

Baseline geochemical data for stream sediment and surface water samples from Panther Creek, the Middle Fork of the Salmon River, and the Main Salmon River from North Fork to Corn Creek, collected prior to the severe wildfires of 2000 in central Idaho

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

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#### INTRODUCTION

During a 3-week period in the summer of 1996, the U.S. Geological Survey conducted a reconnaissance baseline geochemical study in central Idaho. Areas covered include Panther Creek, the Middle Fork of the Salmon River from Boundary Creek to the mouth, and the Main Salmon River from North Fork to Corn Creek (Fig. 1). Stream sediment samples were collected at all sample sites. Filtered and unfiltered stream water samples were collected at most sample sites.

The purpose of the baseline study was to establish a "geochemical snapshot" of the area, as a datum for monitoring future change in the geochemical landscape, whether natural or human-induced. Events that could change the geochemical landscape include, but are not limited to, mining, flood, landslide, wildfire, or resource extraction activities. In the summer of 2000, there were numerous large wildfires in central Idaho. In particular, the Clear Creek (206,000 acres; 83,370 hectares), Little Pistol (74,000 acres; 29,950 hectares), and Shellrock (64,000 acres; 25,900 hectares) fires swept across much of the area that was sampled. Thus, these data represent a pre-fire baseline geochemical dataset. A 2001 post-fire study is planned and will involve re-sampling of the pre-fire baseline sites, to allow for pre- and post-fire comparison.

Sampling was conducted from July 8-28, 1996. Weather during this period was warm and precipitation sparse. Only one rain event occurred during the sampling period—a brief rain storm on the evening of July 16, with about ¼" (0.64 cm) of total precipitation. River levels were lower than normal for this time of year because of lower-than-normal precipitation during the previous winter and spring.

The study area lies within the Salmon River Mountains, and much of the area—particularly along the Middle Fork of the Salmon River—lies within the Frank Church-River of No Return Wilderness (Fig. 1). Topographic relief is high. Peaks at the heads of drainages commonly have elevations above 9000 ft (2740 m), while river-level elevations are typically several thousand feet lower, ranging from around 5700 ft (1740 m) at Boundary Creek on the Middle Fork of the Salmon River, to less than 3000 ft (914 m) at Corn Creek on the Main Salmon River. Terrain ranges from rugged, steep peaks, ridges, and cirques at higher elevations, through tree-covered mountains and meadows at intermediate elevations, to steep, narrow, heavily vegetated canyons at lower elevations. Climatic conditions vary from warm summer days with frequent thunderstorms to cold winter days with heavy snowfall accumulations.

Access to the Panther Creek basin is by U.S. Forest Service gravel roads 030 from North Fork, Idaho, and 055, which runs along Panther Creek. Access to the Main Salmon River from North Fork to Corn Creek is by U.S. Highway 93 and U.S. Forest Service gravel road 030, which runs along the Main Salmon, ending at Corn Creek, and by raft or jet boat. Access to the Middle Fork of the Salmon River is exclusively by oar-powered raft or pack trail.

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#### **GEOLOGIC SETTING**

Precambrian metamorphic rocks, the Late Cretaceous Idaho batholith, and Eocene plutons related to the Challis Volcanics are the predominant rock types in the study area. Faults in the area are mainly northeast and northwest trending.

The metamorphic rocks are principally quartzites and gneisses of the Precambrian Yellowjacket and Apple Creek Formations. Sporadic roof pendants and xenoliths of schist, quartzite, and calc-silicate rocks of uncertain Precambrian to Paleozoic age are found locally in close spatial association with the Idaho batholith.

The Idaho batholith varies from leucocratic granite to biotite granodiorite and hornblende-biotite granodiorite. It is typically salt-and-pepper gray in color and locally porphyritic with large microcline phenocrysts. The batholith is widespread in central Idaho, covering some 25,000 square miles (64,700 sq km).

Eocene plutons found in the study area are generally intermediate to granitic in composition and typically have an overall pink color, such as the large Casto Pluton, which crops out along portions of the Middle Fork of the Salmon River. These plutons are the crystalline, unerupted counterparts to the voluminous Challis Volcanics that are found in much of central Idaho.

