



Memorandum

To	Robert Shoemaker/Chelmsford	Page 1
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409454	
Initial Reviewer	Kristin Rutherford/Chelmsford	
Peer Reviewer	Lori Herberich/Chelmsford	
Date	January 28, 2015	60281242.008.5

SUMMARY

Full validation was performed on the data for 12 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 21, 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 R1409454.

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
P06QANS112114S	P06QASS112114S
P06QBNS112114D (field duplicate of P06QBNS112114S)	P06QBNS112114S (field duplicate of P06QBNS112114D)
P06QBSS112114S	P06QCNS112114S
P06QCSS112114S	P06QDNS112114S
P06QDSS112114S	P29QBNS112114S
P29QBSB112114S	P29QBSS112114S
P06112114B1 (equipment blank)	P29112114B1 (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 0.8°C upon receipt at the laboratory, which was outside the acceptance criteria 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 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	Blank Type	Analyte	Concentration	Units	Actions for Samples	Affected Samples
12/5/14	CCB2	As	3.163 J	ug/L	U@sample result	P06QANS112114S P06QBNS112114S P06QCNS112114S
12/5/14	CCB4	Co	1.466 J	ug/L	U@RL	P06QANS112114S P06QBNS112114S P06QBNS112114D
12/6/14	CCB5	Tl	0.076 J	ug/L	U@RL	P06QANS112114S P06QASS112114S P06QBNS112114S P06QBSS112114S P06QCNS112114S P06QCSS112114S P06QDNS112114S P06QDSS112114S
12/6/14	ICB	U	0.008 J	ug/L	U@RL	P06QANS112114S P06QASS112114S P06QBNS112114S P06QBSS112114S P06QCNS112114S P06QCSS112114S P06QDNS112114S P06QDSS112114S
12/9/14	CCB5	Tl	0.187 J	ug/L	U@RL	P06QBNS112114D P29QBSB112114S
12/5/14	CCB2	Co	2.42 J	ug/L	U@RL	P06112114B1 P29112114B1
12/10/14	PB	Tl	-0.034 J	ug/L	UJ nondetect results	P06112114B1 P29112114B1
12/10/14	PB	U	-0.010 J	ug/L	UJ nondetect results	P06112114B1 P29112114B1

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

[1] Establish an action level (AL) at 5x the blank contamination. If sample result is $< \text{AL}$, 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.

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

In the 6010 analysis, cobalt and arsenic were 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: P06QBSS112114S, P29QBNS112114S, and P29QBSS112114S. The samples did not require qualification since arsenic and cobalt were present in these samples 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, 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 in all soil samples except P06QANS112114S. Therefore, the positive results for chromium, thallium, and vanadium were qualified as estimated biased high (J+) in all soil samples except P06QANS112114S.

MS Results

MS analysis was performed on soil sample P06QANS112114S submitted with this sample set. The recoveries of aluminum and iron did not meet recovery 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 P06QANS112114S submitted with this sample set. The relative percent difference (RPD) and/or absolute difference met the QC acceptance criteria.

Field Duplicate Results

Soil samples P06QBNS112114S and P06QBNS112114D 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 RPD of iron was outside QAPP acceptance limit of $\pm 30\%$. The detected and non-detected results for iron were qualified as estimated (J and UJ, respectively) in all soils. The RPDs of the remaining analytes were within QAPP acceptance limit of $\pm 30\%$ indicating acceptable precision.

Analyte	P14QBNS111814S (mg/kg)	P14QBNS111814D (mg/kg)	RPD (%)
Aluminum	3540	3140	12
Arsenic	1.5	2.0	29
Cobalt	0.70	0.69	1
Iron	1520	2810	60
Thallium	0.037	0.025	39
Chromium	1.9	1.7	11
Uranium	0.138	0.109	23
Vanadium	3.5	3.3	6

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.

Serial Dilution Results

Serial dilution analysis was performed on soil sample P06QANS112114S 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 chromium, thallium, uranium, and vanadium analyzed by Method 6020A. 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.