

Memorandum

To	Robert Shoemaker, Chelmsford	Page	1
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409381		
Initial Reviewer	Linda Adams, Chelmsford		
Peer Reviewer	Lori Herberich, Chelmsford		
Date	February 5, 2015	60281242.008.5	

SUMMARY

Full validation was performed on the data for 17 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 18 and 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 R1409381.

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
P37QBSS111914S	P24QCNS111814S
P37QBNS111914S	P24QCSB111814S
P37QBNS111914D (Field duplicate of P37QBNS111914S)	P14QDSB111814S
P37QBSB111914S	P15QASS111914S

Sample IDs	Sample IDs
P37QCSS111914S	P15QANS111914S
P37QCNS111914S	P15QASB111914S
P37QCSB111914S	P15QBSS111914S
P37QDSS111914S	P15QBNS111914S
P24QCSS111814S	P15111914B1 (equipment blank)
P14111814B1 (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. 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 and 0.8°C upon receipt at the laboratory, which were below 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 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. 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
11/26/14	ICB	Cobalt	2.768 J	ug/L	P14111814B1 P15111914B1
11/26/14	PBW	Iron	8.552 J	ug/L	
12/8/14	PBW	Thallium	0.051 J	ug/L	
12/8/14	CCB2	Uranium	0.008 J	ug/L	
12/4/14	PBS	Aluminum	2.010 J	mg/kg	All soils
12/4/14	CCB3	Aluminum	-19.418 J	ug/L	P37QBSS111914S, P37QBNS111914S P37QBNS111914D, P37QBSB111914S P37QCNS111914S
12/4/14	CCB4	Aluminum	-22.430 J	ug/L	All soils except P37QBSS111914S P37QBNS111914S, P37QBNS111914D P37QBSB111914S, P37QCNS111914S
12/4/14	CCB2	Arsenic	3.526 J	ug/L	P37QBSS111914S, P37QBNS111914S P37QBNS111914D, P37QBSB111914S P37QCNS111914S
12/4/14	CCB4	Arsenic	4.942 J	ug/L	All soils except P37QBSS111914S P37QBNS111914S, P37QBNS111914D P37QBSB111914S, P37QCNS111914S
12/4/14	CCB2	Cobalt	3.027 J	ug/L	P37QBSS111914S, P37QBNS111914S P37QBNS111914D, P37QBSB111914S P37QCNS111914S
12/4/14	ICB	Cobalt	2.168 J	ug/L	All soils except P37QBSS111914S P37QBNS111914S, P37QBNS111914D P37QBSB111914S, P37QCNS111914S
12/5/14	CCB4	Chromium	-0.172 J	ug/L	All soils except P15QBSS111914S, P15QBNS111914S, P15QCNS111914S
12/5/14	CCB5	Chromium	-0.153 J	ug/L	P15QBSS111914S, P15QBNS111914S P15QCNS111914S
12/5/14	CCB3	Thallium	0.054 J	ug/L	P37QBSS111914S, P37QBNS111914S P37QBNS111914D, P37QBSB111914S P37QCNS111914S, P37QCSB111914S P37QDSS111914S, P24QCNS111814S

Date Analyzed	PB/ICB/CCB	Analyte	Concentration Detected	Units	Affected Samples
12/5/14	CCB5	Thallium	0.050 J	ug/L	All soils except P15QBSS111914S P15QBNS111914S, P15QCSS111914S
12/5/14	CCB6	Thallium	0.053 J	ug/L	P15QBSS111914S, P15QBNS111914S P15QCSS111914S
12/5/14	PBS	Vanadium	-0.033 J	mg/kg	All soils
12/5/14	ICB	Uranium	0.006 J	ug/L	All soils

Date Collected	Equipment Blank ID	Analyte	Concentration Detected (ug/L)	Affected Samples
11/18/14	P14111814B1	Chromium	1.2 J	P14QDSB111814S
11/19/14	P15111914B1	Chromium	1.1 J	P15QANS111914S P15QANS111914S P15QASB111914S P15QBSS111914S P15QBNS111914S P37QBSS111914S P37QBNS111914S P37QBNS111914D P37QBSB111914S P37QCSS111914S P37QCNS111914S P37QCSB111914S P37QDSS111914S
11/19//14	P37111914B1	Chromium	1.2 J	
		Iron	1060	
11/18/14	P24111814B1	Chromium	0.852 J	P24QCSS111814S, P24QCNS111814S P24QCSB111814S

Note: Equipment blank P37111914B1 was reported in SDG R1409320 and equipment blank P24111814B1 was reported in SDG R140330.

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])

Blank Type	Blank Result	Sample Result	Action for Samples
PB (Negative)	< (-QL)	< 10x QL	Qualify results \geq 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.

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 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: P37QBSS111914S, P37QDSS111914S, P24QCSS111814S, P24QCNS111814S, P24QCSB111814S, P14QDSB111814S, P15QASS111914S, P15QANS111914S, P15QBSS111914S, and P37QCSS111914S. The results for cobalt in these samples were previously qualified as non-detect due to laboratory blank contamination. The nondetect results for cobalt in these samples were subsequently qualified as estimated (UJ) due to the negative interelement interference.

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 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: P37QBSS111914S, P37QDSS111914S, P24QCSS111814S, P24QCNS111814S, P24QCSB111814S, P14QDSB111814S, P15QASS111914S, P15QANS111914S, P15QBSS111914S, and P37QCSS111914S. These samples did not require qualification since the estimated interference for arsenic was <10% of the results for arsenic and cobalt in the associated samples.

