

Memorandum

To	Robert Shoemaker/Chelmsford	Page 1
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409276	
Initial Reviewer	Linda Adams/Chelmsford	
Peer Reviewer	Lori Herberich/Chelmsford	
Date	January 28, 2015	60281242.008.5

SUMMARY

Full validation was performed on the data for 16 soil samples and one aqueous equipment blank 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 17, 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 R1409276.

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
P34QASS111714S	P34QCSB111714S
P34QANS111714S	P34QDSS111714S
P34QASB111714S	P34QDNS111714S
P34QBSS111714S	P34QDSB111714S
P34QBNS111714S	P34QPSS111714S
P34QBSB111714S	P34QPNS111714S
P34QCSS111714S	P34QPSB111714S
P34QCNS111714S	P34QCNS111714D (field duplicate of P34QCNS111714S)
P34111714B1 (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
- 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 temperature was 2.7°C upon receipt at the laboratory, which was within the acceptance criterion of $4 \pm 2^\circ\text{C}$.

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 criteria 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 and iron were detected in the equipment blanks 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	PBS	Thallium	0.016 J	mg/Kg	All soils
12/4/14	PBS	Uranium	0.003 J	mg/Kg	All soils
11/25/14	ICB	Cobalt	1.42 J	ug/L	All soils
11/25/14	CCB1	Arsenic	2.07 J	ug/L	P34QASS11174S, P34QASB11174S P34QBSS11174S, P34QBNS11174S
11/25/14	CCB3	Arsenic	1.59 J	ug/L	P34QBSB11174S, P34QCSS11174S P34QCNS11174S, P34QDSS11174S P34QDSS11174S, P34QDNS11174S P34QDSB11174S, P34QPSS11174S P34QPNS11174S, P34QPSB11174S
11/25/14	CCB4	Arsenic	1.83 J	ug/L	P34QCNS111714D
12/1/14	ICB	Cobalt	1.79 J	ug/L	P3411I714B1
12/14/14	ICB	Iron	18.68 J	ug/L	
11/26/14	ICB	Thallium	-0.033 J	ug/L	
11/26/14	CCB3	Uranium	-0.011 J	ug/L	

Date Collected	Equipment Blank ID	Analyte	Concentration Detected (ug/L)	Affected Samples
11/17/14	P3411I714B1	Aluminum	363	All soils in this SDG
		Chromium	4.6	
		Iron	937	

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)	$< 10 \times$ QL	Quality results \geq QL as estimated low (J-) and nondetects as estimated (UJ)
		$> 10 \times$ 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+)
		$\geq 10 \times$ 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 and arsenic were detected at concentrations that were greater than 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 soils 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: P34QASS111714S, P34QANS111714S, P34QASB111714S, P34QBSS111714S, P34QBNS111714S, P34QCSS111714S, P34QCNS111714S, P34QCSB111714S, P34QDSS111714S, P34QDNS111714S, P34QPSS111714S, P34QPNS111714S, P34QPSB111714S, and P34QCNS111714S. The 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 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 interferents (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 interferents (aluminum, calcium, iron, and magnesium) from the 6010 analysis of the soil samples 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, 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 imprecision; therefore, the overall qualification for chromium and vanadium was estimated (J). The results for thallium were subsequently qualified due to low matrix spike recovery and field duplicate imprecision; therefore, the overall qualification for thallium was estimated (J).

MS Results

MS analysis was performed on soil sample P34QPSB111714S submitted with this sample set. The unspiked concentrations of aluminum, arsenic, chromium, 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 (71%) fell below the QC acceptance criteria in the MS analysis performed on soil sample P34QPSB111714S. Thallium was within the QC acceptance criteria in the post-digestion spike (PDS) performed on this sample. The detected and non-detected results for thallium were qualified as estimated (J, UJ, respectively) in all soil samples.

The laboratory also performed a MS and PDS analysis on equipment blank P34111714B1. All recoveries were within the acceptance criteria in the MS and PDS. Other than this notation, no validation action was taken on this basis.

Laboratory Duplicate Results

Laboratory duplicate analysis was performed on soil sample P34QPSB111714S submitted with this sample set. The relative percent difference (RPD) of arsenic (39%) exceeded the QC acceptance criteria. The detected and non-detected results for arsenic were qualified as estimated (J, UJ, respectively) in all soil samples.

The laboratory also performed a laboratory duplicate analysis on equipment blank P34111714B1. All RPDs were within the acceptance criteria. Other than this notation, no validation action was taken on this basis.

Field Duplicate Results

Soil samples P34QCNS111714S and P34QCNS111714D 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 aluminum, iron, chromium, uranium, and vanadium were within QAPP acceptance limit of $\pm 30\%$ indicating acceptable precision. The RPD criterion for cobalt was doubled since both the sample and field duplicate results were $<5\times$ the RL. Precision was deemed acceptable for cobalt. The RPDs of arsenic and thallium exceeded the QAPP acceptance criteria of $\leq 30\%$. The detected and non-detect results for arsenic and thallium were qualified as estimated (J, UJ, respectively in all soil samples).

Analyte	P34QCNS111714S (mg/kg)	P34QCNS111714D (mg/kg)	RPD (%)
Aluminum	9710	11400	16
Arsenic	361	114	104
Cobalt	9.5	6.2	42
Iron	23200	23400	0.9
Thallium	5.8	3.8	42
Chromium	28.3	23.2	20
Uranium	2.7	2.2	20
Vanadium	44.4	35.0	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 with the following exceptions. The detected results for chromium and vanadium were qualified as estimated (J) in these samples.

Sample ID	Date	IS out	% compared to ICAL Std.
P34QANS111714S	12/4/14	Sc	146%
P34QASB111714S*	12/4/14	Sc	134%
P34QBSS111714S	12/4/14	Sc	140%
P34QBNS111714S	12/4/14	Sc	138%
P34QCSB111714S	12/4/14	Sc	134%
P34QPSS111714S	12/4/14	Sc	134%
P34QCNS111714D	12/4/14	Sc	134%
Note: *The cumulative qualifier for results affected by equipment blank contamination and internal standard results was "J" and not "J+".			

Serial Dilution Results

Serial dilution analyses were performed on soil sample P34QPSB111714S for all analytes, on soil sample P34QCSB111714S for arsenic, on soil sample P34QCNS111714D for iron, and on equipment blank sample P34111714B1S for all analytes. All criteria were met for the serial dilution analyses performed on soil sample P34QCSB111714S and on equipment blank sample P34111714B1S. 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 P34QPSB111714S. 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	42.47	49.67	17
Uranium	3.573	4.002	12
Vanadium	65.44	76.74	17

The following table summarizes the %D of iron which exceeded the acceptance criteria of $\leq 10\%$ for sample results $>50\times$ the MDL in the serial dilution analysis performed on soil sample P34QCNS111714D. The detected and non-detect results for iron were qualified as estimated (J, UJ, respectively) in all soil samples.

Analyte	Sample Result (ug/L)	Serial Dilution Result (ug/L)	%D
Iron	20100	22600	12

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, MDLs, and RLs were elevated accordingly.

Soil samples P34QANS111714S and P34QCSB111714S were analyzed at 10-fold dilutions for arsenic analyzed by Method 6010C due to elevated levels in the undiluted samples. Sample results, MDLs, and RLs 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, MDLs, and RLs 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.