#### METHODS OF STUDY

#### SAMPLE MEDIA

Geochemical sample media collected include stream-sediment, heavy-mineral-concentrate, and water samples. The chemical composition of a stream-sediment sample is controlled primarily by the major geologic units within the drainage basin and to a lesser degree by metal-scavenging materials such as amorphous iron- and manganese oxides, clays, and organic matter. Minor elemental constituents within the stream sediment, such as elements related to mineral deposits within the drainage basin, may be detected in the sediment analysis, but commonly have a small overall influence on the sample because of dilution by barren material.

Since elements related to mineralized rocks are commonly found in heavy minerals, heavy-mineral-concentrate samples from stream sediment were also collected. Heavy-mineral concentrates provide chemical information about ore-related and rock-forming dense minerals, and permit chemical determination of some elements not easily detected in stream-sediment samples. Further, microscopic identification of nonmagnetic minerals in heavy-mineral-concentrate samples may provide additional useful mineralogical information.

Water samples were collected from available natural water sources, principally from flowing streams and rivers, but also from hot springs. Mineral deposits rich in sulfide minerals (whether mined or unmined), solid waste from mine dumps and mill tailings derived from such deposits, and sulfide-rich rocks from areas of hydrothermally altered bedrock, are possible sources of acid and metal loading in the environment. Locally, hot springs and certain geologic units can contribute high concentrations of dissolved constituents to surface waters.

#### SAMPLE COLLECTION AND PREPARATION

A count of the various types of samples collected from each of the three basins studied is listed in Table 1. In the table, "site dupes" refers to sample quality control site duplicates collected in the field. A sample site map is provided in figure 2.

Table 1. Count of types of samples collected in this study.

	Total	Panther Creek	Middle Fork Salmon	Main Salmon
Water	80	11	48	21
Site dupes	4	1	2	1
Sediment	86	24	42	20
Site dupes	4	1	2	1
Concentrate	34	6	10	18

#### Stream Sediments

Each stream-sediment sample consisted of alluvium from the active stream channel, composited by collecting sediment increments from several places at the sample site, generally along a 30-ft (10-m) stretch of the channel. In order to improve sample representativity, an attempt was made to collect 20 to 30 increments at each site. However, at some sites sparse distribution of available sediment reduced the number of increments to less than 10. The sediment was sieved on site with a 10 mesh (2 mm) stainless steel screen. A 2-lb (0.9-kg) sample of minus-10 mesh sediment was collected in a cloth bag and air-dried.

In the laboratory, stream sediment samples were air-dried and sieved at 80 mesh (0.177 mm), following the method of Peacock and others (1996). The coarse fraction was discarded. The fine sediment fractions were pulverized to a fine flour consistency (minus-100 mesh/0.149 mm), with clean quartz sand pulverized between each sample to reduce risk of cross-contamination. For each sample, an approximate 6.5-oz (185-g) portion was saved for chemical analyses; any remaining material was subsequently archived.

## Heavy Mineral Concentrates

Panned concentrate samples were collected from the same active alluvium as sediment samples, from around boulders and in coarse gravels. The pan concentrate samples were not composited as the stream sediment samples were, but were collected as grab samples in areas where heavy minerals tend to accumulate. A 14-inch stainless steel gold pan was filled with stream sediment sieved to minus-10 mesh (2 mm) with a stainless steel screen, resulting in approximately 16 lb (7.2 kg) of material. This sieved alluvium was panned at the site when running water was available, or collected in a cloth bag for later panning. The alluvium was panned until most of the less-dense minerals (primarily quartz and feldspar), organic materials, and clays were removed. Generally, one to three percent of the original sample remained after panning. The panned sample was bagged, air-dried, and saved for further laboratory preparation.

In the laboratory, panned concentrate samples were sieved to minus-20 mesh (0.84 mm), and then gravity separated using bromoform (specific gravity about 2.85) to remove remaining light minerals, primarily quartz and feldspar. The resultant heavy-mineral-concentrate sample was separated into magnetic, weakly magnetic, and nonmagnetic fractions using a modified Frantz Isodynamic Separator (Taylor and Theodorakos, 1996). The magnetic fraction was extracted at a setting of 0.25 ampere and contains primarily magnetite and ilmenite. The weakly magnetic fraction was extracted at a setting of 1.75 ampere and consists largely of ferromagnesian silicates and iron oxides. The remaining nonmagnetic fraction may contain many ore-related minerals including sulfide minerals, gold and other native metals, and some accessory oxides and silicates. The nonmagnetic heavy-mineral-concentrate samples were split using a Jones splitter. One split was hand ground with an agate mortar and pestle for chemical analysis and the other split was used for microscopic mineralogical analysis. Clean quartz sand

was hand ground between samples to clean the mortar and pestle, thereby reducing the risk of cross-contamination between samples.

