

broadbentinc.com

March 21, 2022 Project No. 14-01-156

Alan Pineda, PE
Professional Engineer
Bureau of Industrial Site Cleanup
Nevada Division of Environmental Protection
375 E. Warm Springs Rd., Ste. 200
Las Vegas, NV 89119

Attn: Mr. Pineda

Re: Data Validation Summary Report, Revision 1

Reporting of Three Kids Mine Background Study Data

Three Kids Mine, Henderson, Nevada

Dear Mr. Pineda,

Broadbent & Associates, Inc. (Broadbent) is pleased to submit this *Data Validation Summary Report:* Reporting of Three Kids Mine Background Study Data, Revision 1 (Background DVSR) for the Three Kids Mine located in Henderson, Nevada. Also included with this submittal is an updated electronic data file of validated background sampling data.

Please do not hesitate to contact us if you should have any questions or require additional information.

Sincerely,

**BROADBENT & ASSOCIATES, INC.** 

Kirk Stowers, CEM Principal Geologist

cc: JD Dotchin, NDEP

James Carlton Parker, NDEP
Joe McGinley, McGinley & Associates, Inc.
Caitlin Jelle, McGinley & Associates, Inc.
Ann Verwiel, ToxStrategies
Robert Unger, Lakemoor Ventures LLC
Mindy Unger-Wadkins, Lakemoor Ventures LLC
Leo Drozdoff, Drozdoff Group LLC
Karen Gastineau, Broadbent & Associates, Inc.
Cynthia Cheatwood, EA Engineering
John Callan, Bureau of Land Management
Elizabeth Moody, Bureau of Land Management
Christene Klimek, City of Henderson

Sean Robertson, City of Henderson Stephanie Garcia-Vause, City of Henderson Anthony Molloy, City of Henderson

#### ATTACHMENT A

#### Responses to NDEP Comments made on November 18, 2021

 General Comment 1 – The Introduction (Section 1) and the Purpose and Objectives section (Section 1.1) of the DVSR only addressed soil analytical data; however, the Sample Validation Summary (Table 1) and the Qualification Tables (Tables 4 through 8) include information for the groundwater samples that were also collected and reported in Job Number L1356341.

The COC lists some samples as "OT" (other) and "W" (water). These designations account for the field equipment blanks "OT" and the profile sample "W." The text was changed to include the reference to each type of aqueous analysis.

2. **General Comment 2** – Section 1.1 of the DVSR indicated that the samples were analyzed by Pace in Las Vegas, Nevada, and Pace Sheridan. However, the data packages were all issued by Pace National, and there was no indication that the samples were ever issued to or analyzed by Pace Las Vegas.

The correct laboratory is Pace Analytical National, Mt Juliet, TN. The text was edited to reflect the correct laboratory.

- 3. General Comment 3 Section 1.1 of the DVSR indicated that Level IV data packages were received for all samples containing sample results, sample quality control (QC) summaries, instrument QC summaries, associated sample receipt information, preparation logs, and analytical raw data. A review of the provided data packages indicated that the data packages provided for the MWMP samples analyzed at Pace Sheridan were not complete Level IV data packages. The following items were provided in the Pace Sheridan data packages:
  - a. The cyanide data included the batch QC samples (i.e., method blank, LCS, MS/MSD, and/or laboratory duplicate) and one continuing calibration blank (CCB).
  - b. The mercury and metals by US EPA Method 200.7 data included the batch QC samples, the initial calibration blank (ICB), one CCB, and the second-source calibration verification.
  - c. The metals by US EPA Method 200.8 data included the batch QC samples, ICB, one CCB, and the second-source calibration verification.
  - d. The fluoride data only included the batch QC samples.
  - e. The anions by US EPA Method 300.0 data included the batch QC samples, one CCB, and the second-source calibration verification.
  - f. The nitrate-nitrite nitrogen data included the batch QC samples and one CCB.
  - g. The pH data included the batch QC samples

The laboratory was contacted to obtain the proper documents to meet the Level IV requirement.

4. General Comment 4 – Section 1.1 of the DVSR did not include reference to all analytical methods. Several SVOC samples were reported for the full list of SVOCs using SW-846 Method 8270C in full scan mode instead of polyaromatic hydrocarbons (PAHs) by SIM. In addition, several samples were reported using by SW-846 Methods 6010B or 6010D, an ICP method.

The laboratory analyzed the first set of samples using the incorrect methods. These samples were reanalyzed by the correct method, where sufficient sample volume remained. Results from the analyses by incorrect methods were not used. The exception to this are the results from analysis of equipment blanks by SVOC 8270C-SIM and Metals 6010B, where the entire sample was consumed or insufficient sample remains for re-analysis. Additionally, two soil samples were analyzed by 8270C-SIM and were not reanalyzed by 8270E-SIM. Where the full list of SVOC were analyzed, only the requested site analytes were reported. Results from 6010D were not reported as all soils were reanalyzed by the correct method. References to SDG packages not used for this analytical effort were removed from the DVSR.

5. **General Comment 5** – Section 1.1 of the DVSR indicated that the metals results, including mercury, were reported as total results; however, the laboratory reported these metals as dissolved results.

The dissolved totals are for the analysis of equipment blanks and/or aqueous profile samples. Text was added to reflect both total and dissolved metals were analyzed.

6. **General Comment 6** – The Validation Process section (Section 1.2) indicated which documents were used as guidance for performing the data validation. Many items in these guidance documents reference the criteria within the associated analytical methods, which are not the methods that were used to analyze these samples. The DVSR was not clear as to what criteria, either the normative reference or the method, was applied for usability in these instances.

The site SAP is the primary document utilized to perform the data verification and validation. Table 5 in Appendix A.2 lists the acceptance criteria. Should acceptance criteria not be addressed in the site SAP the NFGs, NDEP guidance documents, and analytical methods will be referenced for data quality acceptance criteria.

7. **General Comment 7** – Section 1.2 of the DVSR defined the elements of the Level 2B and the Level 4 data validation. Table 1 indicated that a Level IV validation was performed on one of the MWMP samples, Job Number L1359059. As previously indicated, a complete Level IV data package was not received for the MWMP samples; therefore, a

complete Level 4 data validation could not be performed. The following deficiencies were not addressed in the DVSR:

- a. Routine summary forms were not provided.
- b. Instrument-related QC data (e.g., initial calibrations, second-source calibration verifications, continuing calibrations, serial dilution results, post digestion spike recoveries, internal standard recoveries, instrument blanks, interference checks, etc.), with the exceptions indicated above, were not provided.
- c. Raw data (instrument outputs) were not provided for any analyses other than ICP and ICP/MS metals.

The laboratory was contacted to provide the required documentation for a Level IV data package. A 10% Level 4 review of the recovered documents was performed.

- 8. **General Comment 8** The Sample Receipt and Holding Times section (Section 2.1.1) did not address the following sample collection time login discrepancies:
  - a. According to the Chain-of-Custody (COC) Record, laboratory sample L1356341-11, and corresponding field identification of BG-111-18-01, was collected at 14:15 on 5/17/21; however, the laboratory logged the sample collection time as 13:15.
  - b. According to the COC Record, laboratory sample L1363396-17, and corresponding field identification of BG-111-05-01, was collected at 14:47 on 5/19/21; however, the laboratory logged the sample collection time as 14:42.

The request was submitted to the laboratory to correct the login entry error except in cases where samples were rerun and data from a particular SDG were not used (as in the case with BG-111-18-01 in SDG L1356341). There were other dates and/or times that were transposed incorrectly as well. All SDGs were reviewed to verify sample dates and times on sample result forms. These reports were regenerated by the laboratory.

9. General Comment 9 – Section 2.1.1 of the DVSR did not address that the soil samples in Job Numbers L1356341 and L1356719 were initially logged in, prepared, and analyzed using the incorrect methods. These samples were prepared and analyzed using SW-846 Method 8270C with SIM (L1356341), SW-846 Method 8270C full scan (L1356719), and SW-846 Method 6010D. The laboratory relogged these samples, with the exception of the groundwater samples and the SVOC analyses for soil samples BG-122-02-01 and BG-122-01-01 in L1356341, for the correct methods and the laboratory prepared and analyzed the samples using SW-846 Method 8270E with SIM and SW-846 Method 6020. The DVSR did not address this change in sample analytical methods; however, review of the Table 1 and the Table for Qualification Based on Field Blank Contamination (Table 5) indicated that the results from both analyses were validated. In addition, the DVSR did not indicate that the results from the separate analyses were in any way evaluated for comparability across preparation/analytical procedures.

The incorrect analytical methods were identified by client and communicated to the laboratory to rerun the soil samples using the correct analytical methods. Soil results from the incorrect analysis were not reported. The equipment blanks and source blanks were unable to be re-extracted since the entire sample was consumed or insufficient volume remained. Therefore, the results for equipment blanks and source blanks were retained and reported. Reference to SDGs not used for reporting were removed from Table 1 and 5. The DVSR was edited in incorporate the deficiency and the resulting corrective action.

- 10. **General Comment 10** Section 2.1.1 of the DVSR indicated that no data were qualified due to holding time exceedances. The Site Sampling and Analysis Plan (SAP) that was available for review at the time this memorandum was issued did not include the Holding Time Table (Appendix A.2 Table 3). The following issues were noted during evaluation of the holding time based upon the analytical methods:
  - a. Section 1.1 of the DVSR addressed the total solids data; however, these were not included on Table 1. If this method was validated, the samples were analyzed outside of the 7-day holding time included in the referenced analytical method.
  - b. The pH analyses for the MWMP samples were all performed greater than 24 hours after sample creation. The routine holding time for aqueous pH samples is 15 minutes from sample collection and a holding time of up to 24 hours is commonly applied for data usability reviews.
  - a. The total solids method referenced in Section 1.1 was performed to provide dry weight information to be used in conjunction with other analytical methods. The total solids method was not specifically requested (not included on COC) or intended to be reported for any other purpose.
  - b. The time entered is reflective of when the pH reading was entered into the report. The pH of the final effluent was recorded immediately after conclusion of the procedure and is recorded under the heading MWMP General Parameters with the same date and time as the COC.
- 11. **General Comment 11** The Calibration section (Section 2.1.2) of the DVSR indicated that no data were qualified based on calibration nonconformances; however, based upon a review of the data packages provided, the low-level calibration standards in Job Numbers L1356341, L1363396, and L1356719 did not meet the acceptance criteria in the referenced National Functional Guidelines (NFGs; EPA. 2020A. National Functional Guidelines for Inorganic Superfund Methods Data Review. EPA 542-R-20-006 and EPA. 2020B. National Functional Guidelines for Organic Superfund Methods Data Review. EPA 540-R-20-005) for several metals. Any sample results associated with these calibration standards should have been qualified.

The site SAP is the primary document used for the data validation review. The SAP lists method specific DQI/MQO in Appendix A.2 Table 5. This table covers routine laboratory QC and does not specifically call out all method QC measurements such as the low-level standard check. The NFG for inorganics does not specifically address criteria for low-level standard checks. However, in method 6020B (10.5.2), there is a requirement to analyze a mid-level and low-level standard. The criteria listed in 6020B for low-level standard recovery is 80-120%. Therefore, the qualification of data will be re-evaluated based upon the criteria in 6020. Metals results for soils from SDG L1356341 and L1356719 are not reported. References to these SDGs were removed from Tables where applicable.

12. **General Comment 12** – The Field Quality Control Blanks section (Section 2.1.3.2) of the DVSR indicated that one source water blank and four equipment blanks were collected and analyzed in association with these samples. These blanks were not clearly identified in the COC Records or the DVSR.

A tracking spreadsheet was developed to record the equipment blanks, source blank, and the samples associated with each equipment blank separately from this DVSR. Table 1 has been updated to identify the equipment blanks, source blank, and associated samples.

