



**UPDATE AND REVISIONS FOR OPEN-FILE REPORT 98-624,
SYNTHETIC PRECIPITATION LEACHING PROCEDURE
(SPLP) LEACHATE CHEMISTRY DATA FOR SOLID MINE-
WASTE COMPOSITE SAMPLES FROM SOUTHWESTERN
NEW MEXICO, AND LEADVILLE, COLORADO**

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**U.S. DEPARTMENT OF THE INTERIOR
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Abstract

This report supersedes, revises, and updates information and data previously released in Open-File Report 98-624 (Montour and others, 1998). Data for this report were derived from leaching of mine-waste composite samples using a modification of E.P. A. Method 1312, Synthetic Precipitation Leaching Procedure (SPLP). In 1997, members of the U.S. Geological Survey Mine Waste Characterization Project collected four mine-waste composite samples from mining districts near Silverton, Colorado (MAY and YUK), and near Leadville, Colorado (VEN and SUN). This report presents analytical results from these sites.

Introduction

Assessing potential acid and metal mobility from abandoned mine-waste piles is an area of concern for Federal land management agencies and others. Drainage and runoff from these piles may present water-quality concerns similar to acid-mine drainage. Mine-waste piles that contain metal and sulfide reservoirs may become sources of acidic drainage when exposed to surface conditions of oxygen and water. Continuing work on Department of Interior, Abandoned Mine Lands Initiative (AML) projects has demonstrated the need for rapid, accurate characterization of these waste materials in order to aid in the screening, geochemical assessment, and prioritization of a large number of federally owned mine waste piles for remediation. One of the methods currently used to characterize metal mobility from mine-waste material is E.P.A. Method 1312 Synthetic Precipitation Leaching Procedure (SPLP). Because this leach test has been used in many studies involving mine-waste it is the method we chose for the geochemical characterization of the samples in this study.

Purpose of Investigation

The purpose of this investigation is two-fold: First, it provides our collaborators an assessment of EPA Method 1312 leachate chemistry data for solid mine-waste composite samples collected from four different mine waste sites. Second, data from this investigation were used for comparative studies as part of a larger effort to develop a field leach test (Hageman and Briggs, 2000) that was designed and used as a screening tool for evaluation of historic mine-waste piles.

General Methods

Leachate chemistry data for solid mine-waste composite samples were obtained by leaching the mine-waste material using a modification of the EPA Method 1312 Synthetic Precipitation Leaching Procedure (SPLP). For this study, four solid mine-waste composite samples were collected from historic mine-waste piles using the technique described in (Smith and others, 2000). The composite samples were then prepared for use in leach studies. The leachate from the experiments was filtered and aliquots were taken for metals, and anion analysis. Measurements were made for pH and specific conductivity on unfiltered aliquots of leachate.

Sampling Locations

In the summer of 1997, four mine-waste pile composite samples were collected from historic mine dumps in two mining districts in Colorado. The May Day (MAY), and Yukon (YUK) were collected from the Silverton district in southwestern Colorado. The

Venir (VEN) and Sunday #2 (SUN) were collected from the Leadville district in central Colorado.

Geology

May Day (MAY)

The May Day mine-waste dump is located along Cement Creek in the Silverton mining district of San Juan County, in southwestern Colorado. This mining district is developed in the Oligocene, San Juan and Silverton Calderas. The economic mineralization in the region is mainly associated with several younger, Tertiary ore-forming episodes, which post-date caldera formation by a few million years (Lipman and others, 1973; Lipman and others, 1976; Casadevall and Ohmoto, 1977; Bove, and others; 1999).

The May Day waste is situated on United States, Bureau of Land Management (BLM) property and is comprised of a series of northwest oriented benches and is approximately 20,000 cubic yards in volume. The waste resulted from excavation of a northwest trending exploratory tunnel and associated workings. The tunnel was apparently oriented to intercept a northwest-trending, steeply dipping mineralized vein located 50 to 60 m to the west. No obvious mineralization is observed at the tunnel entrance with the exception of weakly, propylitically altered dacite lava flows of the Silverton Volcanics (Lipman and others, 1973). The altered dacite lava flows consist of quartz, sodic plagioclase, orthoclase, epidote, and chlorite. Primary hydrothermal minerals identified in the waste are pyrite, galena, sphalerite, quartz, muscovite, kaolinite, and traces of barite, pyrophyllite, and ferberite. Secondary minerals now present include jarosite and anglesite. It is unknown whether any of the material extracted was processed for metal recovery.

Yukon (YUK)

The Yukon mine-waste dump is located along the east side of Cement Creek in the Silverton mining district of San Juan County, in southwestern Colorado and was developed in the Oligocene, San Juan and Silverton calderas. Hydrothermal mineralization in this district is associated with multiple Tertiary ore-forming periods, which occurred after caldera formation (Lipman and others, 1973; Lipman and others, 1976; Casadevall and Ohmoto, 1977; and Bove and others, 1999).

