



DATA VALIDATION REPORT

Gold King Mine Release Incident

SAMPLE DELIVERY GROUP: 680-117013-4

Prepared by

MEC^x
12269 East Vassar Drive
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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: 680-117013-4
 EPA Project Manager: Steve Way
 Weston Project Manager: Dave Robinson
 TDD No.: 0001/1508-04
 Matrix: Sediment/Water
 QC Level: Stage 2A
 No. of Samples: 2
 No. of Reanalyses/Dilutions: 0
 Laboratory: TestAmerica - Savannah

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_09212015_1300	680-117013-1	Sediment	9/21/15 1:00 PM	8270D
CC06_09212015_1300	680-117013-3	Water	9/21/15 1:00 PM	8270D

II. Sample Management

Anomalies regarding sample management are noted below. The aqueous sample was received within the temperature limits of 4°C ±2°C. The soil sample was received below the temperature limit at 0.6°C. As the sample was not noted to be frozen or damaged, no qualifications were required. The samples were received intact, on ice, and properly preserved. The chains-of-custody (COCs) were 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:

- The COCs requested a number of other analyses. Results of these analyses were reported in related SDGs 117013-1, 117013-3, and 117013-5.
- Some corrections made to one COC were made by overwriting the original entry. These corrections were not initialed or dated.
- The COCs did not list CLP sample IDs, and none were provided. The laboratory logged the samples per the location IDs on the COCs.
- The presence or absence of sample tags was not noted in the case narrative, and sample tags were not listed on the COCs.



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. EPA Method 8270D—Semivolatile Organic Compounds (SVOCs)

Reviewed By: P. Meeks

Date Reviewed: September 30, 2015

The samples listed in Table 1 for this analysis 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 Organic Superfund Methods, EPA Method SW-846 8270D, Contract Laboratory Program Statement of Work for Organic Superfund Methods*, and the *National Functional Guidelines for Inorganic Superfund Data Review* (2008).

- **Holding Times:** The soil sample was extracted within fourteen days of collection and the aqueous sample was extracted within seven days of collection. Both samples were analyzed within 40 days of extraction.
- **Analytical Method Blanks:** The method blanks had no target compound detects.
- **Laboratory Control Sample (LCS)/LCS Duplicate (LCSD):** 2,4-Dinitrophenol was not recovered in the soil LCS/LCSD and 4,6-dinitro-2-methylphenol was recovered below the control limit at 8% and 10%. Nondetected 2,4-dinitrophenol in the soil sample was rejected for the poor recoveries and nondetected 4,6-dinitro-2-methylphenol was qualified as estimated (UJ). The RPD for 4,6-dinitro-2-methylphenol exceeded the control limit at 47%; however, as the analyte was not detected in the soil sample, no qualification was applied. The RPD for 2,4-dinitrophenol exceeded the control limit; however, as the result was not retained, no further qualifications were applied. The remaining recoveries were within laboratory-established control limits and the remaining RPDs were within the QAPP control limit of $\leq 20\%$.
- **Surrogate Recovery:** The surrogate recoveries were within laboratory-established control limits.
- **Matrix Spike/Matrix Spike Duplicate (MS/MSD):** MS/MSD analyses were performed on the soil sample in this SDG. Both recoveries for bis(2-chloroethoxy)methane were below the control limit at 53% and 55%; therefore, the nondetected result for this compound was qualified as estimated (UJ) in the soil sample. Numerous other compounds had recoveries outside the control limits in either the MS or the MSD; however, qualifications were only applied for consistent outliers. Nineteen compounds had RPDs exceeded the control limit; however, as the associated results were nondetects, no qualifications were applied. The reviewer noted two acid fraction surrogates were recovered below the control



limit in the MS only, potentially indicating the generally low MS recoveries were not matrix-related.

MS/MSD analyses were performed on the aqueous sample in this SDG. Several compounds had recoveries outside the control limits in either the MS or the MSD; however, qualifications were only applied for consistent outliers. Eighteen compounds had RPDs exceeded the control limit; however, as the associated results were nondetects, no qualifications were applied.

The remaining recoveries were within the laboratory control limits and the remaining RPDs were within the QAPP control limit of $\leq 20\%$.

- 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: No field blank or equipment rinsate samples were identified in this SDG.
 - Field Duplicates: There were no field duplicate samples identified in this SDG.

