



Memorandum

To	Robert Shoemaker/Chelmsford	Page 1
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409386	
Initial Reviewer	Kristin Rutherford/Chelmsford	
Peer Reviewer	Lori Herberich/Chelmsford	
Date	January 29, 2015	60281242.008.5

SUMMARY

Full validation was performed on the data for eight soil samples analyzed for project specific metals by EPA Methods 6010C and 6020A. The samples were collected at the Pines Area of Investigation in Indiana on November 19, 2014 and were submitted to ALS (formerly Columbia Analytical Laboratories) in Rochester, NY for analysis. ALS processed these samples under sample delivery group (SDG) number R1409386.

The analytical data were evaluated with reference to the "USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Superfund Data Review" (January 2010), the quality control (QC) criteria specified in the analytical method, and the RI/FS QAPP (AECOM, 2005) and the associated QAPP Addendum provided as Appendix B of the SSC Work Plan (AECOM, 2014). Modification of the Functional Guidelines was performed to accommodate the non-CLP methodology.

In general, the data appear valid as reported and may be used for decision making purposes. Qualification of the data was not required.

SAMPLES

The samples included in this review are listed below.

Sample IDs	Sample IDs
P15QBNS111914D (field duplicate of P15QBNS111914S in R1409381)	P15QBSB111914S
P15QCNS111914S	P15QCSB111914S
P15QCSS111914S	P15QDNS111914S
P15QDSB111914S	P15QDSS111914S

REVIEW ELEMENTS

Sample data were reviewed for the following review elements:

- Agreement of analyses conducted with chain-of-custody (COC) requests
- Holding times/sample preservation
- Instrument tuning
- Initial and continuing calibrations
- Laboratory blanks/equipment blanks
- Interference check standard results (ICSAB/ICSA)
- Matrix spike (MS) results
- Laboratory duplicate results
- Field duplicate results
- Laboratory control sample (LCS) results
- Internal standards
- Serial dilution results
- Sample results/reporting issues

DISCUSSION

Agreement of Analyses Conducted With COC Requests

Sample reports were reviewed against the analytical requests as designated on the COC and subsequent communications between AECOM and the laboratory. No issues were noted.

Holding Times/Sample Preservation

All samples were digested and analyzed within the method-specified holding time.

The chemical preservation for all samples was acceptable. The cooler temperatures were 0.7°C and 0.8°C upon receipt at the laboratory, which was outside the acceptance criterion of $4 \pm 2^\circ\text{C}$. Since the samples were received in good condition on ice, no action was required.

Instrument Tuning – ICP-MS

All instrument tuning met QC acceptance criteria.

Initial and Continuing Calibrations

All initial calibrations, initial calibration verification standards (ICVs) and continuing calibration verification standards (CCVs) met QC acceptance criteria. The laboratory analyzed low-level check standards, Contract Required Detection Limit (CRDL) standards, which were spiked with chromium, cobalt, iron, thallium, vanadium, and uranium at the reporting limit (RL) and with aluminum and arsenic at 2x the RL. The recoveries of the CRDL standards were within the acceptance criterion of 70-130%.

Laboratory Blanks/Equipment Blanks

Results for all analytes were reported down to the instrument detection limit (IDL) and nondetects were reported at the IDL. Chromium was detected in the equipment blank associated with the samples in this SDG. Several analytes were detected in the initial and/or continuing calibration blanks (ICBs and/or CCBs) and the laboratory preparation blanks associated with all the samples in this SDG. The following

tables summarize the blank contamination detected and the associated samples. Actions were applied as indicated below.