The Yukon waste is on private property adjacent to a mill known as the Gold Hub, but no mill tailings are visible on the property. The mine waste is estimated to be about 4,000 cubic yards in volume, and is presumed to have come from an adit in propylitically altered dacite lava flows. Primary hydrothermal minerals identified in the waste include pyrite, muscovite, kaolinite, and ferberite. Secondary minerals are jarosite, gypsum, and anglesite. A white efflorescent mineral on the underside of an iron-cemented overhang was identified as gunningite—a highly soluble zinc sulfate.

Venir (VEN)

The Venir mine-waste dump is in the Leadville mining district of central Colorado. Lode ores were mined continuously in this district from 1876 to 1957, producing silver, lead, zinc, and minor copper. The host rock is the Johnson Gulch Porphyry of Tertiary age (43 Ma) in the Breece Hill stock. This quartz monzonite porphyry consists of quartz, plagioclase, and orthoclase phenocrysts, with minor biotite in the groundmass (Thompson and Arehart, 1990).

Venir wastes are distributed in several westward pointing "fingers" or lobes formed

from railcar dumping of the wastes. Hydrothermal minerals in the wastes include pyrite, muscovite, kaolinite, ferberite, barite, and native gold. Quartz, orthoclase, and minor chlorite present in the wastes are from the Johnson Gulch Porphyry host. Jarosite and anglesite are the major secondary minerals.

Sunday (SUN)

The Sunday No. 2 mine-waste dump in the Leadville mining district of Colorado consists of a single conical pile above timberline on the west side of Ball Mountain. The geologic setting is the same as the Venir mine; it is hosted in the Tertiary Johnson Gulch Porphyry (Thompson and Arehart, 1990). At an elevation of about 11,900 feet above mean sea level, it is usually frozen for about six months of the year. During snowmelt runoff, it produces a conspicuous vegetative-kill zone down slope that is barren.

Minerals in the Sunday No. 2 waste composite bulk sample include quartz, muscovite, jarosite, kaolinite, pyrite, galena, and sphalerite. Heavy-mineral concentrates reveal minor cerrusite and barite are present. Acetone-decanted solids from the < 0.090 mm fraction contain gypsum and anglesite as trace constituents.

Methods

Collection of Mine-Waste Grid Composite Samples

At each site, members of the United States Geological Survey's Mine-Waste Characterization Project laid out an informal grid that contained most of the material in the waste pile. The grids consisted of at least 30 evenly-spaced cells for sampling. Approximately 30 sub-samples or increments were randomly collected from each grid cell by sampling the weathered surface material (upper 15 cm) using stainless steel trowels, small hand shovels, and three-prong scrapers. During collection, all rock fragments > 4 cm were discarded. Increments from each cell were combined in a 1-gallon plastic bucket. After collection, the composite from each cell was placed in a plastic washtub and mixed. A sub-sample of the cell composite was placed in a soil bag and saved as a cell composite. The remaining cell composite material was placed into 5-gallon bucket(s) to be mixed with other cell composites to form the mine-waste pile composite sample. This material was then transported back to the laboratory. Each sample was spread out on clean plastic tarps to dry at room temperature. To insure complete drying, samples were completely turned daily with a small plastic scoop until visibly dry (approximately 2 to 3 days depending upon ambient humidity). After drying, each composite was mixed for 5 minutes in a large stainless steel V-Blender to break up friable clods. The composite material was then sieved with a 2 mm screen, with the < 2 mm fraction being recombined and thoroughly homogenized by mixing in the V-Blender for 30 minutes. The > 2 mm fraction was discarded. After homogenization, the < 2 mm composite material was split into 1 gallon cardboard ice cream containers. The tops of the containers were sealed with tape for storage.

Modified E.P.A. Method 1312 (SPLP)

The Synthetic Precipitation Leaching Procedure (SPLP) (U.S. EPA Method 1312; U.S. Environmental Protection Agency, 1986; 1994 update) is a method designed to evaluate the impact of contaminated soils on groundwater. The extraction fluid consists of slightly acidified de-ionized (DI) water that is formulated to simulate natural precipitation. A mixture of 60/40 H₂SO₄/HNO₃ (by weight) is used to achieve the appropriate pH for the extraction fluid. The pH of the de-ionized water is adjusted with the 60/40 H₂SO₄/HNO₃ mixture, depending on which side of the Mississippi River the soils

originate from, to either pH 4.2(+/-) 0.05 (east of the Mississippi) or pH 5.0 (+/-) 0.05 (west of the Mississippi). The SPLP procedure mandates that for mine wastes, the more acidic pH 4.2 extraction fluid be used.

