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Memorandum

To	Robert Shoemaker/Chelmsford	Page 1
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409320	
Initial Reviewer	Kristin Rutherford/Chelmsford	
Peer Reviewer	Lori Herberich/Chelmsford	
Date	January 27, 2015	60281242.008.5

SUMMARY

Full validation was performed on the data for 18 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 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 R1409320.

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 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
P14QASB111814S	P14QASS111814S
P14QBNS111814D (field duplicate of P14QBNS111814S)	P14QBNS111814S (field duplicate of P14QBNS111814D)
P14QBSB111814S	P14QBSS111814S
P14QCNS111814S	P14QCSB111814S
P14QCSS111814S	P14QDSS111814S
P14QANS111814S	P37QDNS111914S
P37QDSB111914S	P37QGNS111914S
P37QGSB111914S	P37QGSS111914S
PM111914S	PXXQX111914S
P37111914B1 (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.

Note the sample ID for sample P14**QA**NS111814S was incorrectly identified as P14**QR**NS111814S in the data package. A revised data package was requested 1/26/15.

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 ± 2°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 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. 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
12/4/14	PB	Tl	-0.009 J	mg/kg	J/UJ results <10X RL	P14QASB111814S P14QASS111814S P14QBNS111814D P14QBSB111814S P14QBSS111814S P14QDSS111814S P14QANS111814S
11/30	CCB	Co	1.75 J	ug/L	U@RL	P14QASB111814S P14QASS111814S P14QBSB111814S P14QANS111814S
12/4/14	CCB	U	0.003 J	ug/L	U@RL	P14QASB111814S
12/5/14	ICB	Co	2.09 J	ug/L	U @ RL	PXXQX111914S
12/5/14	PB	Tl	0.033 J	mg/kg	U @ RL	P37QDNS111914S P37QDSB111914S P37QGNS111914S P37QGSB111914S P37QGSS111914S
12/5/14	PB	U	0.005 J	mg/kg	U@RL	P37QDNS111914S P37QDSB111914S P37QGNS111914S P37QGSB111914S PXXQX111914S
12/4/14	CCB	Co	2.02 J	ug/L	U @ RL	P37111914B1
12/8/14	CCB	Tl	0.050 J	ug/L	U @ RL	P37111914B1
12/8/14	CCB	U	0.008 J	ug/L	U @ RL	P37111914B1

Date Collected	Equipment Blank ID	Analyte	Concentration Detected (ug/L)	Actions for Samples	Affected Samples
11/19/14	P37111914B1	Fe	1060	J+ results <10X EB	P37QDNS111914S P37QDSB111914S P37QGNS111914S P37QGSB111914S P37QGSS111914S
11/18/14	P14111814B1 (in R1409381)	Cr	1.2 J	none, results >AL	P14 samples

January 2010 National Functional Guidelines Blank Actions

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

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 concentration that was greater than the method detection limit (MDL) in the ICSA standards associated with all soil samples. The concentration of the interferents aluminum and magnesium were present in the soil samples at concentrations below the respective concentration in the ICSA standard. However, the interferent calcium was detected at a concentration equal to or greater than that found in the ICSA standard for the following soil samples: P14QASS111814S and P14QBSS111814S. The interferent iron was detected at a concentration equal

to or greater than that found in the ICSA standard for the following soil samples: P14QASS111814S, P14QANS111814S, P14QBSS111814S, P14QBNS111814S, P14QBNS111814D, P14QBSB111814S, P14QDSS111814S, PM111914S, P14QCSS111814S, P14QCNS111814S, and P14QCSB111814S. The samples did not require qualification since arsenic and cobalt were present at a concentration >10% of the results for arsenic and 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 samples P14QASS111814S, P14QANS111814S, P14QASB111814S, P14QBSS111814S, P14QBNS111814S, P14QBNS111814D, P14QBSB111814S, and P14QDSS111814S. 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 samples P14QASS111814S, P14QANS111814S, P14QASB111814S, P14QBSS111814S, P14QBNS111814S, P14QBNS111814D, P14QBSB111814S, and P14QDSS111814S. The results for thallium were subsequently qualified due to matrix spike recovery and field duplicate imprecision; therefore, the overall qualification is estimated (J).

