

# Memorandum

To	Robert Shoemaker/Chelmsford	Page 1
Subject	Data Validation Metals Analysis November 2014 Sampling Pines Area of Investigation, Indiana ALS SDG R1409330	
Initial Reviewer	Linda Adams/Chelmsford	
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Date	February 3, 2015	60281242.008.5

## SUMMARY

Full validation was performed on the data for 14 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, 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 R1409330.

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
P14QDNS111814S	P24QBSS111814S
P14QGSS111814S	P24QBNS111814S
P14QGNS111814S	P24QBNS111814D (field duplicate of P24QBNS111814S)
P14QGSB111814S	P24QBSB111814S
P24QASS111814S	P24QDSS111814S
P24QANS111814S	P24QDNS111814S
P24QASB111814S	P24QDSB111814S
P24111814B1 (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. The following issue was noted. The electronic data deliverable (EDD) was resubmitted by the laboratory due to a discrepancy in the reporting limits for the 6010C and 6020A analyses. The original submission of the EDD had the reporting limits elevated by a factor of 10. The EDD was corrected by the laboratory and resubmitted.

### Holding Times/Sample Preservation

All samples were digested and analyzed within the method-specified holding time.

The chemical preservation for all samples was acceptable. One of two cooler temperatures (0°C) upon receipt at the laboratory was below the acceptance criterion of  $4 \pm 2^\circ\text{C}$ . The remaining cooler temperature was within the acceptance criteria 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 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. Several analytes were detected in the initial and/or continuing calibration blanks (ICBs and/or CCBs), the laboratory preparation blanks, and the equipment 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
12/2/14	PBS	Aluminum	3.566 J	mg/Kg	All soils
12/2/14	PBS	Arsenic	-0.148 J	mg/Kg	All soils
12/2/14	PBS	Cobalt	-0.051 J	mg/Kg	All soils
12/4/14	PBS	Thallium	-0.006 J	mg/Kg	All soils
12/4/14	PBS	Chromium	0.094 J	mg/Kg	All soils
12/4/14	ICB	Cobalt	0.78 J	ug/L	All soils
12/4/14	CCB3	Thallium	0.033 J	ug/L	P14QDNS111814S, P14QGSS111814S P14QGNS111814S, P14QGSB111814S P24QASS111814S
12/4/14	ICB	Thallium	0.022 J	ug/L	All soils except P14QDNS111814S, P14QGSS111814S P14QGNS111814S, P14QGSB111814S P24QASS111814S
12/4/14	CCB3	Uranium	0.005 J	ug/L	P14QDNS111814S, P14QGSS111814S P14QGNS111814S, P14QGSB111814S P24QASS111814S
12/4/14	ICB	Uranium	0.002 J	ug/L	All soils except P14QDNS111814S, P14QGSS111814S P14QGNS111814S, P14QGSB111814S P24QASS111814S
11/26/14	ICB	Cobalt	1.79 J	ug/L	P14111814B1
11/26/14	ICB	Iron	18.68 J	ug/L	
12/1/14	ICB	Thallium	-0.033 J	ug/L	
12/1/14	CCB3	Uranium	-0.011 J	ug/L	

Date Collected	Equipment Blank ID	Analyte	Concentration Detected (ug/L)	Affected Samples
11/18/14	P24111814B1	Chromium	0.852 J	P24QASS111814S, P24QANS111814S P24QASB111814S, P24QBSS111814S P24QBNS111814S, P24QBNS111814D P24QBSB111814S, P24QDSS111814S P24QDNS111814S, P24QDSB111814S
11/18/14	P14111814B1	Chromium	1.2 J	P14QDNS111814S, P14QGSS111814S, P14QGNS111814S, P14QGSB111814S

Note: Equipment blank P14111814B1 was reported in SDG R1409381

### 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
$>$ 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])	
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  $<$ 10x blank result and nondetects (J-/UJ).

If positive and negative blanks are reported for an analyte in the calibration blanks, pick the highest blank (absolute value) and use the table above with the following modifications:

Blank Type	Blank Results	Actions in Table to Use	Modifications
ICB/CCB	Pos. $>$ Neg.	ICB/CCB (Positive)	Estimate nondetects UJ
	Neg. $>$ Pos.	ICB/CCB (Negative)	Use J without +/-

### Interference Check Standard Results (ICSAB and ICSA)

Interference check standard results for the ICSAB solutions met QC acceptance criteria.