The SPLP extraction method for 100% solids and no volatiles was utilized for this study. A sample that is 100% solids requires reduction of particle size to <1 cm (if necessary), and extracting the solid at a 20:1 ratio (100g of mine-waste composite material : 2,000g extraction fluid) on an end-over-end rotary agitator for 18 hours. The solid/liquid slurry is then filtered through a 0.7 urn borosilicate glass fiber filter utilizing a stainless steel pressure filtration unit.

For this study, we used the pH 4.2 extraction fluid and modified the SPLP procedure by sieving and using only the < 2mm fraction of the sample. Because of sieving there was no need for particle size reduction.

Acid-washed extraction vessels (Nalgene® high-density polyethylene 2-liter bottles) were rinsed twice with 10 ml of pH 4.2 extract solution. One hundred grams of each sample (< 2mm fraction) was weighed and placed in an extractor vessel, and two liters of pH 4.2 extraction solution was slowly added to the vessel. Teflon® tape was wrapped around the bottle threads to create a tight seal. The extractor vessels were secured in an Analytical Testing Rotary Agitator (model DC-20B) and rotated end-over-end for eighteen hours at twenty-eight revolutions per minute.

Following extraction, the samples were filtered through a 0.7 µm borosilicate glass fiber filter (Gelman Sciences Inc. P/N 66257JCLP glass fiber filter, 0.7 urn pore size, and 142-mm diameter). The filtration unit used was a Gelman Sciences Hazardous Waste pressure filtration unit (Gelman Sciences Product No. 15046). The filters were placed in position and acid washed with 1 liter of 1N HNO₃ followed by three one liter de-ionized water rinses prior to filtration of the samples. A small aliquot of the unfiltered leachate was taken for pH and specific conductivity measurements. After filtration, aliquots of filtrate were preserved for analysis.

Analytical Methods

Similar analytical methods and procedures were used for all samples. U.S. Geological Survey employees using U.S.G.S. instrumentation and equipment performed all preparation and analyses of samples. Following is a detailed explanation of the analytical techniques and procedures used, the sample requirements, and other key parameters.

pH

Laboratory measurements were taken on unfiltered aliquots of leachate using an Orion Model 230-A pH meter and an Orion Combination pH electrode. (Orion Electrode # 915600). The meter was calibrated prior to each set of measurements and checks were obtained on other buffers between readings to insure all buffers read and recorded to ± 0.02 pH units. The pH analysis requires no sample treatment but the sample solution must be measured immediately.

Specific Conductance

Laboratory measurements of specific conductance (µS/cm) were taken on unfiltered aliquots of leachate using a Myron L Portable Conductivity Meter (Model DC4). Prior to initial use, the meter was calibrated with commercially obtained conductivity standards. Prior to each set of observations, aliquots of commercially obtained 100 µS/cm and 1000 US/cm standards are checked and their values recorded. Conductivity analysis requires no sample treatment and again, the sample should be analyzed immediately.

Anion Analysis

Anions were determined by ion chromatography (IC) using a modification of the method of d'Angelo and Ficklin (1996). Anions measured were fluoride, chloride, nitrate, and sulfate. Preservation of the sample for anion analysis requires filtration and then refrigeration at 4°C. Sample must be analyzed within 28 days.

Metals Analysis

Concentrations of metals were determined by inductively coupled plasma - mass spectroscopy (ICP-MS) (Lamothe and others, 1999) after screening of samples using inductively coupled plasma - atomic emission spectroscopy (ICP-AES) (Briggs and Fey, 1996). Preservation of the samples for metals analysis requires that the sample be filtered and acidified to pH < 1.5 with Ultrex® II Ultrapure HNO₃. Maximum hold time for the samples is 180 days.

Quality Control

Quality Control Checks

All data generated from the laboratory were subject to quality control checks to provide information on the quality and usability of the data. The majority of the quality control procedures used are based on standard USGS QA/QC protocols. Known reference standards were submitted and analyzed with the samples. In addition, a preparation blank and an analytical batch duplicate were included with each analytical set. Table 1 shows the frequency and control limits of laboratory control checks used for leachate samples obtained from leach experiments in this study. A brief description and definition of each type of quality control check follows. Results of quality control checks are presented in Appendix A-1, A-2a, and A2b.

Table 1. Laboratory quality control checks

[IDL = instrument detection limit, RPD = relative percent difference]

Quality Control Check	Frequency	Control Limits
1. Preparation Blank chemistry	one per analytical batch	<IDL or unprocessed extraction fluid
2. Analytical Batch Duplicates	one per analytical batch	± 20% RPD
3. Reference Standards	one set per analytical grouping	±20% RPD

Preparation Blank

A preparation blank is a sample of the appropriate volume of extraction fluid that is carried through the entire experimental process including analysis. The preparation blank is used to determine whether any added reagents, equipment, procedures, or processes introduce any contaminants to the samples. For laboratory studies, at least one preparation blank is run with each analytical batch.