13. General Comment 13 – The Qualifications Due to Blank Contamination section (Section 2.1.3.3) of the DVSR indicated that the NDEP guidance for qualification due to blank contamination (NDEP; 2018. Letter from NDEP to All BMI Companies regarding "Data Validation Guidance." July 13) was followed. This guidance directs the data validator to qualify all sample results with a positive detection in the associated blank as potentially biased high, without a multiplicative factor associated to limit when qualification would be applied. As outlined in the NDEP guidance, Section 2.1.3 indicated that only those samples associated with positive contamination in blanks that were reported as "not-detected" were not qualified; however, the Qualification Based on Laboratory Blank Contamination table (Table 4) did not include all positive sample results associated with contaminated blanks. In addition, manganese results were qualified for CCB contamination in two batches of samples in Job Number L1367629; however, the manganese results in these samples were reported from analyses in different analytical sequences where manganese was not detected in the CCBs.

The qualifications due to blank contamination were re-evaluated based upon the data validation guidance referenced. The Tables were edited to remove reference to manganese not reported as the final result.

14. **General Comment 14** – The Spike Samples section (Section 2.1.4) and the Duplicate Samples section (Section 2.1.7) of the DVSR addressed qualification based upon an out-of-criteria MS/MSD, both recoveries and precision, and the qualified samples were identified on the Qualification Based on Matrix Spike Recovery table (Table 6) and the Qualification Based on Laboratory Duplicate Precision (Table 7). Samples in several Job Numbers were not qualified for select out-of-criteria MS/MSD results; however,

Sections 2.1.4 and 2.1.7 did not include information on what circumstances would lead to results not being qualified.

Text added to further define the reasons for not qualifying data based upon out-of-criteria MS/MSD/LCS/LCSD recoveries and precision. The site samples have high metals content resulting from mining operations. Metals such as manganese and lead are found in the ore body. These high concentrations of metals mask the spike level in MS/MSD. Additionally, complete homogenization is difficult to obtain due to varying grain sizes and potential for presence of manganese ore bodies that cannot be split equally. This makes split sampling nearly impossible if an ore body is captured in one sample but not the other. Therefore RPD values are affected by the presence of elevated metals due to proximity to ore body.

Sample duplicates were not performed for any analysis of soil samples or equipment blanks or source blank. Duplicate analyses were performed for the MWMP profile samples. Only MSD or LCSD were performed.

15. **General Comment 15** – The Other Qualification section (Section 2.1.8) of the DVSR addresses all QC items that are not addressed in previous sections of the DVSR. This section does not detail which additional QC items were reviewed; therefore, it is unclear if interference check samples were reviewed.

The section was added to include serial dilutions, post digestion spikes, and interference checks. No qualifiers were applied based upon serial dilutions and post spikes performed on non-client samples.

16. **General Comment 16** – The Qualification Based on Serial Dilution Percent Difference table (Table 8) indicated that an acceptance criterion of ≤ 10% difference was used to evaluate the serial dilutions; however, the referenced NFGs use a criterion of ≤ 20% difference. Based upon the criterion outlined in the NFGs, the qualification for all sample results, except the lead and zinc sample results for Job Number L1367629, are not applicable. In addition, the serial dilution associated with the qualification of the sample results for Job Number L1367629 was performed on a non-client sample and, therefore, should not have been applied to these background samples.

The SAP Table 5 does not address the control limit for serial dilutions. Method 6020 (Dilution Test) has a guidance level of <=20%. This matches the criterion in the NFG. Qualification based upon serial dilution analysis was re-evaluated. All serial dilutions for client samples were less than 20%. No qualification based upon serial dilutions is necessary. The text of the DVSR was edited and Table 8 was eliminated as there are no qualifications based upon serial dilutions. Additionally, the EDD with qualifiers and reason codes were corrected. The qualifiers were removed if this was the only reason code. The other qualifiers were altered to reflect the remaining data qualifier reason codes.

17. **General Comment 17** – The Evaluation of PARCCS Parameters section (Section 2.2) indicated that the PARCCS (precision, accuracy, representativeness, completeness, comparability, and sensitivity) parameters were reviewed for the groundwater

monitoring samples that were collected. Section 2.2 is where the first discussion of the groundwater samples occurs in the DVSR; however, review of the subsections within Section 2.2 indicated that it was the background soil samples that were actually evaluated.

The "groundwater monitoring" is a relic from another DVSR. Groundwater monitoring was removed and replaced with Phase II sampling event, which includes soil samples and collection of Field QC blanks.

18. **General Comment 18** – The Conclusions and Recommendations section (Section 3) of the DVSR was mostly accurate but inconsistent. This section indicated that > 90% of the data were valid (not rejected); however, previous sections indicated that none of the data were rejected, indicating that 100% of the sample results were valid. In addition, the statements on the usage of the biased data provide examples of what might have led to high or low biased qualification; however, they are presented as actual reasons for qualification within the report.

The text of the DVSR was edited to better describe the actual completeness value, and the language regarding usage of biased data was changed.

#### **Essential Corrections**

1. **Essential Correction 1** – Table 1 should include the matrix of the sample and identification of all field QC samples.

The sample matrix and purpose were added to Table 1. SDG packages not used for reporting were removed.

2. **Essential Correction 2** – The sample results should be summarized on a single table per Job Number with all qualification for each sample addressed. If results are only reviewed in the database, this change is not necessary.

An Excel spreadsheet was generated for this data set that summarizes the sample results with all qualifications and reason codes. Additionally, sample results and qualifications are included in Table 1 of the Background Soil Report, Revision 2.

3. Essential Correction 3 – Complete data packages are required for 2B or 4 review; however, the Pace Sheridan data packages were not complete. It is recommended that Broadbent, or its designee, discuss with its corporate Pace representative or primary Pace contact the level of reporting (Level IV) required to meet Level 2B and 4 validation. Alternatively, it is recommended that either MWMP work be sent to a laboratory that can provide a comprehensive Level IV data package, including Contract Laboratory Program- (CLP-) like summary forms and all raw data, or have the MWMP leachate created at one laboratory and then the leachate shipped to a separate laboratory for analysis.

The laboratory was contacted to provide the Level 4 data package as required per contract. The laboratory is NDEP certified to perform the MWMP analyses. The incomplete SDG package was an oversight.

4. **Essential Correction 4** – The sample qualification for blank contamination appeared to use a multiplicative qualification value, as opposed to the NDEP guidance to qualify all results for any blank detection. The application of a multiplicative qualification value is reasonable; however, the basis and multiplication factor used for this deviation should be discussed in the DVSR.

A discussion regarding the use of qualifications based upon the NDEP guidance is included in the DVSR text.

5. **Essential Correction 5** – It was not apparent from the DVSR if a secondary review was performed for the data validation. All data validation should undergo a second, documented, peer review to ensure qualification is not missing or misapplied and that all electronic data deliverable (EDD) updates are accurate.

A secondary review of the data validation has since been performed. The DVSR text has been revised to state the performance.

6. **Essential Correction 6** – The DVSR did not include any supporting documentation for the qualified sample results. It is recommended that, at a minimum, the pages reviewed that led to qualification be provided with the DVSR and preferably, all pages associated with nonconforming results that might have led to qualification be included with notations for why qualification was or was not necessary.

The SDG packages are attached for reference.

7. **Essential Correction 7** – The NDEP data validation guidance and the referenced NFGs differ on the application of qualification based on out-of-criteria MS/MSD results. It is recommended that the DVSR discuss which referenced guidance was used when qualification is applicable based on MS/MSD and PDS results.

The order of qualifications is as follows: SAP Table 5, NFG, then NDEP or method.

8. **Essential Correction 8** – Non-Client/Job Number matrix-specific QC samples should not be used to qualify client samples. In addition, unless information is available to know that the sample matrix is sufficiently similar, it is recommended that QC only be applied to the source sample and, if applicable, the field duplicate.

References to qualifications based upon non-client samples have been removed. Qualifications based upon matrix spike outliers were changed to only apply to the parent sample.

# Data Validation Summary Report Reporting of Three Kids Mine Background Study Data

Three Kids Mine Henderson, Nevada

#### Submitted to:

Broadbent & Associates, Inc. 8 Pacific Ave, Henderson, NV 89015 702-563-0600

#### Submitted by:

Neptune & Company, Inc. 1435 Garrison St. Suite 201 Lakewood, CO 80215

August 2021 (Revision 1 March 2022)

## **Table of Contents**

1.	Intro	duction	1	1
	1.1	Purpo	se and Objectives	1
	1.2	Valida	ation Process	3
	1.3	Repor	t Organization	4
2.	Data	Validat	ion Summary	6
	2.1	Data V	Validation Findings	6
		2.1.1	Sample Receipt and Holding Times	6
		2.1.2	Calibration	7
			2.1.2.1 Initial and Continuing Calibration Verification	7
		2.1.3	Blank Samples	8
			2.1.3.1 Laboratory Blanks	8
			2.1.3.2 Field Quality Control Blanks	9
			2.1.3.3 Qualifications Due to Blank Contamination	9
		2.1.4	Spike Samples	10
		2.1.5	Surrogate Spikes	11
		2.1.6	Internal Standards	11
		2.1.7	Duplicate Samples	11
		2.1.8	Other Qualifications	12
		2.1.9	Summary of Rejected Data	12
	2.2	Evalua	ation of PARCCS Parameters	13
		2.2.1	Precision	13
		2.2.2	Accuracy	13
		2.2.3	Representativeness	14
		2.2.4	Completeness	14
		2.2.5	Comparability	14
		2.2.6	Sensitivity	15
3.	Cond	lusions	s and Recommendations	16
4.	Refe	rences		17

### **Tables**

- 1. Sample Validation Summary
- 2. Data Validation Criteria for Levels 2B and 4
- 3. Data Validation Qualifiers and Comment Codes
- 4. Qualification Based on Laboratory Blank Contamination
- 5. Qualification Based on Field Blank Contamination
- 6. Qualification Based on Spiked Sample Recovery

7. Qualification Based on Duplicate Precision

## **Appendices**

- A. Analytical Data—Three Kids Mine Background Study (contained on compact disk)
- B. Analytical Data Packages—Pace Laboratories (contained on compact disk)

## **Acronyms and Abbreviations**

APHA American Public Health Association

ASTM American Society for Testing and Materials

CCB Continuing calibration blank

CCV Continuing calibration verification

CFR Code of Federal Regulations

COC Chain of custody

CVAA Cold vapor atomic absorption

DQI Data quality indicator DQO Data quality objective

DVSR Data validation summary report

EB Equipment rinsate blank
EDD Electronic data deliverable

EPA U.S. Environmental Protection Agency GC/MS Gas chromatography/mass spectrometer

ICB Initial calibration blank
ICP Inductively coupled plasma

ICP/MS Inductively coupled plasma/mass spectrometer

LCS Laboratory control sample

LCSD Laboratory control sample duplicate

MD Matrix duplicate

MDL Method detection limit

MS Matrix spike

MSD Matrix spike duplicate

MWMP Meteoric water mobility procedure

NDEP Nevada Division of Environmental Protection

Pace Pace Laboratories, Inc.

PARCCS Precision, accuracy, representativeness, completeness, comparability, and

sensitivity

PQL Practical quantitation limit

QA Quality assurance QC Quality control

RPD Relative percent difference SAP Sampling and analysis plan

SB Source water blank
SDG Sample delivery group
SIM Selective ion monitoring

Site Three Kids Mine Site, Henderson, Nevada

SM Standard Methods

SQL Sample quantitation limit

SVOC Semivolatile organic compound

## 1. Introduction

This data validation summary report (DVSR) was prepared to assess the validity of background soil, quality control (QC) blanks, and soil profile analytical data collected in association with the former Three Kids Mine site in Henderson, Nevada (Site). The sampling event is based on the Phase II Sampling and Analysis Plan, Revision 2, Former Three Kids Mine (SAP), dated November 3, 2021 (Broadbent, 2021). The data summarized herein include those obtained from the Background Study to be used for comparison to the site.

Table 1 lists samples collected during the background sampling event and summarized in this DVSR.