#### Water

This study was reconnaissance in nature, covering a large area in a short period of time. Thus, for collection of water samples, we did not adhere strictly to the rigorous "parts per billion" protocol established by the U.S. Geological Survey Water Resources Division (Horowitz and others, 1994). Rather, procedures were streamlined to facilitate the sample collection process, as described below.

A clean 1-liter polypropylene bottle was used for water sampling. The bottle was rinsed prior to sample collection. While rigorous width- and depth-integrated sampling protocol was not followed, an attempt at collecting integrated samples was made by collecting at intervals across the entire width of the stream or river (width integration). We collected samples below riffles or white water—natural zones of mixing—as a proxy for depth integration. The filled 1-liter bottles were capped and shaken prior to collecting sub-samples from them. Sub-samples were collected in polypropylene bottles rinsed on site with unfiltered water for unfiltered water samples and with filtered water for filtered samples. Bottles for acidified samples were prerinsed in the laboratory with a 10 percent nitric acid solution. In all cases, care was taken at each site to minimize contamination by rinsing bottles and equipment; and by using new, unpowdered vinyl gloves, disposable equipment (filters, syringes, etc.), and clean plastic sheets to cover onsite work areas.

At most sites, three sub-samples were collected: (1) an unacidified, filtered raw water sample for anion analysis, (2) an acidified, unfiltered sample for trace and major cation analysis of both dissolved and suspended species, (3) an acidified, filtered sample for trace and major cation analysis of dissolved species. The unacidified samples were kept in an iced cooler in the field and in a refrigerator in the laboratory prior to analysis. Samples were filtered with sterile 0.45-micron disposable filters and acidified to pH < 2 with ultra-pure, concentrated nitric acid to prevent precipitation of metals and bacterial growth.

At a few sites (principally hot springs) water was collected for ferrous iron content. Sample collection bottles were protected from direct sunlight. The ferrous iron water samples were filtered as described above; collected in opaque, dark brown polypropylene bottles to prevent light penetration; and acidified with ultra-pure, concentrated hydrochloric acid to pH < 2.

Other water data collected and recorded on-site include temperature, pH, conductivity, dissolved oxygen content, an estimate of the water flow rate, and total alkalinity. Conductivity and pH were collected with Orion meters calibrated with standards at each site prior to sample measurement. For pH, two calibration buffer standards that bracketed the sample's pH were used. Dissolved oxygen content was determined using a field-portable CHEMetrics brand colorimetric test kit. Total alkalinity measurements were collected from using a field-portable CHEMetrics titration kit. Alkalinity is a measure of total acid-neutralizing capacity of water and is reported in ppm as CaCO<sub>3</sub>.

Flow rates were not calculated, but rather are ball-park estimates and should be used conservatively. These flow estimates were determined by the 3 river guides on the trip, whose combined river floating experience totaled over 75 years. Later, the flow estimates for the Middle Fork and Main Salmon Rivers were compared with published flow records for the sampling period (Brennan and others, 1996) and with unpublished records maintained by the

Middle Fork Ranger District. The flow estimates were found to be within  $\pm$  25 % at locations where flows are officially recorded.

## **ANALYTICAL TECHNIQUES**

A large number of chemical elements were determined, using a variety of quantitative and semi-quantitative analytical techniques. Table 2 shows the various elements determined and analytical methods used for each of the sample media collected in the study. A brief description and published references for each analytical method is given below. Descriptions and quality assurance/quality control (QA/QC) protocol for most of the analytical methods used in this study are found in Arbogast (1996). U.S. Geological Survey laboratories analyzed all water and heavy-mineral concentrate samples. Stream sediment samples were analyzed by XRAL Laboratories, Inc. of Don Mills, Ontario, Canada, under a contract with the U.S. Geological Survey.