Analytical Batch Duplicates

Analytical batch duplicates consist of two identical splits of a site composite that are carried through the entire experimental process, including analysis in order to

determine the precision of the analytical results. For laboratory studies, at least one duplicate is run with each analytical batch.

Reference Standards

(NIST) water standards were used as a check on ICP analysis. Two Reference standards were submitted with all sample groups undergoing ICP-MS and ICP-AES analysis.

Data Summaries

pH

pH values for the composites ranged from 2.7 (VEN) to 3.4 (MAY). Other pH values were 2.8 (SUN) and 3.1 (YUK). All four mine-waste composite samples showed a decrease in pH from the starting extraction fluid pH of 4.2. The sample pH values ranked VEN < SUN < YUK < MAY. pH results for all composite leachates are found in Table

2.

Specific Conductance

Values for specific conductance measurements ranged from, 202 $\mu\text{S}/\text{cm}$ (MAY) to 1060 $\mu\text{S}/\text{cm}$ (VEN) with (YUK) and (SUN) falling between these values at 650 $\mu\text{S}/\text{cm}$ and 787 $\mu\text{S}/\text{cm}$ respectively. Sample specific conductance values ranked MAY < YUK < SUN < VEN. Specific conductance values for all sites are found in Table 2.

Anions

Anion data reveal that the (VEN) composite had the highest concentration of SO_4^{2-} (280 ppm). The complete set of anion data are found in Table 2.

Table 2 pH, specific conductivity, and anion data (IC)

[data in ppm except Specific Conductivity which is in $\mu\text{S}/\text{cm}$]

	pH	Conductivity	F⁻	C⁻	SO₄²⁻	NO₃⁻
MAY	3.4	202	0.2	0.7	55	0
YUK	3.1	650	15	0.6	210	0.5
VEN	2.7	1060	4.3	3.7	280	0.5
SUN	2.8	787	1.7	1.6	160	0.6

Metals

Following is a summary of selected elements for the four sites. The complete set of ICP data for all elements and all samples analyzed is listed in Appendix A-4.

Aluminum (Al)

Aluminum concentrations ranged from 0.42 mg/L (MAY) to 13.1 mg/L (VEN). Other values were 6.5 mg/L (SUN) and 10.4 mg/L (YUK).

Arsenic (As)

Arsenic was detected in only one sample. (SUN) had a concentration of 4.8 $\mu\text{g}/\text{L}$.

Cadmium (Cd)

Cadmium was detected in all of the samples. Values ranged from 6.1 µg/L (MAY) to 25.4 µg/L (SUN). Other values were 6.4 µg/L (YUK) and 13.3 µg/L (VEN).

Calcium (Ca)

Calcium concentration was highest in the (YUK) composite at 37.8 mg/L. Other values were (VEN) 5.5 mg/L, (SUN) 5.4 mg/L, and (MAY) 2.5 mg/L.

Cobalt (Co)

Cobalt concentrations were 3.7 µg/L (MAY), 8.3 µg/L (SUN), 15.2 µg/L (VEN), and 24.1 µg/L (YUK).

Chromium (Cr)

Chromium was found in three of the samples. Values were 3.5 µg/L (YUK), 9.8 µg/L (SUN), and 30.1 µg/L (VEN). Cr was not detected in the (MAY) sample.

Copper (Cu)

Copper values range from 0.12 mg/L at (MAY) to 4.0 mg/L at (VEN). Other values were 0.22 mg/L at (SUN) and 0.5 mg/L at (YUK).

Iron (Fe)

(MAY) had a total iron concentration of 0.60 mg/L, (SUN) 13.8 mg/L, (YUK) 16.2 mg/L, and (VEN) 28.7 mg/L.

Magnesium (Mg)

Magnesium concentrations ranged from 0.37 mg/L at (SUN) to 6.4 mg/L (VEN). Other values were 0.74 mg/L (MAY) and 3.7 mg/L (YUK).

Manganese (Mn)

Concentrations of manganese ranged from 213 µg/L (MAY) to 1560 µg/L (YUK). Midrange values were 770 µg/L at (SUN) and 1170 µg/L at (VEN). The blank had a manganese concentration of 0.04 µg/L.

Nickel (Ni)

Nickel concentration was lowest in leachate from the (MAY) site at 5.6 µg/L. Nickel concentrations from the other sites were 17.2 µg/L (SUN), 22.7 µg/L (YUK), and 43.8 µg/L at (VEN).

Lead (Pb)

Concentration of lead in the four composites ranged from 0.11 mg/L (YUK) to 2.9 mg/L (MAY). (VEN) had a lead concentration of 0.78 mg/L, and (SUN) 1.2 mg/L.

Zinc (Zn)

Concentrations for zinc ranged from 0.95 mg/L (VEN) to 3.6 mg/L (SUN). Other values were 1.0 mg/L for (MAY), and 1.3 mg/L for the (YUK) composite.

