources, Inc. (ARI) of Seattle, Washington, in accordance with the requirements of the Work Plan. Soil and water samples were analyzed for volatile organic compounds using Method 8260B.

Eleven samples were analyzed for volatile organics, as summarized below:

Sample Type	Primary	Replicate	Trip	Rinsate	Totals
Water	1	0	3	0	4
Soil	7	0	0	0	7
Totals	8	0	3	0	11

The laboratory provided U.S. EPA CLP style deliverables for all sample delivery groups. Sample results are presented with associated data qualifiers in Appendix D.

**Sample Documentation, Custody and Holding Conditions/Times:** All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables were complete. Maximum holding times were specified as 7 days (unpreserved) and 14 days (preserved) for waters and 14 days for soils, at 4° C. ( $\pm 2^\circ$  C.). Analytical holding times were met.

Upon receipt at the laboratory, the transport cooler temperature for SDG FA32 was 14.5 °C, resulting in qualification of sample results for 470E-SS-30, 470E-SS-31, 470E-SS-32 and 470E-TB-021126 (11/29/02 date) as estimates. The elevated temperatures were not expected to significantly affect data

quality and the sample integrity was considered acceptable since the samples were originally placed in coolers containing ice.

The laboratory noted that three vials were received unlabeled, later determined to be 470E-PZ2-01. The chain-of-custody documentation specified several organic analyses for sample 470E-RS-021126, which were later cancelled per instruction from Ridolfi Inc.

**GC/MS Tuning:** GC/MS tune performance was checked using Bromofluorobenzene (BFB) prior to all initial calibrations runs and all subsequent sample analytical runs. All sample analyses were performed within 12 hours of BFB analyses. All ion abundances and relative ion abundances met *Functional Guidelines* acceptance criteria. Review of mass spectral plots and associated mass listings supplied with the raw data, and transcription of mass data to Form V (GC/MS Tuning and Mass Calibration) showed no inconsistencies. No errors were identified during spot checks of calculations.

**Initial Calibration:** Initial multipoint calibrations were performed at 1, 2, 5, 10, 50, 100 and 200 µg/kg, for all target compounds. The USEPA CLP *Functional Guidelines* specify that Average Relative Response Factors (Average RRFs) must be  $\geq 0.05$ , and selected analytes must meet additional minimum RRF and maximum %RSD criteria ( $>30\%$ ). The RRFs for Acrolein in the initial calibrations dated 11/19/02 and 12/5/02 were less than 0.05, resulting in 'R' qualification of Acrolein in sample results for 470E-SB-30, 470E-SB-31, 470E-SB-32, 470E-TB-021126 (both) and 470E-SW-01. The RRF for 2-chloroethylvinyl ether in the initial calibration dated 11/21/02 was less than 0.05, resulting in 'R' qualification of 2-chloroethylvinyl ether in sample results for 470E-SB34-01, 470E-SB34-02 and 470E-TB-021129.

**Continuing Calibration:** Continuing calibrations were performed for all TCL compounds at 50 µg/kg for VOC 8260B analysis. The USEPA CLP *Functional Guidelines* specify that RRFs must be  $\geq 0.05$ , and compounds must meet a maximum percent difference (%D) criteria ( $\leq 25\%$ ). Deviations from the %D criteria were as follows:

Calibration Date	Compound	Samples Affected	Qualifier
11/21/02	2-chloroethylvinyl ether	470E-SB34-01, 470E-SB34-02, 470E-TB-021129	R
11/25/02	Chloromethane, Bromomethane, Acrolein, 2,2'-dichloropropane, 2-chloroethylvinyl ether, Bromoform, t-1,4-dichloro-2-butene, 1,3-dichlorobenzene, 1,4-dichlorobenzene, n-Butylbenzene, 1,2,4-trichlorobenzene, 1,2,3-trichlorobenzene	470E-SB34-01	J, UJ
12/3/02	Bromomethane, acetone, Acrylonitrile, 2-butanone	470E-TB-021126, 470E-SW-01 (11/22/02)	J, UJ
11/23/02 12/6/02	Acrolein	470E-SB-30, 470E-SB-31, 470E-SB-32, 470E-TB-021126 (both), 470E-SW-01	R
12/6/02	2-chloroethylvinyl ether, n-Propylbenzene, n-Butylbenzene	470E-SS-30, 470E-SS-31, 470E-SS-32, 470E-TB-021126	J, UJ

**Blanks:** Analytical method blanks were analyzed at least once for each analytical group and matrix, as specified. Method blank results were acceptable. Three trip blank samples were also analyzed with acceptable results.

