

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 December 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 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

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 was referenced as USEPA Method 8015M [diesel range = C₁₂ - C₂₄, lube or motor oil range = C₂₄ - C₃₈].

Twenty-six samples were analyzed for NWTPH-Dx, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	11	0	0	11
Soil	12	2	1	15
Totals	23	2	1	26

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) 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 FC71 was 7.5 °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 ppm; and motor oil at 50, 100, 250, 500, 2500 and 5000 ppm. 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 $\mu\text{g/mL}$ and 500 $\mu\text{g/mL}$, respectively. The acceptance window for the calibration check is $\pm 15\%$ of the true value. Calibration checks were performed 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 two 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 on each 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: Samples 470E-RS-021219, 470E-SB35-01 and 470E-MW41-01 were analyzed as matrix spike and spike duplicate pairs. Diesel fuel was evaluated at 6.67 and 6.25 mg/L and 205 mg/kg. Laboratory-established control limits applied were 26-132% for soil and 57-151% for water. All recoveries were acceptable.

Laboratory Control Samples: Spiked blanks (LCS) were analyzed in conjunction with the matrix spikes and method blanks. Three 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 (470E-SB36-01/470E-SB36-03). Both diesel and motor oil results exceeded the precision limit of 35%. Data were not qualified due to the field replicate results.

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 were not comparable, however, data were not qualified based on the results of the comparison. 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

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 using Method 8270C.

Five samples were analyzed for semivolatile organics, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	0	0	0	0
Soil	5	0	0	5
Totals	5	0	0	5

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) and 7/40 days for solids and waters, respectively, at 4° C. (±2° C.). All extraction and analytical holding times were met.

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 USEPA CLP *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, for all target compounds and surrogate compounds. The USEPA CLP *Functional Guidelines* specify that Average Relative Response Factors (Average RRFs) must be ≥ 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 with the exception of benzoic acid, 2,4-dinitrophenol, and 4-nitrophenol. No action was taken since these compounds were non-detects in the associated field samples.

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

Calibration Date	Compound	Samples Affected	Qualifier
1/13/03	Benzoic acid, Hexachlorocyclopentadiene, 2,4-dinitrophenol, 4-nitrophenol	All	J, 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 (%)
	Water	Soil
d ₅ -Nitrobenzene	49-118	20-102
2-Fluorobiphenyl	53-106	34-99
d ₁₄ -p-Terphenyl	45-126	43-128
d ₅ -Phenol	43-112	29-93
2-Fluorophenol	47-109	25-92
2,4,6-Tribromophenol	34-142	23 - 131
d ₄ -2-Chlorophenol	51-110	32 - 91
d ₄ -1,2-Dichlorobenzene	39-92	26 - 85

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. All surrogate recoveries were acceptable and no data required qualification.

Matrix Spike/Matrix Spike Duplicate Analyses: Matrix spike (MS) and matrix spike duplicate (MSD) analyses were performed on soil sample 470E-SB36-01. Analyte spike concentrations were 2970 - 4450 µg/kg for low-level soils. 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

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	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

The LCS results were compared to the above criteria. The LCS concentrations were 1670 - 2500 µg/kg for low-level soils. 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 (±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 (±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.

Field Replicates: One field replicate sample pair was submitted and analyzed for petroleum hydrocarbons for determination of analytical variability (470E-SB36-01/470E-SB36-03). Phenanthrene, di-n-butylphthalate, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(b)fluoranthene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, benz(g,h,i)perylene results exceeded the precision limit of 35%. Data were not qualified due to the field replicate results.

Overall Assessment: All deliverables required by the project were present and data packages were complete. The holding time criteria were met. GC/MS tuning requirements were met. Initial and continuing calibration requirements were acceptable with exceptions noted above. 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. Field replicate analyses were not comparable, however, data were not qualified based on the results of the comparison. 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.

Thirty-six samples were analyzed for pesticides, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	14	2	0	16
Soil	18	1	1	20
Totals	32	3	1	36

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. (±2° C). All extraction and analysis holding times were met.

Upon receipt at the laboratory, the transport cooler temperature for SDG FC71 was 7.5 °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%). For SDG FC72, the performance standard for Endrin aldehyde exceeded 40% between the primary and confirmation column for standard DS12. All samples in the SDG were qualified as estimated for Endrin aldehyde.

