

# DATA VALIDATION REPORT

POPLAR POINT  
WASHINGTON, DC

*OCTOBER 22, 2002*

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## 1.0 INTRODUCTION

This report summarizes the validation of analytical results generated from field sampling in August of 2002, in support of the site characterization for Poplar Point. Sampling and analyses were performed according to the *Surface Soil Sampling Work Plan* (Work Plan), (Ridolfi, August 20, 2002). The criteria applied for this validation are 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.

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

Sample Type	Primary	Replicate	Rinsate	Totals
Water	0	0	2	2
Soil	11	1	0	12
Totals	11	1	2	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.). The following sample results were qualified as estimated (J) due to re-extraction holding time exceedance:

- 470E-SS-27 re
- 470E-SS-35 re

Upon receipt at the laboratory, transport cooler temperatures ranged from 17 to 20 °C. Due to the elevated temperatures, all sample results for diesel and motor oil were qualified as estimated (UJ, J). While the data were qualified due to the elevated cooler temperatures, the sample integrity was considered acceptable since the samples were originally placed in coolers containing ice. The elevated temperatures were not expected to significantly affect data quality and the data are considered usable as qualified.

**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 (9/12/02). Linearities were 2.93 - 10.95 and 18.71 %RSD for diesel and motor oil, respectively. 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 checks were performed after every ten sample analyses. These criteria are 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 five method blanks were analyzed, and no analyte responses were reported.

Two equipment rinsate blanks were analyzed for petroleum hydrocarbons to evaluate field sampling technique and potential contributions of analytes. No positive results are reported for the field blanks.

**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: 51-117%, soil 34-113%). All recoveries were within specification with the exception of 470E-SS-27 and 470E-SS-35. The samples were re-extracted and reanalyzed with acceptable surrogate recoveries, but outside the designated holding time. The original analyses were qualified as estimated (UJ, J) due to unacceptable surrogate recovery and the re-extracted analyses were qualified as estimated (J) due to holding time exceedance.

**Matrix Spike/Matrix Spike Duplicate Analyses:** Sample 470E-SS-20s was analyzed as the matrix spike and spike duplicate pair. Diesel fuel was evaluated at 161 mg/kg. Laboratory-established control limits

applied were 26-132% recovery. The recovery of diesel in spike samples was 161%, which exceeds the control limit. Therefore, the result for sample 470E-SS-20s was qualified as estimated (J) for diesel.

**Laboratory Duplicate Analysis:** Sample 470E-SS-20d was analyzed in duplicate by the laboratory. The results of the laboratory duplicate analysis were above the precision limit of 35% RPD (diesel RPD = 49%, motor oil RPD = 82%) . Therefore, results for samples 470E-SS-20d, 470E-SS-20d dup and 470E-SS-20s were qualified as estimated (J) for diesel and motor oil.

**Laboratory Control Samples:** Spiked blanks (LCS) were analyzed in conjunction with the matrix spikes and method blanks. Four solid LCS analyses were performed for diesel with recoveries ranging from 65% to 85%. The LCS spiking level was 150 mg/kg. Performance was considered acceptable and the analytical systems were determined to be in control.

**Compound Quantitation and Reported Detection/Quantitation Limits:** Sample 470E-SS-35 (field replicate) was originally analyzed on 9/05/02 with 8.4 mg/kg diesel and 10U motor oil reported. The sample was re-extracted outside of the holding time and reanalyzed. The reanalysis on 9/14/02 shows significantly higher levels of diesel (3,600 mg/kg) and motor oil (1,700 mg/kg). A third extraction and analysis was performed by the laboratory, which confirmed the second analysis. The third analysis was not reported in the package, but was used by the laboratory to confirm the results of the second extraction/analysis. When compared to the primary sample, 470E-SS-20s, the second analysis of the field replicate meets precision criteria.