**Surrogate Compound Performance:** Surrogate compounds were added to each sample prior to analysis to assess analytical performance on each sample. Surrogate compounds and associated performance criteria for the analysis of TCL analytes in soil and water matrices were those specified in U.S. EPA Method SW-846 8270. Laboratory-established surrogate compound recovery specifications were as follows:

Compound	Acceptance Range (%) Water	Acceptance Range (%) Soil
Dibromofluoromethane	60-140	60-140
d4-1,2-Dichloroethane	74-142	71-143
d8-Toluene	84-129	79-118
4-Bromofluorobenzene	77-122	69-126
d4-1,2-Dichlorobenzene	85-135	79-119

All surrogate recoveries were acceptable.

**Matrix Spike/Matrix Spike Duplicate Analyses:** Matrix spike (MS) and matrix spike duplicate (MSD) analyses were performed on samples 470E-SW-01, 470E-SB34-01 and 470E-SS-30. Analyte spike concentrations were 59 - 60 µg/kg. MS/MSD compounds and associated performance criteria for the analysis of TCL analytes in soil and water matrices were those specified in U.S. EPA Method SW-846 8260B. Laboratory-established spike compound recovery specifications were:

Compound	Recovery Acceptance Range (%) Water	Recovery Acceptance Range (%) Soil
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Vinyl Chloride	31-162	27-187
1,1-Dichloroethene	62-139	48-147
Chloroform	72-123	65-124
1,2-Dichloropropene	80-125	66-131
Trichloroethene	38-170	55-140
Benzene	66-140	63-122
Toluene	80-129	53-129
Chlorobenzene	77-121	60-130
Ethylbenzene	70-136	52-135

The matrix spike recovery for acetone for sample 470E-SB34-01 was slightly high while the RPD was in control. No action was taken. Compound 2-chloroethylvinyl ether was not recovered in the matrix spike samples associated with field sample 470E-SW-01, resulting in qualification of 2-chloroethylvinyl ether for the sample. Vinyl acetate was not recovered in the matrix spike samples associated with field sample 470E-SS-30, resulting in qualification of vinyl acetate for the sample.

**Laboratory Control Samples:** Laboratory control samples (LCSs) were analyzed per batch for the samples. Each of the target compounds were spiked with 50 - 250 µg/kg or µg/L of the LCS mixture. All LCS results were acceptable.

**Internal Standards Performance:** Internal standards were added at a concentration of 50 ng/µL to all sample extracts prior to instrumental analyses. Retention times (RT) for all internal standards were within method specified acceptance limits in all samples (±30 seconds). Internal standard areas were within method specified acceptance limits (0.5 to 2 times the 12-hour calibration standard area) for all samples.

**TCL Compound Identification:** Relative retention times (RRTs) for all reported TCL compounds were within the method specified acceptance limits (±0.06 RRT units). Mass spectra generally show good comparison with library reference spectra.

**Compound Quantitation and Reported Detection/Quantitation Limits:** The detection limits were acceptable as reported. No problems were noted.

**System Performance:** Raw data showed no indication of degradation of system performance during or between analytical runs. Reconstructed ion chromatograms (RICs) showed no abrupt shifts in baseline, high background levels, excessive baseline rise with increased temperature, or other indications of system performance degradation.

**Overall Assessment:** All deliverables required by the project were present and data packages were complete. With the exception of the elevated cooler temperatures, the holding time criteria were met. GC/MS tuning requirements were met. Initial and continuing calibration requirements were acceptable with exceptions as noted. Internal standard (IS) performance was acceptable. Surrogate performance was acceptable. Compound identification and quantitation were acceptable. Raw data showed no indications of system performance degradation. Overall analytical performance was considered acceptable, and data quality sufficient for project use.

## 8.0 METALS ANALYSES - U.S. EPA SW-846 Methods

Metals analyses were performed by Analytical Resources, Inc. (ARI) of Seattle, Washington, in accordance with the requirements of the Work Plan. All samples were analyzed according to the U.S. EPA referenced methods, and calibrations and performance criteria are consistent with the U.S. EPA CLP Statement of Work.