Sulfate (SO₄)

Sulfate concentration ranged from 43 ppm (MAY) to 330 ppm (VEN). Mid-range concentrations were 184 ppm (SUN), and 231 ppm at (YUK). Sulfate concentration in the Blank was 2.0 ppm.

Preparation Blank

Preparation blank analytical results (ICP-MS, and ICP-AES) are presented in Appendix A-1. As we have seen in other leach studies conducted as part of this project, filtration of leachate using borosilicate glass fiber filters have shown enhanced post-filtration concentrations of barium (Ba). Consistent with this finding, the performance blank analyzed with this set of samples had a post-filtration Ba concentration of 44.1 µg/L. Other elements that show moderate increase of concentration in the blank were: bismuth (Bi) 0.06 µg/L, manganese (Mn) 0.04 µg/L, molybdenum (Mo) 1.7 µg/L, antimony (Sb) 0.43 µg/L, Tantalum (Ta) 0.07 µg/L, thallium (Tl) 0.1 µg/L, and tungsten (W) 0.2 µg/L. Elements that show slight increases are: calcium (Ca) 0.11 mg/L, europium (Eu) 0.006 µg/L, sodium (Na) 0.03 mg/L, Niobium (Nb) 0.04 µg/L, Rubidium (Rb) 0.02 µg/L, strontium (Sr) 0.63 µg/L, thorium (Th) 0.005 µg/L, vanadium (V) 0.1 µg/L, and (SO₄²⁻) 2 ppm. All other elements were at or below their detection limits.

Analytical Batch Duplicates

Analytical data (ICP-MS and ICP-AES) for the batch duplicates can be found in Appendix A-3. Gallium (Ga), thulium (Tm), vanadium (V), ytterbium (Yb), and zirconium (Zr) exceeded the relative percent difference (RPD) control limit of 20%.

Reference Standards NIST 1640 and 1643d

The complete set of analytical data for reference standards NIST 1640 and NIST 1643d can be found in Appendix A-2a and A-2b respectively. For reference standard NIST 1640, only silver (Ag) exceeded the relative percent difference (RPD) control limit of 20%. Data for reference standard NIST 1643d showed that only iron (Fe) exceeded the (RPD).

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Appendix A-I, Chemistry Data for Preparation Blank

[Data in bold are ICP-AES, all other data are ICP-MS]

Element:Units	Del Limit	Blank
Ag ug/L	<3	<3
Al mg/L	<0.01	<0.01
As ug/L	<3	<3
Ba ug/L	<0.1	44.1
Be ug/L	<0.05	<0.05
Bi ug/L	<0.005	0.06
Ca mg/L	<0.1	0.11
Cd ug/L	<0.02	<0.02
Ce ug/L	<0.01	<0.01
Co ug/L	<0.02	<0.02
Cr ug/L	<1	<1
Cs ug/L	<0.01	<0.01
Cu mg/L	<0.01	<0.01
Dy ug/L	<0.005	<0.005
Er ug/L	<0.005	<0.005
Eu ug/L	<0.005	0.006
Fe mg/L	<0.05	<0.05
Ga ug/L	<0.02	<0.02
Gd ug/L	<0.005	<0.005
Ge ug/L	<0.02	<0.02
Ho ug/L	<0.005	<0.005
K mg/L	<0.03	<0.03
Li ug/L	<0.1	<0.1
La ug/L	<0.01	<0.01
Lu ug/L	<0.1	<0.1
Mg mg/L	<0.1	<0.1
Mn ug/L	<0.01	0.04
Mo ug/L	<0.2	1.7
Na mg/L	<0.01	0.03
Nb ug/L	<0.02	0.04
Nd ug/L	<0.01	<0.01
Ni ug/L	<0.1	<0.1
P mg/L	<0.01	<0.01
Pb mg/L	<0.08	<0.08
Pr ug/L	<0.01	<0.01
Rb ug/L	<0.01	0.02
Sb ug/L	<0.1	0.43
Sc ug/L	<0.1	<0.1
Se ug/L	<5	<5
Si mg/L	<0.2	<0.2
Sm ug/L	<0.01	<0.01
Sr ug/L	<0.5	0.63
Ta ug/L	<0.02	0.07
Tb ug/L	<0.005	<0.005
Th ug/L	<0.005	0.005
Ti ug/L	<0.1	<0.1
Tl ug/L	<0.05	0.1
Tm ug/L	<0.005	<0.005
U ug/L	<0.005	<0.005
V ug/L	<0.1	0.1
W ug/L	<0.02	0.2
Y ug/L	<0.01	<0.01
Yb ug/L	<0.005	<0.005
Zn mg/L	<0.01	<0.01
Zr ug/L	<0.05	<0.05
SO ₄ ppm	<2	2

Appendix A-2a Analytical Data for Reference Standard NIST 1640 - Relative Percent Difference (RPD)

[ICP-MS]