Chromium and thallium were detected at a concentration that was greater than the MDL in the ICSA standards associated with samples P37QDNS111914S, P37QDSB111914S, P37QGSS111914S, P37QGNS111914S, P37QGSB111914S, PXXQX111914S, PM111914S, P14QCSS111814S, P14QCNS111814S, and P14QCSB111814S. 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 and thallium were qualified as estimated biased high (J+) in samples P37QDNS111914S, P37QDSB111914S, P37QGSS111914S, P37QGNS111914S, P37QGSB111914S, PXXQX111914S, PM111914S, P14QCSS111814S, P14QCNS111814S, and P14QCSB111814S. Some of these results for thallium were subsequently qualified due to field duplicate imprecision; therefore, the overall qualification is estimated (J).

MS Results

MS analyses were performed on soil samples P14QANS1118114S and P37QDSB111914S submitted with this sample set. The recoveries of aluminum, arsenic, iron, and vanadium in sample P14QANS1118114S; and aluminum and iron in sample P37QDSB111914S 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.

The percent recovery of thallium (150%) was above the QC acceptance criteria in the MS analysis performed on soil sample P14QANS1118114S. The post-digestion spike for thallium performed on this sample was within the QC acceptance criteria. The detected results for thallium in site samples P14QASB111814S, P14QASS111814S, P14QBNS111814D, P14QBNS111814S, P14QBSB111814S, P14QBSS111814S, P14QDSS111814S, and P14QANS111814S were qualified as estimated (J).

The percent recovery of arsenic (134%) was above the QC acceptance criteria in the MS analysis performed on soil sample P37QDSB111914S. The post-digestion spike for arsenic performed on this sample was within the QC acceptance criteria. The detected results for arsenic in site samples

P14QCNS111814S, P14QCSB111814S, P14QCSS111814S, P37QDNS111914S, P37QDSB111914S, P37QGNS111914S, P37QGSB111914S, P37QGSS111914S, PM111914S, and PXXQX111914S were qualified as estimated (J).

Laboratory Duplicate Results

Laboratory duplicate analysis was performed on soil sample P14QANS111814S submitted with this sample set. The relative percent difference (RPD) of aluminum (28%) and arsenic (42%) exceeded the QC acceptance criteria. The detected and non-detected results for aluminum and arsenic in site samples P14QASB111814S, P14QASS111814S, P14QBNS111814D, P14QBNS111814S, P14QBSB111814S, P14QBSS111814S, P14QDSS111814S, and P14QANS111814S were qualified as estimated (J) were qualified as estimated (J and UJ, respectively).

Laboratory duplicate analysis was also performed on soil sample P37QDSB111914S submitted with this sample set. The RPD of iron (54%) exceeded the QC acceptance criteria. The detected and non-detected results for iron in site samples P14QCNS111814S, P14QCSB111814S, P14QCSS111814S, P37QDNS111914S, P37QDSB111914S, P37QGNS111914S, P37QGSB111914S, P37QGSS111914S, PM111914S, and PXXQX111914S were qualified as estimated (J and UJ, respectively).

Field Duplicate Results

Soil samples P14QBNS111814S and P14QBNS111814D 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 uranium was doubled since the sample and field duplicate results were both $\leq$ 5x the QL. Precision was deemed acceptable for uranium. The RPDs of arsenic and thallium were outside QAPP acceptance limit of ± 30%. The detected and non-detected results for arsenic and thallium were qualified as estimated (J and UJ, respectively) in all soils. The RPDs of the remaining analytes were within QAPP acceptance limit of ± 30% indicating acceptable precision.

Analyte	P14QBNS111814S (mg/kg)	P14QBNS111814D (mg/kg)	RPD (%)
Aluminum	9920	9160	8
Arsenic	341	249	31
Cobalt	10.2	8.4	19
Iron	30000	23600	24
Thallium	7.2	5.1	34
Chromium	29.6	35.8	19
Uranium	3.1	2.1	38
Vanadium	48.4	37.8	25

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
PM111914S	12/5/14	Sc	150	J Cr and V
P14QCSS111814S	12/5/14	Ge	143	J As
		Sc	139	J Cr and V
P14QCNS111814S	12/5/14	Ge	145	J As
		Sc	156	J Cr and V
P14QCSB111814S	12/5/14	Ge	142	J As
		Sc	155	J Cr and V

Serial Dilution Results

Serial dilution analyses were performed on soil samples P14QANS111814S and P37QDSB111914S 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.