Date Analyzed	Blank Type	Analyte	Concentration	Units	Actions for Samples	Affected Samples
12/1/14	CCB	Co	2.925 J	ug/L	U@RL	P15QBNS111914D P15QBSB111914S P15QCNS111914S P15QCSB111914S P15QDNS111914S P15QDSB111914S P15QDSS111914S
collected 11/19/14	P15111914B1 EB	Cr	1.1 J	ug/L	U @ sample result	P15QBNS111914D P15QBSB111914S P15QDNS111914S P15QDSB111914S
12/5/14	CCB	Cr	-0.178 J	ug/L		
12/5/14	CCB	Tl	0.055 J	ug/L	U@ RL or UJ NDs	P15QBNS111914D P15QBSB111914S P15QCNS111914S P15QCSB111914S P15QCSS111914S P15QDNS111914S P15QDSB111914S P15QDSS111914S
	PB	Tl	-0.004 J	mg/kg		
12/5/14	ICB	U	0.006 J	ug/L	U@RL	P15QBNS111914D P15QBSB111914S P15QCNS111914S P15QCSB111914S P15QCSS111914S P15QDNS111914S P15QDSB111914S
	PB	U	-0.001 J	mg/kg		

January 2010 National Functional Guidelines Blank Actions

Blank Type	Blank Result	Sample Result	Action for Samples
ICB/CCB (Positive)	$\geq \text{IDL/MDL but } \leq \text{QL}$	Nondetect	No action
		$\geq \text{IDL/MDL but } \leq \text{QL}$	Qualify as nondetect (U) at the QL
		$> \text{QL}$	Use professional judgment (see below [1])
	$> \text{QL}$	$\geq \text{IDL/MDL but } \leq \text{QL}$	Qualify as nondetect (U) at the QL
		$> \text{QL but } < \text{Blank Result}$	Qualify as nondetect (U) at the blank level Or qualify result as unusable (R).
		$> \text{Blank Result}$	Use professional judgment (see below [1])
ICB/CCB (Negative)	$\leq (-\text{IDL/MDL}) \text{ but } \geq (-\text{QL})$	$\geq \text{IDL/MDL or nondetect}$	Use professional judgment (see below [2])
	$< (-\text{QL})$	$< 10 \times \text{QL}$	Quality results $\geq \text{QL}$ as estimated low (J-) and nondetects as estimated (UJ)
		$> 10 \times \text{QL}$ (professional judgment)	No action (professional judgment)

Blank Type	Blank Result	Sample Result	Action for Samples
PB / EB / FB (Positive)	> QL	$\geq \text{IDL/MDL but } \leq \text{QL}$	Qualify as nondetect (U) at the QL
		> QL but < 10x Blank Result	Qualify results as unusable (R) or estimated high (J+)
		$\geq 10\text{x Blank Result}$	No action
	$\geq \text{IDL/MDL but } \leq \text{QL}$	Nondetect	No action
		$\geq \text{IDL/MDL but } \leq \text{QL}$	Qualify as nondetect (U) at the QL
		> QL	Use professional judgment (see below [1])
PB (Negative)	< (-QL)	< 10x QL	Qualify results $\geq \text{QL}$ as estimated low (J-), non-detects as estimated (UJ)
		> 10x QL (professional judgment)	No action (professional judgment)

[1] Establish an action level (AL) at 5x the blank contamination. If sample result is <AL, qualify the reported result with a "U".

[2] Estimate positive results and nondetects (J-/UJ).

Interference Check Standard Results (ICSAB and ICSA)

Interference check standard results for the ICSAB solutions met QC acceptance criteria.

Chromium, cobalt, and thallium were detected in the ICSA standards at concentrations greater than the MDL.

In the 6010 analysis, cobalt was detected at a negative concentration that was greater than the absolute value of the method detection limit (MDL) in the ICSA standards associated with all soil samples. The concentration of the interferent aluminum was present in the soil samples at concentrations below the respective concentration in the ICSA standard. However, the interferent iron was detected at a concentration greater than that found in the ICSA standard for soil sample P15QCNS111914S; and calcium and magnesium were detected at a concentration greater than that found in the ICSA standard for soil sample P15QDSS111914S. The samples did not require qualification since cobalt was present at a concentration >10% of the absolute value of the negative result for cobalt in the ICSA standard.

