



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

**Analytical Data for Reconnaissance Geochemical Samples from  
Mine Dumps, Stream Sediments, and Waters at the  
Thompson Creek Tungsten Mine, Custer County, Idaho**

by

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## **Digital Data Files**

### **PDF (Portable Document Format) files that can be viewed with Adobe Acrobat Reader**

LOCATION.pdf	Index map showing the location of the Thompson Creek tungsten mine
GRANITE.pdf	Plutonic rock classification plot showing the composition of granitic rock of the mine waste dumps

### **Digital photographs—TIFF files (Tagged-Image File Format)**

ADIT.tif	View of the collapsed adit of the Thompson Creek tungsten mine
SOUTH.tif	South-facing view of the mine waste dumps
NORTH.tif	North-facing view of the mine waste dumps
99CH006.tif	View of water sample site 99CH006
99CH007.tif	View of water sample site 99CH007
99CH008.tif	View of water sample site 99CH008

### **Microsoft Excel (.xls) format data files**

DUMP.xls	Geochemical data from composite mine dump samples of surface fine material and altered rocks
GRANITE.xls	Geochemical data for a composite sample of granitic rock from the dumps
SEDIMENT.xls	Geochemical data for <80 mesh stream sediments in Thompson Creek upstream and downstream of the Thompson Creek tungsten mine
WATER.xls	Analytical results and field parameters measured in three samples of surface waters
LEACH.xls	Leachate data for a composite sample of fine surface material from the mine dumps

## Introduction

The Thompson Creek mine is an inactive and abandoned tungsten mine located along Thompson Creek about 10.5 miles (17 km) northwest of Clayton, Idaho (see location map in file LOCATION.PDF). The mine consisted initially of open pit mining of outcrops and later of underground workings accessed by a single adit. The mine was worked through 1955 and waste piles presently reside in the flood plain above the east bank of Thompson Creek. A view of the collapsed adit is provided in the TIFF (Tagged-Image File Format) file ADIT.TIF (all photographs taken on 08/13/99). A south-facing view of the mine waste dumps is provided in the TIFF file SOUTH.TIF and a north-facing view is provided in NORTH.TIF.

On August 13, 1999, personnel of the U.S. Geological Survey (USGS) collected four composite samples of the dump material, two stream sediments, and three samples of waters; samples include waters draining from the mine property and waters and sediments from Thompson Creek above and below the mine site. The U.S. Forest Service is currently (2000) considering reclamation of this property. At the request of the U.S. Forest Service, the USGS provided geochemical analyses in this data release for consideration in their reclamation plan. The analytical data are presented in tables in this report as well as in Microsoft Excel (.xls) file format, separated by sample media.

It must be emphasized that when collected the samples described in this report were considered reconnaissance in nature, collected as part of a regional characterization and sampling of a variety of mineral deposit types that occur in east-central Idaho. The methodology used and data provided in this report are *not* intended to represent a complete characterization of the site and do not follow Environmental Protection Agency (EPA) protocol for Site Investigations.

## Mine History

A one-page description of the history and geology of the Thompson Creek mine was provided by Cook (1956) around the time of the mine development. The following paragraph is an excerpt from Cook (1956, p. 22):

“The deposit was discovered by James Clutus in October, 1953, and it has been developed by the Salmon River Scheelite Corporation. Four hundred ninety-three tons of ore containing an average of 0.77 percent  $WO_3$  have been mined and milled, with a recovery of 288 units of  $WO_3$ . The mining has been by open pit methods at the outcrop. During the summer and fall of 1955 an adit was being driven to intersect the ore zone at a depth of 300 feet below the outcrop.”

Note that the Thompson Creek tungsten mine has also been referred to as the “Tungsten Jim mine” and the “Salmon River Scheelite mine”, and is shown on some maps as the “Scheelite Jim mine”. To be clear, the Thompson Creek mine sampled by this study is located on the east bank of Thompson Creek near the southern boundary of section 33, T. 12 N., R. 16 E. (see location map in file LOCATION.PDF).

From 1951-1955 the price of tungsten concentrate reached historically high values of over \$60 per short ton unit (Ross, 1963, fig. 7). But, the price of tungsten concentrate fell sharply after that period and was about \$18.50 per short ton unit by 1958 (Ross, 1963, p. 23). The short life of

the Thompson Creek tungsten mine probably reflects the up and down economics of the 1950's tungsten market.

### **General Geology**

The tungsten skarn deposit exploited by the Thompson Creek mine is mainly in the form of scheelite (calcium tungstate, ideal chemical formula of  $\text{CaWO}_4$ ). It formed along the contacts of bedded argillite and thin interbedded limestone with a large granitic body. The argillite-limestone sequence was mapped by Fisher and others (1992) as part of the Grand Prize Formation of Lower Permian age. This unit is described regionally by Hall (1985, p. 124-125) to consist of "a thick sequence of interbedded light- to medium-gray limestone; fine-grained quartzite; dark-gray, banded siltite; and medium- to dark-gray, carbonaceous, silty to sandy limestone". The granite body appears to be a part of the Cretaceous Pat Hughes intrusive complex that hosts the Thompson Creek molybdenum deposit, site of the active (2000) Thompson Creek molybdenum mine located about 2.5 miles (4 km) to the southeast of the Thompson Creek tungsten mine (LOCATION.PDF). Deposits of molybdenite occur with stockwork veins and veinlets of quartz-biotite-muscovite-orthoclase in the core of the Pat Hughes (or Thompson Creek) intrusive complex (Schmidt and others, 1982; Hall and others, 1984).

Granitic rocks in the dumps of the Thompson Creek tungsten mine are similar to those that outcrop just east of the mine. The igneous rocks in the dump material are mostly weakly silicified, but more silicified samples contain tourmaline (variety elbaite, identified in thin section) and trace amounts of fine-grained, disseminated pyrrhotite and molybdenite. Biotite is abundant in the rock; the biotite grains are mostly unaltered, and in places thinly rimmed by iron oxide minerals. A whole-rock geochemical analysis of a composite sample (74BV99) of igneous rocks from the mine dump material indicates it is granite in composition (see rock classification plot in file GRANITE.PDF). This geochemical analysis also revealed a loss on ignition (when heated above  $925^\circ\text{C}$ ) of 1.1 percent, typical of relatively unaltered granite.

At the Thompson Creek molybdenum deposit the host stock is complexly zoned with a biotite granodiorite forming the border zone and enclosing a core of quartz monzonite (Schmidt and others, 1982). Potassium-argon age determinations of primary biotite from the granodiorite indicate an age of  $88.4 \pm 3$  million years (Schmidt and others, 1982). "Seven potentially distinct phases have been recognized within the outer border sequence, and more may be present," according to Schmidt and others (1982, p. 81). The granite common in the dumps of the Thompson Creek tungsten mine and in outcrops east of the mine likely is genetically related to the Pat Hughes (Thompson Creek) intrusive complex.

The description below of the tungsten-rich, argillite-intrusive contact zone at the Thompson Creek tungsten mine is directly quoted from Cook (1956, p. 22). The "quartz monzonite" described by Cook is the rock our chemical analysis shows is granite in composition.