Validated Sample Result Forms: 680-117013-4

Analysis Method 8270D

Sample Name CC06_09212015_1300

Matrix Type: Solid

Lab Sample Name: 680-117013-1

Sample Date: 9/21/2015 1:00:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
1,1'-Biphenyl	T	92-52-4	9500	9500	9500	ug/Kg	U	U	
2,4,5-Trichlorophenol	T	95-95-4	200	1800	200	ug/Kg	U	U	
2,4,6-Trichlorophenol	T	88-06-2	160	1800	160	ug/Kg	U	U	
2,4-Dichlorophenol	T	120-83-2	200	1800	200	ug/Kg	U	U	
2,4-Dimethylphenol	T	105-67-9	250	1800	250	ug/Kg	U F1	U	
2,4-Dinitrophenol	T	51-28-5	4600	9500	4600	ug/Kg	U *	R	L
2,4-Dinitrotoluene	T	121-14-2	270	1800	270	ug/Kg	U	U	
2,6-Dinitrotoluene	T	606-20-2	230	1800	230	ug/Kg	U	U	
2-Chloronaphthalene	T	91-58-7	200	1800	200	ug/Kg	U	U	
2-Chlorophenol	T	95-57-8	220	1800	220	ug/Kg	U F1	U	
2-Methylnaphthalene	T	91-57-6	210	1800	210	ug/Kg	U	U	
2-Methylphenol	T	95-48-7	150	1800	150	ug/Kg	U F1	U	
2-Nitroaniline	T	88-74-4	250	9500	250	ug/Kg	U	U	
2-Nitrophenol	T	88-75-5	230	1800	230	ug/Kg	U	U	
3 & 4 Methylphenol	T	15831-10-4	430	1800	240	ug/Kg	J	J	
3,3'-Dichlorobenzidine	T	91-94-1	160	3700	160	ug/Kg	U F1 F2	U	
3-Nitroaniline	T	99-09-2	260	9500	260	ug/Kg	U F1 F2	U	
4,6-Dinitro-2-methylphenol	T	534-52-1	950	9500	950	ug/Kg	U *	UJ	L
4-Bromophenyl phenyl ether	T	101-55-3	200	1800	200	ug/Kg	U	U	
4-Chloro-3-methylphenol	T	59-50-7	200	1800	200	ug/Kg	U	U	
4-Chloroaniline	T	106-47-8	290	3700	290	ug/Kg	U F1 F2	U	
4-Chlorophenyl phenyl ether	T	7005-72-3	250	1800	250	ug/Kg	U	U	
4-Nitroaniline	T	100-01-6	270	9500	270	ug/Kg	U F1 F2	U	
4-Nitrophenol	T	100-02-7	1800	9500	1800	ug/Kg	U	U	
Acenaphthene	T	83-32-9	230	1800	230	ug/Kg	U	U	
Acenaphthylene	T	208-96-8	200	1800	200	ug/Kg	U	U	
Acetophenone	T	98-86-2	160	1800	160	ug/Kg	U	U	
Anthracene	T	120-12-7	140	1800	140	ug/Kg	U	U	
Atrazine	T	1912-24-9	130	1800	130	ug/Kg	U	U	
Benzaldehyde	T	100-52-7	320	1800	320	ug/Kg	U	U	
Benzo[a]anthracene	T	56-55-3	150	1800	150	ug/Kg	U	U	