## 1.1 Purpose and Objectives

The purpose of this DVSR is to summarize the validation for chemical soil data collected during this event. The purpose of the background study is to (1) characterize the distribution of inorganic and organic chemicals of site-related chemicals in soils outside the defined Site area; and (2) set a baseline background concentration of site-related chemicals for statistical comparison against samples collected on the Site.

Analyses for soil samples collected during this event were conducted by Pace Analytical National in Mt Juliet, Tennessee and Pace Analytical in Sheridan, Wyoming. All data were delivered as data packages in sample delivery groups (SDG) and accompanied by electronic data deliverables (EDD). For this event, all data packages were Level IV containing sample results, sample quality control (QC) summaries, instrument QC summaries, associated sample receipt information, preparation logs, and analytical raw data. EDDs received from the laboratory were in Excel-compatible format and used for reporting. Sample results were reported along with applicable laboratory qualifiers.

The following types of analyses were conducted on samples collected during this reporting period and analyzed by Pace Analytical National:

- Semivolatile organic compounds (SVOC) by gas chromatography/mass spectrometry (GC/MS) in selective ion monitoring (SIM) mode by U.S. Environmental Protection Agency (EPA) Method 8270C SIM and 8270E SIM SW-846. (EPA 1996 and 2018).
- Total and Dissolved metals by inductively coupled plasma/mass spectrometer (ICP/MS) by EPA Method 6020 SW-846 (EPA 1994B).

- Dissolved metals by inductively coupled plasma-atomic emission spectrometry (ICP) by EPA Method 6010B SW-846 (EPA 1996).
- Physical parameters including total solids by gravimetric determination by Standard Method (SM) 2540G-2011 (APHA 2011).

The following types of analyses were conducted on samples collected during this reporting period and analyzed by Pace-Wyoming:

- Standard Test Method for Column Percolation Extraction of Mine Rock by the Meteoric Water Mobility Procedure (MWMP) by ASTM Method E2242-13 (ASTM 2013).
- Total and Dissolved metals by ICP by EPA Method 200.7 (EPA 1994A).
- Total and Dissolved metals by ICP/MS by EPA Method 200.8 (EPA 1994C).
- Total and Dissolved mercury by cold vapor atomic absorption (CVAA) by EPA Method 245.1 (EPA 1994D).
- Anions by ion chromatography by EPA Method 300.0 (EPA 1993).
- General chemistries, including Nitrate/Nitrite-Nitrogen (EPA Method 353.2),
- Cation/Anion Balance (SM 1030E), Alkalinity (SM 2320B), Total Dissolved Solids (SM 2540), pH (SM 4500-H B), Fluoride (SM 4500-F C), and Cyanide (SM 4500-CN I).

Quantitation limits are critical to the proper evaluation of method sensitivity and non-detect data. Three types of quantitation limits were evaluated as follows:

• Method detection limit (MDL) – This limit was established by the laboratories according to the requirement in 40 CFR 136, Appendix B, and represents the minimum concentration of a substance that can be measured and reported with 99 percent confidence that the analyte concentration is greater than zero. MDLs are established using matrices with little or no interfering species using reagent matrices and are considered the lowest possible reporting limit. Often, the MDL is represented as the instrument detection limit. Because these limits do not reflect sample-specific characteristics and preparation volumes/masses, true MDLs reported in the laboratory EDD are those that have been corrected for sample preparation, dilution, and moisture content factors (see sample quantitation limit below).

- Sample quantitation limit (SQL) The SQL is defined as the MDL adjusted to
  reflect sample-specific actions, such as dilution or use of smaller aliquot sizes,
  and takes into account sample characteristics, sample preparation, and analytical
  adjustments. The SQL represents the sample-specific detection limit and all nondetected results are reported to this level.
- Practical quantitation limit (PQL) This limit is defined as the lowest level at which the entire analytical system gives a recognizable signal and acceptable calibration point for the analyte, and includes the predicted effect of sample matrices with typical interfering species. The PQL is the lowest concentration of an analyte that can be reliably measured within specified limits of precision and accuracy during routine laboratory operating conditions. PQLs are used to estimate or evaluate the minimum concentration at which the laboratory can be expected to reliably measure a specific chemical contaminant during day-to-day analyses of different sample matrices. Detected results greater than the SQL, but less than the PQL, were qualified by the laboratory as estimated. Further qualification based on this scenario is discussed in Section 2.1.8.

Laboratories consistently reported PQLs and SQLs in the EDDs for analytes that have calculated MDLs. Note that parameters reported for the MWMP test by ASTM Method E2242-13 do not have statistically-derived MDLs; and therefore have no MDLs, SQLs, or PQLs reported in the EDD. In addition, MDLs, SQLs, and PQLs were not reported for the surrogate compounds reported by EPA Method 8270 SIM.

### 1.2 Validation Process

There were two levels of validation conducted on the analytical data collected during this investigation as outlined in the Phase II Sampling and Analysis Plan (SAP), Revision 2 (Broadbent, 2021). The summary of the Data Objective Indicators (DQI) and Measurement Quality Objectives (MQO) can be found in Table 6.2 of the SAP. The method specific DQI/MQO are listed in Table 5 of Appendix A.2 of the SAP. Additional guidance for data verification and validation may be obtained from EPA's National Functional Guidelines for Data Review (Inorganic and Organic) (EPA 2020A and 2020B) and in conjunction with current NDEP data validation guidance (NDEP 2018), and analytical methods, where DQI/MQO are not clearly defined in the SAP:

 Level 2B – Verification and validation based on completeness and compliance checks of sample receipt conditions and <u>BOTH sample-related and instrument-related QC results.</u>

• Level 4 - A verification and validation based on completeness and compliance checks of sample receipt conditions, both sample-related and instrument-related QC results, recalculation checks, AND the review of actual instrument outputs.

According to the Site SAP, at least 10 percent of all samples received Level 4 validation; while the remaining 90 percent were validated to Level 2B. Table 1 summarizes samples included in this DVSR and indicates the level of data validation conducted on the results from each sampling location. Table 2 identifies the QC parameters evaluated for each validation level.

Based on data validation and review, data qualifiers were placed in the EDD to signify whether the data were acceptable, acceptable with qualification, or rejected. In addition, for every data validation qualifier, a secondary comment code was entered to indicate the primary reason for qualification. Table 3 provides the definitions for the data validation qualifiers and comment codes used in the EDD. Validation qualifiers and definitions are based on those used by EPA in the current validation guidelines (EPA 2020A and 2020B). Comment codes provide the reason for qualification of data. Results that have any validation qualifier have corresponding reason codes. Laboratory qualifiers are defined in the data packages and are superseded by the data validation qualifiers and accompanying reason codes.

Validated results and comments are contained in the EDD and provided in electronic format as Appendix A to this document.

The data validation hierarchy of qualifiers is as follows:

$$R > J/U/UJ > J+/J-$$

Where: Rejected qualifiers (R) supersede all other qualifiers; Biases (+/-) are superseded by qualifiers without bias; and Conflicting or uncertain biases are also superseded by qualifiers without bias

This data review was peer reviewed by a second data validator to confirm data qualifiers.

## 1.3 Report Organization

After this introductory section, Section 2 summarizes data validation for data collected during this event. Section 3 provides general conclusions about the usability of the data set; however, it does not address a full data usability report that evaluates the data against the overall project data quality objectives (DQO).

References and tables follow the conclusions and recommendations at the end of this document.

## 2. Data Validation Summary

This section describes the data validation findings with regard to the project-data quality indicators (DQI). Section 2.1 summarizes the data validation findings, and Section 2.2 summarizes the evaluation of the following DQI parameters: precision, accuracy, representativeness, completeness, comparability, and sensitivity (PARCCS).

## 2.1 Data Validation Findings

This section summarizes items of the validation process and discusses how these findings affect data quality.

## 2.1.1 Sample Receipt and Holding Times

The condition of the samples upon receipt and holding times are evaluated initially by the laboratory, and then assessed by the reviewer.

Holding time refers to the period of time between sample collection and the preparation and/or analysis of the sample. Sample results were reviewed for compliance with the holding times set forth in Appendix A.2 Table 3 of the approved Site SAP (Broadbent 2021). Note that the expectation for the MWMP test as a whole is for all tests to be completed within 6 months of collection. While individual tests for pH, anions, and others on the MWMP Profile I list typically have shorter holding times less than 6 months, the hold time clock starts upon completion of the fluid extraction (leachate) from the MWMP method. The pH results reported in section MWMP General Parameters were collected immediately upon completion of the final effluent. The pH results reported in section General Parameters is a required measurement (starting point pH) for Total Alkalinity as CaCO3 method.

Several soil, equipment blank, and source blank samples were originally logged in by the laboratory with incorrect analytical methods. Upon communication with the client, the laboratory re-logged samples and assigned the correct analytical methods. All soil analyses by method 6010D were re-logged and re-analyzed by method 6020. Equipment blanks and source blank analyzed by 6010B could not be reanalyzed due to insufficient sample volume remaining. Likewise, two soils, an equipment blank and a source blank analyzed for SVOCs by 8270C-SIM could not be reanalyzed by method 8270E-SIM due to insufficient sample volume remaining. The results from analysis by 6010D are not used and not reported. Results from analysis by 6010B and 8270C-SIM are retained and reported. The lab reported the full SVOC list for some samples, however, only the site specific analytes listed in the SAP are reported.

**Metals:** No data were qualified or rejected due to holding time exceedances.

**SVOCs:** No data were qualified or rejected due to holding time exceedances.

General Chemistries: No data were qualified or rejected due to holding time exceedances.

The Total Dissolved Solids analysis was performed 5 days after generation of leachate sample, which is within the 7 day maximum hold time. The pH listed under the Sample Summary heading "General Parameters" was performed prior to the analysis of Total Alkalinity to obtain a starting point pH. The pH listed under the Sample Summary heading "MWMP General Parameters" was performed immediately after generation of the MWMP leachate. No hold times were exceeded. No qualifiers were applied.

#### 2.1.2 Calibration

Instrument calibration data were included in the laboratory data packages, but not the EDDs (typical of the industry). Review included the instrument setup, operating conditions, initial calibration verifications, and continuing calibration verifications (CCV).

No SVOC or metals data were qualified due to initial calibration non-conformances.

#### 2.1.2.1 Initial and Continuing Calibration Verification

The CCV recovery for manganese were above control limit in SDG L1367629. Results for manganese were not reported for samples bracketed by the CCV outliers. The associated samples were reanalyzed with CCV recoveries for manganese within control limits. No qualifiers were applied for manganese results.

In addition to the CCV, Method 6010 and 6020 require an analysis of a low-level calibration verification (ICVLL or CCVLL). The ICVLL recovery was outside control limits (80-120%) for antimony (high) in SDG L1363384, and for antimony (high), copper (low) and selenium (low) in SDG L1363396. There are 60 total results qualified for the ICVLL outliers. Results for antimony were not qualified for non-detects as a result of high recovery in the ICVLL. The CCVLL recovery was outside control limits for arsenic for method 6010B in SDG L1356341 (equipment blanks, source blank). There are 3 total results qualified for the CCVLL outlier.

The ICV recovery for SVOC surrogates 2-fluorobiphenyl and p-terphenyl-d<sub>14</sub> and the CCV recovery for surrogate nitrobenzene-d<sub>5</sub> were above QC limits. This indicates a high bias for surrogate recoveries in samples. All surrogate recoveries were within control limits in samples except for two p-terphenyl-d<sub>14</sub> recoveries in two samples. The surrogate recoveries in those samples were higher than the recoveries in the ICV and therefore indicate a true

exceedance of the upper control limit. The lowest recoveries of all surrogates are significantly higher (>80%) than their respective lower control limits by an order of magnitude between 2-6 and therefore do not indicate the potential for a biased recovery at the lower control limit (false pass). It is the validator's judgment not to qualify data based upon the high recovery of surrogates in the ICV and CCV. The high recovery of surrogate in samples is discussed later in this document.