"This contact, which is exposed on the east side of Thompson Creek, 300-400 feet above the creek, strikes northerly and dips steeply eastward into the hill. The hanging wall of the ore zone is quartz monzonite. The contact is irregular and shows much evidence of faulting. The granitic rock has been hydrothermally altered (bleached) up to ten feet from the contact. Large pods of tactite, formed mainly by replacement of limy layers in the Wood River formation [now mapped as the Grand Prize Formation] but partly at the

expense of the granitic rock, are found in the contact zone. This tactite consists of fine-grained garnet, pyrite or pyrrhotite, and quartz. Although the scheelite is found in altered granite as well as tactite, the richer concentrations are in tactite which contains vitreous quartz and is therefore hard and resistant. White-fluorescing scheelite is the main ore mineral although yellow-fluorescing powellite coats fractures in the tactite. The actual ore width ranges from almost nothing to 6 feet, although the ore zone averages perhaps 20 ft in width for 800 ft along the contact; this zone contains pods of ore, much barren tactite and considerable unreplaced argillite and limestone. The footwall of the ore zone is siliceous argillite. The quartz monzonite body is wedge-shaped in plan and tapers out at the southern end of the mined area; this may control the limit of ore in that direction.”

Photographs of fluorescing scheelite from the mine are provided in Cookro (1985, fig. Q7, p. 201). The powellite mentioned above is the calcium molybdate ( $\text{CaMoO}_4$ ) end member of the scheelite-powellite solid solution series. Powellite is commonly found with scheelite near molybdenum deposits.

Rock fragments from the dumps below the mine exhibit an array of alteration, including:

- (1) Relatively fresh granite typically, with minor silicification but locally containing moderate silicification, tourmaline, and disseminated fine-grained pyrrhotite and molybdenite;
- (2) Relatively unaltered argillite and thinly bedded limestone cut by thin veins of quartz and calcite;
- (3) Brecciated argillite and limestone replaced with abundant iron-oxide minerals and clusters of sulfide minerals, including pyrite and pyrrhotite.

No attempt was made to quantify the relative proportions of the various types of rock material in the dumps. However, our observations of sulfides and their alteration products at the site (see files SOUTH.TIF and NORTH.TIF) suggest that the amount of sulfide-mineral-rich rock in the mine dumps is greater than is implied by the ore description made by Cook (1956, p. 22).

## **Sample Descriptions, Collection and Preparation Procedures**

### **Dump Samples**

Four composite samples of mine dump material were collected at the Thompson Creek tungsten mine. Sample 61JH99 represents the fine (<2 mm) size material and samples 73BV99, 74BV99, and 75BV99 represent the main varieties of rock materials in the dumps. All mine dump material was collected from the upper 5 cm of the surface of the dumps.

**Sample 61JH99.** This sample was collected to represent the <2 mm-diameter materials on the surfaces of the mine dumps. The mine dump surfaces were sampled with a stainless steel trowel, sieved through a solder-free 10-mesh screen into a plastic pan, and stored in a large plastic bag. At least 1 kg of sample was collected by compositing 60 increments sampled on a random walk over the entire length of the dumps (the entirety of the dumps shown in files NORTH.TIF and SOUTH.TIF). Smith and others (2000) discuss the successful use of this type of sampling strategy to efficiently evaluate the chemistry of the surficial materials of a mine-waste dump.

The composite field sample 61JH99 was mechanically split in the laboratory into two equal parts, one for solid-phase chemical analysis and the second for leaching. The first split was ground

until the particles passed through a 100-mesh (0.149 mm) sieve, and then submitted for chemical analysis.

The second split of sample 61JH99 for leaching was not reduced further in particle size. The <2 mm sample was leached following the procedure of Hageman and Briggs (2000a, 2000b), which is a stream-lined procedure that mimics results of the more rigorous EPA 1312 Synthetic Precipitation Leaching Procedure (Environmental Protection Agency, 1994). Briefly, a representative 50 g sub-sample was collected using a Jones splitter, added to 1 L of de-ionized water, and hand-shaken for 5 minutes. Aliquots of the leachate were then collected for chemical analysis, as described below in the Water Samples section. Minerals identified by X-ray diffraction in the second split of sample 61JH99 include montmorillonite, unidentified clay, albite, orthoclase, gypsum, muscovite, quartz, pyrite, and jarosite. In addition, non-crystalline amorphous material is present.

**Sample 73BV99.** This sample represents the rocks of the mine dumps that contain abundant sulfide and iron-oxide minerals. These mineralized rocks are brecciated. The sulfide minerals are clot-like replacements and the iron-oxide minerals are disseminated throughout the rock. X-ray diffraction analyses on portions of sample 73BV99 identified pyrite, pyrrhotite, quartz, microcline, and minor scheelite and biotite. The sample was collected as 30 increments (separate pieces averaging about the size of a walnut) picked up on a walk along the length of the mine dumps. Each individual increment was chosen because it appeared typical of the sulfide-mineral-rich rocks found throughout the dumps. The field composite sample was crushed in the laboratory. The crushed material was mechanically split into two equal parts. One split was ground until the particles passed through a 100-mesh (0.149 mm) sieve, and then submitted for chemical analysis. A second split was archived.

**Sample 75BV99.** This sample represents the weakly altered argillite and limestone rocks in the mine dumps. These rocks are cross cut by multiple thin veinlets filled with quartz and calcite. This composite sample was collected and processed similar to sample 73BV99.

Dump samples 61JH99 (solid phase), 73BV99, and 75BV99 were chemically analyzed by the following methods:

- (1) inductively coupled plasma-atomic emission spectrometry (ICP-AES) for 40 elements (Ag, Al, As, Au, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cu, Eu, Fe, Ga, Ho, K, La, Li, Mg, Mn, Mo, Na, Nb, Nd, Ni, P, Pb, Sc, Sn, Sr, Ta, Th, Ti, U, V, Y, Yb, and Zn);
- (2) hydride generation atomic absorption spectrometry (hydride AA) for As, Sb, Se, Te, and Tl;
- (3) direct current plasma or absorption spectrophotometry after collection by fire assay to determine Au;
- (4) use of an automated mercury analyzer to determine Hg in separated vapor (cold vapor Hg);
- (5) use of an automated carbon analyzer to determine total carbon; and
- (6) use of an automated sulfur analyzer to determine total sulfur.

These analytical techniques are described in the Appendix. Analytical results for samples 61JH99 (solid phase), 73BV99, and 75BV99 are provided in table 1 as well as in the data file DUMP.xls.

**Sample 74BV99.** This sample represents the igneous rock that is common in the mine dumps. As noted above, the whole rock major-element analysis of this sample indicates a granite composition (see rock classification plot in file GRANITE.PDF). The sample consists of mainly granitic pieces exhibiting minor silicification, with lesser amounts of moderately silicified granite containing tourmaline (variety elbaite) and finely disseminated pyrrhotite and molybdenite. The sample was collected as 30 increments picked up on a random walk along the full length of the



**Table 1.** Geochemical data from composite mine dump samples of surface fine material and altered rocks from the Thompson Creek tungsten mine.