**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 blind field replicate sample pair was submitted and analyzed for petroleum hydrocarbons for determination of analytical variability. Sample pair 470E-SS-20s/470E-SS-35 showed comparable results when using the second set of data for sample 470E-SS-35 (diesel-range RPD = 2%; lube-range RPD = 6%).

**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. All results were qualified as estimated (U, UJ) because of high cooler temperatures. Initial calibration and calibration check requirements were met. Method and field blank performances were within specification. Surrogate compound recoveries were within specification with the exceptions noted above. MS/MSD results were slightly high, resulting in data qualification, while LCS performances 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 good agreement while laboratory duplicate analysis resulted in data qualification. Overall analytical performance is considered acceptable, and data quality is 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 polycyclic aromatic hydrocarbons (PAHs) using Method 8270C modified for selective ion monitoring (SIM) to enhance sensitivities to 0.25 parts per billion.

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

Sample Type	Primary	Replicate	Rinsate	Totals
Water	0	0	3	3
Soil	52	3	0	55
Totals	52	3	3	58

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.). The following sample results were qualified as estimated (UJ, J) because they were not extracted within the recommended holding time:

- 470E-SS-12d re, 470E-SS-03s re, 470E-SS-09d re, 470E-SS-10s re, 470E-SS-25 re, 470E-RS-01

Upon receipt at the laboratory, transport cooler temperatures ranged from 17 to 20 °C. Due to the elevated temperatures, all sample results were qualified as estimated (UJ, J) for all compounds. While the data were qualified due to the elevated cooler temperatures, the sample integrity was considered acceptable since the samples were originally placed in coolers containing ice. The elevated temperatures were not expected to significantly affect data quality and the data are considered usable as qualified.

**GC/MS Tuning:** GC/MS tune performance was checked with 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 meet *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. One error was identified on a DFTPP tune form dated 09/19/02: Sample 470E-SS-21s was analyzed in the set while the lab inadvertently listed 470E-SS-25 on the form. No sample results were affected by this transcription error. No errors were identified during spot checks of calculations.

**Initial Calibration:** Initial multipoint calibrations were performed at 5, 10, 25, 40 and 80 µg/mL for SVOC 8270C analysis and 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 compounds 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 1.0 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 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
9/27/02	Pentachlorophenol	470E-SS-25re	UJ
9/19/02	Benzoic acid, 4-nitrophenol	470E-SS-21s, 470E-SS-21d, 470E-SS-01s, 470E-SS-01d, 470E-SS-02s, 470E-SS-02d, 470E-SS-03s, 470E-SS-03d, 470E-SS-04s, 470E-SS-04d, 470E-SS-05s, 470E-SS-05d,	UJ
9/23/02	Bis(2-Chloroethyl)ether, 2,2'-oxybis(1-chloropropane)	470E-SS-22d dil, 470E-SS-36, 470E-SS-09s, 470E-SS-11s, 470E-SS-11d, 470E-SS-12s, 470E-SS-12d, 470E-SS-13s, 470E-SS-14s, 470E-SS-14d, 470E-SS-15s, 470E-SS-15d, 470E-SS-13d	UJ
9/20/02	2,2'-oxybis(1-chloropropane)	470E-SS-26d, 470E-SS-24s 470E-SS-24d, 470E-SS-34 470E-SS-22s	UJ
9/22/02	2,4-dinitrophenol	470E-SS-22ms, 470E-SS-22msd 470E-SS-22d, 470E-SS-23s 470E-SS-23d, 470E-SS-25 470E-SS-26s, 470E-SS-27 470E-SS-35, 470E-SS-08d, 470E-SS-08msd, 470E-SS-09d, 470E-SS-10s, 470E-SS-10d	UJ
10/05/02	Hexachlorocyclopentadiene, 2,4-dinitrophenol, 4,6-dinitro-2-methylphenol	470E-SS-03s re, 470E-SS-10s re, 470E-SS-09d re, 470E-SS-12d re	UJ
9/23/02	2,2'-oxybis(1-chloropropane), 2,4-dinitrophenol, pentachlorophenol	470E-SS-19s, 470E-SS-19d, 470E-SS-16s, 470E-SS-20d, 470E-SS-20s, 470E-SS-20s ms, 470E-SS-20s msd, 470E-SS-18s, 470E-SS-18d, 470E-SS-17d, 470E-SS-17s, 470E-SS-16d	UJ
9/25/02	Benzoic acid, 2,4-dinitrophenol	470E-SS-19d dil, 470E-SS-20d re	UJ

