

# Memorandum

To	Robert Shoemaker/Chelmsford	Page 2
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409434	
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Date	January 29, 2015	60281242.008.5

## SUMMARY

Full validation was performed on the data for 21 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 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 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. No data were rejected. Select data points were qualified as estimated due to nonconformances of certain QC criteria (see discussion below).

## SAMPLES

The samples included in this review are listed below.

Sample IDs	Sample IDs
P35QANS112014S	P38QBSS112014S
P35QASS112014S	P38QCNS112014S
P37QANS112014S	P38QCSB112014S
P37QASB112014S	P38QCSS112014S
P37QASS112014S	P38QDNS112014S
P38QANS112014S	P38QDSB112014S
P38QASB112014S	P38QDSS112014S

Sample IDs	Sample IDs
P38QASS112014S	P38QGNS112014S
P38QBNS112014D [Field duplicate of P38QBNS112014S]	P38QGSB112014S
P38QBNS112014S	P38QGSS112014S
P38QBSB112014S	P38112014B1 [Equipment blank]

## REVIEW ELEMENTS

Sample data were reviewed for the following parameters:

- 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 temperatures were 0.4 °C and 2.4 °C upon receipt at the laboratory. No action was taken for the slight nonconformance below the QC 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 arsenic, chromium, cobalt, iron, thallium, vanadium, and uranium at the reporting limit (RL) and with aluminum at 2x the RL. 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. Several analytes were detected in the initial and/or continuing calibration blanks (ICBs and/or CCBs), laboratory preparation blanks (PB), and equipment blanks [P38112014B1, P35112014B1 (found in SDG R1409432), and P37111914B1 (found in SDG R1409320)] associated with the samples in this SDG.

Tabulated below is the maximum blank contamination from all associated blanks, the associated samples, and the data validation actions taken. Detected results in blanks are not discussed in this data validation report if the associated results were nondetect or if qualification of sample results was not required.

<b>Blank ID</b>	<b>Analyte</b>	<b>Concentration</b>	<b>Associated Samples</b>	<b>Actions</b>
CCB4	Cobalt	2.806 J µg/L [0.281 mg/kg]	P38QGNS112014S P38QGSB112014S P37QASS112014S P37QANS112014S P37QASB112014S P35QASS112014S	The cobalt results in all associated samples except P35QASS112014S were qualified as nondetect (U) at the sample reporting limit (RL).
ICB	Cobalt	2.529 J µg/L [0.253 mg/kg]	P38QASS112014S P38QANS112014S P38QASB112014S P38QBSS112014S P38QBNS112014S P38QBNS112014S P38QBNS112014D P38QCSS112014S P38QCNS112014S P38QCSB112014S P38QDSS112014S P38QDNS112014S P38QDSB112014S P38QGSS112014S	The cobalt results in all associated samples except P38QASB112014S and P38QBSS112014S were qualified as nondetect (U) at the sample (RL).
ICB	Thallium	0.072 J µg/L [0.0072 mg/kg]	All soils except P35QANS112014S	The thallium results in samples P37QANS112014S, P37QASB112014S, P37QASS112014S, P38QBNS112014D, P38QCNS112014S, P38QCSB112014S, P38QCSS112014S, P38QDNS112014S, P38QDSB112014S, P38QDSS112014S, P38QGNS112014S, P38QGSB112014S, and P38QGSS112014S were qualified as nondetect (U) at the sample RL.
ICB	Uranium	0.008 J µg/L [0.0008 mg/kg]	All soils except P35QANS112014S	The uranium results in samples P37QANS112014S, P37QASB112014S, P38QCNS112014S, P38QCSB112014S, P38QCSS112014S

Blank ID	Analyte	Concentration	Associated Samples	Actions
				P38QDNS112014S, P38QDSB112014S, P38QDSS112014S, P38QGNS112014S, P38QGSB112014S and P38QGSS112014S were qualified as nondetect (U) at the sample RL.
P38112014B1	Chromium	1.4 J µg/L [0.14 mg/kg]	All samples from location P38	The chromium results in samples P38QCNS112014S, P38QCSB112014S, P38QGNS112014S, and P38QGSB112014S were qualified as nondetect (U) at the reported sample concentration.

