

Memorandum

To	Robert Shoemaker/Chelmsford	Page 1
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409432	
Initial Reviewer	Linda Adams/Chelmsford	
Peer Reviewer	Lori Herberich/Chelmsford	
Date	January 27, 2015	60281242.008.5

SUMMARY

Full validation was performed on the data for 14 soil samples and two aqueous equipment blanks 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 20, 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 R1409432.

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
P35QASB112014S	P35QCSB112014S
P35QBSS112014S	P35QDSS112014S
P35QBNS112014S	P35QDNS112014S
P35QBSB112014S	P35QDSB112014S
P35QCSS112014S	P35QPSS112014S
P35QCNS112014S	P35QPNS112014S
P35QCNS112014D (Field duplicate of P35QCNS112014S)	P35QPSB112014S
P37112014B1 (equipment blank)	P35112014B1 (equipment blank)

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- ICP/MS
- 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- ICP/MS
- 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. The following issue was noted.

The sample listed as P35QCSB112014D on the COC was listed in the sampling table for the project as P35QCSB112014S. The laboratory noted on the Cooler Receipt and Preservation Check Form that the jars containing this sample were labeled as P35QCSB112014S. Although the laboratory logged in and reported the sample with a sample ID of P35QCSB112014D, the sample ID listed in the memo and worksheets was corrected to and referred to as P35QCSB112014S as per the sampling table. Other than this notation, no validation action was taken on this basis.

Based on communications with AECOM, equipment blank P37112014B1 was incorrectly listed on the COC and bottle labels as P38112014B1. The EDD was manually corrected to the sample ID of P37112014B1. Other than this notation, no validation action was taken on this basis.

Holding Times/Sample Preservation

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

The chemical preservation for all samples was acceptable. One of two cooler temperatures (0°C) upon receipt at the laboratory was below the acceptance criteria of $4 \pm 2^\circ\text{C}$. The remaining cooler temperature was within the acceptance criterion of $4 \pm 2^\circ\text{C}$. Other than this notation, no validation action was taken on this basis.

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 quantitation limit (QL) and with aluminum and arsenic at 2x the QL. The recoveries of the CRDL standards were within the QC acceptance limits 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. Aluminum, chromium and iron were 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	PB/ICB/CCB	Analyte	Concentration Detected	Units	Affected Samples
12/4/14	CCB4	Cobalt	1.62 J	ug/L	P371I2014B1, P381I2014B1
12/4/14	CCB4	Iron	26.12 J	ug/L	
12/4/14	PBW	Thallium	0.051 J	ug/L	
12/4/14	CCB2	Uranium	0.008 J	ug/L	
12/4/14	CCB2	Aluminum	-17.94 J	ug/L	P35QASB112014S, P35QBSS112014S P35QBNS112014S, P35QBSB112014S P35QCSS112014S, P35QCNS112014S P35QCNS112014D, P35QCSB112014S
12/4/14	CCB3	Aluminum	-15.05 J	ug/L	P35QDSS112014S, P35QDNS112014S P35QDSB112014S, P35QPSS112014S P35QPNS112014S, P35QPSB112014S
12/4/14	CCB3	Arsenic	4.065 J	ug/L	P35QASB112014S, P35QBSS112014S P35QBNS112014S, P35QBSB112014S P35QCSS112014S, P35QCNS112014S P35QCNS112014D, P35QCSB112014S
12/4/14	CCB4	Arsenic	5.063 J	ug/L	P35QDSS112014S, P35QDNS112014S P35QDSB112014S, P35QPSS112014S P35QPNS112014S, P35QPSB112014S
12/4/14	ICB	Cobalt	2.17 J	ug/L	All soils
12/6/14	CCB3	Chromium	-0.097 J	ug/L	P35QCSS112014S, P35QCNS112014S P35QCNS112014D, P35QCSB112014S P35QDSS112014S, P35QDNS112014S P35QDSB112014S, P35QPSS112014S P35QPNS112014S, P35QPSB112014S
12/5/14	PBS	Iron	8.255 J	mg/kg	All soils
12/6/14	PBS	Uranium	0.006 J	mg/kg	All soils
12/6/14	PBS	Thallium	0.047 J	mg/kg	All soils

Date Collected	Equipment Blank ID	Analyte	Concentration Detected (ug/L)	Affected Samples
11/20/14	P351I2014B1	Chromium	2.6 J	Associated with all soils in this SDG
		Iron	829	

January 2010 National Functional Guidelines Blank Actions

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

[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.