[\*, exceeds critical soil values above which toxicity to plants is considered to be likely (Alloway, 1995)]

Sample type			Dump fines	Dump rocks	
Sample number			61JH99	73BV99	75BV99
Lab number			C-138054	C-138911	C-138912
Sample description			Composite dump surface fine (<2 mm ) material	Breccia from dumps with sulfide and iron-oxide minerals	Argillite and limestone from dump; weakly altered
Latitude			44° 20' 0.83	44° 20' 0.83"	44° 20' 0.83"
Longitude			114° 35' 7.58"	114° 35' 7.58"	114° 35' 7.58"
<b>Element</b>	<b>Units</b>	<b>Analytical Method</b>	<b>Results</b>	<b>Results</b>	<b>Results</b>
Major elements					
Al	%	ICP-AES	3.0	2.0	2.2
Ca	%	ICP-AES	3.6	3.2	6.6
Fe	%	ICP-AES	17	12	0.77
K	%	ICP-AES	1.2	1.9	1.9
Mg	%	ICP-AES	1.5	0.47	1.9
Na	%	ICP-AES	0.41	0.12	0.07
P	%	ICP-AES	0.1	0.12	0.02
Ti	%	ICP-AES	0.26	0.10	0.13
Carbon and sulfur					
Total carbon	%	LECO	0.23	0.75	2.83
Total sulfur	%	LECO	7.7	10.9	0.15
Minor and trace elements					
Ag	ppm	ICP-AES	<2	<2	<2
As	ppm	ICP-AES	47	180	<10
As	ppm	Hydride AA	65*	224	12.7
Au	ppm	ICP-AES	<8	<8	<8
Au	ppm	Fire assay	0.56	0.06	0.13
Ba	ppm	ICP-AES	55	32	2700
Be	ppm	ICP-AES	21	6	<1
Bi	ppm	ICP-AES	210	190	<50
Cd	ppm	ICP-AES	7	3	<2
Ce	ppm	ICP-AES	29	29	22
Co	ppm	ICP-AES	28	27	2
Cr	ppm	ICP-AES	77	79	64
Cu	ppm	ICP-AES	570*	540	7
Eu	ppm	ICP-AES	<2	2	<2
Ga	ppm	ICP-AES	17	7	<4
Hg	ppm	Cold Vapor	0.16	0.04	<0.02
Ho	ppm	ICP-AES	10	4	<4
La	ppm	ICP-AES	16	12	11
Li	ppm	ICP-AES	26	26	5
Mn	ppm	ICP-AES	4000*	1000	500
Mo	ppm	ICP-AES	19*	100	6
Nb	ppm	ICP-AES	4	<4	<4
Nd	ppm	ICP-AES	<9	9	<9
Ni	ppm	ICP-AES	<3	<3	4
Pb	ppm	ICP-AES	12	11	5
Sb	ppm	Hydride AA	2.1	5.5	1.1
Sc	ppm	ICP-AES	6	4	<2
Se	ppm	Hydride AA	5.8	7.9	<0.2
Sn	ppm	ICP-AES	<50	<50	<50

**Table 1.** Continued.

Sample number			61JHV99	73BV99	75BV99
<b>Element</b>	<b>Units</b>	<b>Analytical Method</b>	<b>Results</b>	<b>Results</b>	<b>Results</b>
Sr	ppm	ICP-AES	290	300	140
Ta	ppm	ICP-AES	<40	<40	<40
Te	ppm	Hydride AA	1.2	0.6	<0.1
Th	ppm	ICP-AES	9	6	<6
Tl	ppm	Hydride AA	0.7	0.5	0.2
U	ppm	ICP-AES	<100	<100	<100
V	ppm	ICP-AES	150*	50	22
Y	ppm	ICP-AES	13	16	9
Yb	ppm	ICP-AES	1	<1	<1
Zn	ppm	ICP-AES	250	49	31

mine dumps. The increment samples were chosen because they appeared typical of the granitic rock common within the dumps. The field composite sample was crushed in the laboratory. The crushed material was mechanically split into two equal parts. One split was ground until the particles passed through a 100-mesh (0.149 mm) sieve, and then submitted for chemical analysis. A second split was archived.

Sample 74BV99 was analyzed by two methods:

- (1) inductively coupled plasma-atomic emission spectrometry (ICP-AES) for 40 elements; and
- (2) wavelength dispersive X-ray fluorescence spectrometry (WDXRF) to determine 10 major elements.

These analytical techniques are described in the Appendix. The analytical results obtained from sample 74BV99 are provided in table 2 as well as in the data file GRANITE.xls.

**Table 2.** Geochemical data for a composite sample of granitic rock from the Thompson Creek tungsten mine dump. [LOI, loss on ignition indicates amount of total volatiles such as water and carbon dioxide].

Sample number		74BV99	
Lab number		C-139680	
Sample description		Granite in dumps	
Latitude		44° 20' 0.83"	
Latitude (decimal)		44.33356	
Longitude		114° 35' 7.58"	
Longitude (decimal)		114.58544	
<b>Oxide</b>	<b>Units</b>	<b>Analytical Method</b>	<b>Results</b>
SiO <sub>2</sub>	%	WDXRF	72.1
Al <sub>2</sub> O <sub>3</sub>	%	WDXRF	13.2
Fe <sub>2</sub> O <sub>3</sub>	%	WDXRF	2.33
MgO	%	WDXRF	0.96
CaO	%	WDXRF	1.21
Na <sub>2</sub> O	%	WDXRF	1.63
K <sub>2</sub> O	%	WDXRF	6.05
TiO <sub>2</sub>	%	WDXRF	0.50
P <sub>2</sub> O <sub>5</sub>	%	WDXRF	0.31
MnO	%	WDXRF	0.03
LOI	%	WDXRF	1.07
Total	%		99.4

**Table 2.** Continued.

Sample number			74BV99
<b>Element</b>	<b>Units</b>	<b>Analytical Method</b>	<b>Results</b>
Al	%	ICP-AES	6.8
Ca	%	ICP-AES	0.78
Fe	%	ICP-AES	1.1
K	%	ICP-AES	5.2
Mg	%	ICP-AES	0.43
Na	%	ICP-AES	1.2
P	%	ICP-AES	0.06
Ti	%	ICP-AES	0.18
Ag	ppm	ICP-AES	<2
As	ppm	ICP-AES	<10
Au	ppm	ICP-AES	<8
Ba	ppm	ICP-AES	5900
Be	ppm	ICP-AES	3
Bi	ppm	ICP-AES	<50
Cd	ppm	ICP-AES	3
Ce	ppm	ICP-AES	68
Co	ppm	ICP-AES	3
Cr	ppm	ICP-AES	25
Cu	ppm	ICP-AES	17
Eu	ppm	ICP-AES	<2
Ga	ppm	ICP-AES	27
Ho	ppm	ICP-AES	<4
La	ppm	ICP-AES	39
Li	ppm	ICP-AES	14
Mn	ppm	ICP-AES	130
Mo	ppm	ICP-AES	5
Nb	ppm	ICP-AES	6
Nd	ppm	ICP-AES	25
Ni	ppm	ICP-AES	7
Pb	ppm	ICP-AES	8
Sc	ppm	ICP-AES	3
Sn	ppm	ICP-AES	<50
Sr	ppm	ICP-AES	510
Ta	ppm	ICP-AES	<40
Th	ppm	ICP-AES	9
U	ppm	ICP-AES	<100
V	ppm	ICP-AES	32
Y	ppm	ICP-AES	8
Yb	ppm	ICP-AES	<1
Zn	ppm	ICP-AES	34

## Sediment Samples

Two stream sediment samples were collected, one about 400 ft (120 m) upstream of the mine site (sample site 99CH007) and the second about 2000 ft (600 m) downstream of the mine site (site 99CH008). Both samples were composited from active alluvium from pockets of fine sediment along the bank of Thompson Creek. At sample site 99CH007 the sediment was composited as 15 increments over a 60 ft<sup>2</sup> area, while at site 99CH008, 20 increments were composited over a 150 ft<sup>2</sup> area. The sediment samples were sieved on site with a 10 mesh (2 mm) stainless steel screen and 2 lb (1 kg) samples of minus-10 mesh sediment were collected in cloth bags and air dried.