**Blanks:** Analytical method blanks were analyzed at least once for each analytical group and matrix, as specified. Method blank results were acceptable with the exception of one blank, which affects the following samples and results:

Analyte	Affected Samples
Bis(2-Ethylhexyl)phthalate (74 µg/kg)	470E-SS-21d, 470E-SS-22s ms, 470E-SS-22s msd, 470E-SS-22d, 470E-SS-22d dil, 470E-SS-23s, 470E-SS-23d, 470E-SS-24s, 470E-SS-26s, 470E-SS-27, 470E-SS-34, 470E-SS-36

The associated results (positive hits) for the above were less than 10x the blank level and were qualified as non-detects at the associated value with the 'U' qualifier code.

Three equipment rinsate blanks were analyzed. No target compounds were detected above the reporting limits.

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

Compound	Acceptance Range (%)	Acceptance Range (%)
	Water	Soil
d <sub>5</sub> -Nitrobenzene	43-112	11-111
2-Fluorobiphenyl	40-105	10-124
d <sub>14</sub> -p-Terphenyl	32-129	13-125
d <sub>5</sub> -Phenol	29-117	16-116
2-Fluorophenol	38-114	17-110
2,4,6-Tribromophenol	37-127	10 - 124
d <sub>4</sub> -2-Chlorophenol	47-108	20 - 108
d <sub>4</sub> -1,2-Dichlorobenzene	32-89	12 - 108
<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	470E-SS-25	acid	J, UJ
2-Fluorophenol			
d <sub>4</sub> -2-Chlorophenol			

**Matrix Spike/Matrix Spike Duplicate Analyses:** Matrix spike (MS) and matrix spike duplicate (MSD) analyses were performed on soil samples 470E-SS-08s, 470E-SS-20s and 470E-SS-22s. Analyte spike concentrations were 3360 - 5040 µg/kg for low level soils (8270C), 4610 - 6920 µg/kg for medium level soils (8270C) and 554-4610 µg/kg for SIM analysis. MS/MSD compounds and associated performance criteria for the analysis of TCL compounds in soil and water matrices are those specified in U.S. EPA Method SW-846 8270. Laboratory-established spike compound recovery specifications are as follows:

Compound	Recovery Acceptance Range (%)	Recovery Acceptance Range (%)
	Water	Soil
Phenol	41-130	25-116
2-Chlorophenol	52-120	23-113
1,4-Dichlorobenzene	29-80	16-108

N-Nitroso-di-n-propylamine	36-108	12-121
1,2,4-Trichlorobenzene	32-82	23-112
4-Chloro-3-methylphenol	40-139	24-121
Acenaphthene	35-112	15-125
4-Nitrophenol	46-136	10-141
2,4-Dinitrotoluene	42-122	10-139
Pentachlorophenol	18-154	10-141
Pyrene	38-105	10-139
<b>SIM</b>		
Phenanthrene	49-118	10-131
Benzo(k)fluoranthene	38-124	10-132
Chrysene	42-117	10-128

The recoveries of phenanthrene in the matrix spike and matrix spike duplicate samples for 470E-SS-22s were acceptable while the %RPD limit was exceeded. Therefore, sample 470E-SS-22s was qualified as estimated (J) for phenanthrene for the %RPD exceedance. For the MS/MSD SIM analysis of 470E-SS-08s, the %RPDs of phenanthrene, chrysene, benzo(k)fluoranthene were exceeded. The laboratory notes that the higher RPDs may be due to spike levels of some of the PNAs being somewhat high relative to the native levels of PNAs in the sample. No action was taken since the recoveries (%R) of these compounds were acceptable.