**Table 2. Elements determined and analytical methods used for all sample media.** [method codes: MW, water by ICP mass spectrometry; EW, water by ICP atomic emission spectrometry; IC, water by ion chromatography; ET, solid by ICP atomic emission spectrometry; EP, solid by partial-extraction ICP atomic emission spectrometry; GF, solid by graphite furnace atomic absorption; HY, solid by hydride generation atomic absorption; CV, solid by cold-vapor atomic absorption; ES, solid by semi-quantitative emission spectrography]

Sample Media	Method	Elements determined	
Water,	MW	Ag, Al, As, Au, Ba, Be, Bi, Ca, Cd, Ce, Co, Cr, Cs,, Cu, Dy,	
filtered/acidified (FA) and		Er, Eu, Fe, Ga, Gd, Ge, Ho, In, K, La, Li, Mg, Mn, Mo, Na,	
unfiltered/acidified (RA)		Nd, Ni, P, Pb, Pr, Rb, Re, Sb, Se, Sm, Sr, Tb, Th, Ti, Tl, Tm,	
		U, V, W, Y, Yb, Zn	
	EW	Ag, Al, As, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Li, Mg, Mn,	
		Mo, Na, Ni, P, Pb, Sb, Si, Sr, Ti, V, Zn	
Water,	IC	Cl <sup>-</sup> , F <sup>-</sup> , NO <sub>3</sub> <sup>-</sup> , SO <sub>4</sub> <sup>-2</sup>	
filtered/not acidified (FU)			
Stream Sediments	ET	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, Zn	
	EP	Ag, As, Au, Bi, Cd, Cu, Mo, Pb, Sb, Zn	
	GF	Au	
	HY	Se	
	CV	Hg	
Heavy Mineral	ES	Ag, As, Au, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, Ge,	
Concentrates		La, Mg, Mn, Mo, Na, Nb, Ni, P, Pb, Pt, Pt, Sb, Sc, Sn, Sr,	
		Th, Ti, V, W, Y, Zn, Zr	

In this study, quality assurance/quality control (QA/QC) concerns were addressed through the use of internal reference standards, field blanks, sample site duplicates, and analytical duplicates. QA/QC samples comprised approximately 10 percent of the total number of samples analyzed. 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. 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 20 percent. Water samples included field blanks of de-ionized water, used to check for contamination from sampling equipment and

preservatives. Field blanks were collected near the beginning, midway, and near the end of the sampling period, following the same procedures as those used for normal water samples. Sediment and water sample site duplicates were collected randomly. The site duplicates were collected following the same procedures as those used for normal water and sediment samples. Only the sample site duplicate analyses are retained in the published data files.

In the data files for the various sample media, discrepancies in element concentration for the same sample determined by different analytical methods (for example, gold) may be attributable to the particulate nature of certain elements, different sample weights used, different dissolution and extraction procedures, and to instrumental bias. For gold in particular, the graphite furnace atomic absorption spectrophotometric (GF) analytical method provides the most statistically representative results, due to the larger sample weight analyzed.

For simplicity, reporting units for all sample media are percent (%), parts per million (ppm), or parts per billion (ppb). Water sample analyses, which are commonly reported in the literature as milligrams per liter or micrograms per liter, are given here as ppm or ppb, respectively.

# Techniques Used on Stream Sediment and Concentrate Samples Inductively Coupled Plasma-Atomic Emission Spectrometry—Stream Sediments

Two ICP-AES methods were used in the study for multi-element analyses of stream sediments: (1) a 40-element total digestion method (designated "ET" in Table 2 and in the data files), and (2) a 10-element, partial-extraction method (designated "EP" in Table 2 and in the data files).

In the first multi-element method (ET), 40-element ICP-AES, samples were digested and analyzed following the procedure of Briggs (1996). Samples (0.2 g) were digested using a mixture of hydrochloric, nitric, perchloric, and hydrofluoric acids, and the solutions were heated at  $110^{\circ}$  C until dry. Additional perchloric acid and water were added to the residue and the mixture was then taken to dryness at  $150^{\circ}$  C. Aqua regia and dilute nitric acid were added to the residue to bring the solution to a final volume, the solution was heated at  $95^{\circ}$  C for an hour, and then, after cooling, the sample was aspirated into the argon plasma and element concentrations were determined simultaneously with a multi-channel ICP-AES instrument. Calibration is performed by standardizing with digested rock reference materials and with a series of multi-element solution standards. Limits of determination for 40-element ICP-AES are shown in Appendix 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%.