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 interferents aluminum, calcium, and magnesium were present in the soil samples at concentrations below the respective concentration in the ICSA standard. However, the interferent iron was detected at a concentration equal to or greater than that found in the ICSA standard for the following soil samples: P35QASB112014S, P35QBNS112014S, P35QBSB112014S, P35QCSS112014S, P35QCNS112014S, P35QCNS112014D, P35QCSB112014S, P35QPSS112014S, P35QPNS112014S, and P35QPSB112014S. These samples did not require qualification since the estimated interference for arsenic and cobalt was <10% of the results for arsenic and cobalt in the associated samples.

In the 6010 analysis, arsenic was detected at a concentration that was greater than the MDL in the ICSA standards associated with all soil samples. The concentration of the interferents aluminum, calcium, and magnesium were present in the soil samples at concentrations below the respective concentration in the ICSA standard. However, the interferent iron was detected at a concentration equal to or greater than that found in the ICSA standard for the following soil samples: P35QASB112014S, P35QBNS112014S, P35QBSB112014S, P35QCSS112014S, P35QCNS112014S, P35QCNS112014D, P35QCSB112014S, P35QPSS112014S, P35QPNS112014S, and P35QPSB112014S. These samples did not require qualification since since the calculated estimated interferences for arsenic were <10% of the sample results for arsenic.

In the 6010 analysis, arsenic was detected at a concentration that was greater than the MDL and also at a negative concentration that was greater than the absolute value in the ICSA standards associated with the diluted analyses of soil samples P35QCSB112014S and P35QPNS112014S. The concentration of the interferences aluminum, calcium, and magnesium were present in these 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 samples P35QCSB112014S and P35QPNS112014S. These samples did not require qualification since the calculated estimated interferences for arsenic were <10% of the sample results for arsenic and >10x the absolute value of the negative result for arsenic in the ICSA standards.

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. Although the aluminum results from the 6020A analysis exceeded the calibration range, results were comparable to those reported from the 6010 analysis. Consequently, professional judgment was applied to use the results for the interferences (aluminum, calcium, iron, and magnesium) reported in the 6010 analysis to evaluate the potential for interelement interferences in the 6020A analysis.

Chromium, thallium, and vanadium 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 interferences (aluminum, calcium, iron, and magnesium) from the 6010 analysis of the soils samples were present at a concentration that was equal to or greater than the true value concentration of the interferences spiked in the 6020A analysis of the ICSA standards. Therefore, the positive results for chromium, thallium, and vanadium were qualified as estimated biased high (J+) in all soil samples due to interelement interferences. The results for chromium and vanadium were subsequently qualified due to serial dilution and laboratory duplicate imprecision; therefore, the overall qualification for chromium and vanadium was estimated (J). Thallium was qualified as non-detect (U) in soil sample P35QBSS112014S due to laboratory blank contamination. The non-detect result for thallium in this sample was not further qualified on the basis of interelement interferences. The results for thallium in the remaining soil samples were qualified as estimated biased high (J+) due to interelement interferences. The results for thallium were subsequently qualified due to low matrix spike recovery and laboratory duplicate imprecision; therefore, the overall qualification for thallium was estimated (J).

MS Results

MS analysis was performed on soil sample P35QCSB112014S submitted with this sample set. The unspiked concentrations of aluminum, arsenic, iron, and vanadium exceeded 4x the concentration spiked. Other than this notation, no validation action was taken on this basis.

