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# U.S. DEPARTMENT OF THE INTERIOR U.S. GEOLOGICAL SURVEY

#### EPA METHOD 1312 (SYNTHETIC PRECIPITATION LEACHING PROCEDURE) LEACHATE CHEMISTRY DATA FOR SOLID MINE WASTE COMPOSITE SAMPLES FROM SILVERTON AND LEADVILLE, COLORADO

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## Abstract

The EPA Method 1312 Synthetic Precipitation Leaching Procedure (SPLP) leachate chemistry data for mine waste samples is presented. Four mine waste composite samples were collected from Silverton (YUK and MAY) and Leadville (SUN and VEN), Colorado and were extracted following the EPA Method 1312 procedure. Leachate pH values ranged from 2.68 to 3.37 and the samples were ranked by increasing pH: VEN < SUN < YUK < MAY. Specific conductivity values ranged from 203  $\mu$ S to 1080  $\mu$ S and the samples were ranked by increasing specific conductivity: MAY < YUK < SUN < VEN. For selected metal concentrations, no consistent pattern presented itself. MAY is the sample that typically has the lowest metal concentrations with the exception of nickel, zinc, and lead. The samples had SPLP leachate concentrations below the EPA Method 1311 Toxicity Characteristic Leaching Procedure (TCLP) regulatory levels.

## Introduction

## Metal Contamination from Mining Waste

Accurately assessing metal mobility from abandoned mine waste piles is an area of concern for land management agencies. Drainage and runoff from mine waste piles can present water quality concerns similar to acid mine drainage. Mine waste piles that contain potential metal and sulfide reservoirs can become sources of acidic drainage when exposed to the surface conditions of oxygen and water. Work that has begun on abandoned mine lands projects have demonstrated a need for a method to aid land management agencies in characterizing and prioritizing mine waste piles for remediation. Current leach methods suggested by the U.S. Environmental Protection Agency, Toxicity Characteristic Leaching Procedure (TCLP) and Synthetic Precipitation Leaching Procedure (SPLP), were developed to determine toxicity for disposal purposes and not intended to accurately predict surface runoff chemistry from mine waste piles. The effects of particle size reduction, endover-end rotary agitation, and extended extraction time (18 hours) are believed to remove most realistic approximations of simulating runoff from mine waste. However, the SPLP (EPA Method 1312) is often used as one of several tools in prediction estimates for water chemistry associated with solid mine wastes.

#### Purpose of Investigation

The purpose of the original leach study was to conduct a comparison of solid mine waste leachate chemistry utilizing several different extraction, also known as leach, methods. The leachate chemistry results for different methods were to be compared with each other for a particular location and compared with actual rainfall runoff chemistry when available. The results of this comparative study were to be utilized in the development of a simpler, more realistic leach procedure. This leach procedure would be a modification of the EPA Method 1312 method and, while developed for laboratory use, could be quickly modified for field use in preliminary characterization of solid mine waste. This paper presents the solid mine waste composite samples' leachate chemistry derived from one leach method, the EPA Method 1312.

#### General Methods of Study

The main goal of this study was to determine the resulting leachate chemistry from solid mine wastes that were subjected to the EPA Method 1312 Synthetic Precipitation Leaching Procedure (SPLP). Four solid mine waste sample composites were prepared for use in leach studies. The resulting leachate from the experiments was filtered and aliquots of the filtrate were taken for metals, anion, and acidity analyses. Measurements were made for pH, specific conductivity, and temperature on both unfiltered and filtered aliquots of the leachate.

## Methods

#### Sampling

#### Sampling Locations

Four samples were collected from two mining districts in Colorado. Two samples, Mayday (MAY) and Yukon (YUK), were collected from the Silverton district in southwestern Colorado in July, 1997. The sampling sites are located along Cement Creek north of Silverton in San Juan County (37°15'00" 107°40'00"). Two samples, Sunday No. 2 (SUN) and Venir (VEN), were collected from the eastern side of the Leadville mining district in Lake County (39°15'00" 106°17'30"), near the middle of the northeast-trending Colorado mineral belt during August, 1997.

#### **Collection of Mine Waste Composite Samples**

At each location, the Mine Waste Characterization Project members delineated a grid of approximately 30 cells for sampling on the mine waste pile. Thirty subsamples were taken randomly from the pile surface (upper 6 inches/15 cm) within each individual grid cell. The cell composite subsamples were collected with a steel trowel, placed in a plastic bucket, and then mixed together in a plastic dishpan to produce the grid cell composite. A subsample of the grid cell composite was stored in a paper soil sampling bag to aid in drying of the sample. All of the grid cell composite samples were then composited into one large mine waste pile sample composite representing all of the grid cells.

Upon return to the laboratory, the mine waste pile composite samples were spread out on plastic sheets and air dried. Samples were turned once daily with plastic shovels until it was visually determined that the samples were completely dry. The dried composite samples were then mixed in a V-blender to break up solidified clods. Each composite was then sieved with a 2 mm screen, with the < 2 mm fraction being re-homogenized in the V-blender for use in these experiments. The mine waste pile sample composites were the samples used for the SPLP leach experiments (P. Hageman, unpublished notes).

#### EPA Method 1312 (SPLP) Leach Test

The Synthetic Precipitation Leaching Procedure (SPLP) (U.S. EPA Method 1312; U.S. Environmental Protection Agency, 1986; 1994 update)) is a method designed by the Environmental Protection Agency to evaluate the impact of contaminated soils on groundwater. Although the SPLP method closely parallels the Toxicity Characteristic

Leaching Procedure (TCLP), the extraction fluid consists of slightly acidified deionized water that is designed to simulate precipitation. A mixture of 60/40  $H_2SO_4/HNO_3$  (by weight) is used to achieve the appropriate pH for the extraction fluid. The pH of the deionized water is adjusted with the 60/40  $H_2SO_4/HNO_3$  mixture, depending on which side of the Mississippi River the soils originate from, to either pH 4.2  $\pm$  0.05 (east of the Mississippi) or pH 5.0  $\pm$  0.05 (west of the Mississippi). The SPLP procedure mandates that for wastes, the more acidic pH 4.2 extraction fluid be used. For this study, the pH 4.2 extraction fluid was used for all samples because they were all mine wastes.