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

Compound	Recovery Acceptance Range (%)	Recovery Acceptance Range (%)
	Water	Soil
Phenol	49-115	45-123
2-Chlorophenol	59-112	49-116
1,4-Dichlorobenzene	27-86	30-115
N-Nitroso-di-n-propylamine	41-101	26-102
1,2,4-Trichlorobenzene	31-88	35-111
4-Chloro-3-methylphenol	54-112	31-119
Acenaphthene	50-98	37-102
4-Nitrophenol	43-124	28-111
2,4-Dinitrotoluene	53-112	33-118
Pentachlorophenol	10-129	18-125
Pyrene	42-103	29-95
<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, 1670-2500 µg/kg for medium soils (8270C) and 200-1670 µg/kg for SIM analysis. LCS performance indicates the analytical systems are in control. The recovery for 4-nitrophenol was above the control limit in one LCS sample for SDG ES36. No action was taken since 4-nitrophenol was not detected in the associated samples.



**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 with the exception of the following:

Internal Standard	Sample	Compounds	Qualifier
Phenanthrene-d10 Chrysene-d12 Perylene-d12 Di-n-octylphthalate-d4	470E-SS-20d, 470E-SS-20s, 470E-SS-20s msd	Compounds associated with noncompliant internal standards	J, UJ
Perylene-d12	470E-SS-34	Compounds associated with noncompliant internal standard	J, UJ

For the above samples, the laboratory notes in the case narrative that there was matrix interference effecting sample 470E-SS-20s since the spike and spike duplicate samples for 470E-SS-20s had similar internal problems. Sample 470E-SS-34 was reanalyzed with similar results. Compounds quantitated from the noncompliant internal standards were qualified as estimated (UJ, J).

**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. Data qualified by the laboratory with an 'M' were considered detected and qualified as estimated. The 'M' indicates an estimated value of an analyte with a low spectral match as determined by the GC analyst.

**Compound Quantitation and Reported Detection/Quantitation Limits:** The detection limits were acceptable as reported and no were problems noted. For the SIM PAH analysis, the laboratory noted that several samples were analyzed at a two-fold dilution due to dark extract color, elevating the reporting limit.

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

**Field Replicates:** Three blind field replicates were collected and analyzed for determination of analytical variability. Sample results for replicate pairs are presented in the sample results table. The field pairs were 470E-SS-34/08s, 470E-SS-35/20s and 470E-SS-36/22s.

The percent moisture for sample 470E-SS-36 was suspect based on comparison to its primary (470E-SS-22s). The moisture value for this sample also did not compare well with the independent determination reported in the inorganics data set. The laboratory was contacted and reviewed the moisture data and visually examined the moisture from the sample containers. The laboratory acknowledged that a mistake had been made in the moisture determination for sample 470E-SS-36 for the organics fractions (semivolatiles, pesticides, PCBs). The laboratory used the inorganic moisture value of 9.8% in the organic forms and submitted corrected results.

The following results were outside the precision acceptance range for soils of ± 35%RPD:

- Pair 470E-SS-36/22s : phenanthrene, fluoranthene, butylbenzylphthalate, indeno(1,2,3-cd)pyrene, and benzo(g,h,i)perylene.
- Pair 470E-SS-35/20s: 2-methylnaphthalene.
- Pair 470E-SS-34/08s: naphthalene, 2-methylnaphthalene, phenanthrene, anthracene, fluoranthene, pyrene, benzo(a)anthracene, chrysene, benzo(k)fluoranthene, benzo(a)pyrene, indeno(1,2,3-cd)pyrene, dibenz(a,h)anthracene and benzo(g,h,i)perylene.

Associated results have not been qualified.