In the second multi-element method (EP), concentrations of Ag, As, Au, Bi, Cd, Cu, Mo, Pb, Sb, and Zn were determined on samples by a 10-element ICP-AES partial extraction procedure developed by Motooka (1996). This procedure solubilizes metals not tightly bound in the silicate lattice of rocks, soils, and stream sediments; metals tightly bound in highly resistant minerals are not extracted. Samples (1 g) were decomposed with concentrated hydrochloric acid and hydrogen peroxide in a hot-water bath. Metals were extracted in diisobutyl ketone (DIBK)/Aliquat 336 in the presence of ascorbic acid and potassium iodide. The DIBK/Aliquat 336 phase was then aspirated directly into the argon plasma and element concentrations were determined simultaneously with a multi-channel ICP-AES instrument. Limits of determination for 10-element ICP-AES are shown in Appendix Table A2. It is important to note that this procedure is a partial digestion and depending on element availability, results may be biased low

when compared to other methods of analyses. In addition, high Cu content in samples can cause interferences for elements determined by this method; for samples with high Cu content, data by this method should be used cautiously (J. M. Motooka, U.S. Geological Survey, oral commun., 1995). *Analytical Performance:* Data were deemed acceptable if recovery for all 10 elements was  $\pm 20\%$  at five times the LOD and the calculated RSD of duplicate samples was no greater than 15%.

## **Atomic Absorption Spectrophotometry—Stream Sediments**

Two atomic absorption spectrophotometric (AAS) methods were used for determining selenium and gold in stream sediment samples. Determination limits for these techniques are given in Appendix Table A2.

Sediment samples were analyzed for selenium using a continuous-flow hydride generation AAS (Hageman and Welsch, 1996). The samples (0.25 g) were digested by adding concentrated nitric, perchloric, and hydrofluoric acids and heating. After cooling, hydrochloric acid was added to form Se (IV), necessary for determination by hydride generation. A mixture of hydrochloric acid, sodium borohydride, and sodium hydroxide was added to produce selenium hydride. The selenium hydride gas was then stripped off the liquid using a phase separator and transported with inert gas to the atomizer of the atomic absorption spectrophotometer where selenium concentration was determined. This method is designated HY in Table 2 and in the data files. *Analytical Performance:* 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%.

Gold was determined in stream sediments by graphite furnace atomic absorption spectrophotometry following the method of O'Leary and Meier (1996). Samples were digested using a hydrobromic acid-bromine digestion, an MIBK extraction, and then gold was determined on the solutions by graphite-furnace atomic absorption. This method is designated GF in Table 2 and in the data files. *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 was measured in sediment samples using a modification of the atomic absorption method of O'Leary and others (1996). This method is designated CV in Table 2 and in the data files. Mercury was determined by weighing out 0.1 g of sample and digesting with a mixture of sulfuric acid, nitric acid, 5% potassium permanganate, and 5% potassium peroxydisuflate in a water bath for one hour. The excess of potassium permanganate was reduced with hydroxylamine sulfate solution and then Hg (II) was reduced with stannous chloride. The Hg vapor was separated and measured using a LEEMAN PS200 automated mercury analyzer. Samples exceeding the working range of 0.02 to 1.8 ppm mercury required dilution. Determination limits for this technique are given in Appendix Table A2. *Analytical Performance:* Data were deemed acceptable if recovery of mercury was ±20% at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%.

## **Semiquantitative Emission Spectrography—Concentrates**

The minus-20-mesh nonmagnetic heavy-mineral-concentrate samples were analyzed for 37 major, minor, and trace elements by a direct-current arc, semiquantitative emission spectrographic (SES) technique (Adrian and others, 1996). Spectrographic results were determined by visually comparing spectra derived from the sample and recorded on photographic film against spectra obtained from laboratory reference standards. Standard concentrations are

geometrically spaced over any given order of magnitude as follows: 100, 50, 20, 10, 5, 2 etc. Samples whose concentrations were estimated to fall between those values were assigned values of 70, 30, 15, 7, 3, 1.5 etc. Elements determined by SES are Ag, As, Au, B, Ba, Be, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, Ge, La, Mg, Mn, Mo, Na, Nb, Ni, P, Pb, Pd, Pt, Sb, Sc, Sn, Sr, Th, Ti, V, W, Y, Zn, and Zr. This method is designated ES in Table 2 and in the data files. Limits of determination for elements determined by SES are listed in Appendix Table A3. Analytical Performance: The precision of this analytical technique is approximately ± one reporting interval at the 83 percent confidence level and ± two reporting intervals at the 96 percent confidence level (Motooka and Grimes, 1976).