Blank actions were applied based on the following guidance:

#### January 2010 National Functional Guidelines Blank Actions

Blank Type	Blank Result	Sample Result	Action for Samples
<b>ICB/CCB (Positive)</b>	≥ 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])
<b>ICB/CCB (Negative)</b>	≤ (-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)
		<b>PB / EB / FB (Positive)</b>	> 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])
<b>PB (Negative)</b>	< (-QL)	< 10x QL	Qualify results ≥ QL as estimated low (J-), nondetects 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: P38QANS112014S, P38QASB112014S, P38QBSS112014S, P38QBNS112014S, P38QCSS112014S, P38QCSB112014S, P38QDSS112014S, P38QDNS112014S, P38QGSS112014S, P35QASS112014S, and P35QANS112014S. The nondetect results for cobalt in samples P38QANS112014S, P38QBNS112014S, P38QCSS112014S, P38QCSB112014S, P38QDSS112014S, P38QDNS112014S, and P38QGSS112014S 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. These results were subsequently qualified due to laboratory and field duplicate imprecision; therefore, the overall qualification is estimated (J).

### **MS Results**

MS analysis was performed on soil sample P38QBSB112014S submitted with this sample set. The concentrations of aluminum, arsenic, chromium, iron, and vanadium were present in the unspiked parent sample at concentrations >4x the spike concentration. No validation action was taken on this basis. The percent recoveries for the remaining target analytes were within the QC acceptance criteria.

### **Laboratory Duplicate Results**

Laboratory duplicate analysis was performed on soil sample P38QBSB112014S submitted with this sample set. The relative percent differences (RPDs) for aluminum (34), arsenic (26), chromium (58), iron (29), uranium (113), and vanadium (36) exceeded the QAPP QC acceptance criterion of <20%RPD. The positive and nondetect results for these analytes were qualified as estimated (J, UJ, respectively) in all soil samples.

### **Field Duplicate Results**

Soil samples P38QBNS112014S and P38QBNS112012D were as the field duplicate pair submitted with this sample set. The following table summarizes the RPDs of the detected analytes in these samples. The RPDs for all target analytes exceeded the QAPP QC acceptance criterion of  $\leq 30\%$ RPD. The positive and nondetect results for aluminum, arsenic, chromium, cobalt, iron, thallium, uranium, and vanadium were qualified as estimated (J, UJ, respectively).

Analyte	P38QBNS112014S (mg/kg)	P38QBNS112014D (mg/kg)	RPD (%)
Aluminum	4830	2110	78
Arsenic	65.8	20.2	106
Chromium	11.3	6.3	57
Cobalt	5.3 J	2.3 J	79
Iron	18600	11400	48
Thallium	2.5	0.437 J	140
Uranium	3.8	0.592	146
Vanadium	29.3	15.1	64

### **LCS Results**

The LCS recoveries met the QC acceptance criteria for all LCS analyses.

### **Internal Standards - ICP/MS**

All internal standards met the QC acceptance criteria with the following exception tabulated below.

Sample ID	IS out	% compared to ICAL Std.
P35QANS112014S	SC	152%

Chromium was the only target analyte reported in association with this internal standard; therefore, the positive chromium result in sample P35QANS112014S was qualified as estimated (J).

### **Serial Dilution Results**

Serial dilution analyses were performed on soil sample P35QANS112014S for aluminum, cobalt, and iron and on soil sample P38QBSB112014 for all analytes. Additionally, serial dilution analysis was performed on equipment blank sample P38112014B1 for all analytes. All QC acceptance criteria were met.

### **Sample Results/Reporting Issues**

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

Soil samples P38QASB112014S and P38QBNS112014S were analyzed at 10-fold dilutions for arsenic analyzed by method 6020A and soil samples P35QANS112014 and P35QASS112014S were analyzed at 50-fold dilutions for arsenic by Method 6020A. All remaining soil samples were analyzed at a 5-fold dilution for arsenic analyzed by Method 6020A. All soil samples were analyzed at 5-fold dilutions 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 samples. Sample results, MDLs, and RLs were elevated accordingly.