	Det. Limit	NIST-1640 found	NIST-1640 true	RPD
Ag ug/L	<3	3.8	7.6	67%
Al ug/L	<0.1	55.8	52	7.1%
As ug/L	<3	27.2	26.7	1.9%
Ba ug/L	<0.1	154	148	4.0%
Be ug/L	<0.05	36.1	34.9	3.4%
Bi ug/L	< 0.005	0.0		
Ca mg/L	<0.05	7.2	7.1	1.4%
Cd ug/L	<0.02	23.7	22.8	3.9%
Ce ug/L	<0.01	0.4		
Co ug/L	<0.02	21.4	20.3	5.3%
Cr ug/L	< 1	38.7	38.6	0.3%
Cs ug/L	<0.01	0.2		
Cu ug/L	<0.5	87.6	85.2	2.8%
Dy ug/L	< 0.005	0.0		
Er ug/L	< 0.005	0.0		
Eu ug/L	< 0.005	0.0		
Fe ug/L	<50	<50	34.3	
Ga ug/L	<0.02	<0.02		
Gd ug/L	< 0.005	0.1		
Ge ug/L	<0.02	<0.02		
Ho ug/L	< 0.005	0.0		
Kmg/L	<0.03	0.94	0.9	4.3%
La ug/L	<0.01	0.3		
Li ug/L	<0.1	52.7	50.7	3.9%
Lu ug/L	<0.1	<0.1		
Mg mg/L	<0.01	6.2	5.8	6.7%
Mn ug/L	<0.01	127	122	4.0%
Mo ug/L	<0.2	45.8	46.8	2.2%
Na mg/L	<0.01	30.1	29.4	2.4%
Nb ug/L	<0.02	0.1		
Nd ug/L	<0.01	0.4		
Ni ug/L	<0.1	28.0	27.4	2.2%
Pt ug/L	<0.01	<0.01		
Pb ug/L	<0.05	28.6	27.9	2.5%
Pr ug/L	<0.01	0.1		
Rb ug/L	<0.01	2.1	2	6.3%
Sb ug/L	<0.01	13.9		0.7%
Sc ug/L	<0.1	0.9		
Se ug/L	<5	26.3		
Si mg/L	<0.2	5.0		6.2%
Sm ug/L	<0.01	0.1		
Sr ug/L	<0.5	130		8.0%
Ta ug/L	<0.02	0.1		
Tb ug/L	< 0.005	0.0		
Th ug/L	< 0.005	0.2		
Tiug/L	<0.1	0.8		
Tl ug/L	<0.05	<0.05		
Tm ug/L	< 0.005	< 0.005		
U ug/L	< 0.005	0.9		
V ug/L	<0.1	13.7	13	5.2%
W ug/L	<0.02	0.3		
Yug/L	<0.01	0.2		
Ybug/L	< 0.005	0.0		
Z nuq/L	<0.5	56.1		5.3%
Zr ug/L	<0.05	0.3		
SO4 mg/L	<2	10.0		

Appendix A-2b Analytical Data for Reference Standard NIST 1643d - Relative Percent Difference (RPD)

[ICP-MS]

	Det. Limit	NIST-1643d found	NIST-1643d true	RPD
Ag ug/L	<3	<3	1.27	
Al ug/L	<0.1	140	128	9.0%
As ug/L	<3	56.7	56	1.2%
Ba ug/L	<0.1	520	510	1.9%
Be ug/L	<0.05	12.9	12.5	3.1%
Bi ug/L	< 0.005	13.6		
Ca mg/L	<0.05	31.8	31	2.5%
Cd ug/L	<0.02	6.5	6.5	0.0%
Ce ug/L	<0.01	0.02		
Co ug/L	<0.02	26.8	25	6.9%
Cr ug/L	<1	19.9	18.5	7.3%
Cs ug/L	<0.01	5		
Cu ug/L	<0.5	21.7	20.5	5.7%
Dy ug/L	< 0.005	< 0.005		
Er ug/L	< 0.005	< 0.005		
Eu ug/L	< 0.005	0.05		
Fe ug/L	<50	125	91.2	31.3%
Ga ug/L	<0.02	<0.02		
Gd ug/L	<0.005	<0.005		
Ge ug/L	<0.02	0.1		
Ho ug/L	<0.005	<0.005		
K mg/L	<0.03	2.4	2.4	0.0%
La ug/L	<0.01	0.02		
Li ug/L	<0.1	17.3	16.5	4.7%
Lu ug/L	<0.1	<0.1		
Mg mg/ L	<0.01	8.6	8	7.2%
Mn ug/L	<0.01	41	37.7	8.4%
Mo ug/L	<0.2	120	113	6.0%
Na mg/L	<0.01	22.1	22.1	0.0%
Nb ug/L	<0.02	0.1		
Nd ug/L	<0.01	<0.01		
Ni ug/L	<0.1	60.8	58.1	4.5%
P mg/L	<0.01	<0.01		
Pb ug/L	<0.05	18.6	18.2	2.2%
Pr ug/L	<0.01	<0.01		
Rb ug/L	<0.01	12.7	13	2.3%
Sb ug/L	<0.1	55	54.1	1.6%
Sc ug/L	<0.1	0.7		
Se ug/L	<5	11.4		
Si mg/L	<0.2	3.1	2.7	13.8%
Sm ug/L	<0.01	<0.01		
Sr ug/L	<0.5	289	295	2.1%
Ta ug/L	<0.02	0.1		
Tb ug/L	<0.005	<0.005		
Th ug/L	<0.005	0.1		
Ti ug/L	<0.1	0.5		
Tl ug/L	<0.05	7.1	7.3	2.8%
Tm ug/L	<0.005	<0.005		
U ug/L	<0.005	0.0		
V ug/L	<0.1	38.7	35.1	9.8%
W ug/L	<0.02	0.28		
Y ug/L	<0.01	0.0		
Yb ug/L	<0.005	<0.005		
Zn ug/L	<0.5	74.2	72.5	2.3%
Zr ug/L	<0.05	0.05		
SO4 mg/L	<2	<2		