# Techniques Used on Water Samples

## **Inductively Coupled Plasma-Atomic Emission Spectrometry**

Acidified water samples were analyzed for major (Al, Ca, Fe, K, Mg, Na, and Si) and selected trace elements following the ICP-AES method of Briggs and Fey (1996) (designated "EW" in Table 2 and in the data files). Water samples were aspirated directly into an argon plasma and element concentrations were determined by ICP-AES. Limits of determination for the multi-element ICP-AES method for water samples are shown in Appendix Table A4. *Analytical Performance:* Data were deemed acceptable if recovery was ±20% at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%.

## Ion Chromatography

The anions Cl<sup>2</sup>, F<sup>2</sup>, NO<sub>3</sub>, and SO<sub>4</sub><sup>2</sup>-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 an ion 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 calibration standards to determine sample concentrations. This method is designated IC in Table 2 and in the data files. Limits of determination for anions in raw water samples are shown in Appendix Table A5. *Analytical Performance:* Data were deemed acceptable if recovery was ±20% at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%.

## **Inductively Coupled Plasma-Mass Spectrometry**

Acidified-filtered and acidified-unfiltered waters were analyzed to determine 51 elements by ICP-MS using a method developed by the U.S. Geological Survey (A.L. Meier, personal commun., 1995; Meier and others, 1994). This method is designated MW in Table 2 and in the data files. 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 ICP-MS are shown in Appendix Table A6. *Analytical Performance:* Data were deemed acceptable if recovery was ±20% at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%.

## **Ferrous Iron by Colorimetry**

Ferrous iron was determined by colorimetry, using a microprocessor-controlled, single beam Hach spectrophotometer (Hach Company, 1996). 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. The limit of determination for this method is 0.01 ppm. *Analytical Performance*: Data were deemed acceptable if recovery was ±20% at five times the LOD and the calculated percent RSD of duplicate samples was no greater than 20%.

## Which Technique Should I Use?

In some instances, the same elements were determined by more than one analytical technique (Table 2). This section is provided as guidance for those cases where multiple values are listed for a given element in a sample. Filtered/acidified and unfiltered acidified water samples were analyzed by both ICP-MS and ICP-AES. In general, the ICP-AES method is more quantitative and data from this method is preferred over ICP-MS in those instances where values are given by both methods. This is particularly true for the major elements Al, Ca, Fe, K, Mg, Na, and Si, where concentrations commonly exceeded the dynamic range of the ICP-MS instrument. For solid sample media, gold analyses by graphite furnace atomic absorption are in all cases preferred over gold values reported by ICP-AES methods. As described above, high copper content in stream sediment samples can cause interferences on other elements in the 10-element, partial extraction ICP-AES method (EP). In these instances, the total extraction ICP-AES method (ET) is preferred for these elements.

## **DESCRIPTION OF DATA FILES**

The analytical data files are provided in two formats, as a Microsoft Access 2000 database and as separate Microsoft Excel .XLS files (version 3.0). The data in the Access database are arranged as a series of tables. The tables and their brief descriptions follow below. For those not using Microsoft Access 2000, the tables are saved as separate .XLS files with the same name and fields. The following abbreviations are used in the tables: FeOx, iron oxide; MnOx, manganese oxide; ppt, precipitate.

## FIELD NUMBERS

The field number coding scheme is as follows: The first two numbers indicate the year the sample was collected. The next two letters (SA) indicate samples from Salmon National Forest. The next 3 digits indicate the sample site number. Collectively, these three parameters comprise the Site ID. Following the 3-digit number are suffixes indicating sample media type and, if applicable, QA/QC samples. The Site ID and media suffix together comprise the sample field number.

Media	Suffix
Stream sediment	S
Heavy-mineral concentrate	C
Filtered/unacidified water	FU
Unfiltered/acidified water	RA

Filtered/acidified water FA Water for ferrous iron analysis FE

For QA/QC samples, a "D" suffix immediately following the 3-digit number indicates a sample site duplicate. These suffixes precede the sample media type suffix. Thus, field number 96SA072S indicates a sediment sample from site 96SA072, while field number 96SA072DS indicates a site duplicate of the same.

#### **COORDINATES**

Sample site locations are given in the SampleSiteInfo table as both degrees-minutes-decimal seconds and decimal degrees. The coordinates were determined by digitizing sample locations as plotted on U.S. Geological Survey 1:24,000-scale topographic maps. The datum was 1927 CONUS and the spheroid was Clarke 1866.

