



DATA VALIDATION REPORT

Gold King Mine Release Incident

SAMPLE DELIVERY GROUP: D75708

Prepared by

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I. INTRODUCTION

Task Order Title: Gold King Mine Release Incident
 Project No.: 20408.012.001.0274.00
 20408.012.001.0267.00
 Sample Delivery Group: D75708
 EPA Project Manager: Steve Way
 Weston Project Manager: Dave Robinson
 TDD No.: 0001/1508-04
 Matrix: Water and Sediment
 QC Level: Stage 2A
 No. of Samples: 2
 No. of Reanalyses/Dilutions: 0
 Laboratory: Accutest

Table 1. Sample Identification

<i>Location ID</i>	<i>Lab Sample Name</i>	<i>Matrix Type</i>	<i>Collection Date</i>	<i>Method</i>
CC06_092915_1800	D75708-2	Water	9/29/2015	SM3500C, SW9056
CC06_092915_1813	D75708-1	Sediment	9/29/2015	ASTM1498-76M, SW9045D, SW9056, SW3060A/7196A

II. Sample Management

Anomalies regarding sample management are noted below. The samples were received within the temperature limits of 4°C ±2°C. The samples were received intact, on ice, and properly preserved. The chain-of-custody (COC) was appropriately signed and dated by field and laboratory personnel. The presence or absence of custody seals on the cooler was not specifically noted.

The following issues were noted:

- Redox potential and pH were not requested on the COC.
- The COC did not list CLP sample IDs, and none were provided. The laboratory logged the samples per the location IDs on the COC.
- The presence or absence of sample tags was not noted in the case narrative, and sample tags were not listed on the COC.



Data Qualifier Reference Table

Qualifier	Organics	Inorganics
U	The analyte was analyzed for, but was not detected above the reported sample quantitation limit. The associated value is the quantitation limit or the estimated detection limit for dioxins or PCB congeners.	The material was analyzed for, but was not detected above the level of the associated value. The associated value is either the sample quantitation limit or the sample detection limit. The associated value is the sample detection limit or the quantitation limit for perchlorate only.
UB	The analyte was detected in the sample and in either the associated laboratory blank or field blank. If detected below the reporting limit (RL) the analyte result was reported as non-detected at the RL due to blank contamination. If detected above the RL, the analyte result was reported as non-detected at the reported result due to blank contamination.	The analyte was detected in the sample and in either the associated laboratory blank or field blank. If detected below the reporting limit (RL) the analyte result was reported as non-detected at the RL due to blank contamination. If detected above the RL, the analyte result was reported as non-detected at the reported result due to blank contamination.
J	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample.
J+	Not applicable	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample, and may have a potential positive bias.
J-	Not applicable	The analyte was positively identified; the associated numerical value is the approximate concentration of the analyte in the sample, and may have a potential negative bias.



Qualifier	Organics	Inorganics
UJ	The analyte was not deemed above the reported sample quantitation limit. However, the reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.	The material was analyzed for, but was not detected. The associated value is an estimate and may be inaccurate or imprecise.
UJB	The analyte was detected in the sample and in either the associated laboratory blank or field blank; the analyte result was reported as non-detected at either the RL or the reported result. The reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.	The analyte was detected in the sample and in either the associated laboratory blank or field blank; the analyte result was reported as non-detected at either the RL or the reported result. The reported quantitation limit is approximate and may or may not represent the actual limit of quantitation necessary to accurately and precisely measure the analyte in the sample.
N	The analysis indicates the presence of an analyte for which there is presumptive evidence to make a "tentative identification."	Not applicable.
NJ	The analysis indicates the presence of an analyte that has been "tentatively identified" and the associated numerical value represents its approximate concentration.	Not applicable.
R	The data are unusable. The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.	The data are unusable. The sample results are rejected due to serious deficiencies in the ability to analyze the sample and to meet quality control criteria. The presence or absence of the analyte cannot be verified.