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 then extracting the solid at a 20:1 liquid to solid ratio on an end-over-end rotary agitator for 18 hours. The solid/liquid slurry is then filtered through a 0.7  $\mu$ m borosilicate glass fiber filter utilizing a pressure filtration unit. Since the mine waste composites samples were sieved to utilize the <2 mm fraction, there was no need for particle size reduction.

Clean extraction vessels (Nalgene® high density polyethylene 2-liter bottles) were rinsed twice with 10 mL of pH 4.2 extraction solution and the rinses discarded. One hundred grams of each sample were weighed out and placed in extractor vessels. Two liters of pH 4.2 extraction solution were slowly added to the vessel. The extractor vessels had Teflon® tape wrapped around the bottle threads to create a tight seal and minimize leaks when the caps were closed. The extractor vessels were secured in an Analytical Testing Rotary Agitator (Model DC-20B) and rotated for 18 hours at 28 r.p.m.

After 18 hours of extraction, the samples were filtered through a new 0.7  $\mu$ m borosilicate glass fiber filter (Gelman Sciences Inc. P/N 66257, TCLP glass fiber filter, 0.7  $\mu$ m pore size, 142 mm diameter). The filtration unit was a Gelman Sciences Hazardous Waste pressure filtration unit (Gelman Sciences Product No. 15046). The filters were acid-washed with one liter of 1 N HNO<sub>3</sub> followed by three one liter deionized water rinses prior to filtration of the samples. A small aliquot of unfiltered leachate was taken for pH and specific conductivity measurements. After filtration, the pH, specific conductivity, and the temperature of the filtrates were measured and recorded.

Aliquots of the filtrate (SPLPA) were taken for four different analyses. An aliquot designated for cations and metals measurement by inductively coupled plasma-atomic emission spectrometry (ICP-AES) and inductively coupled plasma mass spectrometry (ICP-MS) was preserved by acidification to  $pH \leq 1.5$  with Ultrex® II Ultrapure nitric acid. An unacidified aliquot designated for ion chromatography (IC) analysis to determine selected anion concentrations was preserved by refrigeration. An aliquot was taken for ferrous iron concentration measurement. Approximately 60 mL of filtrate, stored in an amber container, was acidified with 10 drops of concentrated HCl to prevent oxidation of ferrous iron during storage. Another unacidified aliquot of filtrate was designated for acidity titrations. Approximately 125 mL of filtrate was refrigerated until time of analysis.

## Analytical Methods

Methods employed to analyze the various parameters are discussed below. Table 1 lists the methods, sample preservation techniques, and recommended holding times for the analytes. Analyses for this study were performed by U.S. Geological Survey personnel in the Central Region Mineral Resources Program, Denver, CO.

Parameter/		Recommended	
Component	Sample Treatment	Holding Time	Type of Analysis
pН	None	Immediate Analysis	pH meter
Conductivity	None	Immediate Analysis	Conductivity meter
Temperature	None	Immediate Analysis	Temp. probe on Cond. meter
Metals	Filter, Acidify with HNO3	180 days	ICP-MS, ICP-AES
Anions	Filter, 4°C	28 days (SO4)	Ion Chromatography
Acidity	Filter, 4°C	as soon as possible	Titration
Ferrous Iron	Filter, Acidify with HCl	2 weeks	Colorimetric

Table 1. List of parameters and components measured

### **Project Laboratory**

#### pH

pH was measured using an Orion Model 230A pH meter and Orion Combination pH electrode (Orion electrode no. 915600). At the beginning of each set of measurements the instrument was calibrated with commercial buffer solutions, bracketing the expected range of the samples to be measured (either pH 4 and 7 or pH 1.68 and 4). A pH buffer check on a third buffer (either pH 6 or 3 depending on calibration range) was performed to check the calibration of the instrument prior to beginning measurements and prior to measurements for each sample

### Specific Conductance

Specific Conductance ( $\mu$ S) was measured with Orion Conductivity Meter Model 126 and an Orion conductivity cell (Orion No. 012210). The calibration of the instrument was checked with commercial conductivity standards prior to each set of measurements.

#### Temperature

Temperature was measured in degrees Centigrade (°C) by the Orion Conductivity Meter Model 126 and an Orion conductivity cell (Orion No. 012210). Temperature was measured in conjunction with the specific conductivity measurements.

#### Metals

Concentrations of cations and metals in the leachates were determined by inductively coupled plasma - mass spectroscopy (ICP-MS) (Meier, et al., 1994) and/or inductively coupled plasma - atomic emission spectroscopy (ICP-AES) (Briggs and Fey, 1996). The analyses were performed using U.S. Geological Survey analytical equipment by USGS personnel. Results are expressed as ug/L (ppb) or mg/L (ppm)

#### Anions

Concentrations (ppm) of selected anions were determined by ion chromatography (IC) (d'Angelo and Ficklin, 1996). Anions measured were fluoride, chloride, nitrate, and sulfate. The analyses were performed using U.S. Geological Survey analytical equipment by USGS personnel.

### Ferrous Iron (Fe<sup>2+</sup>)

Dissolved ferrous iron concentrations (ppm) were determined using the Hach D/R 2000 portable spectrophotometer and Hach AccuVac<sup>™</sup> Ferrous Iron reagent ampoules. Ferrous iron was determined by 1,10-phenanthroline utilizing Hach procedure 8146 (©Hach Company, 1988).