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Benzo[a]pyrene	T	50-32-8	290	1800	290	ug/Kg	U	U
Benzo[b]fluoranthene	T	205-99-2	210	1800	210	ug/Kg	U	U
Benzo[g,h,i]perylene	T	191-24-2	120	1800	120	ug/Kg	U	U
Benzo[k]fluoranthene	T	207-08-9	360	1800	360	ug/Kg	U	U
bis (2-chloroisopropyl) ether	T	108-60-1	170	1800	170	ug/Kg	U	U
Bis(2-chloroethoxy)methane	T	111-91-1	220	1800	220	ug/Kg	U F1	UJ Q
Bis(2-chloroethyl)ether	T	111-44-4	250	1800	250	ug/Kg	U	U
Bis(2-ethylhexyl) phthalate	T	117-81-7	160	1800	160	ug/Kg	U	U
Butyl benzyl phthalate	T	85-68-7	150	1800	150	ug/Kg	U	U
Caprolactam	T	105-60-2	370	1800	370	ug/Kg	U F1	U
Carbazole	T	86-74-8	170	1800	170	ug/Kg	U F1 F2	U
Chrysene	T	218-01-9	120	1800	120	ug/Kg	U	U
Dibenz(a,h)anthracene	T	53-70-3	220	1800	220	ug/Kg	U	U
Dibenzofuran	T	132-64-9	180	1800	180	ug/Kg	U	U
Diethyl phthalate	T	84-66-2	210	1800	210	ug/Kg	U	U
Dimethyl phthalate	T	131-11-3	190	1800	190	ug/Kg	U	U
Di-n-butyl phthalate	T	84-74-2	170	1800	170	ug/Kg	U	U
Di-n-octyl phthalate	T	117-84-0	160	1800	160	ug/Kg	U	U
Fluoranthene	T	206-44-0	180	1800	180	ug/Kg	U	U
Fluorene	T	86-73-7	200	1800	200	ug/Kg	U	U
Hexachlorobenzene	T	118-74-1	220	1800	220	ug/Kg	U	U
Hexachlorobutadiene	T	87-68-3	200	1800	200	ug/Kg	U	U
Hexachlorocyclopentadiene	T	77-47-4	230	1800	230	ug/Kg	U	U
Hexachloroethane	T	67-72-1	160	1800	160	ug/Kg	U	U
Indeno[1,2,3-cd]pyrene	T	193-39-5	160	1800	160	ug/Kg	U	U
Isophorone	T	78-59-1	180	1800	180	ug/Kg	U	U
Naphthalene	T	91-20-3	170	1800	170	ug/Kg	U	U
Nitrobenzene	T	98-95-3	150	1800	150	ug/Kg	U	U
N-Nitrosodi-n-propylamine	T	621-64-7	180	1800	180	ug/Kg	U	U
N-Nitrosodiphenylamine	T	86-30-6	180	1800	180	ug/Kg	U F1	U
Pentachlorophenol	T	87-86-5	1800	9500	1800	ug/Kg	U	U
Phenanthrene	T	85-01-8	150	1800	150	ug/Kg	U	U
Phenol	T	108-95-2	190	1800	190	ug/Kg	U F1 F2	U
Pyrene	T	129-00-0	150	1800	150	ug/Kg	U	U

Analysis Method 8270D

Sample Name CC06_09212015_1300

Matrix Type: Water

Lab Sample Name: 680-117013-3

Sample Date: 9/21/2015 1:00:00 PM

Analyte	Total/Dissolved	CAS No	Result Value	Reporting Limit	MDL	Result Units	Lab Qualifier	Validation Qualifier	Validation Notes
1,1'-Biphenyl	T	92-52-4	0.57	9.8	0.57	ug/L	U	U	
2,4,5-Trichlorophenol	T	95-95-4	1.2	9.8	1.2	ug/L	U	U	
2,4,6-Trichlorophenol	T	88-06-2	0.84	9.8	0.84	ug/L	U	U	
2,4-Dichlorophenol	T	120-83-2	1.1	9.8	1.1	ug/L	U	U	
2,4-Dimethylphenol	T	105-67-9	3.9	9.8	3.9	ug/L	U	U	
2,4-Dinitrophenol	T	51-28-5	9.8	49	9.8	ug/L	U	U	
2,4-Dinitrotoluene	T	121-14-2	1.2	9.8	1.2	ug/L	U	U	
2,6-Dinitrotoluene	T	606-20-2	1.1	9.8	1.1	ug/L	U	U	
2-Chloronaphthalene	T	91-58-7	0.79	9.8	0.79	ug/L	U F1	U	
2-Chlorophenol	T	95-57-8	0.85	9.8	0.85	ug/L	U	U	
2-Methylnaphthalene	T	91-57-6	0.77	9.8	0.77	ug/L	U	U	
2-Methylphenol	T	95-48-7	0.87	9.8	0.87	ug/L	U	U	
2-Nitroaniline	T	88-74-4	1.3	49	1.3	ug/L	U	U	
2-Nitrophenol	T	88-75-5	0.75	9.8	0.75	ug/L	U	U	
3 & 4 Methylphenol	T	15831-10-4	1.3	9.8	1.3	ug/L	U	U	
3,3'-Dichlorobenzidine	T	91-94-1	29	59	29	ug/L	U	U	
3-Nitroaniline	T	99-09-2	4.9	49	4.9	ug/L	U	U	
4,6-Dinitro-2-methylphenol	T	534-52-1	9.8	49	9.8	ug/L	U	U	
4-Bromophenyl phenyl ether	T	101-55-3	0.76	9.8	0.76	ug/L	U F1	U	
4-Chloro-3-methylphenol	T	59-50-7	0.98	9.8	0.98	ug/L	U	U	
4-Chloroaniline	T	106-47-8	2.2	20	2.2	ug/L	U	U	
4-Chlorophenyl phenyl ether	T	7005-72-3	0.83	9.8	0.83	ug/L	U F1	U	
4-Nitroaniline	T	100-01-6	4.9	49	4.9	ug/L	U	U	
4-Nitrophenol	T	100-02-7	1.9	49	1.9	ug/L	U	U	
Acenaphthene	T	83-32-9	0.75	9.8	0.75	ug/L	U	U	
Acenaphthylene	T	208-96-8	0.84	9.8	0.84	ug/L	U	U	
Acetophenone	T	98-86-2	0.56	9.8	0.56	ug/L	U	U	
Anthracene	T	120-12-7	0.68	9.8	0.68	ug/L	U	U	
Atrazine	T	1912-24-9	1.2	9.8	1.2	ug/L	U	U	
Benzaldehyde	T	100-52-7	1.1	9.8	1.1	ug/L	U	U	
Benzo[a]anthracene	T	56-55-3	0.54	9.8	0.54	ug/L	U	U	
Benzo[a]pyrene	T	50-32-8	0.7	9.8	0.7	ug/L	U	U	
Benzo[b]fluoranthene	T	205-99-2	2.6	9.8	2.6	ug/L	U	U	