## **GEOCHEMICAL ANALYSES**

All geochemical data are given in the following format. Field identifiers consist of a single line. The first one or two letters give the chemical element symbol, then units of measurement, and finally a code for the analytical method used for the element in that particular column. These three items are separated by underscores. Element symbols and associated names are shown in the appendix. Units of measurement are: PPM, parts per million; PPB, parts per billion; and PCT, percent. The analytical methods and associated code letters are:

CV cold-vapor atomic absorption spectrophotometry for mercury

CO colorimetric method for ferrous iron

EP inductively coupled plasma-atomic emission spectrometry (partial extraction)

ET inductively coupled plasma-atomic emission spectrometry (total digestion)

ES semiquantitative emission spectrography

EW inductively coupled plasma-atomic emission spectrometry (water samples)

GF graphite-furnace atomic absorption spectrophotometry

HY hydride generation atomic absorption spectrophotometry

IC ion chromatography

MW inductively coupled plasma-mass spectrometry (water samples)

For example, As\_PPM\_ET indicates arsenic, in parts per million, determined by total digestion inductively coupled plasma-atomic emission spectrometry. For all geochemical data except heavy mineral concentrates, the symbol "<" indicates that an element was not observed at the lower limit of determination shown. A ">" indicates that an element was detected but in concentration above the upper limit of determination shown. A blank entry indicates that the sample was not analyzed for that particular element. For heavy mineral concentrate geochemical analyses, the following symbols are used: N(x), not detected at the limit of determination given in parentheses; L(x), detected, but less than the limit of determination given in parentheses; C(x), greater than the upper determination limit given in parentheses. A dash indicates no analysis.

#### DESCRIPTIONS OF TABLES AND FIELDS WITHIN TABLES

Below are brief descriptions of the various tables and fields contained within the tables. First are tables containing sample and site descriptive information, next are tables containing chemical analyses. Fields common to all or most tables are listed together. Fields unique to specific tables are listed below those tables.

## Tables containing sample and site descriptive information

Table Description

tblSampLog table of all sample sites and the sample media collected at each site

Fields: Field Type: Contents:

Site ID text sample site identifier (field is common to all tables)

Water\_FU number filtered (0.45 micron), unacidified water Water RA unfiltered, acidified (HNO<sub>3</sub>) water

Water\_FA number filtered (0.45 micron), acidified (HNO<sub>3</sub>) water Water FE2+ number filtered (0.45 micron), acidified (HCl) water for

Fe<sup>2+</sup> analysis

Stream Sed number stream sediment sample (minus 80 mesh)

Concentrate number heavy-mineral concentrate sample (minus 20 mesh)

Table Description

**tblSiteInfo** information relevant to all sample sites

Fields: Field Type: Contents:

Site ID text sample site identifier (field is common to all tables)

LatDeg number latitude, degrees
LatMin number latitude, minutes

LatSec number latitude, decimal seconds

LongDeg number longitude, degrees LongMin number longitude, minutes

LongSec number longitude, decimal seconds
LatitudeDD number latitude in decimal degrees
LongitudeDD number longitude in decimal degrees

Elev ft number elevation at sample site, in feet; from 1:24,000

topographic map

Quadrangle text 7.5' USGS quadrangle on which the site is located

County text county that site is located in Date\_Coll date/time date of sample collection at site Time\_Coll date/time time of sample collection at site Location\_Info text brief sample location descriptor Site Descript text brief sample site descriptor

Deposit Type text brief description of mineral deposit(s) located

upstream of sample site

Deposit Name text name of deposit(s) located upstream of site

Mine District text mining district name

Weather text weather at time of sample collection

Air\_Temp\_C text air temperature (Celsius) at time of collection Geol Bedrock memo description of bedrock geology at the site

Contamination text description of anthropogenic contamination at site

Table Description

**tblWatSiteI** information relevant specifically to water sample sites

Fields: Field Type: Contents:

Site ID text sample site identifier (field is common to all tables)

Temp_C	number	water temperature at collection site, degrees Celsius
Flow_Rate	number	estimated water flow rate (ball-park estimate)
Flow_Units	text	units of measurement for flow (gpm, gallons per
		minute; cfs, cubic feet per second)
pН	number	water pH at collection site
Conduct_uS/cm	number	water conductivity at collection site, in
		microsiemens per centimeter
Diss_O2_ppm	number	dissolved oxygen in water at collection site, in ppm
Alkalin_ppm	number	water alkalinity at collection site, as parts per
		million equivalent CaCO <sub>3</sub>
Water_Source	text	source of water samples collected
Type_of_Site	text	water sample site descriptor
Water_Color	text	color of sampled water
Water_Odor	text	odor of sampled water
Turbidity	text	qualitative estimate of water turbidity
Channel_Bed	text	brief description of water channel bed
Other_Notes	text	additional descriptive information for sample or site