## 2.1.3 Blank Samples

Field and laboratory blanks consisting of contaminant-free water were prepared and analyzed as part of standard quality assurance/quality control (QA/QC) procedures to monitor for potential contamination of field equipment, laboratory process reagents, and sample containers. For this program, two types of blanks were prepared and analyzed: (1) laboratory blanks, and (2) field QC blanks. Each blank type is discussed in the following sections (Sections 2.1.3.1 and 2.1.3.2). The assignment of validation qualifiers associated with blank contamination is discussed in Section 2.1.3.3.

#### 2.1.3.1 Laboratory Blanks

Two types of laboratory blanks were prepared and analyzed: calibration blanks and method blanks. Both types were prepared in the laboratory using high-grade, contaminant-free water.

Calibration Blanks (for metals only) – Calibration blanks are acidified high-grade contaminant-free water analyzed at the beginning (initial calibration blank [ICB]), end (continuing calibration blank [CCB]), and every 10 runs during analysis of metals by ICP, ICP/MS, and CVAA. Their primary function is to initially set the calibration curve (along with calibration standards) and continually monitor the background for possible variations in instrument electronic signal or cross-contamination. ICB and CCB data are included in the laboratory data packages, but not the EDDs.

Method Blanks – Method blanks are laboratory QC samples that are prepared and analyzed with each batch of environmental samples. Method blanks are high-grade contaminant-free water that is carried though all preparation procedures in batches with field samples (including the addition of all reagents and QC monitoring compounds). Method blanks monitor potential contaminants in laboratory processes, reagents, and containers, and were analyzed for each analytical method used on field samples.

The following analytes were detected in one or more laboratory blanks and qualified in at least one sample result in the EDD; qualified data are discussed in Section 2.1.3.3:

- Metals-soils The ICB and CCB for 6020 analyses contained one or more of antimony, arsenic, lead, manganese and zinc above reporting limits. The method blank contained zinc, lead, and manganese. Where associated sample positive results are greater than 10 times the blank value, when comparing raw data, no qualifiers were applied. Where associated positive results are less than 10 times the blank value, when comparing raw data, qualifiers were applied. All affected samples contained positive results.
- Metals-aqueous The CCB contained lead above the reporting limit for 6010B analysis (equipment blanks, source blank). The method blank contained zinc.
   Qualifiers were applied to all positive results. Where the laboratory assigned a U qualifier, the result is qualified as estimated (UJ) at the MDL.
- PAHs-soils Benzo(b)fluoranthene, indeno(1,2,3-cd)pyrene, phenanthrene, and pyrene. All affected sample contained positive results.
- PAHs-aqueous No analytes were detected above the MDL.

Table 4 provides a summary of results qualified due to laboratory blank contamination.

#### 2.1.3.2 Field Quality Control Blanks

Two types of field QC blanks were collected for this event: source water blanks (SB); and equipment rinsate blanks (EB). One SB and four EBs were collected for this event.

SBs are "clean" decontamination water used during the sampling event and acts as the baseline for evaluating EBs. When an analyte is detected in the SB, all detections of that analyte in EBs are qualified and coded in the EDD as described in Section 2.1.3.3 below.

EBs are decontamination water (source water) passed over the field sampling equipment at a given location after decontamination of that equipment. The sample collected immediately previous to and the sample collected immediately after the EB are associated with that blank for evaluation. Analytes detected in the EBs are evaluated against the same analytes in the associated samples. If the same analyte is detected in both the EB and its associated sample(s), that analyte is qualified in associated sample(s) as described in Section 2.1.3.3.

Table 5 shows the analytes detected in each EB blank for comparison with the SB.

#### 2.1.3.3 Qualifications Due to Blank Contamination

According to the revised NDEP guidance for qualifying data based on blank contamination (NDEP 2018), no data are censored when associated blanks contain measurable analytes. If

associated blanks contain measurable analytes, the associated sample data are merely qualified as estimated (J) and assigned the comment code ("b" for laboratory blank contamination and "f" for field blank contamination) in the EDD. Tables 4 and 5 present data that were qualified as estimated (J) based on laboratory and field blank contamination, respectively. Bias (+ or -) for estimated samples was assigned based on the blank value reported by the laboratory. If the blank value was positive, then associated positive sample results may be biased high. If the blank value was negative (only reported for metals), then associated sample results may be biased low.

Note that not every compound detected in laboratory and field blanks resulted in qualification of sample results, because associated results may already be reported as non-detects from the laboratory.

## 2.1.4 Spike Samples

Spiked samples are environmental matrices spiked with a subset of target compounds at known concentrations. These QC samples were analyzed with project samples to measure laboratory accuracy and potential interference from the matrix. Two types of spike samples were analyzed with the project samples to monitor for potential interferences during analysis:

- Matrix spike (MS) and matrix spike duplicate (MSD) samples; these samples
  consist of aliquots of environmental samples spiked with a subset of target
  compounds. MS/MSD samples monitor potential interference from the sites
  specific sample matrix and its effect on target compounds.
- Blank spike samples, also known as laboratory control samples (LCS); these samples are an aliquot of reagent soil or water spiked with a subset of target compounds. The LCS monitors laboratory accuracy without the bias of a sample matrix.

The reviewer evaluated both the spike and duplicate recoveries of the MS pairs and LCS. Data were qualified if either recovery in the pair failed to meet criteria. Data that were qualified based on MS/MSD and/or LCS recoveries that were outside the QC limits are presented in Table 6. Analytical bias was determined, and qualified data were assigned bias codes (- or +) as necessary. Qualified results were assigned the validation comment code "e" in the EDD. Note that no data were qualified based on LCS recoveries.

The laboratory on occasion selected a non-client sample to perform the MS/MSD analysis. The results are not qualified based upon another client's sample. Only MS/MSD results from this client's sample set are evaluated for precision.

No qualifiers are applied to samples where the native concentration is greater than four times the spike level.

The soil samples contain high metals content that make homogenization of samples difficult to achieve, which directly impacts precision. Additionally, the metals content for mined metals is high and often mask the spike concentration added to samples, which directly impacts accuracy. These are the main reasons for MS/MSD to fail criteria. Given the variability of sample composition, qualifiers are only applied to the parent sample.

## 2.1.5 Surrogate Spikes

Surrogate spikes were prepared by adding compounds similar to target compounds of interest to sample aliquots and associated QC samples for organic analyses only. Surrogate spike recoveries monitor the efficiency of contaminant extraction from the sample medium into the instrument measuring system and measure possible interferences from the sample matrix that may affect the data quality of target compound results.

Surrogate spikes were added to each of the samples submitted for SVOC analysis to monitor potential interferences from the matrix. Surrogates were added to the sample aliquot during preparation of the sample for analysis, and surrogate recoveries were compared with QC acceptance limits. Surrogate recoveries outside of the acceptable limits indicate interference from the sample matrix for the detection of target compounds. There were two surrogates above the control limits for SVOCE-SIM analysis. The surrogates are not associated with the target analytes for this project. The surrogate is an early eluter and the target analytes elute after the second surrogate which was in control. No data were qualified based upon the early eluting surrogate. As such, no data were qualified or rejected based on surrogate recoveries.

#### 2.1.6 Internal Standards

Internal standards were used for quantitation of SVOCs and ICP/MS by adding compounds similar to target compounds of interest to sample aliquots. Internal standards are used in the quantitation of target compounds in the sample or sample extract. Internal standard responses were presented in full data packages received from the laboratory. All internal standard criteria were met; no data were qualified or rejected based on these criteria.

## 2.1.7 Duplicate Samples

Duplicate samples involved the preparation and analysis of an additional aliquot of a field sample. Results from duplicate sample analysis measure laboratory precision as well as homogeneity of contaminants in the field matrix. Spiked duplicates such as MS/MSD pairs

and/or LCSDs for organic analyses and metals, and matrix duplicates (MD) for inorganic analyses (anions) were used to evaluate laboratory precision and provide insight into sample matrix homogeneity.

At least one duplicate analysis was performed with each batch of field samples processed in the laboratory. The laboratory calculated the relative percent difference (RPD) between the two detected values for duplicate analyses. RPD values within the acceptable limits indicate both laboratory precision and minimal matrix heterogeneity of compounds detected in the samples. Results associated with elevated RPD values were qualified as estimated to indicate the variability in detected concentrations or poor laboratory precision.

Table 7 lists sample results qualified based on duplicate precision.

#### 2.1.8 Other Qualifications

This section is reserved to address qualification scenarios and comment codes that were not addressed in main sections above. For this event, sample results reported greater than the SQL, but less than the PQL were reported and qualified as discussed below.

Quantitation less than the practical quantitation limit for stable chemistries — The laboratory evaluated the SQL and PQL for each sample result. In cases where sample results were greater than the SQL, but less than the PQL, the laboratory qualified the results as estimated. Specifically, results with this scenario were qualified by the laboratory as "J". During data validation, positive results less than the PQL but greater than or equal to the SQL were qualified as estimated (J). Qualitatively, the results are acceptable; however, these results were considered estimated, because as the value approaches the SQL, the accuracy of the measurement is less certain. In these cases, bias cannot be determined. All results qualified as estimated (J) for this reason were assigned the validation comment code "j" in the EDD.

There are additional QC elements included for metals analysis including serial dilutions, post spikes, and interference checks. All serial dilutions are within the control limit of <=20%. All interference checks for target analytes were within acceptable limits. The post spike recovery for arsenic in SDG L1367629 was outside control limits due to the large amount of arsenic in the sample. No qualification of data is applied for this outlier.

## 2.1.9 Summary of Rejected Data

For this sampling event, no data were qualified as rejected.

### 2.2 Evaluation of PARCCS Parameters

Overall data quality was acceptable based on the critical indicator parameters. PARCCS parameters were reviewed for laboratory analytical results obtained during the Phase II sampling event conducted during this reporting period. The sections below discuss the results of the evaluation for each indicated parameter.

#### 2.2.1 Precision

Precision is the measure of the variability associated with an entire sampling and analysis process. It is the comparison among independent measurements as the result of repeated application of the same process under similar conditions. It is determined by analyzing spike sample pairs (MS/MSD and LCS/LCSD), and MD pairs. Precision is expressed as the RPD of a pair of values (or results).

The frequency criterion for MS/MSD or MD pairs is 5 percent of the samples collected (per matrix) or one per each analytical batch of 20 or less per matrix. MS/MSD or MD samples were collected, analyzed, and evaluated for each analysis performed on every sample matrix. The frequencies in which MS/MSDs or MDs were prepared and analyzed by the laboratory met the frequency requirement as stated above. Table 7 provides a list of results qualified for duplicate precision. Data qualified for duplicate precision may indicate the heterogeneity of the sample matrix. No data were rejected based on laboratory duplicate precision.

## 2.2.2 Accuracy

Accuracy is the degree to which a measurement agrees with its true value and is expressed as percent recovery. Accuracy is assessed by evaluating instrument calibrations and comparing MS, MSD, LCS, LCSD, and surrogate recoveries with associated QC limits.

Instrument calibrations were evaluated against acceptable QC limits and found acceptable.

The frequency criterion for MS/MSD and LCS/LCSD is 5 percent of the samples per matrix or one per each analytical batch of 20 samples or less per matrix. MS/MSD and LCS/LCSD samples were prepared, analyzed, and evaluated for each analysis performed at the proper frequency. The criteria for MS/MSD and LCS/LCSD accuracy are based on SAP requirements.

Positive or negative signs were assigned to qualifiers to indicate the expected bias, as indicated by the QC result. In some cases, the biases were in conflict and the hierarchy of validation was used. Table 6 provides a list of results qualified for spike sample accuracy.

Surrogate recoveries were acceptable. No data were rejected based on these QC nonconformances.

In addition, matrix interferences that may result in inaccurate results were identified from serial dilutions conducted during ICP analysis. No results were qualified based on serial dilution results.

#### 2.2.3 Representativeness

Representativeness is a qualitative parameter and is defined by the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or a process or environmental condition. Sample results were evaluated for representativeness by examining items related to sample collection, including COC documentation, sample labeling, collection dates, and condition of the samples upon receipt at the laboratory. Laboratory procedures also were examined, including anomalies reported by the laboratory, either upon receipt of the samples at the laboratory or during analytical processes, adherence to recommended holding times of samples prior to analysis, calibration of laboratory instruments, adherence to analytical methods, and completeness of data package documentation.