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Benzo[g,h,i]perylene	T	191-24-2	0.85	9.8	0.85	ug/L	U	U
Benzo[k]fluoranthene	T	207-08-9	1.2	9.8	1.2	ug/L	U	U
bis (2-chloroisopropyl) ether	T	108-60-1	0.77	9.8	0.77	ug/L	U	U
Bis(2-chloroethoxy)methane	T	111-91-1	0.92	9.8	0.92	ug/L	U	U
Bis(2-chloroethyl)ether	T	111-44-4	1.1	9.8	1.1	ug/L	U	U
Bis(2-ethylhexyl) phthalate	T	117-81-7	1.6	9.8	1.6	ug/L	U	U
Butyl benzyl phthalate	T	85-68-7	1.2	9.8	1.2	ug/L	U	U
Caprolactam	T	105-60-2	0.78	9.8	0.78	ug/L	U	U
Carbazole	T	86-74-8	0.7	9.8	0.7	ug/L	U	U
Chrysene	T	218-01-9	0.5	9.8	0.5	ug/L	U	U
Dibenz(a,h)anthracene	T	53-70-3	0.98	9.8	0.98	ug/L	U	U
Dibenzofuran	T	132-64-9	0.78	9.8	0.78	ug/L	U	U
Diethyl phthalate	T	84-66-2	0.86	9.8	0.86	ug/L	U	U
Dimethyl phthalate	T	131-11-3	0.97	9.8	0.97	ug/L	U	U
Di-n-butyl phthalate	T	84-74-2	0.82	9.8	0.82	ug/L	U F1	U
Di-n-octyl phthalate	T	117-84-0	1.4	9.8	1.4	ug/L	U	U
Fluoranthene	T	206-44-0	0.73	9.8	0.73	ug/L	U	U
Fluorene	T	86-73-7	0.94	9.8	0.94	ug/L	U F1	U
Hexachlorobenzene	T	118-74-1	0.78	9.8	0.78	ug/L	U	U
Hexachlorobutadiene	T	87-68-3	0.61	9.8	0.61	ug/L	U	U
Hexachlorocyclopentadiene	T	77-47-4	2.5	9.8	2.5	ug/L	U	U
Hexachloroethane	T	67-72-1	0.75	9.8	0.75	ug/L	U	U
Indeno[1,2,3-cd]pyrene	T	193-39-5	0.98	9.8	0.98	ug/L	U	U
Isophorone	T	78-59-1	0.88	9.8	0.88	ug/L	U	U
Naphthalene	T	91-20-3	0.69	9.8	0.69	ug/L	U	U
Nitrobenzene	T	98-95-3	0.72	9.8	0.72	ug/L	U	U
N-Nitrosodi-n-propylamine	T	621-64-7	0.71	9.8	0.71	ug/L	U	U
N-Nitrosodiphenylamine	T	86-30-6	0.9	9.8	0.9	ug/L	U	U
Pentachlorophenol	T	87-86-5	2	49	2	ug/L	U	U
Phenanthrene	T	85-01-8	0.76	9.8	0.76	ug/L	U	U
Phenol	T	108-95-2	0.82	9.8	0.82	ug/L	U	U
Pyrene	T	129-00-0	0.62	9.8	0.62	ug/L	U	U