Qualification Code Reference Table

Qualifier	Organics	Inorganics
H	Holding times were exceeded.	Holding times were exceeded.
S	Surrogate recovery was outside QC limits.	The sequence or number of standards used for the calibration was incorrect
C	Calibration %RSD or %D was noncompliant.	Correlation coefficient is <0.995 or calibration was noncompliant.
R	Calibration RRF was <0.05.	%R for calibration is not within control limits.
B	Presumed contamination as indicated by the preparation (method) blank results.	Presumed contamination as indicated by the preparation (method) or calibration blank results.
L	Laboratory Blank Spike/Blank Spike Duplicate %R was not within control limits.	Laboratory Control Sample %R was not within control limits.
L1	LCS/LCSD RPD was outside control limits.	LCS/LCSD RPD was outside control limits.
Q	MS/MSD recovery was poor.	MS recovery was poor.
Q1	MS/MSD RPD was outside control limits.	MS/MSD RPD was outside control limits.
E	Not applicable.	Duplicates showed poor agreement.
I	Internal standard performance was unsatisfactory.	ICP ICS results were unsatisfactory.
A	Not applicable.	ICP Serial Dilution %D were not within control limits.
M	Tuning (BFB or DFTPP) was noncompliant.	ICPMS tune was not compliant.
T	Presumed contamination as indicated by the trip blank results.	Not applicable.
+	False positive – reported compound was not present.	Not applicable.
-	False negative – compound was present but not reported.	Not applicable.
F	Presumed contamination as indicated by the FB or ER results.	Presumed contamination as indicated by the FB or ER results.
F1	Field duplicate results were outside the control limit.	Field duplicate results were outside the control limit.
\$	Reported result or other information was incorrect.	Reported result or other information was incorrect.



Qualifier	Organics	Inorganics
?	TIC identity or reported retention time has been changed.	Not applicable.
D	The analysis with this flag should not be used because another more technically sound analysis is available.	The analysis with this flag should not be used because another more technically sound analysis is available.
P	Instrument performance for pesticides was poor.	Post Digestion Spike recovery was not within control limits.
*II, *III	Unusual problems found with the data that have been described in Section II, "Sample Management," or Section III, "Method Analyses." The number following the asterisk (*) will indicate the report section where a description of the problem can be found.	Unusual problems found with the data that have been described in Section II, "Sample Management," or Section III, "Method Analyses." The number following the asterisk (*) will indicate the report section where a description of the problem can be found.



III. Method Analyses

A. VARIOUS EPA METHODS—General Chemistry

Reviewed By: P. Meeks

Date Reviewed: October 8, 2015

The samples listed in Table 1 for these analyses were validated based on the guidelines outlined in the *Sampling and Analysis Plan/Quality Assurance Project Plan for Gold King Mine Release, Silverton, San Juan County, Colorado* (2015), *United States Environmental Protection Agency Contract Laboratory Program Statement of Work for Inorganic Superfund Methods, SW-846 3060A/7196, 9045D, and 9056, ASTM Method 1498-76M, Standard Methods for the Examination of Water and Wastewater 3500CR B-2011*, and the *National Functional Guidelines for Superfund Inorganic Data Review* (2010).

- Holding Times: A footnote on the result summary page for sample CC06_092915_1800 indicated the laboratory preserved the hexavalent chromium aliquot within 24 hours of collection. Nitrate-N and nitrite-N were analyzed beyond the holding time in sample CC06_092915_1813; therefore, these results, nondetects, were qualified as estimated (UJ). The pH and redox potential measurements were performed in a fixed laboratory rather than on-site; therefore the pH result and redox potential results were qualified as estimated (J), as the analyses were not conducted in the field. No bias was assigned as the effect on the results could not be ascertained. The remaining holding times, as listed below, were met.
 - Redox Potential Vs. H₂ - ASAP
 - Hexavalent chromium (3060A/7196A) – 24-hours from extraction
 - Hexavalent chromium (3500 Cr) - 30 days to extract and 7 days to analyze
 - Nitrate-N and nitrite-N (9056) - 48 hours
 - pH (9045D) – ASAP
- Analytical Method Blanks: There were no detects in the method blanks.
- Laboratory Control Samples: The pH recovery was within the laboratory control limits of 63-158% and the EPA Method 150.1 check standard control limit of ± 0.05 . All other recoveries were within the laboratory control limits of 90-110%.
- Laboratory Duplicates: Laboratory duplicate analyses were performed on sample CC06_092915_1813 for hexavalent chromium and redox potential. Hexavalent chromium was not detected in either sample. The redox potential relative percent difference (RPD) was within the QAPP control limit of $\leq 20\%$.