## 2.2.4 Completeness

Completeness is defined as the percentage of measurements judged to be valid. The validity of sample results is determined through the data validation process. All rejected sample results are considered to be incomplete. Data that are qualified as undetected (U), undetected at estimated reporting limits (UJ), and estimated (J) are considered to be valid. The number of valid results divided by the number of possible individual analyte results, expressed as a percentage, determines the completeness of the data set. No data were rejected, so validation completeness is 100%. Project completeness will be addressed in the data usability study at a later date.

## 2.2.5 Comparability

Comparability of the data is a qualitative parameter that expresses the confidence with which one data set may be compared with another. Comparability of the data is achieved by using standard methods for sampling and analysis, reporting data in standard units, normalizing results to standard conditions, and using standardized reporting formats and data validation procedures.

#### 2.2.6 Sensitivity

Sensitivity is the measure of the signal from an instrument that represents an actual deflection or response above instrument noise. Analytical sensitivity is measured by the MDL and is reported with the necessary dilution factors, preparation factors, and dry-weight factors of an individual sample as the SQL. The sensitivity requirements were based on the laboratory's ability to detect and report consistent and reliable limits. Prior to solicitation and final selection of laboratories to conduct these analyses, expected MDLs and PQLs were reviewed against project objectives and found to be acceptable.

It is expected that when a direct comparison to approved applicable screening levels is conducted, some SQLs will exceed the corresponding levels. Procedures for handling nondetects (whether above the screening level or not) will be addressed in future risk assessment work plans.

Scenarios involving dilutions, high moisture content, and matrix interference affect the SQL by raising it according to the dilution factor or percent moisture content. Dilutions were required for numerous metal, anions, and SVOC analyses because of high concentrations. Whenever the concentration exceeded the linear range of the instrumentation, dilutions were analyzed. All reportable dilution results have been provided.

## 3. Conclusions and Recommendations

This section summarizes the conclusions and recommendations regarding usability of the data for the project objectives. Based on the evaluation of each data set, 100 percent of the data obtained during this event are valid (that is, not rejected). Biased data will be used as follows:

- Biased high results are due to high spike and surrogate recoveries, and blank contamination, which indicates the reported results are at the upper limit of concentration for the analyte, recognizing that the actual value may be lower.
- Biased low results are due to low spike and surrogate recoveries and negative blank values, which indicates the reported results are at the lower limit of concentration for the analyte, recognizing that the actual value may be higher.

All validated data points may be considered for use in other purposes that extend beyond the original project objectives, including evaluation against screening levels and risk assessment with the appended qualifiers noted in this document.

## 4. References

- American Public Health Association (APHA). 2011. Standard Methods for the Examination of Water and Wastewater. "Standard Method 2540G Total, Fixed, and Volatile Solids in Solid and Semisolid Samples".
- American Society for Testing and Materials International (ASTM). 2013. "Standard Test Method for Column Percolation Extraction of Mine Rock by the Meteoric Water Mobility Procedure." October.
- Broadbent & Associates, Inc. (Broadbent). 2021. "Phase II Sampling and Analysis Plan, Revision 2. Former Three Kids Mine Lakemoor Ventures LLC." Henderson, Nevada. November 3.
- Nevada Division of Environmental Protection. 2018. Letter from NDEP to All BMI Companies regarding "Data Validation Guidance." July 13.
- U.S. Environmental Protection Agency (EPA). 1993. "Method 300.0: Determination of Inorganic Anions by Ion Chromatography" Revision 2.1.
- EPA. 1994A. "Method 200.7, Revision 4.4: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry" Revision 4.4.
- EPA. 1994B. "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods." SW-846. Update III. Revision 0. September.
- EPA. 1994C. "Method 200.8: Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry" Revision 5.4.
- EPA. 1994D. "Method 245.1: Determination of Mercury in Water by Cold Vapor Atomic Absorption Spectrometry" Revision 3.0.
- EPA. 1996. "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods." SW-846. Update III. Revision 3. December.
- EPA. 2002. Guidance on Environmental Data Verification and Data Validation. Washington, DC. November.

  Available from http://www.epa.gov/QUALITY/qs-docs/g8-final.pdf

EPA. 2018. "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods." SW-846. Update VI. Revision 6. June.

EPA. 2020A. National Functional Guidelines for Inorganic Superfund Methods Data Review. EPA 542-R-20-006. Washington, DC. November.

Available from <a href="https://www.epa.gov/sites/default/files/2021-03/documents/nfg">https://www.epa.gov/sites/default/files/2021-03/documents/nfg</a> for inorganic superfund methods data review november 2020.pdf

EPA. 2020B. National Functional Guidelines for Organic Superfund Methods Data Review. EPA 540-R-20-005. Washington, DC. November. Available from https://www.epa.gov/sites/default/files/2021-

03/documents/nfg for organic superfund methods data review november 2020.pdf

# **Tables**

# Appendix A

Three Kids Mine Electronic Validated Data Deliverable—Background Soil Study 2021 (electronic format only)

## **Appendix B**

Three Kids Mine Analytical Data Packages—Background Soil Study 2021 (electronic format only)

# **Tables**

							V	alidation Le	vel
Field Sample ID	Date Sampled	Time Sampled	Matrix	Sample Association	Sample Type	SDG(s)	Metals	svocs	MWMP
BG-111-01-01	5/19/21	14:10	Soil	ASSOCIATION	Normal	L1363396	2B		
BG-111-02-01	5/19/21	15:33	Soil	BG-111-25-00	Normal	L1363396	2B		
BG-111-03-01	5/18/21	11:05	Soil		Normal	L1363396	2B		
BG-111-04-01	5/19/21	13:25	Soil		Normal	L1363396	2B		
BG-111-05-01	5/19/21	14:47	Soil		Normal	L1363396	2B		
BG-111-06-01	5/17/21	11:08	Soil		Normal	L1363396	2B		
BG-111-07-01	5/19/21	9:04	Soil		Normal	L1363396	2B		
BG-111-08-01	5/19/21	10:10	Soil		Normal	L1363396	2B		
BG-111-09-01	5/20/21	7:44	Soil	BG-111-25-00	Normal	L1363396	4		
BG-111-10-01	5/18/21	10:25	Soil		Normal	L1363396	2B		
BG-111-11-01	5/18/21	13:10	Soil		Normal	L1363396	2B		
BG-111-12-01	5/19/21	8:06	Soil		Normal	L1363396	2B		
BG-111-13-01	5/18/21	15:15	Soil		Normal	L1363396	2B		
BG-111-14-01	5/20/21	9:37	Soil	BG-13-14-00	Normal	l1363384	4		
BG-111-15-01	5/20/21	8:34	Soil		Normal	l1363384	4		
BG-111-16-01	5/18/21	9:30	Soil		Normal	L1363396	2B		
BG-111-17-01	5/18/21	14:30	Soil		Normal	L1363396	2B		
BG-111-18-01	5/17/21	13:15	Soil		Normal	L1363396	2B		
BG-111-19-01	5/18/21	8:40	Soil		Normal	L1363396	2B		
BG-111-20-01	5/18/21	13:50	Soil		Normal	L1363396	2B		
BG-111-21-01	5/17/21	13:15	Soil		Normal	L1363396	2B		
BG-111-22-01	5/20/21	13:25	Soil		Normal	11363384	4		
BG-111-23-01	5/20/21	13:53	Soil		Normal	l1363384	4		
BG-111-24-COMP	5/19/21	14:50	Water		MWMP	L1359059			4
BG-112-01-01	5/20/21	15:00	Soil		Profiile Normal	1361061, L1363384	 4	 2B	4
BG-112-01-01 BG-112-02-01	5/21/21	7:34	Soil		Normal	1361061, L1363384	4	2B	
BG-112-03-01	5/21/21	8:47	Soil		Normal	1361061, L1363384	4	2B	<del></del>
BG-112-04-01	5/21/21	8:14	Soil		Normal	1361061, L1363384	4	2B	
BG-112-05-01	5/21/21	9:23	Soil		Normal	1361061, L1363384	2B	2B	
BG-121-01-01	5/19/21	8:22	Soil		Normal	L1367629	2B		
BG-121-02-01	5/19/21	9:38	Soil		Normal	L1367629	2B		
BG-121-03-01	5/18/21	8:24	Soil		Normal	L1367629	2B		
BG-121-04-01	5/18/21	10:50	Soil		Normal	L1367629	2B		
BG-121-05-01	5/17/21	13:02	Soil		Normal	L1367629	2B		
BG-121-06-01	5/19/21	7:39	Soil		Normal	L1367629	2B		
BG-121-07-01	5/19/21	10:10	Soil		Normal	L1367629	2B		
BG-121-08-01	5/18/21	13:08	Soil		Normal	L1367629	2B		
BG-121-09-01	5/18/21	14:42	Soil		Normal	L1367629	2B		
BG-121-10-01	5/18/21	14:15	Soil		Normal	L1367629	2B		
BG-121-11-01	5/18/21	9:05	Soil		Normal	L1367629	2B		
BG-121-12-01	5/19/21	15:48	Soil	BG-121-30-00	Normal	L1367629	2B		
BG-121-13-01	5/18/21	12:35	Soil		Normal	L1367629	2B		
BG-121-14-01	5/20/21	8:26	Soil		Normal	L1367629	2B		
BG-121-15-01	5/20/21	7:37	Soil	BG-121-30-00	Normal	L1367629	2B		
BG-121-16-01	5/17/21	15:35	Soil		Normal	L1367629	2B		
BG-121-17-01	5/17/21	14:14	Soil		Normal	L1367629	2B		
BG-121-18-01	5/18/21	15:35	Soil		Normal	L1367629	2B		
BG-121-19-01	5/19/21	14:56	Soil		Normal	L1367629	2B		
BG-121-20-01	5/19/21	14:27	Soil		Normal	L1367629	2B		
BG-121-21-01	5/20/21	9:00	Soil		Normal	L1367629	2B		
BG-121-22-01	5/19/21	15:28	Soil		Normal	L1367629	2B		
BG-121-23-01	5/19/21	13:56	Soil		Normal	L1367629	2B		
BG-121-24-01	5/18/21	7:55	Soil		Normal	L1367629	2B		<del></del>
BG-121-25-01	5/19/21	13:28	Soil		Normal	L1367629	2B		
BG-121-26-01	5/19/21	12:55	Soil		Normal	L1367629	2B		
BG-121-27-01	5/18/21	9:53	Soil		Normal	L1367629	2B		