#### Acidity

Acidity titrations were performed on the samples because all pH values were less than 4.5. Acidity was determined using a titration method modified from ASTM test method C (ASTM, 1991) which is intended specifically for mine drainage and other similar waters. If necessary, the sample pH is lowered to less than 4 with sulfuric acid ( $H_2SO_4$ ) to neutralize volatile components. Hydrogen peroxide ( $H_2O_2$ ) is added to the sample prior to boiling to accelerate the complete oxidation of iron, along with hydrolysis of ferric and other metal salts. The cooled sample is then titrated with sodium hydroxide (NaOH) to endpoints pH 3.7 and 8.3. The titration end-points were determined electrometrically, using the same equipment as for leachate pH measurements.

### **Quality Control**

Table 2 shows the frequency and control limits of laboratory quality control checks used for leachate samples obtained from the leach methods in this study. A brief description and definition of each type of quality control check during the project follows. Results of quality control checks are presented in Appendix A.

Quality Control Check	Frequency*	Control Limits
Preparation Blank	one per experimental batch	<idl chemistry<="" extraction="" fluid="" or="" td="" unprocessed=""></idl>
Matrix Spikes	one per experimental batch	75 - 125%
Analytical Batch Duplicates	one per experimental batch	± 20% RPD

Table 2. Laboratory Quality Control Ch	ecks.
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\*U.S. EPA, 1990, Document ILMO1.0 Statement of Work for Inorganic Analysis

#### 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, or procedures introduce any contaminants to the samples. One preparation blank was run with the SPLP analytical batch.

#### Matrix Spikes

Matrix spikes are filtrate samples that are spiked with selected aqueous metal standards prior to preservation to provide information about any matrix effects during the analytical methods. Matrix spikes for lead (5 ppm Pb) were prepared for the SPLP analytical batch.

#### Analytical Batch Duplicates

Analytical batch duplicates are duplicate samples that are carried through the entire experimental process including analysis to determine the precision of the analytical results. One duplicate was run with the SPLP analytical batch.

## EPA Method 1312 Leachate Chemistry Data

Selected leachate chemistry data for the different extractions are listed in Table 3. The complete data set for all elements analyzed is listed in Appendix B. The data in Appendix B lists all elements run on either ICP-AES, ICP-MS, or IC. Several ICP-AES elements (Ag, As, Be, Li, P, Ti, V) that were below detection for the majority of the samples and had ICP-MS data available were omitted from Appendix B. ICP-AES concentrations were used for major cations and selected metals (Al, Ca, Fe, K, Mg, Na). ICP-MS concentrations for lead (Pb) were utilized for this study.

## pН

pH values for the samples ranged from 2.68 (VEN) to 3.37 (MAY). Other pH values were 2.77 (SUN) and 3.09 (YUK). All mine waste samples had a decrease in pH from the starting extraction fluid pH of 4.23. The pH of the blank (EF1) remained at the starting pH of the extraction fluid (4.23). The sample pH values ranked VEN < SUN < YUK < MAY.

### Specific Conductivity

Values for specific conductivity measurements ranged from 203  $\mu$ S (MAY) to 1080  $\mu$ S (VEN). Both YUK and SUN had significant specific conductivity measurements also, with 657  $\mu$ S and 801  $\mu$ S, respectively. The blank had a specific conductivity measurement of 25  $\mu$ S. The sample specific conductivity values ranked opposite of pH: MAY < YUK < SUN < VEN.

## Metals

#### Aluminum (Al)

Aluminum concentrations ranged from 0.43 mg/L (MAY) to 14 mg/L (VEN). SUN and YUK had aluminum concentrations of 6.6 mg/L and 13 mg/L, respectively. The blank did not have detectable aluminum.

#### Arsenic (As)

Arsenic was detected in SUN (2.5  $\mu$ g/L), VEN (0.6  $\mu$ g/L), and YUK (0.6  $\mu$ g/L) but was not detected in MAY and the blank (EF1).

#### Cadmium (Cd)

Cadmium was below detection (<10  $\mu$ g/L) for MAY, YUK, and the blank. Detectable cadmium concentrations were 12  $\mu$ g/L (VEN) and 24  $\mu$ g/L (SUN).

#### Cobalt (Co)

Cobalt concentrations were 14  $\mu$ g/L (SUN), 21  $\mu$ g/L (VEN), and 32  $\mu$ g/L (YUK). Cobalt was not detected in the blank and MAY.

#### Chromium (Cr)

Chromium was detected for one sample. VEN had a chromium concentration of 24 µg/L.

			Extraction					Specific	Specific						
		Leach	Fluid	Sample	Temperature	pН	pН	Conductance	Conductance	Acidity as	F	CI	SO4	NO3	Fe2+
Sample	Date	Method	рН	Weight (g)	degrees C	pre-filter	post-filter	pre-filter (uS)	post-filter (uS)	mg/L CaCO3	ppm	ppm	ppm	ppm	mg/L
EFISPLPA	3/4/98	SPLP	4.23	0	23.3	4.25	4.23	24.0	25.0	5	<0.1	<0.1	23	1.3	0.01
MAYSPLPA	3/4/98	SPLP	4.23	100.00	23.5	3.37	3.38	202	203	31	0.4	0.2	110	1.4	0.48
SUNSPLPA	3/4/98	SPLP	4.23	100.00	23.2	2.77	2.78	803	801	176	1.3	0.6	330	1.6	10.5
VENSPLPA	3/4/98	SPLP	4.23	100.00	23.3	2.67	2.68	1077	1080	273	0.9	0.9	740	2.6	19.5
YUKSPLPA	3/4/98	SPLP	4.23	100.00	22.8	3.09	3.10	657	658	151	20	0.7	530	1.7	16.7