Visual estimates of the sample collected were made at each site. The alluvium at site 99CH007 consisted of 80% intermediate to felsic porphyritic rocks of the Challis Volcanics, 10% argillite and quartzite, 10% granite; and alluvium at site 99CH008 consisted of 85% porphyritic rocks of the Challis Volcanics, 10% granite, 3% argillite, 2% vein quartz with local FeOx coatings. Minor iron oxide and manganese oxide staining on alluvium and low organic content were estimated at both sites. Bedrock was not present at either sample site, but a talus slope at site 99CH007 contained quartzite and argillite, locally cut by pyrite-bearing quartz veins. The metasedimentary rocks exhibited strong cleavage with iron oxide on joints.

In the laboratory, the composite sediment samples were sieved to minus-80 mesh (0.177 mm) and the fine fraction was pulverized until the particles passed through a 100-mesh (0.149 mm) sieve. The samples were split, one for chemical analysis and the second for archival. The sediment samples were analyzed by the same six methods listed above for rock samples 61JH99 (solid phase), 73BV99, and 75BV99. The analytical results are provided in table 3 as well as in the data file SEDIMENT.xls.

**Table 3.** Geochemical data for <80 mesh stream sediments in Thompson Creek upstream and downstream of the Thompson Creek tungsten mine.

Sample number			99CH007	99CH008
Location relative to mine			upstream	downstream
Element	Units	Analytical method		
Ag	ppm	ICP-AES	<2	<2
Al	%	ICP-AES	7.7	7.7
As	ppm	ICP-AES	17	<10
As	ppm	HY	2.4	2.3
Au	ppm	ICP-AES	<8	<8
Au	ppm	FA	<0.005	<0.005
Ba	ppm	ICP-AES	900	1,000
Be	ppm	ICP-AES	<1	<1
Bi	ppm	ICP-AES	<50	<50
C (total)	%	LECO	0.21	0.48
Ca	%	ICP-AES	5.6	5.1
Cd	ppm	ICP-AES	<2	<2
Ce	ppm	ICP-AES	85	85
Co	ppm	ICP-AES	33	29
Cr	ppm	ICP-AES	220	170
Cu	ppm	ICP-AES	18	19
Eu	ppm	ICP-AES	<2	<2
Fe	%	ICP-AES	7.5	6.7
Ga	ppm	ICP-AES	22	25
Hg	ppm	CV	<0.02	<0.02
Ho	ppm	ICP-AES	<4	5
K	%	ICP-AES	1.4	1.5
La	ppm	ICP-AES	42	40
Li	ppm	ICP-AES	14	15
Mg	%	ICP-AES	3.4	2.9
Mn	ppm	ICP-AES	1,300	1,200
Mo	ppm	ICP-AES	2	<2
Na	%	ICP-AES	1.3	1.4
Nb	ppm	ICP-AES	37	35
Nd	ppm	ICP-AES	40	35
Ni	ppm	ICP-AES	17	15
P	%	ICP-AES	0.12	0.13
Pb	ppm	ICP-AES	<4	7
S (total)	%	LECO	<0.05	<0.05
Sb	ppm	HY	<0.6	<0.6
Sc	ppm	ICP-AES	35	29
Se	ppm	HY	<0.2	<0.2
Sn	ppm	ICP-AES	<50	<50
Sr	ppm	ICP-AES	540	570
Ta	ppm	ICP-AES	<40	<40
Te	ppm	HY	<0.1	<0.1
Th	ppm	ICP-AES	<6	<6
Ti	%	ICP-AES	1.5	1.3
Tl	ppm	HY	0.1	0.3
U	ppm	ICP-AES	<100	<100
V	ppm	ICP-AES	400	320
Y	ppm	ICP-AES	24	23
Yb	ppm	ICP-AES	2	2
Zn	ppm	ICP-AES	110	110

## Water Samples

Three surface water samples were collected from the area, two from the stream sediment sample sites (99CH007 and 99CH008) and one from a small pool (99CH006) about 100 ft (30 m) directly below the collapsed adit at the Thompson Creek Tungsten mine (see ADIT.TIF). Scanned photographs of these three sites are included as 99CH006.TIF, 99CH007.TIF and 99CH008.TIF. The sample from the small pool is believed to be adit drainage; the source of the water is a buried 6 inch (15 cm) rusted steel pipe that apparently drains the adit. The other two samples are stream water from Thompson Creek above and below the mine, as described above in the Sediment Samples section. Water samples were collected in a clean 1 L polypropylene bottle. At the two stream sites, width-integrated samples were collected below riffles for better sample representativity. Below the adit, water was collected directly from the steel pipe from which it issued.

Parameters measured in the field at each of the three sites include pH, conductivity, field alkalinity, turbidity, dissolved oxygen, acidity, an estimate of flow, water color, and water odor. Conductivity, pH, and turbidity meters were calibrated at each site. Field alkalinity and acidity were measured using portable titration kits available from CHEMetrics and Hach, respectively. Dissolved oxygen was measured using a portable colorimetric kit from CHEMetrics. Six separate water samples were collected at each site, and analyzed as follows:

- (1) a filtered (0.45 micron disposable filter), acidified ( $\text{HNO}_3$ ) sample for major and trace cation analysis, collected in a new, acid-rinsed ( $\text{HNO}_3$ ) clear polypropylene bottle; analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) and inductively coupled plasma-mass spectrometry (ICP-MS),
- (2) an unfiltered, acidified ( $\text{HNO}_3$ ) raw sample for major and trace cation analysis, collected in a new, acid-rinsed ( $\text{HNO}_3$ ) clear polypropylene bottle; analyzed by inductively coupled plasma-atomic emission spectrometry (ICP-AES) and inductively coupled plasma-mass spectrometry (ICP-MS),
- (3) a filtered (0.45 micron disposable filter), acidified (HCl) sample for  $\text{Fe}^{2+}$  analysis, collected in a new, acid-rinsed (HCl) dark brown polypropylene bottle, protected from sunlight; analyzed by colorimetry,
- (4) a filtered (0.45 micron disposable filter), preserved ( $\text{KCr}_2\text{-HNO}_3$ ) sample for Hg analysis, collected in an acid-rinsed ( $\text{HNO}_3$ ) glass bottle with teflon lid; analyzed by atomic fluorescence,
- (5) a filtered (0.45 micron disposable filter), unacidified sample (refrigerated) for anion analysis; analyzed by ion chromatography,
- (6) an unfiltered, unacidified sample (refrigerated) for alkalinity analysis by laboratory titration.

Collection of samples (1) and (2) above allows for comparison of dissolved versus suspended chemical constituents. The analytical results and field parameters measured for the three surface waters are provided in table 4 as well as the data file WATER.xls.

Dump sample 61JH99 was leached as described in the Dump Samples section. Water samples collected from the leachate include numbers (1), (4), and (5) above. The analytical results and parameters measured for this sample are listed in table 5 and in data file LEACH.xls.