							V	alidation Le	vel
	Date	Time		Sample	Sample				
Field Sample ID	Sampled	Sampled	Matrix	Association	Type	SDG(s)	Metals	SVOCS	MWMP
BG-121-28-COMP	5/19/21	11:00	Soil		MWMP Profile	L1359064			2B
BG-121-29-COMP	5/20/21	9:35	Soil		MWMP Profile	L1359064			2B
BG-122-01-01	5/20/21	10:43	Soil	BG-122-11-00	Normal	L1367629	2B	2B	
BG-122-02-01	5/20/21	10:00	Soil		Normal	L1367629	2B	4	
BG-122-03-01	5/20/21	13:24	Soil		Normal	L1361061, L1367629	2B	2B	
BG-122-04-01	5/20/21	13:54	Soil		Normal	L1361061, L1367629	2B	2B	
BG-122-05-01	5/20/21	14:35	Soil		Normal	L1361061, L1367629	2B	2B	
BG-122-06-01	5/21/21	7:43	Soil		Normal	L1361061, L1367629	2B	2B	
BG-122-07-01	5/20/21	13:02	Soil	BG-122-11-00	Normal	L1361061, L1367629	2B	2B	
BG-122-08-01	5/20/21	15:02	Soil		Normal	L1361061, L1367629	2B	2B	
BG-122-09-01	5/21/21	7:18	Soil		Normal	L1361061, L1367629	2B	2B	
BG-122-10-COMP	5/20/21	6:50	Soil		MWMP Profile	L1359064			2B
BG-13-01-01	5/20/21	12:36	Soil	BG-13-14-00	Normal	L1367629	2B		
BG-13-02-01	5/21/21	10:35	Soil		Normal	L1367629	2B		
BG-13-03-01	5/21/21	10:05	Soil		Normal	L1367629	2B		
BG-13-04-01	5/21/21	8:42	Soil		Normal	L1367629	2B		
BG-13-05-01	5/21/21	9:46	Soil		Normal	L1367629	2B		
BG-13-06-01	5/21/21	9:03	Soil		Normal	L1367629	2B		
BG-13-07-01	5/21/21	9:27	Soil		Normal	L1367629	2B		
BG-13-08-01	5/21/21	10:07	Soil		Normal	L1367629	2B		
BG-13-09-01	5/19/21	8:51	Soil		Normal	L1367629	2B		
BG-13-10-01	5/19/21	8:04	Soil		Normal	L1367629	2B		
BG-13-11-01	5/19/21	9:38	Soil		Normal	L1367629	2B		
BG-13-12-01	5/19/21	8:18	Soil		Normal	L1367629	2B		
BG-13-13-01	5/19/21	10:57	Soil		Normal	L1367629	2B		
BG-111-25-00	5/19/21	16:36	Water	BG-111-02-01, BG-111-09-01	Equipment Blank	L1356341	2B		
BG-111-26-00	5/20/21	11:24	Water		Source Blank	L1356341	2B	2B	
BG-13-14-00	5/20/21	11:53	Water	BG-111-14-01, BGB-13-01-01	Equipment Blank	L1356341	2B		
BG-121-30-00	5/19/21	16:38	Water	BG-121-12-01, BG-121-15-01	Equipment Blank	L1356341	2B		
BG-122-11-00	5/20/21	11:22	Water	BG-122-01-01, BG-122-07-01	Equipment Blank	L1356341	2B	2B	

# TABLE 2 DATA VALIDATION CRITERIA BACKGROUND STUDY DATA VALIDATION SUMMARY REPORT Three Kids Mine Site, Henderson, Nevada

Inorganics Validation Criteria	Level 4	Level 2B
Holding times	Х	X
Calibration (initial and continuing verifications)	Х	Х
Blanks (laboratory and field)	X	X
Inductively coupled plasma (ICP) interference check sample	Х	X
Laboratory control sample	X	X
Duplicate sample analysis	X	X
Matrix spike analysis	Х	X
ICP serial dilution	X	X
Sample results verification	X	
Field duplicate	X	X
Overall assessment of data set	Х	X
Organics Validation Criteria		-
Holding times	Х	Х
Gas chromatography/mass spectrometry (GC/MS) instrument performance check	X	X
Calibration (initial and continuing verifications)	Х	Х
Blanks (laboratory and field)	Х	Х
Surrogate recovery	Х	Х
Laboratory control sample	Х	Х
Duplicate sample analysis	Х	Х
Matrix spike/matrix spike duplicate analysis	Х	Х
Field duplicate	Х	Х
Internal standard performance	Х	X
Target compound identification	Х	
Sample results verification	X	
Overall assessment of data set	X	X

#### Notes:

Criterion not evaluatedCriterion evaluated

# TABLE 3 DATA VALIDATION QUALIFIERS AND COMMENT CODES BACKGROUND STUDY DATA VALIDATION SUMMARY REPORT Three Kids Mine Site, Henderson, Nevada

Validation Qualifier	Definition
U	The analyte was analyzed for, but was not detected above the level of
U	the reported sample quantitation limit.
	The analyte was analyzed for, but was not detected. The reported
UJ	sample quantitation limit is approximate and may be inaccurate or
	imprecise.
J	The result is an estimated quantity. The associated numerical value is an
	approximate concentration of the analyte in the sample.
	The sample result is rejected and unusable due to serious deficiencies in
R	meeting quality control criteria. The analyte may or may not be present
	in the sample.
+	Estimated results are possibly biased high based on associated quality
•	control
_	Estimated results are possibly biased low based on associated quality
	control
X	Result is not used for reporting because a more accurate and precise
	result is reported in its place.
Comment Code	Definition
a	Surrogate recovery exceeded
b	Laboratory method blank and common blank contamination
С	Calibration criteria exceeded
d	Duplicate precision criteria exceeded
е	Matrix spike recovery criteria exceeded
f	Field blank contamination
g	Quantification below practical quantitation limit
h	Holding time exceeded or other sample receipt issues
i	Internal standard criteria exceeded
j	Analyte detected above SQL, but below PQL
k	Serial dilution and/or post-digestion spike criteria exceeded
I	Laboratory control sample recovery exceeded
m	Result is not recommended for use, because a result from a more
m	sensitive technique was available
n	Indicates that the analyte result was undetected (U) in the sample and
11	was reported by the laboratory as less than the sample quantitation limit

#### Note:

Other comment codes may be defined as the project encounters additional issues that may impact data quality.