Table 3. Selected Method 1312 Leachate Chemistry Data

	Al	As	Ba	Be	Ca	Cd	Ce	Co	Cr	Cu	Fe	к	La	Li	Mg
Sample	mg/L	ug/L	ug/L	ug/L	mg/L	ug/L	ug/L	ug/L	ug/L	ug/L	mg/L	mg/L	ug/L	ug/L	mg/L
EFISPLPA	< 0.01	< 0.2	21	< 0.05	<0.1	<10	< 0.01	<10	<10	<10	<0.05	<0.5	< 0.01	< 0.1	<0.1
MAYSPLPA	0.43	< 0.2	73	0.2	2.3	<10	0.2	<10	<10	110	0.47	1.5	< 0.01	4.8	0.79
SUNSPLPA	6.6	2.5	53	1.3	6.6	24	4.4	14	<10	220	13	0.89	< 0.01	3.3	0.42
VENSPLPA	14	0.6	52	1.4	6.2	12	17	21	24	3900	27	1.1	6.7	7.5	7.2
YUKSPLPA	13	0.6	29	0.9	47	<10	5.0	32	<10	620	17	1.3	< 0.01	14	4.6

Anions by IC. Concentrations in bold by ICP-AES. Concentrations in italics by ICP-MS.

## Table 3. Selected Method 1312 Leachate Chemistry Data (continued)

	Mn '	Na	Ni	Pb	Si	Sr	Th	Ti	U	V	Zn
Sample	ug/L	mg/L	ug/L	ug/L	mg/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
EFISPLPA	<10	<0.1	<10	< 0.05	<0.1	<10	< 0.005	< 0.1	< 0.005	< 0.1	11
MAYSPLPA	190	0.21	<10	2200	1.9	26	< 0.005	0.2	0.12	< 0.1	980
SUNSPLPA	700	<0.1	17	1100	1.4	50	0.09	1.2	1.6	< 0.1	3800
VENSPLPA	990	<0.1	38	820	1.3	39	5.5	2.1	8.4	0.5	970
YUKSPLPA	1500	0.10	22	86	2.0	50	1.7	4.2	0.73	0.1	1400

Anions by IC. Concentrations in bold by ICP-AES. Concentrations in italics by ICP-MS.

### Copper (Cu)

Concentrations for copper ranged from 110  $\mu$ g/L (MAY) to 3900  $\mu$ g/L (VEN). SUN and YUK had copper concentrations of 220  $\mu$ g/L and 620  $\mu$ g/L, respectively. Copper was not detected in the blank.

## Ferrous Iron (Fe<sup>2+</sup>)

The blank concentration for ferrous iron registered at the detection limit of the spectrophotometer, 10  $\mu$ g/L. Ferrous iron was detected for the other samples and concentrations ranged from 0.48 mg/L (MAY) to 19.5 mg/L (VEN). SUN and YUK had concentrations of 10.5 mg/L and 16.7 mg/L, respectively.

### Iron (Fe)

Total iron concentrations ranged from 0.47 mg/L (MAY) to 27 mg/L (VEN). YUK and SUN had midrange concentrations of 13 mg/L and 17 mg/L, respectively. Iron was not detectable in the blank.

### Manganese (Mn)

Concentrations for manganese ranged from 190  $\mu$ g/L (MAY) to 1500  $\mu$ g/L (YUK). Midrange values of 700  $\mu$ g/L and 990  $\mu$ g/L were detected for SUN and VEN, respectively. Manganese was not detected in the blank.

### Nickel (Ni)

Nickel was not detected for MAY and the blank. YUK and SUN had nickel concentrations of 22  $\mu$ g/L and 17  $\mu$ g/L, respectively. VEN had a nickel concentration of 38  $\mu$ g/L.

### Lead (Pb)

Lead concentrations ranged from 86  $\mu$ g/L (YUK) to 2200  $\mu$ g/L (MAY). SUN and VEN had lead concentrations of 1100  $\mu$ g/L and 820  $\mu$ g/L, respectively. Lead was not detected in the blank.

#### Zinc (Zn)

Concentrations for zinc ranged from 970  $\mu$ g/L (VEN) to 3800  $\mu$ g/L (SUN). Values for MAY and YUK were 980  $\mu$ g/L and 1400  $\mu$ g/L, respectively. A zinc concentration of 11  $\mu$ g/L was detected in the blank.

## Sulfate (SO<sub>4</sub>)

Sulfate concentrations ranged from 110 ppm (MAY) to 740 ppm (VEN). Concentrations for SUN and YUK were 330 ppm and 530 ppm, respectively. The blank had a sulfate concentration of 23 ppm.

## Acidity

Acidity concentrations ranged from 31 mg/L CaCO<sub>3</sub> (MAY) to 273 mg/L CaCO<sub>3</sub> (VEN). Values for SUN and YUK were 176 and 151, respectively. The blank had an acidity value of 5 mg/L CaCO<sub>3</sub>. The sample values for acidity ranked MAY < YUK < SUN < VEN.

## Summary

The four mine waste composite samples that underwent the EPA Method 1312 (SPLP) procedure had leachate pH values that ranged from 2.68 to 3.37. The samples were ranked by increasing pH: VEN < SUN < YUK < MAY. Specific conductance values ranged from 203  $\mu$ S to 1080  $\mu$ S and the samples were ranked by increasing specific conductance: MAY < YUK < SUN < VEN. Increasing specific conductance rankings were opposite of increasing pH rankings, thus samples with the lowest pH had the highest specific conductance. For selected metal concentrations, no consistent sample ranking pattern presented itself. MAY (highest pH and lowest specific conductance) typically had the lowest metal concentrations with the exception of nickel (Ni), zinc (Zn), and lead (Pb). All samples had SPLP leachate concentrations below the TCLP regulatory levels.