**Table 4.** Analytical results and field parameters measured in three samples of surface waters.

Field Parameters									
			Mine effluent		Thompson Creek				
			Upstream	Downstream					
Water sample site			99CH006	99CH007	99CH008				
Collection date			8/13/99	8/13/99	8/13/99				
pH			5.7	5.5	5.8				
Conductivity (microseimens/cm)			980	88	93				
Dissolved oxygen (ppm)			9	7	7				
Turbidity (FTU)			0	0.56	0.34				
Water temperature (°C)			5	8.9	10				
Flow rate			2 gal/min	10 cubic ft/sec	10 cubic ft/sec				
Color			colorless	colorless	colorless				
Odor			none	none	none				
Filter size (um)			0.45	0.45	0.45				
Alkalinity (as ppm CaCO <sub>3</sub> )			110	40	35				
Acidity (as ppm CaCO <sub>3</sub> )			40	<20	<20				
<b>Water sample</b>			<b>99CH006FA</b>	<b>99CH007FA</b>	<b>99CH008FA</b>		<b>99CH006RA</b>	<b>99CH007RA</b>	<b>99CH008RA</b>
Lab number			C-137525	C-137540	C-137545		C-137478	C-137493	C-137498
			<i>Dissolved concentrations (filtered)</i>			<i>Total acid soluble concentrations (unfiltered)</i>			
Alkalinity (as ppm CaCO <sub>3</sub> )-Lab measurement						110	40	42	
Element	Units	Method							
Fe <sup>2+</sup>	ppm	CO	0.02	0.01	0.01				
F	ppm	IC	0.6	0.2	0.2				
Cl	ppm	IC	1.9	0.3	0.3				
SO <sub>4</sub>	ppm	IC	530	4	5.6				
NO <sub>3</sub>	ppm	IC	0.4	<0.1	<0.1				
Ag	ppb	ICP-MS	<0.01	<0.01	<0.01	0.03	0.01	<0.01	
Ag	ppb	ICP-AES	<10	<10	<10	<10	<10	<10	
Al	ppb	ICP-MS	<0.01	9.2	4.9		56	45	
Al	ppb	ICP-AES	<10	12	<10	27	61	50	
As	ppb	ICP-MS	1	0.4	0.4	1	0.5	0.4	
As	ppb	ICP-AES	<100	<100	<100	<100	<100	<100	
Au	ppb	ICP-MS	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
B	ppb	ICP-AES	<5	<5	<5	<5	<5	<5	
Ba	ppb	ICP-MS	13	8.3	8.1	12	8.3	8.7	
Be	ppb	ICP-MS	<0.05	<0.05	<0.05	0.1	<0.05	<0.05	
Be	ppb	ICP-AES	<5	<5	<5	<5	<5	<5	
Bi	ppb	ICP-MS	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	
Ca	ppm	ICP-MS	160	9.1	10	130	9	10	
Ca	ppm	ICP-AES	180	9.9	11	180	10	11	
Cd	ppb	ICP-MS	0.5	<0.02	<0.02	0.5	<0.02	<0.02	
Cd	ppb	ICP-AES	<5	<5	<5	<5	<5	<5	
Ce	ppb	ICP-MS	0.01	0.03	0.03	0.04	0.2	0.2	
Co	ppb	ICP-MS	<0.02	<0.02	<0.02	<0.02	<0.02	0.02	
Co	ppb	ICP-AES	<10	<10	<10	<10	<10	<10	
Cr	ppb	ICP-MS	<1	<1	<1	<1	<1	<1	
Cr	ppb	ICP-AES	<10	<10	<10	<10	<10	<10	
Cs	ppb	ICP-MS	0.2	<0.02	<0.02	0.2	<0.02	<0.02	
Cu	ppb	ICP-MS	2	<0.5	<0.5	2	<0.5	<0.5	
Cu	ppb	ICP-AES	<10	<10	<10	<10	<10	<10	
Dy	ppb	ICP-MS	0.009	0.008	<0.005	0.01	0.02	<0.005	
Er	ppb	ICP-MS	<0.005	<0.005	<0.005	<0.005	0.01	0.01	
Eu	ppb	ICP-MS	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	
Fe	ppb	ICP-MS		<30			31	<30	

**Table 4. Continued**

Water sample			99CH006FA	99CH007FA	99CH008FA	99CH006RA	99CH007RA	99CH008RA
Element	Units	Method						
Fe	ppb	ICP-AES	<20	<20	<20	62	35	29
Ga	ppb	ICP-MS	<0.02	<0.03	<0.03	<0.02	0.03	<0.03
Gd	ppb	ICP-MS	0.008	0.01	0.006	<0.005	0.009	0.02
Ge	ppb	ICP-MS	0.04	<0.09	<0.09	0.02	<0.09	<0.09
Hg	ppb	CV AA	<0.005	<0.005	<0.005			
Ho	ppb	ICP-MS	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
In	ppb	ICP-MS	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
K	ppm	ICP-MS	3.2	0.57	0.61	2.7	0.51	0.55
K	ppb	ICP-AES	3.6	0.6	0.63	3.7	0.64	0.66
La	ppb	ICP-MS	0.1	0.04	0.04	0.2	0.07	0.09
Li	ppb	ICP-MS	21	0.7	0.7	20	0.5	0.5
Li	ppb	ICP-AES	22	<10	<10	22	<10	<10
Mg	ppm	ICP-MS	28	1.6	1.8	27	1.6	1.8
Mg	ppm	ICP-AES	34	1.6	1.7	34	1.6	1.8
Mn	ppb	ICP-MS	190	0.32	0.22	170	0.82	0.58
Mn	ppb	ICP-AES	220	<10	<10	220	<10	<10
Mo	ppb	ICP-MS	3.3	0.84	0.73	3.1	0.82	0.65
Mo	ppb	ICP-AES	<20	<20	<20	<20	<20	<20
Na	ppm	ICP-MS	24	5.7	5.8	24	5.6	5.8
Na	ppm	ICP-AES	30	5.3	5.4	32	5.7	5.5
Nd	ppb	ICP-MS	0.03	0.03	0.04	0.07	0.08	0.09
Ni	ppb	ICP-MS	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Ni	ppb	ICP-AES	<10	<10	<10	<10	<10	<10
P	ppb	ICP-MS	<1	24	27	<1	26	24
P	ppb	ICP-AES	<100	<100	<100	<100	<100	<100
Pb	ppb	ICP-MS	<0.05	<0.05	<0.05	0.3	<0.05	<0.05
Pb	ppb	ICP-AES	<50	<50	<50	<50	<50	<50
Pr	ppb	ICP-MS	<0.01	<0.01	<0.01	0.01	0.02	0.02
Rb	ppb	ICP-MS	9.4	0.6	0.62	8.5	0.63	0.66
Re	ppb	ICP-MS	0.08	<0.02	<0.02	0.08	<0.02	<0.02
Sb	ppb	ICP-MS	0.2	<0.03	<0.03	0.1	<0.03	<0.03
Sb	ppb	ICP-AES	<50	<50	<50	<50	<50	<50
Se	ppb	ICP-MS	0.7	<0.8	<0.8	0.4	<0.8	<0.8
Si	ppm	ICP-AES	6.9	9.8	9.8	7	10	9.9
Sm	ppb	ICP-MS	<0.01	<0.01	0.02	0.01	0.01	0.01
Sr	ppb	ICP-MS	3600	71	78	3200	72	77
Sr	ppb	ICP-AES	3300	70	74	3400	71	75
Tb	ppb	ICP-MS	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Th	ppb	ICP-MS	0.06	<0.03	<0.03	0.05	<0.03	<0.03
Ti	ppb	ICP-MS	<0.05	<0.05	<0.05	0.07	<0.05	<0.05
Ti	ppb	ICP-AES	<50	<50	<50	<50	<50	<50
Tl	ppb	ICP-MS	<0.05	<0.05	<0.05	0.07	<0.05	<0.05
Tm	ppb	ICP-MS	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
U	ppb	ICP-MS	8.8	0.17	0.19	9.6	0.2	0.22
V	ppb	ICP-MS	0.6	2	2	0.7	2	2
V	ppb	ICP-AES	<10	<10	<10	<10	<10	<10
W	ppb	ICP-MS	0.5	0.03	0.06	0.5	0.05	0.03
Y	ppb	ICP-MS	0.3	0.05	0.04	0.3	0.08	0.06
Yb	ppb	ICP-MS	<0.01	<0.01	<0.01	<0.01	0.02	0.02
Zn	ppb	ICP-MS	170	<0.5	<0.5	170	0.5	<0.5
Zn	ppb	ICP-AES	190	<10	<10	200	<10	<10