The percent recovery of thallium (27%) fell below the QC acceptance criteria in the MS analysis performed on soil sample P35QCSB112014S. Thallium was within the QC acceptance criteria in the post-digestion spike performed on this sample. The detected and non-detected results for thallium were qualified as estimated (J, UJ, respectively) in all soil samples.

Laboratory Duplicate Results

Laboratory duplicate analysis was performed on soil sample P35QCSB112014S submitted with this sample set. The following table summarizes the relative percent differences (RPDs) of the detected analytes which exceeded the QAPP acceptance criteria of $\leq 20\%$ for sample results $> 5x$ the QL. The detected and non-detected results for arsenic, chromium, thallium, uranium, and vanadium were qualified as estimated (J, UJ, respectively) in all soil samples.

Analyte	Sample Result (mg/kg)	Lab Duplicate Result (mg/kg)	%RPD
Arsenic	429	530	21
Chromium	20.5	35	52
Thallium	4.65	11.4	84
Uranium	1.75	4.45	87
Vanadium	32.4	63.9	65

Field Duplicate Results

Soil samples P35QCNS112014S and P37QCNS112014D were the field duplicate pair submitted with this sample set. The following table summarizes the RPDs of the detected analytes in these samples. The RPDs of all analytes were within QAPP acceptance limit of $\pm 30\%$ indicating acceptable precision.

Analyte	P35QCNS112014S (mg/kg)	P35QCNS112014D (mg/kg)	RPD (%)
Aluminum	12800	10500	20
Arsenic	389	355	9.1
Cobalt	13.0	10.4	22
Iron	31400	29300	6.9
Thallium	7.5	9.5	24
Chromium	31.1	33.2	6.5
Uranium	3.4	4.3	23
Vanadium	51.4	56.4	9.3

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 with the following exceptions. Results were qualified as indicated.

Sample ID	Date	IS out	% compared to ICAL Std.	Action
P35QBNS112014S	12/6/14	Sc	135%	J chromium and vanadium
P35QBSB112014S	12/6/14	Sc	133%	J chromium and vanadium
P35QCNS112014S	12/6/14	Sc	134%	J chromium and vanadium
P35QPSS112014S	12/6/14	Sc	137%	J chromium and vanadium
P35QPNS112014S	12/6/14	Sc	144%	J chromium and vanadium

Serial Dilution Results

Serial dilution analyses were performed on soil sample P35QCSB112014S and on equipment blank sample P35112014B1S for all analytes. All criteria were met for the serial dilution analyses performed on equipment blank sample P35112014B1S. No validation action was taken on this basis.

The following table summarizes the percent differences (%Ds) of the analytes which exceeded the acceptance criteria of $\leq 10\%$ for sample results $>50\times$ the MDL for Method 6010C and $>100\times$ the MDL for Method 6020A in the serial dilution analysis performed on soil sample P35QCSB112014S. The detected and non-detect results for chromium, uranium, and vanadium were qualified as estimated (J, UJ, respectively) in all soil samples.

Analyte	Sample Result (ug/L)	Serial Dilution Result (ug/L)	%D
Chromium	37.9	45.9	21
Uranium	3.23	3.66	13
Vanadium	60.0	41.9	20

Sample Results/Reporting Issues

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

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

Soil samples P35QCSB112014S and P35QPNS112014S were analyzed at 10-fold dilutions for arsenic analyzed by Method 6010C due to elevated levels in the undiluted samples. Sample results, IDLs, and QLs were elevated accordingly.

All soil samples were analyzed at 10-fold dilutions for iron analyzed by Method 6010C due to elevated levels in the undiluted samples. Sample results, IDLs, and QLs were elevated accordingly.

The QAPP indicates that arsenic should be analyzed by Method 6020A in order to obtain an RL of 0.10 mg/kg for the soil samples. The laboratory analyzed arsenic in the soil samples by Method 6010C resulting in a RL of 1.0 mg/kg. Other than this notation, no validation action was taken on this basis.