Table A-3. Analytical Batch Duplicates by ICP - Relative Percent Difference (RPD)

[Data in bold ICP-AES, all other ICP-MS, ND=not detected]

Element:units	Det.Limit	YUK	YUK DUP.	RPD
Ag ug/L	<3	<3	<3	ND
Al mg/L	<0.01	10.4	10.9	4.7
Asug/L	<3	<3	<3	ND
Ba ug/L	<0.1	35.5	34	0.0
Be ug/L	<0.05	0.9	1.0	10.5
Bi ug/L	<0.005	0.01	0.009	10.5
Ca mg/L	<0.1	37.8	40.3	6.4
Cd ug/L	<0.02	6.4	6.4	0.0
Ce ug/L	<0.01	6.6	6.3	4.7
Co ug/L	<0.02	24.1	24.4	1.2
Cr ug/L	<1	3.5	3.3	5.9
Cs ug/L	<0.05	0.38	0.34	11.1
Cu mg/L	<0.01	0.49	0.52	5.9
Dy ug/L	<0.005	0.62	0.65	4.7
Er ug/L	<0.005	0.21	0.24	13.3
Eu ug/L	<0.005	0.2	0.19	5.1
Fe mg/L	<0.05	16.2	16.9	4.2
Ga ug/L	<0.02	0.04	0.06	40.0
Gd ug/L	<0.005	0.82	0.85	3.6
Ge ug/L	<0.02	<0.02	<0.02	ND
Ho ug/L	<0.005	0.098	0.097'	1.0
K mg/L	<0.03	1.2	1.3	8.0
La ug/L	<0.01	2.9	2.8	3.5
Li ug/L	<0.1	11	10.8	1.8
Lu ug/L	<0.1	<0.1	<0.1	ND
Mg mg/L	<0.1	3.7	3.9	5.3
Mn ug/L	<0.01	1600	1600	0.0
Mo ug/L	<0.2	0.46	0.43	6.7
Na mg/L	<0.1	<0.1	0.1	ND
Nb ug/L	<0.02	0.1	0.1	0.0
Nd ug/L	<0.01	3.3	3.2	3.1
Ni ug/L	<0.1	22.7	22.3	1.8
P mg/L	<0.01	<0.01	<0.01	ND
Pb mg/L	<0.08	0.11	0.09	10.5
Pr ug/L	<0.01	0.85	0.83	2.4
Rb ug/L	<0.01	2.9	2.9	0.0
Sb ug/L	<0.1	2.4	2.4	0.0
Sc ug/L	<0.1	2.6	2.7	3.8
Se ug/L	<5	<5	<5	ND
Si mg/L	<0.2	1.5	1.5	0.0
Sm ug/L	<0.01	0.73	0.73	0.0
Sr ug/L	<0.5	52.8	54.6	3.4
Ta ug/L	<0.02	0.22	0.21	4.7
Tb ug/L	<0.005	0.12	0.13	8.0
Th ug/L	<0.005	2.6	2.3	12.2
Ti ug/L	<0.1	6.4	5.8	9.8
Ti ug/L	<0.05	<0.05	<0.05	ND
Tm ug/L	<0.005	0.03	0.02	40.0
U ug/L	<0.005	1.0	1.0	0.0
V ug/L	<0.1	0.4	0.5	22.0
W ug/L	<0.02	0.2	0.2	0.0
Yug/L	<0.01	3	3	0.0
Yb ug/L	<0.005	0.12	0.15	22.0
Zn mg/L	<0.01	1.3	1.3	0.0
Zr ug/L	<0.05	0.5	0.2	85.7
SO4 mg/L	<2	230	230	0.0