**Table 4.** Continued.

Water sample site descriptions:

99CH006	Small pool below collapsed adit at latitude 44° 20' 0.4", longitude 114° 35' 10.88" Channel bed: heavy FeOx (and MnOx?) coating granite and tactite boulders; salts locally on pyritic boulders; abundant brown organic sediment and moss; pool surrounded by willows, grass, and trees.
99CH007	Thompson Creek 400 ft (120 m) upstream of the mine site at latitude 44° 20' 6.74", longitude 114° 35' 10.80" Channel bed: orange and brown stained cobbles to boulders; well-vegetated banks; minor FeOx and MnOx, most of the staining is organic in origin.
99CH008	Thompson Creek 2,000 ft (600 m) downstream of the mine site at latitude 44° 19' 47.22", longitude 114° 35' 17.41" Channel bed: orange and brown stained cobbles to boulders; well-vegetated banks; minor FeOx and MnOx, most of the staining is organic in origin.

**Table 5.** Leachate data for a sample of fine surface material from the mine dumps. [See text for leach procedure; see table 1 for sample information].

Sample type			Leachate				
Sample number			61JH99				
Leach pH			3.1				
Leach conductivity (microseimens/cm)			980				
Leach acidity (ppm)			100				
Element	Units	Analytical method		Element	Units	Analytical method	
F	ppm	IC	0.4	La	ppb	ICP-MS	6.1
Cl	ppm	IC	0.3	Li	ppb	ICP-MS	21
SO <sub>4</sub>	ppm	IC	410	Li	ppb	ICP-AES	23
NO <sub>3</sub>	ppm	IC	<0.07	Mg	ppm	ICP-MS	10
Ag	ppb	ICP-MS	<0.2	Mg	ppm	ICP-AES	14
Ag	ppb	ICP-AES	<10	Mn	ppb	ICP-MS	3,800
Al	ppb	ICP-MS	1,300	Mn	ppb	ICP-AES	4,300
Al	ppb	ICP-AES	1,800	Mo	ppb	ICP-MS	<1
As	ppb	ICP-MS	2	Mo	ppb	ICP-AES	<20
As	ppb	ICP-AES	<100	Na	ppm	ICP-MS	0.02
Au	ppb	ICP-MS	<0.01	Na	ppm	ICP-AES	<0.1
B	ppb	ICP-AES	<5	Nd	ppb	ICP-MS	5.4
Ba	ppb	ICP-MS	47	Ni	ppb	ICP-MS	54
Ba	ppb	ICP-AES	46	Ni	ppb	ICP-AES	65
Be	ppb	ICP-MS	23	P	ppb	ICP-MS	<1
Be	ppb	ICP-AES	23	P	ppb	ICP-AES	<100
Bi	ppb	ICP-MS	<0.1	Pb	ppb	ICP-MS	1.1
Ca	ppm	ICP-MS	86	Pb	ppb	ICP-AES	<50
Ca	ppm	ICP-AES	26	Pr	ppb	ICP-MS	1.6
Cd	ppb	ICP-MS	18	Rb	ppb	ICP-MS	8.5
Cd	ppb	ICP-AES	17	Re	ppb	ICP-MS	<0.02
Ce	ppb	ICP-MS	12	Sb	ppb	ICP-MS	<0.2
Co	ppb	ICP-MS	45	Sb	ppb	ICP-AES	<50
Co	ppb	ICP-AES	50	Se	ppb	ICP-MS	0.7
Cr	ppb	ICP-MS	1	Si	ppm	ICP-AES	0.38
Cr	ppb	ICP-AES	<10	Sm	ppb	ICP-MS	0.93
Cs	ppb	ICP-MS	2.6	Sr	ppb	ICP-MS	380
Cu	ppb	ICP-MS	220	Sr	ppb	ICP-AES	370
Cu	ppb	ICP-AES	260	Tb	ppb	ICP-MS	0.11
Dy	ppb	ICP-MS	0.55	Th	ppb	ICP-MS	0.9
Er	ppb	ICP-MS	0.24	Ti	ppb	ICP-AES	<50
Eu	ppb	ICP-MS	0.29	Tl	ppb	ICP-MS	<0.2
Fe	ppb	ICP-MS	3,200	Tm	ppb	ICP-MS	0.04
Fe	ppb	ICP-AES	4,400	U	ppb	ICP-MS	17
Ga	ppb	ICP-MS	0.04	V	ppb	ICP-MS	0.3
Gd	ppb	ICP-MS	0.68	V	ppb	ICP-AES	<10
Ge	ppb	ICP-MS	<0.02	W	ppb	ICP-MS	<0.2
Hg	ppb	CV	<0.1	Y	ppb	ICP-MS	3.5
Ho	ppb	ICP-MS	0.11	Yb	ppb	ICP-MS	0.23
In	ppb	ICP-MS	0.08	Zn	ppb	ICP-MS	910
K	ppm	ICP-MS	0.16	Zn	ppb	ICP-AES	920
K	ppm	ICP-AES	0.2				

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## APPENDIX. Summary of Analytical Methods

In this study, Quality Assurance/Quality Control concerns for solid media were addressed through the use of blind reference materials (accuracy) and analytical duplicates (precision). Reference standards were interspersed with batches of samples and the analyses of the reference standards were checked to assure that the reported values were within  $\pm 20$  percent of accepted values. Analytical duplicates were interspersed with batches of samples and the analyses of the duplicates were checked to assure that the relative standard deviation (RSD) between duplicates was no greater than  $\pm 20$  percent.

Chemical analyses of the stream sediment and mine dump materials were performed by XRAL Laboratories of Don Mills, Ontario, Canada, under a contract with the USGS. The analytical methods and techniques that were used are described below. In the discussion below and the attached data files:

- “ppm” signifies parts per million,
- “ppb” is parts per billion,
- “%” is weight percent of the sample, and
- “<” represents a less than value below the specified lower limit of determination.

### ***METHODS USED ON SOLID MEDIA (ROCK, MINE WASTE, AND SEDIMENTS)***

#### **40 Element ICP-AES**

Forty major, minor, and trace elements were determined in geological materials by inductively coupled plasma-atomic emission spectrometry (ICP-AES). The sample (0.2 g) is decomposed using a mixture of hydrochloric, nitric, perchloric, and hydrofluoric acids at low temperature. The digested sample is aspirated into the ICP-AES discharge where the elemental emission signal is measured simultaneously for the forty elements. Calibration is performed by standardizing with digested rock reference materials and with a series of multi-element solution standards. Upper and lower determination limits for this method are listed below in table A1.

*Analytical Performance:* Data were deemed acceptable if recovery for all 40 elements was  $\pm 15\%$  at five times the Lower Limit of Determination (LOD) and the calculated Relative Standard Deviation (RSD) of duplicate samples was no greater than 15%.