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# Appendices

Appendix A. Quality Control Checks

Appendix B. EPA Method 1312 Leachate Chemistry Data

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#### Appendix A List of Tables

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The data generated from the laboratory was subjected to quality control checks to provide information on the quality and usability of the data. The majority of quality control procedures used were based upon procedures listed in the U.S. Environmental Protection Agency Document ILMO1.0 (1990) Statement of Work for Inorganic Analysis and upon quality control measures specified in the leach procedures.

### **Performance Blanks**

Performance Blank Analytical Results are presented in Table A-1. Samples filtered with a borosilicate glass fiber filter had elevated barium (Ba) concentrations. Extraction fluid 1 (EF1) for the SPLP has pre-filtration barium concentrations below the 0.02  $\mu$ g/L instrument detection limit (M. Montour, unpublished leach parameter study data), but has a post-filtration concentration of 21  $\mu$ g/L.

Molybdenum (Mo) and zinc (Zn) exhibit moderate increases in concentration for the borosilicate glass fiber filtrate relative to the pre-filtration concentrations (M. Montour, unpublished leach parameter study data). In general, all other elements are below instrument detection levels.

## Matrix Spike Recovery

Matrix spike recovery results for the SPLP method are summarized in Table A-2. Matrix spikes were performed only for lead (Pb) because preliminary analyses indicated that this metal from the EPA TCLP regulatory list would be of most interest to the study. If the matrix spike lies outside of the range of 75 to 125 percent spike recovery, the samples need to be flagged (U.S. EPA, 1990). All matrix spikes had recoveries within the acceptable limits.

## **Analytical Batch Duplicates**

Analytical batch duplicate results are presented in Table A-3. Many duplicate samples were below the instrument detection limit of the elements. The samples were generally within the relative percent difference (RPD) control limits of 20 percent. Elements with concentrations near detection limits sometimes exceeded the control limit (F, Ag, Bi, Ce, Er, Gd, In, Re, Sc, Se, Ti, Y). The control limit was exceeded by molybdenum (Mo), with concentrations above the control limit and above the detection limit (0.02  $\mu$ g/L) (M. Montour, unpublished leach parameter study data), and lead (Pb), which slightly exceeded the control limit. It cannot be determined whether the discrepancy between the sample and duplicate is a result of heterogeneity between laboratory subsamples or analytical uncertainty.

Performance Blank	EF1SPLPA	Performance Blank	EF1SPLPA
Analytical Batch	CB3	Analytical Batch	CB3
Date	3/4/98	Date	3/4/98
Leach Method	SPLP	Leach Method	SPLP
Extraction Fluid	60/40	Extraction Fluid	60/40
	H2SO4/HNO3		H2SO4/HNO3
		K (mg/L)	< 0.5
Extraction Fluid pH	4.23	La (ug/L)	< 0.01
Temperature, C	23.3	Li (ug/L)	< 0.1
pH (pre-filter)	4.25	Mg (mg/L)	< 0.1
pH (post-filter)	4.23	Mn (ug/L)	< 1 0
Spec. Cond. (uS, pre-Filter)	24.0	Mo (ug/L)	1.8
Spec. Cond. (uS, post-Filter)	25.0	Na (mg/L)	< 0.1
F (ppm)	<0.1	Nb (ug/L)	< 0.02
Cl (ppm)	<0.1	Nd (ug/L)	< 0.01
SO4 (ppm)	23	Ni (ug/L)	< 1 0
NO3 (ppm)	1.3	P (ug/L)	< 1
Ag (ug/L)	< 0.01	Pb (ug/L)	< 0.05
Al (mg/L)	< 0.01	Pr (ug/L)	< 0.01
As (ug/L)	< 0.2	Rb (ug/L)	< 0.01
Au (ug/L)	< 0.01	Re (ug/L)	< 0.02
B (mg/L)	< 0.01	Sb (ug/L)	0.1
Ba (ug/L)	21	Sc (ug/L)	< 0.1
Be (ug/L)	< 0.05	Se (ug/L)	< 0.2
Bi (ug/L)	< 0.01	Si (mg/L)	< 0.1
Ca (mg/L)	< 0.1	Sm (ug/L)	< 0.01
Cd (ug/L)	< 1.0	Sn (ug/L)	< 0.05
Ce (ug/L)	< 0.01	Sr (ug/L)	< 1 0
Co (ug/L)	< 1.0	Ta (ug/L)	< 0.02
Cr (ug/L)	< 1.0	Tb (ug/L)	< 0.005
Cs (ug/L)	< 0.01	Te (ug/L)	< 0.1
Cu (ug/L)	< 1.0	Th (ug/L)	< 0.005
Dy (ug/L)	< 0.005	Ti (ug/L)	< 0.1
Er (ug/L)	< 0.005	Tl (ug/L)	< 0.05
Eu (ug/L)	< 0.005	Tm (ug/L)	< 0.005
Fe (mg/L)	< 0.05	U (ug/L)	< 0.005
Ga (ug/L)	< 0.02	V (ug/L)	< 0.1
Gd (ug/L)	< 0.005	W (ug/L)	< 0.02
Ge (ug/L)	< 0.02	Y (ug/L)	< 0.01
Hf (ug/L)	< 0.05	Yb (ug/L)	< 0.01
Ho (ug/L)	< 0.005	Zn (ug/L)	11
In (ug/L)	< 0.01	Zr (ug/L)	< 0.05

# Table A-1. Performance Blank Analytical Results

ICP-AES in bold. ICP-MS in italics.