**Overall Assessment:** All deliverables required by the project were present and data packages were complete. Sample holding times were met with the exceptions noted above. All results were qualified as estimated (U, UJ) because of high cooler temperatures. GC/MS tuning requirements were met. Initial and continuing calibration requirements were acceptable. Method blanks showed some low-level detections of bis(2-Ethylhexyl)phthalate requiring the qualification of some results. Internal standard (IS) performance was acceptable with the exception of a few internals where matrix interference was noted by the laboratory, necessitating data qualification. Compound identification and quantitation are acceptable. Raw data show no indications of system performance degradation. Overall analytical performance was considered acceptable, and data quality is 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.

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

Sample Type	Primary	Replicate	Rinsate	Totals
Water	0	0	3	3
Soil	60	3	0	63
Totals	60	3	3	66

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 at 4° C. (±2° C.). All extraction and analysis holding times were met with the exception of sample 470E-RS-01, which was extracted one day outside the holding time. All results for the sample were qualified as estimated (UJ).

Upon receipt at the laboratory, transport cooler temperatures ranged from 17 to 20 °C. The sample integrity was considered acceptable since the samples were originally placed in coolers containing ice. The elevated temperatures are not expected to significantly affect data quality. The data are considered usable and are not qualified.

**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 was above the limit of 20 percent and/or the combined breakdown of 4,4'-DDT and Endrin exceeded 30 percent in several standards. The following compounds were qualified due to the breakdown of 4,4'-DDT exceeding 20%:

Breakdown Check Date	Sample	Compound	Qualifier
9/29/02	470E-SS-08s dil	4,4'-DDD	NJ
9/23/02	470E-SS-24s	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-25	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-26s	4,4'-DDT	R
		DDE	NJ
	470E-SS-26d	4,4'-DDT	R
		DDE	NJ
	470E-SS-34	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-24d	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-35	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
9/23/02	470E-SS-11s	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-12s	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-12d	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-13s	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-13d	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-14s	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-14d	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-15s	4,4'-DDT	J
		DDE	NJ
		DDD	NJ
	470E-SS-15d	4,4'-DDT	J
		DDE	NJ
		DDD	NJ

470E-SS-19d	4,4'-DDT	J
	DDE	NJ
	DDD	NJ
470E-SS-20s	4,4'-DDT	J
	DDD	NJ

In accordance with the *Functional Guidelines*, sample results for 470E-SS-26s and 470E-SS-26d were qualified as 'R' for 4,4'-DDT since breakdown products DDD or DDE were present while 4,4'-DDT was undetected.