- Matrix Spike/Matrix Spike Duplicate (MS/MSD): MS/MSD analyses are applicable only to the anion analysis. MS/MSD analyses were performed on sample CC06_092915_1813 for hexavalent chromium, nitrate-N, and nitrite-N. MSD recoveries were not reported. As the MSD results were reported, the reviewer calculated the recoveries. The nitrate-N and nitrite-N recoveries were within the method control limits of 80-120% and the RPDs were within the QAPP control limit of $\leq 20\%$.

A footnote on the MS result summary indicated the digested MS/MSD recoveries for hexavalent chromium were low at 22% and 35%. The reported MS/MSD results were actually post digestion spikes. As these recoveries were within the laboratory control limit of 75-125%; the nondetected parent sample result for hexavalent chromium was estimated (UJ) rather than rejected for the recoveries below 30%. As the digested MS/MSD RPD exceeded the control limit at 46%, the result was further qualified as estimated (UJ).

- Field QC Samples: Field QC samples were evaluated, and if necessary, qualified based on method blanks and other laboratory QC results affecting the usability of the field QC data. Any remaining detects were used to evaluate the associated site samples. Following are findings associated with field QC samples:
 - Field Blanks and Equipment Rinsates: This SDG had no identified field blank or equipment rinsate samples.
 - Field Duplicates: There were no field duplicate samples identified in this SDG.

Validated Sample Result Forms: D75708

Analysis Method ASTM1498-76M

Sample Name CC06_092915_1813

Matrix Type: Soil

Lab Sample Name: D75708-1

Sample Date: 9/29/2015

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Redox Potential Vs H2	T		704			mv		J	H

Analysis Method D3500C

Sample Name CC06_092915_1800

Matrix Type: Water

Lab Sample Name: D75708-2

Sample Date: 9/29/2015

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Chromium, Hexavalent	T	18540-29-9	0.1	0.1	0.005	mg/l	U	U	

Analysis Method SW3060A

Sample Name CC06_092915_1813

Matrix Type: Soil

Lab Sample Name: D75708-1

Sample Date: 9/29/2015

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Chromium, Hexavalent	T	18540-29-9	1	1	0.27	mg/kg	U	UJ	Q, Q1

Analysis Method SW9045D

Sample Name CC06_092915_1813

Matrix Type: Soil

Lab Sample Name: D75708-1

Sample Date: 9/29/2015

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
pH	T		2.78			su		J	H

Analysis Method SW9056

Sample Name CC06_092915_1813

Matrix Type: Soil

Lab Sample Name: D75708-1

Sample Date: 9/29/2015

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Nitrogen, Nitrate	T	14797-55-8	3.2	3.2	1.4	mg/kg	U	UJ	H
Nitrogen, Nitrite	T	14797-65-0	4.3	4.3	2.1	mg/kg	U	UJ	H

Analysis Method *SW9056*

Sample Name CC06_092915_1800

Matrix Type: Water

Lab Sample Name: D75708-2

Sample Date: 9/29/2015

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
Nitrogen, Nitrate	T	14797-55-8	0.05	0.05	0.03	mg/l	U	U	
Nitrogen, Nitrite	T	14797-65-0	0.02	0.02	0.015	mg/l	U	U	