# Table A-2. Matrix Spike Recovery Results

		Leach	Pb	% Recovery
Sample	Date	Method	ug/L	Pb
EF1SPLPA	3/4/98	SPLP	0.05	
EF1SPLPMPB	3/4/98	SPLP	4800	96.0
MAYSPLPA	3/4/98	SPLP	2200	
MAYSPLPMPB	3/4/98	SPLP	7200	100.0
SUNSPLPA	3/4/98	SPLP	1100	
SUNSPLPMPB	3/4/98	SPLP	5700	92.0
VENSPLPA	3/4/98	SPLP	820	
VENSPLPMPB	3/4/98	SPLP	5200	87.6
YUKSPLPA	3/4/98	SPLP	86	
YUKSPLPMPB	3/4/98	SPLP	4800	94.3

	MAYSPLPA	MAYSPLPD	RPD	
Temperature, C	23.5	23.1	1.7	In (ug
pH pre-filter	3.37	3.34	0.9	K (mg
pH post-filter	3.38	3.37	0.3	La (ug
Spec. Cond.	202	205	1.5	Li (ug
pre-filter (uS)				Mg (n
Spec. Cond.	203	205	1.0	Mn (u
post-filter (uS)				Mo (u
Acidity, mg/L CaCO3	31	32	3.2	Na (m
F (ppm)	0.4	0.3	28.6	Nb (u
Cl (ppm)	0.2	0.3	40.0	Nd (u
SO4 (ppm)	110	98	11.5	Ni (ug
NO3 (ppm)	1.4	1.3	7.4	P (ug/
Fe2+(mg/L)	0.48	0.48	0.0	Pb (ug
Ag (ug/L)	0.02	0.04	66.7	Pr (ug
Al (mg/L)	0.43	0.45	4.5	Rb (u
As (ug/L)	< 0.2	< 0.2	ND	Re (ug
Au (ug/L)	< 0.01	< 0.01	ND	Sb (ug
B (mg/L)	< 0.01	< 0.01	ND	Sc (ug
Ba (ug/L)	73	72	1.4	Se (ug
Be (ug/L)	0.2	0.2	0.0	Si (m
Bi (ug/L)	0.04	0.05	22.2	Sm (u
Ca (mg/L)	2.3	2.4	4.3	Sn (ug
Cd (ug/L)	<10	<10	ND	Sr (ug
Ce (ug/L)	0.2	0.3	40.0	Ta (ug
Co (ug/L)	<10	<10	ND	Tb (u
Cr (ug/L)	<10	<10	ND	Te (ug
Cs (ug/L)	0.46	0.46	0.0	Th (u
Cu (ug/L)	110	110	0.0	Ti (ug
Dy (ug/L)	0.04	0.04	0.0	Tl (ug
Er (ug/L)	0.02	0.01	66.7	Tm (u
Eu (ug/L)	0.01	0.01	0.0	U (ug
Fe (mg/L)	0.47	0.51	8.2	V (ug
Ga (ug/L)	< 0.02	< 0.02	ND	W (ug
Gd (ug/L)	0.03	0.04	28.6	Y (ug
Ge (ug/L)	< 0.02	< 0.02	ND	Yb (u
Hf (ug/L)	< 0.05	< 0.05	ND	Zn (u
Ho (ug/L)	0.006	0.006	0.0	Zr (ug

Table A-3. Analytical Batch Duplicates - Re	elative Percent Difference
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	MAYSPLPA	MAYSPLPD	RPD
In (ug/L)	0.02	0.01	66.7
K (mg/L)	1.5	1.4	6.9
La (ug/L)	< 0.01	< 0.01	ND
1.i (ug/1.)	4.8	4.1	15.7
Mg (mg/l.)	0.79	0.79	0.0
Mn (ug/L)	190	190	0.0
Mo (ug/L)	0.4	2.9	151.5
Na (mg/L)	0.21	0.21	0.0
Nb (ug/L)	< 0.02	< 0.02	ND
Nd (ug/L)	0.1	0.1	0.0
Ni (ug/L)	<10	<10	ND
P (ug/L)	3	3	0.0
Pb (ug/L)	2200	2700	20.4
Pr (ug/L)	0.03	0.03	0.0
Rb (ug/L)	2.2	2.3	4.4
Re (ug/L)	0.03	0.04	28.6
Sb (ug/L)	1.9	2.1	10.0
Sc (ug/L)	0.2	0.3	40.0
Se (ug/L)	0.8	1	22.2
Si (mg/L)	1.9	1.8	5.4
Sm (ug/L)	0.04	0.04	0.0
Sn (ug/L)	< 0.05	< 0.05	ND
Sr (ug/L)	26	25	3.9
Ta (ug/L)	< 0.02	< 0.02	ND
Tb (ug/L)	0.007	0.007	0.0
Te (ug/L)	< 0.1	< 0.1	ND
Th (ug/L)	< 0.005	< 0.005	ND
Ti (ug/L)	0.2	0.1	66.7
Tl (ug/L)	< 0.05	0.2	ND
Tm (ug/L)	< 0.005	< 0.005	ND
U (ug/L)	0.12	0.12	0.0
V (ug/L)	< 0.1	< 0.1	ND
W (ug/L)	< 0.02	< 0.02	ND
Y (ug/L)	0.2	0.1	66.7
Yb (ug/L)	< 0.01	< 0.01	ND
Zn (ug/L)	980	1000	2.0
Zr (ug/L)	< 0.05	< 0.05	ND



## Appendix B. EPA Method 1312 Leachate Chemistry Data

Sample	Analytical Batch	Date	Leach Method	Liquid to Solid Ratio	Extraction Fluid Chemistry	Method Extraction Fluid	Particle Size Reduction	Length of Extraction Time (hours)	Method of Agitation	Filtration	Filter Type	Filter Pore Size (um)	Filter Membrane Preparation	Amount of Extraction Fluid (mL)
СВЗАВ	CB3	3/4/98	Acid Blank											
EFISPLPA	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
EFISPLPMPB	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
MAYSPLPA	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
MAYSPLPD	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
MAYSPLPMPB	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
SUNSPLPA	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
SUNSPLPMPB	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
VENSPLPA	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
VENSPLPMPB	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
YUKSPLPA	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000
YUKSPLPMPB	CB3	3/4/98	SPLP	20:1	60/40 H2SO4/HNO3	1	no	18	rotary agitator	positive pressure	borosilicate glass	0.7	acid wash	2000