#### **Hydride Generation AA for As, Sb, Se, Te, and Tl**

Arsenic, antimony, and thallium are determined by weighing 0.1 g of sample into a zirconium crucible. Approximately 0.75 g of sodium peroxide is added and mixed. The mixture is heated in a muffle furnace set at 750°C for four minutes. The sample is cooled, then 15 ml of water and 5 ml of concentrated HCl is added. The mixture is shaken and 0.25 ml of an ascorbic acid KI solution is added, then diluted with 20% HCl and let to stand overnight. Arsenic, selenium, and thallium are then measured using hydride generation atomic absorption spectrometry (Hydride AA). The optimum concentration ranges without sample dilution for these elements in various solid phase sample media are: As—0.6 ppm to 20 ppm, Sb—0.6 ppm to 20 ppm, and thallium—0.1 ppm to 10 ppm.

**Table A1.** Reporting limits for 40 elements by ICP-AES.

Element	Lower Determination Limit	Upper Determination Limit
<b>Weight Percent</b>		
Al	0.005	50
Ca	0.005	50
Fe	0.02	25
K	0.01	50
Mg	0.005	5
Na	0.005	50
P	0.005	50
Ti	0.005	25
<b>Parts Per Million</b>		
Ag	2	10,000
As	10	50,000
Au	8	50,000
Ba	1	35,000
Be	1	5,000
Bi	10	50,000
Cd	2	25,000
Ce	5	50,000
Co	2	25,000
Cr	2	25,000
Cu	2	15,000
Eu	2	5,000
Ga	4	50,000
Ho	4	5,000
La	2	50,000
Li	2	50,000
Mn	4	50,000
Mo	2	50,000
Nb	4	50,000
Nd	9	50,000
Ni	3	50,000
Pb	4	50,000
Sc	2	50,000
Sn	5	50,000
Sr	2	15,000
Ta	40	50,000
Th	6	50,000
U	100	100,000
V	2	30,000
Y	2	25,000
Yb	1	5,000
Zn	2	15,000

Selenium and tellurium are determined by weighing 0.25 g of sample into a test tube, adding a mixture of nitric and perchloric acids and heating. After the solution is cooled, hydrochloric and nitric are added, heated again and cooled. The samples are diluted and analyzed using hydride generation atomic absorption spectrometry. The expected analytical range for selenium is 0.2 to 4 ppm, and the lower reporting limit for tellurium is 0.1 ppm.

*Analytical Performance:* Data were deemed acceptable if recovery of As and Sb was  $\pm 20\%$  at five times the LOD and the calculated percent RSD of duplicate samples was no greater than

20%. Data for selenium were deemed acceptable if recovery of that element was  $\pm 20\%$  at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%. Data for tellurium were deemed acceptable if recovery of that element was  $\pm 15\%$  at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 15%.

### **Gold by Fire Assay**

Gold was determined by DCP or atomic absorption spectrophotometry after collection by fire assay. An assay fusion consists of heating a 15 g mixture of the finely pulverized sample with about three parts of a flux until the product is molten. One of the ingredients of the flux is a lead compound, which is reduced by other constituents of the flux or sample to metallic lead. The latter collects all the gold, together with silver, platinum metals, and small quantities of certain base metals present in the sample and falls to the bottom of the crucible to form a lead button. The gangue of the ore is converted by the flux into a slag sufficiently fluid so that all particles of lead may fall readily through the molten mass. The choice of a suitable flux depends on the character of the ore. The lead button is cupelled to oxidize the lead, leaving behind a dore bead containing the precious metals. The dore bead is then transferred to a test tube, dissolved with aqua regia, diluted to a specific volume and determined by DCP or atomic absorption spectrophotometry. The lower reporting level for a 15 g sample charge is 5 ppb by DCP and atomic absorption. The upper reporting limit is 10,000 ppb.

*Analytical Performance:* Data were deemed acceptable if recovery of gold was  $\pm 20\%$  at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%.

### **Mercury Analyses of Separated Cold Vapor (Cold Vapor Hg)**

Mercury was determined by weighing out 0.1 g of sample, which was digested with a mixture of sulfuric acid, nitric acid, 5% potassium permanganate, and 5% potassium peroxydisulfate in a water bath for one hour. The excess of potassium permanganate is reduced with hydroxylamine sulfate solution and then Hg (II) is reduced with stannous chloride. The Hg vapor is separated and measured using a LEEMAN PS200 automated mercury analyzer. The technique offers a lower reporting limit of 0.02 ppm mercury in solid-phase samples. Samples exceeding the working range of 0.02 - 1.8 ppm mercury require dilution.

*Analytical Performance:* Data were deemed acceptable if recovery of mercury was  $\pm 20\%$  at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%.

### **Total Carbon Analyses**

Total carbon was determined by the use of an automated carbon analyzer. A weighed sample (0.25 g sample used) is combusted in an oxygen atmosphere at 1370°C to oxidize carbon to carbon dioxide. Moisture and dust are removed and the carbon dioxide gas is measured by a solid state infrared detector. The operating range for total carbon is from 0.05% to about 30%.

*Analytical Performance:* Data were deemed acceptable if recovery of total carbon was  $\pm 15\%$  at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 15%.

## Total Sulfur Analyses

Total sulfur was determined by using an automated sulfur analyzer. Approximately 0.25 g of sample is mixed with iron chips and LECOCEL and is heated in a combustion tube in a stream of oxygen at high temperature. Sulfur is oxidized to sulfur dioxide. Moisture and dust are removed and then the sulfur dioxide gas is measured with a CS-244 infrared detector. The reporting range for total sulfur is from 0.05% to about 35%.

*Analytical Performance:* Data were deemed acceptable if recovery of total sulfur was  $\pm 15\%$  at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 15%.

## Wavelength Dispersive X-ray Fluorescence Spectrometry (WDXRF) by USGS Laboratories

Wavelength dispersive X-ray fluorescence spectrometry (WDXRF) was conducted by personnel in the USGS Laboratories in Lakewood, Colorado, to determine 10 major elements in granitic sample 74BV99 (results provided in GRANITE.xls). The sample was fused with lithium tetraborate and the resultant glass disc was introduced into a wavelength dispersive X-ray spectrometer. The disc was irradiated with X-rays from an X-ray tube. X-ray photons emitted by the elements in the sample were counted and concentrations determined using previously prepared calibration standards. In addition to 10 major elements, the method provides a gravimetric loss-on-ignition. The WDXRF procedures and calibration techniques used by the USGS Laboratories are described in Taggart and others (1981, 1987).

**Table A2.** Reporting limits (calibration range) for 10 elements by WDXRF.

Element	Lower Determination Limit	Upper Determination Limit
<b>Percent</b>		
SiO <sub>2</sub>	0.10	99.0
Al <sub>2</sub> O <sub>3</sub>	0.10	58.0
Fe <sub>2</sub> O <sub>3</sub>	0.04	28.0
MgO	0.10	60.0
CaO	0.02	60.0
Na <sub>2</sub> O	0.15	30.0
K <sub>2</sub> O	0.02	30.0
TiO <sub>2</sub>	0.02	10.0
P <sub>2</sub> O <sub>5</sub>	0.05	50.0
MnO	0.01	15.0
Loss-on-ignition	0.01	100.0

*Analytical Performance:* Long-term instrument drift is corrected using drift monitor analyses. Monitor intensity values obtained during the analyses are compared with monitor intensity values from the original spectrometer calibration. Corrections are calculated by the spectrometer's software. Long-term drift monitoring cannot correct for short-term efforts or significant changes in the operating parameters.

In order to keep track of instrumental short-term drift, at least every twelfth disc is one of five instrument check standards. The standards represent the average, high and low for the 10 analyzed elements. If the analyzed disc exceeds three times the standard deviation of the counting statistics, analysis is halted and the instrument is checked using other discs. If the disc is corrupt, it



is removed and another is made. If the instrument shows signs of drift, then a recalibration is performed.

