



## Memorandum

To	Robert Shoemaker/Chelmsford	Page 1
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409459	
Initial Reviewer	Linda Adams/Chelmsford	
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### SUMMARY

Full validation was performed on the data for 10 soil samples 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 R1409459.

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
P29QASS112114S	P29QDNS112114S
P29QANS112114S	P29QDNS112114D(Field duplicate of P29QDNS112114S)
P29QASB112114S	P29QCSS112114S
P29QDSS112114S	P29QCNS112114S
P29QDSB112114S	P29QCSB112114S

## 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 temperature (0.8°C) upon receipt at the laboratory was 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 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 blank 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
12/6/14	ICB	Cobalt	1.66 J	ug/L	All soil samples
12/6/14	PBS	Aluminum	5.16 J	mg/kg	All soil samples
12/9/14	PBS	Thallium	0.009 J	mg/kg	All soil samples
12/9/14	PBS	Uranium	-0.001 J	mg/kg	All soil samples

Date Collected	Equipment Blank ID	Analyte	Concentration Detected (ug/L)	Affected Samples
11/21/14	P291I2114B1	Chromium	1.5 J	Associated with all soils in this SDG
		Iron	115	

### **January 2010 National Functional Guidelines Blank Actions**

Blank Type	Blank Result	Sample Result	Action for Samples
<b>ICB/CCB (Positive)</b>	$\geq$ 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])
<b>ICB/CCB (Negative)</b>	$\leq$ (-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)
<b>PB / EB / FB (Positive)</b>	> 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])

[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 <10x the negative blank result 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 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 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: P29QASS112114S, P29QANS112114S, P29QDSS112114S, and P29QCSS112114S. These samples did not require qualification since the estimated interference for cobalt was <10% of the results for 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.

Arsenic, 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 with the exception of soil samples P29QDSB112114S and P29QCSB112114S. Therefore, the positive results for arsenic, chromium, thallium, and vanadium were qualified as estimated biased high (J+) in all soil samples except samples P29QDSB112114S and P29QCSB112114S due to interelement interferences. Thallium was qualified as non-detect (U) in soil samples P29QASB112114S, P29QCNS112114S, P29QDNS112114S, and P29QDNS112114D due to laboratory blank contamination. The non-detect results for thallium in these samples were not further qualified on the basis of interelement interferences. Chromium and vanadium were subsequently qualified as estimated (J) in samples P29QCSS112114S and P29QDSS112114S due to internal standard nonconformances.

### **MS Results**

MS analysis was performed on soil sample P29QCSB112114S submitted with this sample set. The unspiked concentrations of aluminum and iron exceeded 4x the concentration spiked. Other than this notation, no validation action was taken on this basis.

The percent recoveries of the remaining analytes were all within the QC acceptance criteria.

### **Laboratory Duplicate Results**

Laboratory duplicate analysis was performed on soil sample P29QCSB112114S submitted with this sample set. The relative percent differences (RPDs) of the detected analytes were all within the QAPP acceptance criteria of  $\leq 20\%$  for sample results  $> 5x$  the QL.

### **Field Duplicate Results**

Soil samples P29QDNS112114S and P29QDNS112114D 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 all analytes were within QAPP acceptance limit of  $\pm 30\%$  indicating acceptable precision.

Analyte	P29QDNS112114S (mg/kg)	P29QDNS112114D (mg/kg)	RPD (%)
Aluminum	9150	6930	28
Arsenic	4390	3600	20
Cobalt	1.9	1.5	24
Iron	0.355	0.384	7.8

Analyte	P29QDNS112114S (mg/kg)	P29QDNS112114D (mg/kg)	RPD (%)
Thallium	5.0	5.6	11
Chromium	7.8	6.3	21
Uranium	0.481	0.542	12
Vanadium	8.3	8.3	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
P29QDSS112114S	12/6/14	Sc	133%	J chromium and vanadium
P29QCSS112114S	12/6/14	Sc	134%	J chromium and vanadium

### **Serial Dilution Results**

Serial dilution analysis was performed on soil sample P29QDNS112114S for all analytes. The percent differences (%Ds) of all analytes were within the acceptance criteria of  $\leq 10\%$  for sample results  $>50\times$  the MDL for Method 6010C and  $>100\times$  the MDL for Method 6020A.

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

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

All soil samples were analyzed at a 5-fold dilution for arsenic, chromium, thallium, uranium, and vanadium analyzed by Method 6020A. Sample results, MDLs, 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, MDLs, and QLs were elevated accordingly.