	Extraction					Specific	Specific													
	Fluid	Sample	Temperature	pН	pН	Conductance	Conductance	Acidity as	F	CI	SO4	NO3	Fe2+	Ag	Al	As	Au	В	Ba	Be
Sample	рН	Weight (g)	degrees C	pre-filter	post-filter	pre-filter (uS)	post-filter (uS)	mg/L CaCO3	ppm	ppm	ppm	ppm	mg/L	ug/L	mg/L	ug/L	ug/L	mg/L	mg/L	ug/L
CB3AB									nm	nm	nm	nm	nm	0.02	<0.01	< 0.2	< 0.01	<0.01	<10	< 0.0.
EFISPLPA	4.23	0	23.3	4.25	4.23	24.0	25.0	5	<0.1	<0.1	23	1.3	0.01	< 0.01	<0.01	< 0.2	< 0.01	<0.01	21	< 0.05
EFISPLPMPB	4.23	0	23.3	4.25	4.23	24.0	25.0	nm	nm	nm	nm	nm	nm	0.03	0.012	< 0.2	< 0.01	< 0.01	21	< 0.05
MAYSPLPA	4.2.3	100.00	23.5	3.37	3.38	202	203	31	0.4	0.2	110	1.4	0.48	0.02	0.43	< 0.2	< 0.01	<0.01	73	0.2
MAYSPLPD	4.23	100.00	23.1	3.34	3.37	205	205	32	0.3	0.3	98	1.3	0.48	0.04	0.45	< 0.2	< 0.01	< 0.01	72	0.2
MAYSPLPMPB	4.23	100.00	23.5	3.37	3.38	202	203	nm	nm	nm	nm	nm	nm	< 0.01	0.43	< 0.2	< 0.01	< 0.01	71	0.2
SUNSPLPA	4.23	100.00	23.2	2.77	2.78	803	801	176	1.3	0.6	330	1.6	10.5	0.02	6.6	2.5	< 0.01	< 0.01	53	1.3
SUNSPLPMPB	4.23	100.00	23.2	2.77	2.78	803	801	nm	nm	nm	nm	nm	nm	0.06	6.5	3.0	< 0.01	< 0.01	52	1.2
VENSPLPA	4.23	100.00	23.3	2.67	2.68	1077	1080	273	0.9	0.9	740	2.6	19.5	0.1	14	0.6	< 0.01	<0.01	52	1.4
VENSPLPMPB	4.23	100.00	23.3	2.67	2.68	1077	1080	nm	nm	nm	nm	nm	nm	0.2	15	0.8	< 0.01	< 0.01	55	1.5
YUKSPLPA	4.23	100.00	22.8	3.09	3.10	657	658	151	20	0.7	530	1.7	16.7	< 0.01	13	0.6	< 0.01	< 0.01	29	0.9
YUKSPLPMPB	4.23	100.00	22.8	3.09	3.10	657	658	nm	nm	nm	nm	nm	nm	0.04	12	0.4	< 0.01	< 0.01	28	0.7

													T	T	T	T	1	1	1	1	1
	Bi	Ca	Cd	Ce	Co	Cr	Cs	Cu	Dy	Er	Eu	Fe	Ga	Gd	Ge	Hf	Но	In	к	La	L
Sample	ug/L	mg/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	mg/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	mg/L	ug/L	ug/
CB3AB	< 0.01	<0.1	<10	< 0.01	<10	<10	< 0.01	<10	< 0.005	< 0.005	< 0.005	< 0.05	< 0.02	< 0.005	< 0.02	< 0.05	< 0.005	< 0.01	< 0.5	< 0.01	0.4
EF1SPLPA	< 0.01	<0.1	<10	< 0.01	<10	<10	< 0.01	<10	< 0.005	< 0.005	< 0.005	< 0.05	< 0.02	< 0.005	< 0.02	< 0.05	< 0.005	< 0.01	<0.5	< 0.01	< 0.1
EFISPLPMPB	0.13	<0.1	<10	< 0.01	<10	<10	< 0.01	<10	< 0.005	< 0.005	< 0.005	< 0.05	< 0.02	< 0.005	< 0.02	< 0.05	< 0.005	< 0.01	<0.5	< 0.01	0.7
MAYSPLPA	0.04	2.3	<10	0.2	<10	<10	0.46	110	0.04	0.02	0.01	0.47	< 0.02	0.03	< 0.02	< 0.05	0.006	0.02	1.5	< 0.01	4.8
MAYSPLPD	0.05	2.4	<10	0.3	<10	<10	0.46	110	0.04	0.01	0.01	0.51	< 0.02	0.04	< 0.02	< 0.05	0.006	0.01	1.4	< 0.01	4.1
MAYSPLPMPB	0.08	2.3	<10	0.2	<10	<10	0.47	110	0.04	0.01	0.01	0.48	< 0.02	0.05	< 0.02	< 0.05	0.007	0.02	1.4		
SUNSPLPA	0.05	6.6	24	4.4	14	<10	0.50	220	1.6	0.94	0.39	13	0.07	1.3	0.03	< 0.05	0.35	0.02	0.89	< 0.01	4.8
SUNSPLPMPB	0.05	6.6	26	4.5	12	<10	0.48	230	1.6	0.97	0.36	13	0.08	1.2	0.03	< 0.05	0.35			< 0.01	3.3
ENSPLPA	0.03	6.2	12	17	21	24	0.2	3900	2.4	1.3	0.64	27	0.3	2.6	0.03	< 0.05	0.32	0.1	0.88	< 0.01	3.3
ENSPLPMPB	0.11	6.5	12	16	22	25	0.2	4200	2.4	1.3	0.61	28	0.4					0.31	1.1	6.7	7.5
UKSPLPA	< 0.01	47	<10	5.0	32	<10	0.30	620	0.48	0.19	0.18			2.4	0.08	< 0.05	0.44	0.30	1.3	6.6	7.5
UKSPLPMPB	0.06	44	<10	5.1	28	<10	0.30	580	0.40	0.19		17	0.08	0.66	0.03	0.4	0.083	0.1	1.3	< 0.01	14
						10	0.00	580	0.45	0.19	0.18	17	0.07	0.59	0.03	0.2	0.082	0.1	1.2	< 0.01	13