**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 low standard was 0.005 µg/mL, 0.01 µg/mL or 0.05 µg/mL depending on the compound. The high standards were 0.08 µg/mL, 0.16 µg/mL or 0.8 µg/mL depending on the compound. 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
9/22/02 09:34	a-bhc, Heptachlor	470E-SS-11d, 470E-SS-14d, 470E-SS-15d, 470E-SS-16s	J, UJ
9/22/02 16:53	Heptachlor, Endrin, Endosulfan I, 4,4'-DDD, Endosulfan Sulfate, Endrin ketone, Endrin aldehyde	470E-SS-17d	J, UJ
10/03/02 16:34	4,4'-DDD	470E-SS-20s ms, 470E-SS-20s msd	J, UJ
9/23/02 00:12	All compound s except Endosulfan I, methoxychlor	470E-SS-17d, 470E-SS-20d, 470E-SS-19s	J, UJ
9/23/02 07:30	Endrin, Endosulfan II, 4,4'-DDD, 4,4'-DDT	470E-SS-11s, 470E-SS-12d, 470E-SS-13s, 470E-SS-13d, 470E-SS-14s	J, UJ
9/23/02 14:49	b-bhc, Hepthachlor epoxide, Endosulfan I, Dieldrin, 4,4'-DDE, Endrin, Endosulfan II, 4,4' -DDD, Endosulfan Sulfate, Endrin ketone, Endrin Aldehyde, g-chlordane, a-chlordane	470E-SS-14d, 470E-SS-15s, 470E-SS-15d, 470E-SS-19d, 470E-SS-20s	J, UJ
9/27/02 07:26, 15:39 9/28/02 00:54, 09:06	d-bhc	470E-SS-01s, 470E-SS-01d, 470E-SS-02s, 470E-SS-02d, 470E-SS-03s, 470E-SS-03d, 470E-SS-04s, 470E-SS-04d, 470E-SS-05s, 470E-SS-05d, 470E-SS-06s, 470E-SS-06d, 470E-SS-07 dil, 470E-SS-08d dil, 470E-SS-09s dil, 470E-SS-09d dil, 470E-SS-08s dil, 470E-SS-10s dil, 470E-SS-10d dil, 470E-SS-10s, 470E-SS-10d	J, UJ
9/28/02 17:19	d-bhc, 4,4'-DDT	470E-SS-09s, 470E-SS-09d, 470E-SS-07, 470E-SS-08s, 470E-SS-08s ms, msd, 470E-SS-08d	J, UJ
9/21/02 15:28	d-bhc	470E-SS-21s dil, 470E-SS-21d dil, 470E-SS-22s dil, 470E-SS-22d dil, 470E-SS-23s dil, 470E-SS-23d dil, 470E-SS-24s dil	J,UJ
9/22/02 07:53, 16:06	d-bhc, Endosulfan sulfate	470E-SS-34 dil, 470E-SS-21s, 470E-SS-21d, 470E-SS-2s, 470E-SS-22s ms, msd, 470E-SS-22d, 470E-SS-23s, 470E-SS-23d, 470E-SS-24s, 470E-SS-24d, 470E-SS-25, 470E-SS-26s, 470E-SS-26d, 470E-SS-27, 470E-SS-35, 470E-SS-34	J, UJ
9/28/02 19:17 9/29/02 01:32	d-bhc, 4,4'-DDD, 4,4'-DDT, Methoxychlor	470E-SS-08s dil	J, UJ

**Blanks:** Method blanks were analyzed for each analytical group. Method blanks showed no detections of target compounds 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 for the

sample. Laboratory-established acceptance limits were 24-111% for tetrachloro-m-xylene and 20-119% for decachlorobiphenyl. The following samples were qualified as estimated due to surrogate recovery:

Surrogate	Sample	Compounds	Qualifier
Decachlorobiphenyl	470E-SS-22s dil	4,4'-DDE, 4,4'-DDT, 4,4'-DDD and gamma chlordane	J
Decachlorobiphenyl	470E-SS-15s dil	4,4'-DDE, 4,4'-DDT, 4,4'-DDD, methoxychlor, alpha-chlordane and gamma chlordane	J
Decachlorobiphenyl	470E-SS-17d dil	4,4'-DDE, 4,4'-DDT, 4,4'-DDD	J

**Matrix Spike/Matrix Spike Duplicate Analyses:** Matrix spike and matrix spike duplicate analyses were performed on 470E-SS-20s, 470E-SS-08s and 470E-SS-22s. Analyte spike concentrations ranged from 18.6 to 37 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

The MS/MSD was not spiked for 4,4'-DDT for samples 470E-SS-08s and 470E-SS-22s. Since the associated LCSs for 4,4'-DDT were acceptable, no further action was taken. For spike pair 470E-SS-20s, only an MS was provided. Since the recoveries were acceptable, no further action was taken. MS/MSD performance was considered acceptable for all samples.

**Laboratory Control Samples:** Three 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 several 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. The laboratory reported a 'C' qualifier for alpha-chlordane for sample 470E-SS-08d to indicate probable detection of alpha-chlordane for the sample, but with matrix interference. Since a concentration was not assigned to the 'C' qualification, the result was considered non-reportable. There were several dilutions for the majority of the samples due to the presence of high concentrations of target compounds. 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. No problems were identified during review of the raw data.