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% and the individual compounds were < 25%.

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 29-110% (soil) and 38-108% (water) for tetrachloro-m-xylene; and 18-151% (soil) and 30-115% (water) for decachlorobiphenyl. All surrogate recoveries were acceptable.

Matrix Spike/Matrix Spike Duplicate Analyses: Matrix spike and matrix spike duplicate analyses were performed on 470E-SB35-01, 470E-MW39-01 and 470E-MW40-01. Analyte spike concentrations ranged from 1 to 2 µg/L or 22.8 to 45.6 µg/kg. The following is a summary of the compounds and laboratory-established acceptance limits for the spiked samples:

Compound	Water Acceptance Limits (%)	Soil Acceptance Limits (%)
Lindane	46-115	10-130
Heptachlor	56-101	10-127
Aldrin	46-93	10-112
Dieldrin	55-118	10-144
Endrin	62-118	10-147
DDT	57-121	10-161

MS/MSD performances were acceptable for all samples.

Laboratory Control Samples: Four 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. A 'P' qualifier, indicating the difference between the primary and confirmation columns exceeded 40%, was reported for alpha-chlordane in samples 470E-SB30-01 and 470E-SB30-02. Therefore, alpha-chlordane was qualified with a 'J' in these two samples. 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.

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. 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.

Thirty-two samples were analyzed for polychlorinated biphenyls, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	16	0	0	16
Soil	15	1	0	16
Totals	31	1	0	32

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 FC71 was 7.5 °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	Qualification
12/24/02	Aroclor 1260	470E-SB30-01, 470E-SB31-02	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-SB35-01, 470E-MW51-01 and 470E-MW-34-01. Analyte spike concentrations for aroclor 1242 were 457 µg/kg and 20.2 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 10.1 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. 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 - C₁₂]).

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

Sample Type	Primary	Replicate	Rinsate	Trip Blanks	Totals
Water	9	0	0	3	12
Soil	18	0	0	0	18
Totals	27	0	0	3	30

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. (±2° C.).

Upon receipt at the laboratory, the transport cooler temperature for SDG FC71 was 7.5 °C. Data were not qualified due to the slightly elevated cooler temperature. 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 ppm. 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 checks were performed after every ten sample analyses. Calibration check frequencies for all analyses were conducted after every ten instrumental runs. The following results required qualification based on out-of-compliance calibration:

Calibration Date	Samples Affected	Qualification
12/31/02	470E-TB-021219, MB, 470E-SB36-01, 470E-SB36-02, 470E-SB36-03, 470E-SB35-01, 470E-SB35-02	UJ

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.

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 with the exception of BFB for sample 470E-SB35-02RE, which was qualified as estimated.

Matrix Spike/Matrix Spike Duplicate Analyses: Samples 470E-SB36-01 and 470E-MW34-01 were analyzed as the matrix spike/spike duplicate pairs. Gasoline range hydrocarbons were evaluated at 421 mg/kg and 2.5 mg/L. Laboratory-established control limits applied were 41-148% recovery for soil and 66-135% recovery for water. The results were acceptable.

Laboratory Control Samples: Spiked blanks (LCS) were analyzed in conjunction with the matrix spikes and method blanks. Two soil and two liquid LCS analyses were performed for gasoline range hydrocarbons with acceptable recoveries. The LCS spiking level was 2.5 mg/L for waters and 250 mg/kg for soils. The laboratory-established acceptance limits for the LCS were 61-122% for soil and 64-118% for water. 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. Recommended sample holding times and conditions were met with the exception noted above. Initial calibration and calibration check requirements were met with the exception noted above. Method and field blank performances were within specification. Surrogate compound recoveries were within specification with the exception noted above. 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.

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

Sample Type	Primary	Replicate	Trip	Rinsate	Totals
Water	5	0	1	0	6
Soil	0	0	1	0	1
Totals	5	0	2	0	7

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 FC71 was 7.5 °C. No data were qualified due to the slightly elevated cooler temperature. 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 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 ppb for all target compounds. The USEPA CLP *Functional Guidelines* specify that Average Relative Response Factors (Average RRFs) must be ≥ 0.05 , and selected analytes must meet additional minimum RRF and maximum %RSD criteria ($>30\%$). The RRF for Acrolein in the initial calibration dated 12/18/02 was less than 0.05, resulting in 'R' qualification of Acrolein in all samples in SDGs FC69 and FC71.