Appendix A-4. Leachate Chemistry Data for MAY, YEN, YUK, and SUN

[Data in bold ICP-AES, all other ICP-MS, ND=not detected]

Element:units	Det. Limit	Blank	MAY	VEN	SUN	YUK
Aq ug/L	<3	<3	<3	<3	<3	<3
Al mg/L	<0.01	<0.01	0.42	13.1	6.6	10.4
As ug/L	<3	<3	<3	<3	4.8	<3
Ba ug/L	<0.1	44.1	79.5	54.4	64.6	35.5
Be ug/L	<0.05	<0.05	0.2	1.4	1	0.9
Bi ug/L	< 0.005	0.06	0.12	0.03	0.01	0.01
Ca mg/L	<0.1	0.11	2.5	S.5	5.4	37.8
Cd ug/L	<0.02	< 0.02	6.12	13.3	25.4	6.41
Ce ug/L	<0.01	<0.01	0.46	21.2	5.84	6.62
Co ug/L	<0.02	<0.02	3.69	15.2	8.27	24.1
Cr ug/L	<1	<1	<1	30.1	9.8	3.5
Cs ug/L	<0.01	<0.01	0.54	0.21	0.57	0.38
Cu mg/L	<0.01	<0.01	0.12	4	0.22	0.5
Dy ug/L	< 0.005	< 0.005	0.05	2.97	2.15	0.62
Er ug/L	< 0.005	< 0.005	0.02	1.63	1.24	0.21
Eu ug/L	< 0.005	0.006	0.03	0.76	0.49	0.2
Fe mg/L	<0.05	<0.06	0.6	28.7	13.8	16.2
Ga ug/L	<0.02	<0.02	<0.02	0.41	0.05	0.04
Gd ug/L	< 0.005	< 0.005	0.063	3.25	1.63	0.82
Ge ug/L	<0.02	>:0.02	<0.02	0.07	<0.02	<0.02
Ho ug/L	< 0.005	< 0.005	0.009	0.58	0.46	0.098
K mg/L	<0.03	<0.03	1.5	1.1	0.96	1.2
Li ug/L	<0.1	<0.1	1.1	4.7	<0.1	11
La ug/L	<0.01	<0.01		11.3	2.82	2.93
Lu ug/L	<0.1	<0.1	<0.1	0.2	0.2	<0.1
Mg mg/L	<0.1	<0.1	0.74	6.4	0.37	3.7
Mn ug/L	<0.01	0.04	213	1170	770	1560
Mo ug/L	<0.2	1.74	0.74	0.59	0.44	0.46
Na mg/L	<0.01	0.03	0.26	0.08	0.09	0.1
Nb ug/L	<0.02	0.04	0.04	0.07	0.05	0.1
Nd ug/L	<0.01	<0.01	0.23	10.3	3.29	3.34
Ni ug/L	<0.1	<0.1	5.6	43.8	17.2	22.7
P mg/L	<0.01	<0.01	<0.01	<0.01	0.02	<0.01
Pb mg/L	<0.08	<0.08	2.9	0.78	1.2	0.11
Pr ug/L	<0.01	<0.01	0.06	2.56	0.74	0.85
Rb ug/L	<0.01	0.02	3.07	5.96	3.75	2.88
Sb ug/L	<0.1	0.43	2.62	0.41	5.02	2.43
Sc ug/L	<0.1	<0.1	<0.1	3.2	0.4	2.6
Se ug/L	<5	<5	<5	<5	<5	<5
Si mg/L	<0.2	<0.2	1.5	1.1	0.9	1.5
Sm ug/L	<0.01	<0.01	0.06	2.67	1	0.73
Sr ug/L	<0.5	0.63	31.3	43.3	52.6	52.8
Ta ug/L	<0.02	0.07	0.08	0.1	0.07	0.22
Tb ug/L	< 0.005	< 0.005	0.01	0.54	0.32	0.12
Th ug/L	< 0.005	0.005	0.03	7.79	0.32	2.63
Ti ug/L	<0.1	<0.1	0.2	5	2.2	6.4
Tl ug/L	<0.05	0.1	0.08	0.07	<0.05	<0.05
Tm ug/L	< 0.005	< 0.005	< 0.005	0.19	0.17	0.03
U ug/L	< 0.005	< 0.005	0.17	11.5	2.24	1.02
V ug/L	<0.1	0.1	<0.1	0.5	0.1	0.4
W ug/L	<0.02	0.2	0.2	0.25	0.1	0.2
Y ug/L	<0.01	<0.01	0.22	16.1	13.4	2.96
Yb ug/L	< 0.005	< 0.005	0.01	1.31	1.12	0.12
Zn mg/L	<0.01	<0.01	1	0.96	3.6	1.3
Zr ug/L	<0.05	<0.05	0.06	0.1	0.06	0.5
SO4 ppm	<2	2	43	330	184	231