In addition to the instrument standards, a sample preparation check standard disc is prepared for every 20 samples produced and analyzed along with the samples. If this disc shows a deviation of three standard deviations or more, and the instrument standards show no deviation, then another check standard disc is prepared. If it again shows deviation, then sample preparation is halted and the problem is located. If both the sample preparation standard and the instrument standard exceed control limits, then the instrument recalibration is performed.

## ***METHODS USED ON WATER AND LEACHATE SAMPLES***

In this study, Quality Assurance/Quality Control concerns for water samples were addressed through the use of internal reference standards, field blanks, site duplicates, and analytical duplicates. Reference standards were interspersed with batches of samples and the analyses of the reference standards were checked to assure that reported values were within  $\pm 20$  percent of the accepted values. Water samples included field blanks of de-ionized water, used to check for contamination from sampling equipment and preservatives. Field blanks were collected following the same procedures as those used for normal water samples. For all analytical methods used on water samples listed below, data were deemed acceptable if recovery was  $\pm 20\%$  at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%.

### **27 Elements by ICP-AES**

Acidified water samples were analyzed for major (Al, Ca, Fe, K, Mg, Na, and Si) and selected trace elements following the inductively coupled plasma-atomic emission spectrometry (ICP-AES) method of Briggs and Fey (1996). Water samples were aspirated into a plasma and element concentrations were determined directly by ICP-AES. Limits of determination for the multi-element ICP-AES method for water samples are shown in table A3 below.

### **Ion Chromatography**

The anions  $\text{Cl}^-$ ,  $\text{F}^-$ ,  $\text{NO}_3^-$ , and  $\text{SO}_4^{2-}$  were determined sequentially by ion chromatography on unfiltered, unacidified water samples following a modification (d'Angelo and Ficklin, 1996) of the procedure of Fishman and Pyen (1979). The raw water samples were kept cool from the time of collection until they were analyzed. The samples were injected into a chromatograph where ions of interest separate along an ion exchange separator column at different rates, depending on the affinity of each species for the ion-exchange resin. Samples then passed into a flow-through conductivity cell where the anions were detected and their peak heights were recorded. Unknown samples were compared with peak heights of reference standards to determine sample concentrations. Limits of determination for anions in raw water samples are shown in table A4 below.

**Table A3.** Limits of determination for the multi-element ICP-AES method for water samples.

Element	Lower Determination Limit	Upper Determination Limit
<b>Parts Per Million</b>		
Al	0.01	1,000
Ca	1	1,000
Fe	0.02	1,000
K	1	1,000
Mg	1	1,000
Na	1	1,000
P	0.1	1,000
Si	1	1,000
<b>Parts Per Billion</b>		
Ag	10	10,000
As	100	10,000
B	5	10,000
Ba	5	10,000
Be	5	10,000
Cd	5	10,000
Cr	10	10,000
Co	10	10,000
Cu	10	10,000
Li	10	10,000
Mn	10	10,000
Mo	20	10,000
Ni	10	10,000
Pb	50	10,000
Sb	50	10,000
Sr	1	10,000
Ti	50	10,000
V	10	10,000
Zn	10	10,000

**Table A4.** Limits of determination for anions in raw water samples by sequential ion chromatography.

Anion	Lower Determination Limit	Upper Determination Limit <sup>1</sup>
<b>Parts Per Million</b>		
Cl <sup>-</sup>	0.1	4
F <sup>-</sup>	0.05	2
NO <sub>3</sub> <sup>-</sup>	0.1	10
SO <sub>4</sub> <sup>2-</sup>	0.5	20

### Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)

Acidified-filtered and acidified-unfiltered waters, and leachates were analyzed to determine 52 elements by ICP-MS using a method developed by the U.S. Geological Survey (A.L. Meier, personal commun., 1995; Meier and others, 1994). The method is used to determine numerous elements directly in the water sample without the need for preconcentration or dilution. Element detection limits are in the sub-part-per-billion range and the working linear range is six orders of

magnitude or more. By using derived response curves, percent of ionization, and natural isotopic abundances, estimates of concentrations for the elements can be determined in samples without the need of a calibration standard for every element. The method is most useful for trace elements in the parts-per-billion range; analyses for major elements in the parts-per-million range are less accurate and ICP-AES data should be used. Limits of determination for elements by ICP-MS are shown in table A5 below.

**Table A5.** Limits of determination for elements in water samples by the ICP-MS method.

Element	Lower Determination Limit <sup>1</sup>	Element	Lower Determination Limit <sup>1</sup>
Parts per million (ppm)		Parts per billion (ppb)	
Ca	0.05	K	0.30
Mg	0.01	La	0.01
Na	0.01	Li	0.10
Si	0.25	Mn	0.01
Parts per billion (ppb)		Mo	0.02
Ag	0.01	Nd	0.01
Al	0.01	Ni	0.10
As	0.20	P	1
Au	0.01	Pb	0.05
Ba	0.02	Pr	0.01
Be	0.05	Rb	0.01
Bi	0.01	Re	0.02
Cd	0.02	Sb	0.03
Ce	0.01	Se	0.8
Co	0.02	Sm	0.01
Cr	1.0	Sr	0.02
Cs	0.02	Tb	0.005
Cu	0.50	Th	0.03
Dy	0.005	Ti	0.05
Er	0.005	Tl	0.05
Eu	0.005	Tm	0.005
Fe	30	U	0.01
Ga	0.02	V	0.10
Gd	0.005	W	0.02
Ge	0.02	Y	0.01
Ho	0.005	Yb	0.01
In	0.01	Zn	0.50

### Mercury by Cold Vapor Atomic Absorption

Mercury was measured in water samples using the cold-vapor AAS technique of O'Leary and others (1996). Preserved water samples were analyzed directly. Mercury (II) was reduced in the solutions to elemental mercury gas with hydroxylamine hydrochloride and stannous chloride in a continuous flow system, releasing mercury into the quartz cell of an atomic absorption spectrophotometer where the mercury concentration was determined. Limits of determination are shown in table A6 below.

## Ferrous Iron by Colorimetry

Ferrous iron was determined by colorimetry, using a microprocessor-controlled, single beam Hach spectrophotometer. Samples were introduced into an AccuVac Ampul and mixed quickly. Phenanthroline in the ampul reacts with ferrous iron in the sample to form an orange color in proportion to the ferrous iron concentration. Ferric iron does not react. The ampul was then placed into the spectrophotometer and concentration was measured. For concentrations higher than 3 ppm, solutions were diluted and re-analyzed. Limits of determination are shown in table A6 below.

## Alkalinity by Titration

On-site alkalinity tests were done on all samples collected throughout the study, using a field-portable CHEMetrics titration kit. For comparison, water samples were also collected for laboratory alkalinity determination. For the laboratory alkalinity, an Orion 960 Autochemistry System was used for endpoint titration analysis. The titrant was added to 50 ml of sample until a pH of 4.5 was achieved. Alkalinity was then calculated and reported in units of ppm as CaCO<sub>3</sub>. Limits of determination are shown in table A6 below.

**Table A6.** Lower and upper determination limits for mercury by the cold-vapor AAS technique, ferrous iron by colorimetry, and alkalinity by titration.

Element	Method	Lower Determination Limit	Upper Determination Limit
Hg	CV	0.1 ppb	None <sup>1</sup>
Fe <sup>2+</sup>	CO	0.01 ppm	None <sup>1</sup>
Alkalinity	TI	1.0 ppm	None <sup>1</sup>

<sup>1</sup> Samples with high concentrations were diluted and re-analyzed.