								1	1	T	1	T	T	1	1	1	1	1		-	-	
Sample	Mg mg/L	Mn	Mo	Na	Nb	Nd	Ni	Р	РЪ	Pr	Rb	Re	Sb	Sc	Se	Si	Sm	Sn	Sr	Та	ть	т
Sample	mg/L	ug/L	ug/L	mg/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	mg/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug
CB3AB	<0.1	<10	1.6	<0.1	< 0.02	< 0.01	<10	< /	< 0.05	< 0.01	0.02	< 0.02	0.07	< 0.1	< 0.2	<0.1	< 0.01	< 0.05	<10	< 0.02	< 0.005	< 0.
EFISPLPA	<0.1	<10	1.8	<0.1	< 0.02	< 0.01	<10	< 1	< 0.05	< 0.01	< 0.01	< 0.02	0.1	< 0.1	< 0.2	<0.1	< 0.01	< 0.05	<10	< 0.02	< 0.005	< 0.
EFISPLPMPB	<0.1	<10	0.4	<0.1	< 0.02	< 0.01	<10	< 1	4800	< 0.01	0.01	< 0.02	< 0.02	< 0.1	< 0.2	<0.1	< 0.01	< 0.05	<10	< 0.02	< 0.005	< 0
MAYSPLPA	0.79	190	0.4	0.21	< 0.02	0.1	<10	3	2200	0.03	2.2	0.03	1.9	0.2	0.8	1.9	0.04	< 0.05	26	< 0.02	0.007	< 0
MAYSPLPD	0.79	190	2.9	0.21	< 0.02	0.1	<10	3	2700	0.03	2.3	0.04	2.1	0.3	1	1.8	0.04	< 0.05	25	< 0.02	0.007	< 0.
MAYSPLPMPB	0.77	190	1.0	0.23	< 0.02	0.1	<10	8	7200	0.03	2.2	0.04	2.0	0.1	0.8	1.9	0.05	< 0.05	25	< 0.02	0.008	< 0.
SUNSPLPA	0.42	700	0.89	<0.1	< 0.02	2.6	17	20	1100	0.60	2.7	< 0.02	4.9	0.4	0.8	1.4	0.83	< 0.05	50	< 0.02	0.25	< 0.
SUNSPLPMPB	0.42	700	3.2	<0.1	< 0.02	2.5	19	24	5700	0.56	2.7	0.02	5.0	0.4	2	1.3	0.80	< 0.05	48	< 0.02	0.24	< 0.
VENSPLPA	7.2	990	2.0	<0.1	< 0.02	8.6	38	12	820	2.1	4.5	0.02	0.3	2	2	1.3	2.4	< 0.05	39	< 0.02	0.42	< 0.
VENSPLPMPB	7.6	1000	2.7	<0.1	< 0.02	8.2	43	19	5200	2.0	4.4	< 0.02	0.4	2	2	1.4	2.3	< 0.05	41	< 0.02	0.43	< 0.
YUKSPLPA	4.6	1500	1.8	0.10	< 0.02	2.6	22	24	86	0.65	2.1	< 0.02	2.5	2	2	2.0	0.68	< 0.05	50	0.04	0.11	< 0.
YUKSPLPMPB	4.3	1400	1.3	0.15	< 0.02	2.5	22	12	4800	0.65	1.9	< 0.02	2.3	1	1	1.9	0.67	< 0.05	48	0.05	0.10	< 0.

							1	T	1		T
	Th	Ti	ті	Tm	U	v	w	Y	Yb	Zn	Zr
Sample	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L	ug/L
CB3AB	< 0.005	< 0.1	0.06	< 0.005	< 0.005	< 0.1	< 0.02	< 0.01	< 0.01	<10	< 0.05
EFISPLPA	< 0.005	< 0.1	< 0.05	< 0.005	< 0.005	< 0.1	< 0.02	< 0.01	< 0.01	11	< 0.05
EFISPLPMPB	< 0.005	< 0.1	< 0.05	< 0.005	< 0.005	< 0.1	< 0.02	< 0.01	< 0.01	14	< 0.05
MAYSPLPA	< 0.005	0.2	< 0.05	< 0.005	0.12	< 0.1	< 0.02	0.2	< 0.01	980	< 0.05
MAYSPLPD	< 0.005	0.1	0.2	< 0.005	0.12	< 0.1	< 0.02	0.1	< 0.01	1000	< 0.05
MAYSPLPMPB	< 0.005	0.5	0.09	< 0.005	0.12	< 0.1	< 0.02	0.1	0.01	950	0.3
SUNSPLPA	0.09	1.2	< 0.05	0.12	1.6	< 0.1	< 0.02	9.4	0.76	3800	< 0.05
SUNSPLPMPB	0.12	1	0.1	0.12	1.6	< 0.1	< 0.02	9.4	0.76	3800	< 0.05
VENSPLPA	5.5	2.1	0.08	0.17	8.4	0.5	< 0.02	12	0.98	970	< 0.05
VENSPLPMPB	5.5	2.0	0.1	0.16	8.8	0.5	< 0.02	11	0.94	1000	0.05
YUKSPLPA	1.7	4.2	< 0.05	0.02	0.73	0.1	0.08	2.2	0.1	1400	11
YUKSPLPMPB	1.8	3.2	0.08	0.02	0.76	< 0.1	< 0.02	1.9	0.1	1300	15