In the 6010 analysis, arsenic was detected at a negative concentration that was greater than the absolute value of the MDL in the ICSA standards associated with soil samples P24QCNS111814S and P14QDSB111814S. 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 associated with soil samples P24QCNS111814S and P14QDSB111814S. The nondetect results for cobalt in samples P37QBNS111914S, P37QBNS111914D, P37QBSB111914S, P37QCNS111914S, and P37QCSB111914S were qualified as estimated (UJ) due to the negative interelement interference. The remaining samples did not require qualification since cobalt was present in these samples at a concentration >10x of the absolute value of the negative result for cobalt 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 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 soils 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). Thallium was qualified as non-detect (U) in soil samples P15QANS111914S, P15QASB111914S, P15QBNS111914S, P15QBSS111914S, P37QBNS111914S, P37QBNS111914D, P37QBSS111914S, P37QCNS111914S, P37QCSB111914S, P37QCSS111914S, and P37QDSS111914S due to laboratory blank contamination. Thallium was not detected in soil sample P37QBSB111914S. The non-detect results for thallium in these sample were not further qualified on the basis of interelement interferences. The detected results for thallium in soil samples P14QDSB111814S, P15QASS111914S, P24QCNS111814S, P24QCSB111814S, and P24QCSS111814S were qualified as estimated biased high (J+) on the basis of interelement interferences but were then subsequently qualified as estimated due to low matrix spike recovery, laboratory duplicate imprecision, and serial dilution imprecision resulting in an overall qualification of estimated (J).

MS Results

MS analysis was performed on soil sample P14QDSB111814S 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 (27%) fell below the QC acceptance criteria in the MS analysis performed on soil sample P14QDSB111814S. 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.

The percent recovery of uranium (73%) fell below the QC acceptance criteria in the MS analysis performed on soil sample P14QDSB111814S. Uranium was within the QC acceptance criteria in the post-digestion spike performed on this sample. The detected and non-detected results for uranium were qualified as estimated (J, UJ, respectively) in all soil samples.

Laboratory Duplicate Results

Laboratory duplicate analysis was performed on soil sample P14QDSB111814S submitted with this sample set. The relative percent differences (RPDs) of the detected analytes were all within the QAPP acceptance criterion of <20% for sample results >5x the QL with the following exception. The RPD of thallium (26%) exceeded the QAPP acceptance criteria in the laboratory duplicate analysis. The detected and non-detected results for thallium were qualified as estimated (J, UJ, respectively) in all soil samples.

Field Duplicate Results

Soil samples P37QBNS111914S and P37QBNS111814D were 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 chromium was doubled since the sample and field duplicate results were both < 5x the QL. Precision was deemed acceptable for chromium. The RPDs of the remaining analytes were within QAPP acceptance limit of $\pm 30\%$ indicating acceptable precision.

Analyte	P37QBNS111814S (mg/kg)	P37QBNS111814D (mg/kg)	RPD (%)
Aluminum	2940	2940	0
Arsenic	1.2	1.1	8.7
Cobalt	0.970	0.969	0.10
Iron	2710	2720	0.4
Thallium	0.009	0.012	28.6
Chromium	4.8	2.6	59.5
Uranium	0.112	0.110	1.8
Vanadium	3.2	3.2	0

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
P37QBSS111914S	12/5/14	Sc	136%	J chromium and vanadium
P37QBNS111914D	12/5/14	Sc	133%	J chromium and vanadium
P37QCNS111914S	12/5/14	Sc	132%	J chromium and vanadium
P37QDSS111914S	12/5/14	Sc	138%	J chromium and vanadium
P24QCSS111814S	12/5/14	Ho	131%	J Thallium
		Sc	159%	J chromium and vanadium
		Bi	134%	J Uranium
P24QCNS111814S	12/5/14	Sc	166%	J chromium and vanadium
		Bi	136%	J Uranium
P24QCSB111814S	12/5/14	Sc	157%	J chromium and vanadium
		Bi	136%	J Uranium
P14QDSB111814S	12/5/14	Ho	131%	J Thallium
		Sc	163%	J chromium and vanadium
		Bi	137%	J Uranium
P15QASS111914S	12/5/14	Sc	145%	J chromium and vanadium
P15QANS111914S	12/5/14	Sc	139%	J chromium and vanadium
P15QASB111914S	12/5/14	Sc	131%	J chromium and vanadium
P15QBSS111914S	12/5/14	Sc	141%	J chromium and vanadium

Sample ID	Date	IS out	% compared to ICAL Std.	Action
P15QBNS111914S	12/5/14	Sc	132%	J chromium and vanadium

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 analyses were performed on soil sample P14QDSB111814S and on equipment blank sample P15111914B1S for all analytes. All criteria were met for the serial dilution analyses performed on equipment blank sample P15111914B1S. No validation action was taken on this basis.

The following table summarizes the percent differences (%Ds) of the analytes which exceeded the acceptance criterion of <10% for sample results >50x the MDL for Method 6010C and >100x the MDL for Method 6020A in the serial dilution analysis performed on soil sample P14QDSB111814S. The detected and non-detect results for chromium, thallium, 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	47.7	65.1	37
Thallium	11.8	13.7	17
Uranium	5.32	6.3	38
Vanadium	84.5	116.6	38

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 P14QDSB111814S and P24QCNS111714S 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.