In the 6020A analysis, the only interferent reported in the raw data was aluminum. Aluminum is a target compound reported from the 6010 analysis. During data validation, the aluminum results from the 6010 analysis were compared to those in the 6020A analysis for all soil samples. Consequently, professional judgment was applied to use the results for the interferents (aluminum, calcium, iron, and magnesium) reported in the 6010 analysis to evaluate the potential for interelement interferences in the 6020A analysis.

Chromium and thallium were detected at a concentration that was greater than the MDL in the ICSA standards associated with all soil samples. One or more of the interferents (aluminum, calcium, iron, and magnesium) from the 6010 analysis of soil samples P15QCNS111914S and P15QDSS111914S were present at a concentration that was equal to or greater than the true value concentration of the interferents spiked in the 6020A analysis of the ICSA standards. Therefore, the positive results for chromium were qualified as estimated biased high (J+) in soil samples P15QCNS111914S and P15QDSS111914S. The result for chromium in sample P15QCNS111914S was subsequently qualified due to internal standard recovery; therefore, the overall qualification is estimated (J). Since the results for thallium in samples P15QCNS111914S and P15QDSS111914S were negated due to laboratory blank contamination, no qualifications were required due to positive bias in the ICSA.

MS Results

MS analysis was performed on soil sample P15QDSB111914S submitted with this sample set. The recovery of iron did not meet QC acceptance criteria since the unspiked sample concentration exceeded 4x the spiked concentration. Other than this notation, no validation action was taken on this basis.

Laboratory Duplicate Results

Laboratory duplicate analysis was performed on soil sample P15QDSB111914S submitted with this sample set. The relative percent difference (RPD) and/or absolute difference met the QC acceptance criteria.

Field Duplicate Results

Soil samples P15QBNS111914S (reported in R1409381) and P15QBNS111914D were collected as the field duplicate pair submitted with this sample set. The following table summarizes the RPDs of the detected analytes in these samples. The RPD criterion of thallium was doubled since the sample and field duplicate results were both $\leq 5x$ the QL. Precision was deemed acceptable for thallium. The RPDs of the remaining analytes were within QAPP acceptance limit of $\pm 30\%$ indicating acceptable precision.

Analyte	P15QBNS111914S (mg/kg)	P15QBNS111914D (mg/kg)	RPD (%)
Aluminum	1610	1910	17
Arsenic	1.9	1.6	17
Cobalt	1.2	1.2	0
Iron	2400	2960	21
Thallium	0.021	0.037	55
Chromium	2.4	2.8	15
Uranium	0.180	0.202	12
Vanadium	4.1	5.2	24

LCS Results

The LCS recoveries met the QC acceptance criteria for all LCS analyses.

Internal Standards - ICP/MS

All internal standards met QC acceptance criteria (70-130%) with the following exceptions. Results were qualified as indicated.

Sample ID	Date	IS out	% compared to ICAL Std.	Action
P15QCSS111914S	12/5/14	Ge	131	J As
		Sc	132	J Cr and V
P15QCNS111914S	12/5/14	Ge	134	J As
		Sc	132	J Cr and V
P15QCSB111914S	12/5/14	Ge	133	J As
		Sc	131	J Cr and V
P15QDSB111914S	12/5/14	Ge	131	J As
		Sc	132	J Cr and V
P15QBSB111914S	12/5/14	Ge	131	J As

Note: If the result was previously negated due to laboratory blank contamination, no further action was taken on the basis of high internal standard recoveries.

Serial Dilution Results

Serial dilution analysis was performed on soil sample P15QDSB111914S for this sample set. The percent difference criteria were met.

Sample Results/Reporting Issues

Sample results were spot-checked. No issues were noted.

All soil samples were analyzed at a 5-fold dilution for arsenic, chromium, thallium, uranium, and vanadium analyzed by Method 6020A. Sample results, MDLs, and RLs were elevated accordingly.

Sample P15QCNS111914S was analyzed at a 50-fold dilution for iron by Method 6010C due to an elevated level in the undiluted sample. Sample results, MDLs, and RLs were elevated accordingly.