**Field Replicates:** Three blind field replicates were collected and analyzed for determination of analytical variability. Sample results for replicate pairs are presented with the sample results in Appendix D. Sample 470E-SS-36 was corrected for moisture by the laboratory as noted above. The field pairs were 470E-SS-34/08s, 470E-SS-35/20s and 470E-SS-36/22s. The following results were outside the precision acceptance range for soils of  $\pm 35\%$ :

- Pair 470E-SS-36/22s: 4,4'-DDD
- Pair 470E-SS-35/20s: 4,4'-DDE, 4,4'-DDT,  $\alpha$ -chlordane

**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 exceptions noted above. Calibration requirements were met and acceptable with the exception of data that were qualified due to the percent difference exceedances. Method blanks showed no target compounds to be present. Compound identification and quantitation are acceptable as reported. Overall analytical performance is considered acceptable, and data quality is 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.

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

Sample Type	Primary	Replicate	Rinsate	Totals
Water	0	0	3	3
Soil	61	3	0	64
Totals	61	3	3	67

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 at 4° C. ( $\pm 2^\circ$  C.). All extraction and analysis holding times were met with the exception of the extraction holding times for samples 470E-RS-01, 470E-SS-08s and 470E-SS-20s. All associated results were qualified as estimated (UJ).

Upon receipt at the laboratory, transport cooler temperatures ranged from 17 to 20 °C. The sample integrity was considered acceptable since the samples were originally placed in coolers containing ice. The elevated temperatures are not expected to significantly affect data quality. The data are considered usable and are not qualified.

**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%, demonstrating acceptable linearity.

**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 compounds 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 of 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-20s, 470E-SS-08s and 470E-SS-22s. Analyte spike concentrations for Aroclor 1242 were 372 µg/kg, 924 µg/kg, 8570 µg/kg and 22200/22100 µg/kg. The laboratory-established acceptance window for Aroclor 1242 for soil was 15-143%. The laboratory performed the MS/MSD analysis within the holding time for sample 470E-SS-08s with 'Y' qualifications, indicating unreportable results for Aroclor 1242, due to matrix interference. The sample, MS and MSD were re-extracted outside of the holding time but with acceptable recovery of Aroclor 1242. The MS/MSD performance was considered acceptable for all samples as qualified.

**Laboratory Control Samples:** LCS samples were analyzed with acceptable results. The laboratory-established acceptance windows for the analysis are 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,

which indicated an elevated detection limit. The 'Y' qualifier was considered a 'U' for data qualification purposes.

**Field Replicates:** Three blind field replicates were collected and analyzed for determination of analytical variability. Sample results for replicate pairs are presented with the sample results in Appendix D. The field pairs were 470E-SS-34/08s, 470E-SS-35/20s and 470E-SS-36/22s. All compound pairs with positive results were within the precision acceptance range for soils of  $\pm 35\%$ RPD. It is noted that for pair 470E-SS-34/08s sample 470E-SS-34 was reported as 2,800  $\mu\text{g/kg}$  AR1248 while replicate sample 470E-SS-08s was reported as 2,900 'Y'.

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

**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 exceptions noted above. Initial and continuing calibration requirements were acceptable. Method blanks showed no target compounds 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 was 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 -  $\text{C}_{12}$ ]) - Volatile Petroleum Products Method for Soil and Water Analyses, as established by the Washington State Department of Ecology.

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

Sample Type	Primary	Replicate	Rinsate	Trip Blanks	Totals
Water	0	0	2	1	3
Soil	12	1	0	0	13
Totals	12	1	2	1	16

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) and 7/40 days for solids and waters, respectively, at 4° C. ( $\pm 2^\circ$  C.).

Upon receipt at the laboratory, transport cooler temperatures ranged from 17 to 20 °C. Due to the elevated temperatures, all sample results were qualified as estimated. While the data were qualified due to the elevated cooler temperatures, the sample integrity was considered acceptable since the samples

were originally placed in coolers containing ice. The elevated temperatures are not expected to significantly affect data quality and the data are considered usable as qualified.