Continuing Calibration: Continuing calibrations were performed for all TCL compounds at 50 $\mu\text{g/kg}$ for VOC 8260B analysis. The USEPA CLP *Functional Guidelines* specify that RRFs must be ≥ 0.05 , and compounds must meet a maximum percent difference (%D) criteria ($\leq 25\%$). All criteria were met.

Blanks: Analytical method blanks were analyzed at least once for each analytical group and matrix, as specified. Method blank results were acceptable. Two 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 (%)	Acceptance Range (%)
	Water	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 was performed on sample 470E-MW39-01. Analyte spike concentrations were 50 $\mu\text{g/L}$. MS/MSD compounds and associated performance criteria for the analysis of TCL analytes were those specified in U.S. EPA Method SW-846 8260B. Laboratory-established spike compound recovery specifications were:

Compound	Recovery Acceptance Range (%)	Recovery Acceptance Range (%)
	Water	Soil
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 recoveries were acceptable with the exception of 2-chloroethylvinyl ether, which was not recovered in the spike samples, resulting in estimated qualification of 2-chloroethylvinyl ether for all samples in SDGs FC69 and FC71. Additionally, trans-1,4-dichloro-2-butene and vinyl acetate recoveries were below control limits, resulting in estimated qualification of these compounds for all samples in SDGs FC69 and FC71.

Laboratory Control Samples: Laboratory control samples (LCSs) were analyzed per batch for the samples. Each of the target compounds was spiked with 50 - 250 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. 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. MS/MSD results were acceptable with exceptions noted above. 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).

Thirty-eight samples were analyzed for metals, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Water	20	4	1	25
Soil	12	1	0	13
Totals	32	5	1	38

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 1.5 – 7.5° 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 ≥ 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. Therefore no data are qualified.

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 soil sampling technique and potential contributions of analytes. Positive results were reported for aluminum, calcium, iron and manganese. All associated results for these analytes are greater than 5 times the blank result. No results require qualification.

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 D034 reference material. Control limits for target analytes for this LCS are ERA's certified advisory limits. All analytes are within the ERA D034 advisory range. 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). 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). 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 with the exception of antimony (57.5%R). All associated antimony results are non-detects and are qualified as estimated (UJ). Soil sample matrix spike recoveries are acceptable with the exception of antimony (18.5%R). All associated antimony results are non-detects and are rejected (R), in accordance with the *Functional Guidelines*.

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 within project-specified limits (85–115%R) for arsenic and lead. Two thallium recoveries are outside acceptance limits (one soil, one water); the associated results are non-detects and are qualified as estimated (UJ). All soil selenium spike recoveries are outside acceptance limits and most water selenium spike recoveries are low; the associated results are qualified as estimated (J, 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-SB36-01, 470E-SB36-03) 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 barium, beryllium, cobalt, magnesium, manganese, mercury, potassium and thallium exceed these guidelines and overall comparability is considered poor. No results were qualified based on field replicate performance.

Four blind field replicate water sample pairs were submitted and analyzed for metals for determination of analytical variability (two field-filtered, two unfiltered). Sample results for replicate pairs (470E-MW41-01, 470E-MW50-01 (filtered and unfiltered; 470E-MW40-01, 470E-MW51-01 (filtered and unfiltered)) are presented in Appendix D. For results that are $\geq 5X$ the reporting limit, RPDs $\leq 20\%$ are considered to indicate good comparability. For results $< 5X$ the reporting limit, results that are $\pm 1X$ the reporting limit are considered to indicate good comparability. With the exception of aluminum and lead in one unfiltered sample (470E-MW40-01, 470E-MW51-01), results for all detected metals meet these guidelines and thus exhibit good comparability.

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. Several analytes were detected in the rinsate blank, but all associated results were greater than 5 times the blank results. 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 soil antimony results and qualification of water antimony results as estimated (UJ). Graphite furnace QC was acceptable with the exception that several selenium and two 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 poor comparability for soils and good comparability for waters. Overall analytical performance is considered acceptable and the data quality is sufficient for project use.