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 interferences 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: P24QDNS111814S, P24QGSS111814S, P24QGNS111814S, P24QGSSB111814S, P24QDSS111814S, P24QDNS111814D, and P24QDSB111814S. These samples did not require qualification since the estimated interference for arsenic and cobalt was  $<$ 10% of the results for arsenic and 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 interferences (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. With the exception of soil sample P24QBSB111814S, one or more of the interferences (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 interferences 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 vanadium were subsequently qualified due to serial imprecision; therefore, the overall qualification for vanadium was estimated (J). Thallium was qualified as non-detect (U) in soil samples P24QASB111814S, P24QBNS111814S, P24QBNS111814D, and P24QBSS111814S due to laboratory blank contamination. The non-detect results for thallium in these samples were not further qualified on the basis of interelement interferences. The results for thallium in the soil samples P14QDNS111814S, P14QGNS111814S, P14QGSSB111814S, P14QGSS111814S, P24QANS111814S, P24QASS111814S, P24QDNS111814S, P24QDSB111814S, and P24QDSS111814S were qualified as estimated biased high (J+) due to interelement interferences. The result for thallium in soil sample P24QBSB111814S was previously qualified as estimated biased low (J-) due to negative blank actions therefore, the overall qualification for thallium was estimated (J). Chromium was qualified as non-detect (U) in soil samples P24QABNS111814S, P24QBNS111814S, and P24QBNS111814D due to laboratory blank contamination. The non-detect results for chromium in these three samples were not further qualified on the basis of interelement interferences. The results for chromium in the remaining soil samples were subsequently qualified due to serial dilution imprecision; therefore, the overall qualification for chromium in these samples was estimated (J).

### **MS Results**

MS analysis was performed on soil sample P24QDSB111814S 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.

### **Laboratory Duplicate Results**

Laboratory duplicate analysis was performed on soil sample P24QDSB111814S submitted with this sample set. The relative percent difference (RPDs) of were within the QC acceptance criteria for all analytes.

### **Field Duplicate Results**

Soil samples P24QBNS111814S and P24QBNS111814D 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 RPD criterion was doubled for thallium since the detected results were both <5x the RL. Precision was deemed acceptable for thallium. The RPDs of the remaining analytes were within QAPP acceptance limit of  $\pm 30\%$  indicating acceptable precision.

Analyte	P24QBNS111814S (mg/kg)	P24QBNA111814D (mg/kg)	RPD (%)
Aluminum	2230	2280	2.2
Arsenic	6.2J	8.0 J	25
Cobalt	0.587 J	0.642 J	9.0
Iron	2690	2880	6.8
Thallium	0.032 J	0.044 J	32
Chromium	2.0	2.1	4.9

Analyte	P24QBNS111814S (mg/kg)	P24QBNA111814D (mg/kg)	RPD (%)
Uranium	0.094 J	0.096 J	2.1
Vanadium	3.2	3.5	9.0

### **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. The detected results for chromium and vanadium were qualified as estimated (J) in these samples.

Sample ID	Date	IS out	% compared to ICAL Std.
P14QDNS111814S	12/4/14	Sc	145%
P14QGSB111814S	12/4/14	Sc	142%

### **Serial Dilution Results**

Serial dilution analyses were performed on soil sample P24QDSB111814S and on equipment blank sample P24111814B1 for all analytes. All criteria were met for the serial dilution analyses performed on equipment blank sample P24111814B1. No validation action was taken on this basis.

The following table summarizes the percent differences (%Ds) of the analytes which exceeded the acceptance criteria of  $\leq 10\%$  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 P24QDSB111814S. The detected and non-detect results for chromium 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	28.29	33.31	18
Vanadium	48.05	56.69	18

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

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

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.

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.

The following soil samples were analyzed for iron by Method 6010C at dilutions due to elevated levels in the undiluted samples. Sample results, MDLs, and RLs were elevated accordingly.

Sample	Dilution
P24QANS111814S, P24QASS111814S	10X

<b>Sample</b>	<b>Dilution</b>
P14QGSS111814S, P24QDNS111814S, P24QDSB111814S, P24QDSS111814S	20X
P14QDNS111814S, P14QGNS111814S, P14QGSB111814S	50X