All samples were analyzed according to the U.S. EPA Target Analyte List, which includes 21 metals and 2 metalloids (antimony and arsenic). Aluminum, antimony, barium, beryllium, cadmium, calcium, chromium, cobalt, copper, iron, magnesium, manganese, nickel, potassium, silver, sodium, vanadium, and zinc were analyzed by the Inductively Coupled Plasma–Atomic Emission Spectrometric method (ICP–AES, Method 6010B). Arsenic, lead, selenium, and thallium were analyzed by the Graphite Furnace Atomic Absorption method (GFAA, Methods 7060A, 7421, 7740, and 7841, respectively). Mercury was analyzed by the Cold Vapor Atomic Absorption method (CVAA, Methods 7470A/7471A).

Fifty samples were analyzed for metals, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	26	0	1	27
Soil	22	1	0	23
Totals	48	1	1	50

Water analyses were performed on both unfiltered and field-filtered samples (0.45 µm filter).

The laboratory provided U.S. EPA CLP style deliverables for all sample delivery groups. Sample results are presented with associated data qualifiers in Appendix D.

**Sample Documentation, Custody and Holding Conditions / Times:** All samples were handled and delivered to the laboratory according to chain-of-custody procedure. Laboratory data deliverables are complete and consistent with the specifications of the U.S. EPA CLP SOW. Nitric acid was added to water sample containers by the laboratory as a preservative. No preservatives were added to the soil samples. Upon receipt at the laboratory, transport coolers temperatures ranged from 0 – 14.5° C. Maximum holding times for waters and soils are specified as 28 days for mercury and 6 months for other metals. All sample analyses were performed within the recommended holding times and are acceptable. No results require qualification due to holding times and conditions.

**Initial Calibration:** The laboratory performed initial instrumental calibrations daily using at least the minimum required number of data points to establish the analytical curve for each method: a blank and one standard for ICP analyses, a blank and three standards for GFAA analyses and a blank and five standards for mercury analyses. Correlation coefficients for all GFAA and mercury initial calibrations are  $\geq 0.995$ , as required.

**Initial Calibration Verification:** The laboratory performed initial calibration verification checks (ICVs) immediately after initial instrumental calibrations during all ICP, GFAA and mercury analytical sequences, as required. All ICV recoveries are within acceptance limits (90–110% for ICP/GFAA; 80–120% for mercury). A spot check of ICV recoveries showed no calculation errors.

**Continuing Calibration Verification:** The laboratory analyzed continuing calibration verification standards (CCVs) at the required frequency for all ICP, GFAA and mercury analytical sequences (at the beginning and end of each run; at a frequency of  $\geq 10\%$  or every two hours, whichever is more frequent).

All CCV recoveries are within acceptance limits (90–110% for ICP/GFAA; 80–120% for mercury). A spot check of CCV recoveries showed no calculation errors.

**Blanks:** Initial calibration blanks (ICBs) were analyzed immediately after ICVs, and continuing calibration blanks (CCBs) were analyzed immediately after CCVs during all ICP, GFAA and mercury analytical sequences, as required. The CLP *Functional Guidelines* require that positive sample results less than 5 times the amount in any blank should be qualified as "U" (quantitation limit). All positive sample results associated with blank positive results are greater than 5 times the amount in an associated calibration blank, with the exception of three thallium analyses on 12/3/02 associated with CCB7. Thallium results for the three samples were qualified as non-detects (U).

Preparation blanks were analyzed for all target analytes at the required frequency (one per matrix per preparation batch). The CLP *Functional Guidelines* require that positive sample results less than 5 times the amount in the preparation blank should be qualified as "U" (quantitation limit). No positive results were reported for any preparation blanks.

One equipment rinsate blank was collected and analyzed to evaluate field ground water sampling technique and potential contributions of analytes. No positive results were reported for the rinsate blank.

**Interference Check Samples:** ICP interference check solutions (ICS) were analyzed for the target analytes at the beginning of each ICP analytical run, as required by the method. Recoveries for all required target analytes in all check samples are within acceptance limits (80–120%). A spot check of ICS recoveries showed no calculation errors.

**Laboratory Control Samples:** Laboratory control samples (LCS) were analyzed at the required frequency (at least one sample per matrix per preparation batch). The aqueous LCSs are spiked method blanks. All target analytes are within laboratory established control limits (80–120% recovery) for the reported analyses of aqueous LCSs (dissolved and total metals). The solid LCS is ERA 247 reference material. Control limits for target analytes for this LCS are ERA's certified advisory limits. All analytes are within the ERA 247 advisory limits. A spot check of LCS recoveries showed no calculation errors.