TABLE 4: Qualification Based on Laboratory Blank Contamination

1356341-12 1356341-12 1356341-12 1356341-12 1356341-12 1356341-13 1356341-13	BG-122-11-00 BG-122-11-00 BG-122-11-00 BG-122-11-00 BG-122-11-00 BG-111-26-00 BG-111-26-00	BENZO(B)FLUORANTHENE INDENO(1,2,3-CD)PYRENE PHENANTHRENE PYRENE ZINC BENZO(B)FLUORANTHENE INDENO(1,2,3-CD)PYRENE	0.00005 0.00005 0.00005 0.00005 0.05 0.	0.000017 0.000018 0.000017 0.00652 0.000017	0.0000601 0.0000621 0.0000408 0.0000479 0.0285	B B BJ BJ	J+ J+ J+ J+	b,f b,f b,f,j b,f,j b,f,j	0.0221 0.0182 0.027 0.0287 0.0084	MB MB MB	mg/l mg/l mg/l mg/l
1356341-12 1356341-12 1356341-12 1356341-13 1356341-13	BG-122-11-00 BG-122-11-00 BG-122-11-00 BG-111-26-00 BG-111-26-00	PHENANTHRENE PYRENE ZINC BENZO(B)FLUORANTHENE	0.00005 0.00005 0.05	0.000018 0.000017 0.00652	0.0000408 0.0000479	B1	J+	b,f,j b,f,j	0.027	MB MB	mg/l
1356341-12 1356341-12 1356341-13 1356341-13	BG-122-11-00 BG-122-11-00 BG-111-26-00 BG-111-26-00	PYRENE ZINC BENZO(B)FLUORANTHENE	0.00005 0.05	0.000017 0.00652	0.0000479	ВЈ	J+	b,f,j	0.0287	MB	
1356341-12 1356341-13 1356341-13	BG-122-11-00 BG-111-26-00 BG-111-26-00	ZINC BENZO(B)FLUORANTHENE	0.05	0.00652							mg/l
1356341-13 1356341-13	BG-111-26-00 BG-111-26-00	BENZO(B)FLUORANTHENE			0.0285	BJ	J+	hfi	0.0084		
1356341-13	BG-111-26-00	, ,	0.00005	0.000017				~,',]	3.000 T	MB	mg/l
		INDENO(1 2 3-CD)PVRENE		0.000017	0.0000265	ВЈ	J+	b,j	0.0221	MB	mg/l
1356341-13		INDLINO(1,2,3-CD)F INLINE	0.00005	0.000018	0.000023	ВЈ	J+	b,j	0.0182	MB	mg/l
	BG-111-26-00	PHENANTHRENE	0.00005	0.000018	0.0000376	ВЈ	J+	b,j	0.027	MB	mg/l
1356341-13	BG-111-26-00	PYRENE	0.00005	0.000017	0.0000419	ВЈ	J+	b,j	0.0287	MB	mg/l
1356341-13	BG-111-26-00	ZINC	0.05	0.00652	0.0077	ВЈ	J+	b,j	0.0084	MB	mg/l
1356341-14	BG-13-14-00	ZINC	0.05	0.00652	0.00752	ВЈ	J+	b,f,j	0.0084	MB	mg/l
1356341-15	BG-121-30-00	ZINC	0.05	0.00652	0.0136	ВЈ	J+	b,f,j	0.0084	MB	mg/l
1367629-22	BG-121-19-01	MANGANESE	2.56	0.274	174		J	b	0.1376 <sup>b</sup>	ССВ	mg/kg
367629-25	BG-122-07-01	ARSENIC	1.02	0.102	3.79	J	J	b	0.5778	ССВ	mg/kg
367629-26	BG-122-03-01	ARSENIC	1.02	0.102	3.67	J	J	b	0.5778	ССВ	mg/kg
367629-30	BG-122-09-01	ARSENIC	1.02	0.102	4.59	J	J	b	0.5778	ССВ	mg/kg
1367629-45	BG-121-15-01	ZINC	25.1	0.743	28.7	В	J	b,f,k	5.07	MB	mg/kg
1367629-46	BG-121-14-01	ZINC	25	0.741	19.7	BJ	J	b,j,k	5.07	MB	mg/kg
1367629-47	BG-121-21-01	ZINC	25.1	0.742	11.3	ВЈ	J	b,j,k	5.07	MB	mg/kg
1367629-48	BG-122-02-01	ZINC	25	0.741	11.4	ВЈ	J	b,j,k	5.07	MB	mg/kg
363384-06	BG-112-01-01	ANTIMONY	3.17	0.175	0.369	J	J	b	0.102	ССВ	mg/kg
13 13 13 13 13 13 13 13	356341-13 356341-13 356341-14 356341-15 367629-22 37629-25 37629-26 37629-30 367629-45 367629-47	856341-13         BG-111-26-00           856341-13         BG-111-26-00           856341-14         BG-13-14-00           856341-15         BG-121-30-00           867629-22         BG-121-19-01           87629-25         BG-122-07-01           87629-26         BG-122-03-01           87629-30         BG-122-09-01           867629-45         BG-121-15-01           867629-46         BG-121-14-01           867629-47         BG-121-21-01           867629-48         BG-122-02-01	856341-13         BG-111-26-00         PYRENE           856341-13         BG-111-26-00         ZINC           856341-14         BG-13-14-00         ZINC           856341-15         BG-121-30-00         ZINC           867629-22         BG-121-19-01         MANGANESE           87629-25         BG-122-07-01         ARSENIC           87629-26         BG-122-03-01         ARSENIC           87629-30         BG-122-09-01         ARSENIC           867629-45         BG-121-15-01         ZINC           867629-46         BG-121-14-01         ZINC           867629-47         BG-121-21-01         ZINC           867629-48         BG-122-02-01         ZINC	856341-13       BG-111-26-00       PYRENE       0.00005         856341-13       BG-111-26-00       ZINC       0.05         856341-14       BG-13-14-00       ZINC       0.05         856341-15       BG-121-30-00       ZINC       0.05         867629-22       BG-121-19-01       MANGANESE       2.56         87629-25       BG-122-07-01       ARSENIC       1.02         87629-26       BG-122-03-01       ARSENIC       1.02         87629-30       BG-122-09-01       ARSENIC       1.02         867629-45       BG-121-15-01       ZINC       25.1         867629-46       BG-121-14-01       ZINC       25         867629-47       BG-121-21-01       ZINC       25.1         867629-48       BG-122-02-01       ZINC       25.1	856341-13         BG-111-26-00         PYRENE         0.00005         0.000017           856341-13         BG-111-26-00         ZINC         0.05         0.00652           856341-14         BG-13-14-00         ZINC         0.05         0.00652           856341-15         BG-121-30-00         ZINC         0.05         0.00652           867629-22         BG-121-19-01         MANGANESE         2.56         0.274           87629-25         BG-122-07-01         ARSENIC         1.02         0.102           87629-26         BG-122-03-01         ARSENIC         1.02         0.102           87629-30         BG-122-09-01         ARSENIC         1.02         0.102           867629-45         BG-121-15-01         ZINC         25.1         0.743           867629-46         BG-121-14-01         ZINC         25         0.741           867629-47         BG-121-21-01         ZINC         25.1         0.742           867629-48         BG-122-02-01         ZINC         25         0.741           8384-06         BG-112-01-01         ANTIMONY         3.17         0.175	856341-13         BG-111-26-00         PYRENE         0.00005         0.000017         0.0000419           856341-13         BG-111-26-00         ZINC         0.05         0.00652         0.0077           856341-14         BG-13-14-00         ZINC         0.05         0.00652         0.00752           856341-15         BG-121-30-00         ZINC         0.05         0.00652         0.0136           867629-22         BG-121-19-01         MANGANESE         2.56         0.274         174           87629-25         BG-122-07-01         ARSENIC         1.02         0.102         3.79           87629-26         BG-122-03-01         ARSENIC         1.02         0.102         3.67           87629-30         BG-122-09-01         ARSENIC         1.02         0.102         4.59           867629-45         BG-121-15-01         ZINC         25.1         0.743         28.7           867629-46         BG-121-14-01         ZINC         25         0.741         19.7           867629-47         BG-121-21-01         ZINC         25.1         0.742         11.3           867629-48         BG-122-02-01         ZINC         25         0.741         11.4	856341-13         BG-111-26-00         PYRENE         0.00005         0.000017         0.0000419         BJ           856341-13         BG-111-26-00         ZINC         0.05         0.00652         0.0077         BJ           856341-14         BG-13-14-00         ZINC         0.05         0.00652         0.00752         BJ           856341-15         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ           867629-22         BG-121-19-01         MANGANESE         2.56         0.274         174           87629-25         BG-122-07-01         ARSENIC         1.02         0.102         3.79         J           87629-26         BG-122-03-01         ARSENIC         1.02         0.102         3.67         J           87629-30         BG-122-09-01         ARSENIC         1.02         0.102         4.59         J           867629-45         BG-121-15-01         ZINC         25.1         0.743         28.7         B           867629-46         BG-121-14-01         ZINC         25         0.741         19.7         BJ           867629-48         BG-122-02-01         ZINC         25         0.741         11.4         BJ	856341-13         BG-111-26-00         PYRENE         0.00005         0.000017         0.0000419         BJ         J+           856341-13         BG-111-26-00         ZINC         0.05         0.00652         0.0077         BJ         J+           856341-14         BG-13-14-00         ZINC         0.05         0.00652         0.00752         BJ         J+           856341-15         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ         J+           867629-22         BG-121-19-01         MANGANESE         2.56         0.274         174         J           87629-25         BG-122-07-01         ARSENIC         1.02         0.102         3.79         J         J           87629-26         BG-122-03-01         ARSENIC         1.02         0.102         3.67         J         J           87629-30         BG-122-09-01         ARSENIC         1.02         0.102         4.59         J         J           867629-45         BG-121-15-01         ZINC         25.1         0.743         28.7         B         J           867629-46         BG-121-14-01         ZINC         25         0.741         19.7         BJ         J	856341-13         BG-111-26-00         PYRENE         0.00005         0.000017         0.0000419         BJ         J+         b,j           856341-13         BG-111-26-00         ZINC         0.05         0.00652         0.0077         BJ         J+         b,j           856341-14         BG-13-14-00         ZINC         0.05         0.00652         0.00752         BJ         J+         b,f,j           856341-15         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ         J+         b,f,j           867629-22         BG-121-19-01         MANGANESE         2.56         0.274         174         J         b           87629-25         BG-122-07-01         ARSENIC         1.02         0.102         3.79         J         J         b           87629-26         BG-122-03-01         ARSENIC         1.02         0.102         3.67         J         J         b           87629-30         BG-122-09-01         ARSENIC         1.02         0.102         4.59         J         J         b           87629-45         BG-121-15-01         ZINC         25.1         0.743         28.7         B         J         b,f,k <tr< td=""><td>856341-13         BG-111-26-00         PYRENE         0.00005         0.000017         0.0000419         BJ         J+         b,j         0.0287           856341-13         BG-111-26-00         ZINC         0.05         0.00652         0.0077         BJ         J+         b,j         0.0084           856341-14         BG-13-14-00         ZINC         0.05         0.00652         0.00752         BJ         J+         b,f,j         0.0084           856341-15         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ         J+         b,f,j         0.0084           867629-22         BG-121-19-01         MANGANESE         2.56         0.274         174         J         b         0.1376<sup>b</sup>           87629-25         BG-122-07-01         ARSENIC         1.02         0.102         3.79         J         J         b         0.5778           87629-26         BG-122-03-01         ARSENIC         1.02         0.102         3.67         J         J         b         0.5778           867629-45         BG-121-15-01         ZINC         25.1         0.743         28.7         B         J         b,f,k         5.07           867629-46</td><td>356341-13         BG-111-26-00         PYRENE         0.00005         0.000017         0.0000419         BJ         J+         b,j         0.0287         MB           356341-13         BG-111-26-00         ZINC         0.05         0.00652         0.0077         BJ         J+         b,j         0.0084         MB           356341-14         BG-13-14-00         ZINC         0.05         0.00652         0.00752         BJ         J+         b,f,j         0.0084         MB           366341-15         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ         J+         b,f,j         0.0084         MB           367629-22         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ         J+         b,f,j         0.0084         MB           367629-22         BG-121-19-01         MANGANESE         2.56         0.274         174         J         b         0.1376b         CCB           37629-25         BG-122-07-01         ARSENIC         1.02         0.102         3.67         J         J         b         0.5778         CCB           37629-26         BG-122-09-01         ARSENIC         1.02         0.102</td></tr<>	856341-13         BG-111-26-00         PYRENE         0.00005         0.000017         0.0000419         BJ         J+         b,j         0.0287           856341-13         BG-111-26-00         ZINC         0.05         0.00652         0.0077         BJ         J+         b,j         0.0084           856341-14         BG-13-14-00         ZINC         0.05         0.00652         0.00752         BJ         J+         b,f,j         0.0084           856341-15         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ         J+         b,f,j         0.0084           867629-22         BG-121-19-01         MANGANESE         2.56         0.274         174         J         b         0.1376 <sup>b</sup> 87629-25         BG-122-07-01         ARSENIC         1.02         0.102         3.79         J         J         b         0.5778           87629-26         BG-122-03-01         ARSENIC         1.02         0.102         3.67         J         J         b         0.5778           867629-45         BG-121-15-01         ZINC         25.1         0.743         28.7         B         J         b,f,k         5.07           867629-46	356341-13         BG-111-26-00         PYRENE         0.00005         0.000017         0.0000419         BJ         J+         b,j         0.0287         MB           356341-13         BG-111-26-00         ZINC         0.05         0.00652         0.0077         BJ         J+         b,j         0.0084         MB           356341-14         BG-13-14-00         ZINC         0.05         0.00652         0.00752         BJ         J+         b,f,j         0.0084         MB           366341-15         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ         J+         b,f,j         0.0084         MB           367629-22         BG-121-30-00         ZINC         0.05         0.00652         0.0136         BJ         J+         b,f,j         0.0084         MB           367629-22         BG-121-19-01         MANGANESE         2.56         0.274         174         J         b         0.1376b         CCB           37629-25         BG-122-07-01         ARSENIC         1.02         0.102         3.67         J         J         b         0.5778         CCB           37629-26         BG-122-09-01         ARSENIC         1.02         0.102

Notes:		Qualifiers/C	Codes:
ССВ	Continuing calibration blank	+/-	Result may be biased high/low, respectively
ICB	Initial calibration blank	b	Comment code for laboratory blank contamination
MB	Method blank	В	Same analyte is found in associated laboratory blank
mg/kg	Milligram per kilogram	С	Laboratory calibration criteria not met
mg/L	Milligram per liter	f	Field blank contamination
PQL	Practical quantitation limit	j	Analyte detected above SQL, but less than PQL
SDG	Sample delivery group	J	Identification of analyte is acceptable; reported value is estimate
SQL	Sample quantitation limit	J6	Matrix interfered with accurate quantitation; spike value is low.
		k	Serial dilution and/or post-spike criteria not met
<sup>a</sup> Denotes th	ne highest blank value associated with samples.	01	Analyte failed serial dilution and/or post-spike; matrix interference
<sup>b</sup> Highest ass	sociated CCB reported in mg/L; not mg/kg.	V	Sample concentration too high to evaluate spike recovery