**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 ≤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.

Two equipment rinsate blanks and one trip blank were analyzed for NWTPH-G to evaluate field sampling technique, potential contributions of analytes and introduction of contaminants during transport. No positive results were reported for the field or 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-SS-20s was analyzed as the matrix spike/spike duplicate pair. Gasoline range hydrocarbons were evaluated at 270 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 solid LCS analyses were performed for gasoline range hydrocarbons with recoveries ranging from 100% to 144%. The laboratory-established acceptance limits for the LCS are 61 - 122%. The LCS recovery was above the laboratory-established acceptance limit for SDG ES36 (140%R, 144 %R). Samples 470E-SS-20s and 470E-SS-20d were qualified as estimated. The LCS spiking level was 250 mg/kg. Performance was considered acceptable and the analytical systems were determined to be in control as qualified.

**Compound Quantitation and Reported Detection/Quantitation Limits:** No were problems 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.

**Field Replicates:** One blind field replicate sample pair was submitted and analyzed for petroleum hydrocarbons for determination of analytical variability. Results for sample pair 470E-SS-20s/470E-SS-35 were non-detects for gasoline range hydrocarbons.

**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 performance was acceptable with the exceptions noted above. 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 was sufficient for project use.

## 7.0 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-seven samples were analyzed for metals, as summarized below:

Sample Type	Primary	Replicate	Rinsate	Totals
Soil	51	3	3	57

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. No preservatives were added to the soil samples. 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.

Upon receipt at the laboratory, transport coolers temperatures ranged from 17 – 20° C. The sample integrity was considered acceptable since the samples were originally placed in coolers containing ice.

The elevated temperatures are not expected to significantly affect data quality. The data are considered usable and are not qualified.

**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 six thallium analyses on 9/20/02 associated with CCB1 and CCB2. Thallium results for the six 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.

Three equipment rinsate blanks were collected and analyzed to evaluate field soil sampling technique and potential contributions of analytes. Positive results were reported for calcium, lead and sodium in one or more blanks. Associated results for calcium and lead are greater than 5 times the blank results, and no data require qualification. Sodium results for ten samples are less than 5 times the blank amount and are qualified as non-detects (U).

**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 LCS is a spiked method blank. All target analytes are within laboratory-established control limits (80–120% recovery) for the reported analyses of aqueous LCS (rinsate blanks). 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. 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). 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. Soil sample matrix spike recoveries are acceptable with the exception of antimony (all SDGs), arsenic (SDG ES37), calcium (SDG ES36, SDG ES37), and manganese (SDG ES36). All antimony recoveries are less than 30% and all associated results are non-detects, requiring their rejection (R). All arsenic, calcium and manganese results 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 within project-specified limits (85–115%R) for arsenic, lead and thallium. Seventeen selenium spike recoveries are outside acceptance limits; the associated results are qualified as estimated (UJ, J).

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:** Three blind field replicate soil sample pairs were submitted and analyzed for metals for determination of analytical variability:

- 470E-SS-08s/470E-SS-34
- 470E-SS-20s/470E-SS-35
- 470E-SS-22s/470E-SS-36

The replicate pairs are splits of homogenized soil. Sample results for replicate pairs 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. With the exception of thallium in the first pair, and calcium and iron in the third pair, results meet these guidelines and overall comparability is considered good. 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 two thallium CCBs, requiring

qualification of six associated results as non-detected (U). Sodium was reported in three rinsate blanks; associated results for 10 samples are less than 5 times the blank result and are qualified as non-detects (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 of all soil antimony results (R). Some arsenic, calcium and manganese results were qualified as estimated (J) because matrix spike recoveries were outside acceptance limits. Graphite furnace QC was acceptable with the exception that seventeen selenium results were qualified as estimated because post digestion spikes were outside acceptance limits. Reported quantitation or lower reporting limits are acceptable. Soil replicate analyses showed good comparability. Overall analytical performance is considered acceptable and the data quality is sufficient for project use.