**Duplicate Sample Analyses:** Laboratory duplicate samples were analyzed for the target analytes at the required frequency (at least one sample per matrix per preparation batch), with the exception that no duplicates were analyzed for water for the combined QC batch for SDGs EZ56 and EZ97. Acceptance limits applied in this evaluation of duplicate sample analyses are in accordance with the requirements of the U.S. EPA *Functional Guidelines* (results  $\geq 5X$  the reporting limit,  $\leq 35\%$  Relative Percent Difference (RPD) for soil,  $\leq 20\%$  RPD for water; results  $< 5X$  the reporting limit  $\pm 2X$  the reporting limit for soil,  $\pm$  the reporting limit for water). Results of all duplicate analyses meet these criteria. A comparison of raw data and reporting forms showed no transcription errors. A recalculation of RPDs showed no calculation errors.

**Matrix Spike Sample Analyses:** Matrix spike samples were analyzed for the target analytes at the required frequency (at least one sample per matrix per preparation batch), with the exception that no duplicates were analyzed for water for the combined QC batch for SDGs EZ56 and EZ97. Samples were spiked at CLP-specified concentrations. CLP acceptance limits for matrix spike recovery are 75–125% and are applicable only to those samples in which the sample concentration does not exceed four times the spike concentration. Water sample matrix spike recoveries are acceptable. Soil sample matrix spike recoveries are acceptable with the exception of antimony (EZ56/EZ97 = 33.2%R) and antimony, calcium, and potassium (FA17/FA32 = 19.1%R, 143%R, and 70.5%R, respectively). All associated antimony

results are non-detects. Results associated with EZ56/EZ97 are qualified as estimated (UJ); results associated with FA17/FA32 are rejected (R), in accordance with the *Functional Guidelines*. All calcium and potassium results associated FA17/FA32 are positive and are qualified as estimated (J).

**Graphite Furnace QC:** Duplicate injections were performed for all analyses. Examination of raw data shows that the duplicate injections agree within  $\pm 20\%$  Relative Standard Deviation (%RSD). Post digestion spikes were analyzed for all samples at a spike level of 20  $\mu\text{g/L}$  for all four methods. Post digestion spike recoveries are outside project-specified limits (85–115%R) for the following: One arsenic recovery is outside acceptance limits (water); the associated result is positive and qualified as estimated (J). Three lead recoveries are outside acceptance limits (water); the associated results are qualified as estimated (J, UJ). Five thallium recoveries are outside acceptance limits (water); the associated results are non-detects and are qualified as estimated (UJ). Eighteen selenium spike recoveries are outside acceptance limits (four soils, fourteen waters); the associated results are non-detects and are qualified as estimated (UJ).

At least 10% of post digestion spike recoveries were verified for each SDG by recalculation using raw data. The U.S. EPA CLP Furnace Atomic Absorption Scheme was followed, as required.

**Reported Detection/Quantitation Limits:** Reported quantitation or lower reporting limits are acceptable.

**Field Replicates:** One blind field replicate soil sample pair was submitted and analyzed for metals for determination of analytical variability. The replicate pair is a split of homogenized soil. Sample results for replicate pair (470E-SB32-01, 470E-SB32-03) are presented in Appendix D. For results that are  $\geq 5X$  the reporting limit, RPDs  $\leq 35\%$  are considered to indicate good comparability. For results  $< 5X$  the reporting limit, results that are  $\pm 2X$  the reporting limit are considered to indicate good comparability. Results for arsenic, iron, lead and zinc exceed these guidelines and overall comparability is considered fair. No results were qualified based on field replicate performance.

**Overall Assessment:** All deliverables required by the project are present and data packages are complete. All analyses meet recommended sample holding times. Initial and continuing calibration verification standards and blanks are acceptable, with the exception of one thallium CCB, requiring qualification of three associated results as non-detected (U). Recoveries for interference check samples and laboratory control samples are acceptable. Laboratory duplicate sample analyses are acceptable. Low matrix spike recoveries for antimony required rejection (R) of several soil antimony results and qualification of other soil antimony results as estimated (UJ). For two SDGs, all calcium and potassium results were qualified as estimated (J) because matrix spike recoveries were outside acceptance limits. Graphite furnace QC was acceptable with the exception that one arsenic, 3 lead, 18 selenium, and 5 thallium results were qualified as estimated because post digestion spikes were outside acceptance limits. Reported quantitation or lower reporting limits are acceptable. Replicate analyses showed fair comparability for soils and good comparability for waters. Overall analytical performance is considered acceptable and the data quality is sufficient for project use.