	Lab	Field					Lab	Val	Reason		Field	Blank	
SDG	Sample ID	Sample ID	Analyte	PQL	SQL	Result	Qual	Qual	Code	Blank Value a	Blank ID	Туре	Units
L1363396	L1363396-19	BG-111-02-01	ARSENIC	1.01	0.101	11	,	J+	f	0.00471	BG-111-25-00	ER	mg/kg
L1363396	L1363396-19	BG-111-02-01	MANGANESE	2.52	0.271	1350		J	d,f	0.00961	BG-111-25-00	ER	mg/kg
L1363384	L1363384-01	BG-111-09-01	ARSENIC	1.02	0.102	3.69	01	J	f,k	0.00471	BG-111-25-00	ER	mg/kg
L1363384	L1363384-01	BG-111-09-01	MANGANESE	2.55	0.275	137	J5 O1	J	e,f,k	0.00961	BG-111-25-00	ER	mg/kg
L1363384	L1363384-03	BG-111-14-01	LEAD	2.1	0.104	13		j	f,k	0.00517	BG-13-14-00	ER	mg/kg
L1363384	L1363384-03	BG-111-14-01	MANGANESE	2.62	0.281	597		j	e,f,k	0.065	BG-13-14-00	ER	mg/kg
L1361061	L1361061-06	BG-122-07-01	BENZO(G,H,I)PERYLENE	0.00607	0.00179	0.0019	J	J+	f,j	0.0000642	BG-122-11-00	ER	mg/kg
L1361061	L1361061-06	BG-122-07-01	CHRYSENE	0.00607	0.00235	0.00443	J	J+	f,i	0.0000438	BG-122-11-00	ER	mg/kg
L1361061	L1361061-06	BG-122-07-01	PYRENE	0.00607	0.00202	0.00208	J	J+	f,j	0.0000479	BG-122-11-00	ER	mg/kg
L1367629	L1367629-24	BG-121-12-01	ARSENIC	1	0.1	5.6		J+	f	0.00608	BG-121-30-00	ER	mg/kg
L1367629	L1367629-24	BG-121-12-01	LEAD	2	0.0992	7.77		J+	f	0.0158	BG-121-30-00	ER	mg/kg
L1367629	L1367629-24	BG-121-12-01	MANGANESE	2.51	0.269	232		J	b,f	0.219	BG-121-30-00	ER	mg/kg
L1367629	L1367629-24	BG-121-12-01	ZINC	25.1	0.742	22.6	J	J+	f, j	0.0136	BG-121-30-00	ER	mg/kg
L1367629	L1367629-25	BG-122-07-01	ARSENIC	1.02	0.102	3.79		J+	f	0.0154	BG-122-11-00	ER	mg/kg
L1367629	L1367629-25	BG-122-07-01	LEAD	2.04	0.101	7.39		J+	f	0.031	BG-122-11-00	ER	mg/kg
L1367629	L1367629-25	BG-122-07-01	MANGANESE	2.55	0.274	109		J	b,f	0.427	BG-122-11-00	ER	mg/kg
L1367629	L1367629-25	BG-122-07-01	ZINC	25.5	0.756	80.4		J+	f	0.0285	BG-122-11-00	ER	mg/kg
L1367629	L1367629-37	BG-13-01-01	LEAD	203	10	9410		J+	f	0.00517	BG-13-14-00	ER	mg/kg
L1367629	L1367629-37	BG-13-01-01	MANGANESE	254	27.2	207000		J	b,f	0.065	BG-13-14-00	ER	mg/kg
L1367629	L1367629-37	BG-13-01-01	ZINC	25.4	0.751	646		J+	f	0.00752	BG-13-14-00	ER	mg/kg
L1367629	L1367629-45	BG-121-15-01	ARSENIC	1	0.1	3.65		J+	f	0.00608	BG-121-30-00	ER	mg/kg
L1367629	L1367629-45	BG-121-15-01	LEAD	2.01	0.0995	7.03		J	f,k	0.0158	BG-121-30-00	ER	mg/kg
L1367629	L1367629-45	BG-121-15-01	MANGANESE	2.51	0.269	195		J	b,f,k	0.219	BG-121-30-00	ER	mg/kg
L1367629	L1367629-45	BG-121-15-01	ZINC	25.1	0.743	28.7	В	J	b,f,k	0.0136	BG-121-30-00	ER	mg/kg
L1367629	L1367629-49	BG-122-01-01	ARSENIC	1.01	0.101	3.53		J+	f	0.0154	BG-122-11-00	ER	mg/kg
L1367629	L1367629-49	BG-122-01-01	LEAD	2.02	0.1	11.5		J	f,k	0.031	BG-122-11-00	ER	mg/kg
L1367629	L1367629-49	BG-122-01-01	MANGANESE	2.53	0.271	190		J	b,f,k	0.427	BG-122-11-00	ER	mg/kg
L1367629	L1367629-49	BG-122-01-01	ZINC	25.3	0.748	106		J	b,f,k	0.0285	BG-122-11-00	ER	mg/kg
L1356341	L1356341-12	BG-122-11-00	LEAD	0.006	0.00299	0.031		J+	f	0.00376	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	MANGANESE	0.01	0.000934	0.427		J+	f	0.0156	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	ZINC	0.05	0.00652	0.0285	ВJ	J+	b,f,j	0.0077	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	BENZO(A)ANTHRACENE	0.00005	0.00002	0.0000425	J	J+	f, j	0.0000273	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	BENZO(A)PYRENE	0.00005	0.000018	0.0000311	J	J+	f,j	0.0000188	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	BENZO(B)FLUORANTHENE	0.00005	0.000017	0.0000601	В	J+	b,f	0.0000265	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	BENZO(G,H,I)PERYLENE	0.00005	0.000018	0.0000642		J+	f	0.0000197	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	CHRYSENE	0.00005	0.000018	0.0000438	J	J+	f,j	0.0000214	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	DIBENZ(A,H)ANTHRACENE	0.00005	0.000018	0.0000654		J+	f	0.0000186	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	INDENO(1,2,3-CD)PYRENE	0.00005	0.000018	0.0000621	В	J+	b,f	0.000023	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	PHENANTHRENE	0.00005	0.000018	0.0000408	BJ	J+	b,f,j	0.0000376	BG-111-26-00	SB	mg/l
L1356341	L1356341-12	BG-122-11-00	PYRENE	0.00005	0.000017	0.0000479	ВJ	J+	b,f,j	0.0000419	BG-111-26-00	SB	mg/l
L1356341	L1356341-14	BG-13-14-00	LEAD	0.006	0.00299	0.00517	J	J+	f,j	0.00376	BG-111-26-00	SB	mg/l
L1356341	L1356341-14	BG-13-14-00	MANGANESE	0.01	0.000934	0.065		J+	f	0.0156	BG-111-26-00	SB	mg/l
L1356341	L1356341-14	BG-13-14-00	ZINC	0.05	0.00652	0.00752	ВJ	J+	b,f,j	0.0077	BG-111-26-00	SB	mg/l
L1356341	L1356341-15	BG-121-30-00	LEAD	0.006	0.00299	0.0158		J+	f	0.00376	BG-111-26-00	SB	mg/l
L1356341	L1356341-15	BG-121-30-00	MANGANESE	0.01	0.000934	0.219		J+	f	0.0156	BG-111-26-00	SB	mg/l
L1356341	L1356341-15	BG-121-30-00	ZINC	0.05	0.00652	0.0136	ВJ	J+	b,f,j	0.0077	BG-111-26-00	SB	mg/l
L1356341	L1356341-23	BG-111-25-00	MANGANESE	0.01	0.000934	0.00961	J	J+	f,j	0.0156	BG-111-26-00	SB	mg/l

TABLE 5: Qualification Based on Field Blank Contamination

	Lab	Field					Lab	Val	Reason		Field	Blank	
SDG	Sample ID	Sample ID	Analyte	PQL	SQL	Result	Qual	Qual	Code	Blank Value <sup>a</sup>	Blank ID	Type	Units
L1356341	L1356341-25	BG-122-01-01	BENZO(A)ANTHRACENE	0.00605	0.00175	0.00513	J	J+	f,j	0.0000425	BG-122-11-00	ER	mg/kg
L1356341	L1356341-25	BG-122-01-01	BENZO(A)PYRENE	0.00605	0.00181	0.00202	J	J+	f,j	0.0000311	BG-122-11-00	ER	mg/kg
L1356341	L1356341-25	BG-122-01-01	BENZO(B)FLUORANTHENE	0.00605	0.00154	0.0098		J+	f	0.0000601	BG-122-11-00	ER	mg/kg
L1356341	L1356341-25	BG-122-01-01	BENZO(G,H,I)PERYLENE	0.00605	0.00179	0.00884		J+	f	0.0000642	BG-122-11-00	ER	mg/kg
L1356341	L1356341-25	BG-122-01-01	CHRYSENE	0.00605	0.00234	0.0166		J+	f	0.0000438	BG-122-11-00	ER	mg/kg
L1356341	L1356341-25	BG-122-01-01	DIBENZ(A,H)ANTHRACENE	0.00605	0.00174	0.00367	J	J+	f,j	0.0000654	BG-122-11-00	ER	mg/kg
L1356341	L1356341-25	BG-122-01-01	INDENO(1,2,3-CD)PYRENE	0.00605	0.00183	0.00644		J+	f	0.0000621	BG-122-11-00	ER	mg/kg
L1356341	L1356341-25	BG-122-01-01	PHENANTHRENE	0.00605	0.00233	0.00406	J	J+	f,j	0.0000408	BG-122-11-00	ER	mg/kg
L1356341	L1356341-25	BG-122-01-01	PYRENE	0.00605	0.00202	0.0106		J+	f	0.0000479	BG-122-11-00	ER	mg/kg

Notes:		Qualifier	rs/Codes:
		+/-	Result may be biased high/low, respectively
ER	Equipment rinsate blank	b	Comment code for laboratory blank contamination
mg/kg	Milligram per kilogram	В	Same analyte is found in associated laboratory blank
mg/L	Milligram per liter	d	Result qualified based on duplicate precision
PQL	Practical quantitation limit	e	Result qualified based on matrix spike recovery
Qual	Qualifier	f	Comment code for field blank contamination
SB	Source water blank	j	Analyte detected above SQL, but less than PQL
SDG	Sample delivery group	J	Identification of analyte is acceptable; reported value is estimate
SQL	Sample quantitation limit	J5	Matrix interfered with accurate quantitation; spike value is low.
<sup>a</sup> Denote	es the associated field blank value reported in mg/L.	k	Serial dilution and/or post-spike criteria not met
		01	Analyte failed serial dilution and/or post-spike; matrix interference

TABLE 6: Qualification Based on Matrix Spike Recovery

	Lab	Field					Lab	Val	Reason	Result
SDG	Sample ID	Sample ID	Analyte	PQL	SQL	Result	Qual	Qual	Code	Unit
MS/MSD Reco	overies Lead = 133,	/254 %R (QC Limit 60	)-140 %R)							
L1363396	L1363396-01	BG-111-19-01	LEAD	2.03	0.1	41.9	J3 J5	J	d,e	mg/kg
MS/MSD Reco	overies Antimony =	= 61.2/56.4 %R; Man	ganese = 147/127 %	R (QC Limit	60-140 %R)					
L1363384	L1363384-01	BG-111-09-01	ANTIMONY	3.06	0.169	0.202	J J6	J-	e,j,k	mg/kg
L1363384	L1363384-01	BG-111-09-01	MANGANESE	2.55	0.275	137	J5 O1	J	e,f,k	mg/kg
MS/MSD Reco	overies Antimony =	= 58.0/62.3 %R (QC L	imit 60-140 %R)							
L1367629	L1367629-01	BG-121-05-01	ANTIMONY	3.01	0.166	0.494	J J3 J6	J	d,e,j	mg/kg
L1367629	L1367629-30	BG-122-09-01	ANTIMONY	3.01	0.166	0.166	U	UJ	e,n	mg/kg
Notes:				Qualifiers	/Codes:					
%R	Percent recovery			+/-	Result may	be biased high,	/low, respecti	vely		
mg/kg	Milligram per kilogram d Duplicate precision criteria exceeded									
MS	Matrix spike			е	Matrix spik	e recovery crite	eria exceeded			
MSD	Matrix spike dupli	icate		f	Field blank	contamination	1			
PQL	Practical quantita	ation limit		j	Analyte det	ected above SC	QL, but less th	an PQL		
QC	Quality control			J	Identificati	on of analyte is	acceptable; r	eported valu	e is estimate	
Qual	Qualifier			J3	Associated	batch QC outsi	de QC range fo	or precision		
SDG	Sample delivery gr	roup		J5	Matrix inte	rfered with acc	urate quantit	ation; spike	/alue is low.	
SQL	Sample quantitati	ion limit		J6	Matrix inte	rfered with acc	urate quantit	ation; spike	alue is low.	
	k Serial dilution and/or post-spike criteria not met									
				n	Analyte und	detected (U) at	the SQL			
				01	Analyte fail	ed serial dilutio	on and/or pos	t-spike; matr	ix interferenc	ce
				U	Analyte rep	orted as undet	ected at the S	QL		
				UJ	Analyte rep	orted as undet	ected, but SQ	L is an estima	ite	
				V	Sample con	centration too	high to evalu	ıate spike rec	overy	

TABLE 7: Qualification Based on Laboratory Duplicate Precision

	Lab	Field					Lab	Val	Reason	Result
SDG	Sample ID	Sample ID	Analyte	PQL	SQL	Result	Qual	Qual	Code	Unit
MS/MSD Du	plicate Precision L	ead = 51.9% RPD; Ma	anganese = 69.6%	RPD (QC Lin	nit ≤20% RPD)			•	•	
L1363396	L1363396-01	BG-111-19-01	LEAD	2.03	0.1	41.9	J3 J5	J	d,e	mg/kg
L1363396	L1363396-01	BG-111-19-01	MANGANESE	2.53	0.271	747	J3 O1 V	J	d	mg/kg
MS/MSD Du	plicate Precision A	ntimony = 33.7% RP	D; Selenium = 20.	1% RPD (QC	Limit ≤20% RP	D)		•	•	
L1367629	L1367629-01	BG-121-05-01	ANTIMONY	3.01	0.166	0.494	11316	J	d,e,j	mg/kg
L1367629	L1367629-01	BG-121-05-01	SELENIUM	2.51	0.18	0.692	113	J	d,j	mg/kg
Notes:				Qualifiers/	Codes:			•	•	
mg/kg	Milligram per kilo	gram		+/-	Result may b	e biased high/lo	w, respectively			
MS	Matrix spike			d	Duplicate pr	ecision criteria	exceeded			
MSD	Matrix spike dupli	icate		e	Matrix spike	recovery criteri	a exceeded			
PQL	Practical quantita	ation limit		j	Analyte dete	cted above SQL,	but less than PQ	L		
QC	Quality control			J	Identificatio	n of analyte is a	cceptable; report	ed value is e	stimate	
RPD	Relative percent d	lifference		J3	Associated ba	atch QC outside	QC range for pre	cision		
SDG	Sample delivery gr	roup		J5	Matrix interf	ered with accur	ate quantitation;	; spike value	is low.	
SQL .	Sample quantitati	ion limit		J6	Matrix interf	ered with accur	ate quantitation	; spike value	is low.	
				01	Analyte faile	d serial dilution	and/or post-spik	e; matrix int	erference	
				V	Sample conc	entration too h	igh to evaluate sp	ike recovery	/	

# **Appendix A**

Three Kids Mine Electronic Validated Data Deliverable—Background Soil Study 2021 (electronic format only)

# **Appendix B**

Three Kids Mine Analytical Data Packages—Background Soil Study 2021 (electronic format only)