## Tables containing geochemical analyses

The following fields are common to all or most tables containing geochemical analyses:

Fields:	Field Type:	Contents:
Site_ID	text	sample site identifier (field is common to all tables)
Field_No	text	sample field number; this is the site identifier with
		applicable QA/QC suffix and sample media suffix;
		for example 96SA072DFA
Lab_No	text	laboratory-assigned sample number; this is the
		sample record ID in the USGS National
		Geochemical Database
Actual_Sample	text	description of actual sample analyzed, following
		sample preparation
QA/QC_Info	text	quality assurance/quality control information

For the following tables, geochemical analyses follow the common fields described above. Format for the geochemical analyses are as described above in the **GEOCHEMICAL ANALYSES** section. All geochemical analyses are text fields due to the presence of the qualifiers "<", ">", N, L, and G.

Table Description

**tblWaterFU** anion analyses, filtered/unacidified water **tblWaterRA** cation analyses, filtered/acidified water **tblWaterFE** tblWaterFE Fe<sup>2+</sup> analyses, filtered/acidified water

The following fields are unique to specific tables:

Table Description

**tblConcentr** heavy-mineral concentrate descriptive and analytical data

Fields:	Field Type:	Contents:
Sieve_Size	text	sample sieve size as prepared in laboratory
Sample_Descr	text	brief sample description
Character	text	character of sample: single grab or composite of several increments
Source	text	sample collected from outcrop, float, alluvium, etc.
Mineral_Scan	text	on-site hand lens examination of panned concentrate

Geochemical analyses follow at this point.

Table	Description
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sediment describitive and analytical d	tblSediment	sediment descriptive and analytical data
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ument	scument acscriptive	and anarytical data
Fields:	Field Type:	Contents:
Sample_Desc	r text	brief sample description
Character	text	character of sample: single grab or composite of
		several increments
Source	text	sample collected from outcrop, float, alluvium, etc.
Organic_Cont	t text	relative organic content in sample collected
Sieve_Size	text	sample sieve size as prepared in laboratory
Stain_Alluv	text	iron oxide or manganese oxide staining observed on
		alluvium
Geology_Allu	ıv text	geologic description of alluvium at sample site
Geoch	emical analyses follow	v at this point.

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#### APPENDIX

Table A1. Limits of determination for stream sediment samples analyzed by 40-element inductively coupled plasma-atomic emission spectrometry, total extraction (ET).

muuctively cot	ipicu piasilia		sion spectrome	ii y, totai cati	action (ET).
Element		Upper Determination	Element		Upper Determination
	Limit	Limit	_	Limit	Limit
	Percent			rts Per Million	
Al, aluminum	0.005	50	Ga, gallium	4	50,000
Ca, calcium	0.005	50	Ho, holmium	4	5,000
Fe, iron	0.02	25	La, lanthanum	2	50,000
K, potassium	0.01	50	Li, lithium	2	50,000
Mg, magnesium	0.005	5	Mn, manganese	4	50,000
Na, sodium	0.005	50		2	50,000
P, phosphorus	0.005	50	Nb, niobium	4	50,000
Ti, titanium	0.005	25	Nd, neodymium	9	50,000
	arts Per Million		Ni, nickel	3	50,000
Ag, silver	2	10,000	Pb, lead	4	50,000
As, arsenic	10	50,000	Sc, scandium	2	50,000
Au, gold	8	50,000	Sn, tin	5	50,000
Ba, barium	1	35,000	Sr, strontium	2	15,000
Be, beryllium	1	5,000	Ta, tantalum	40	50,000
Bi, bismuth	10	50,000	Th, thorium	6	50,000
Cd, cadmium	2	25,000	U, uranium	100	100,000
Ce, cerium	5	50,000	V, vanadium	2	30,000
Co, cobalt	2	25,000	Y, yttrium	2	25,000
Cr, chromium	2	25,000	Yb, ytterbium	1	5,000
Cu, copper	2	15,000	Zn, zinc	2	15,000
Eu, europium	2	5,000		<u> </u>	<u> </u>

**Table A2.** Limits of determination for selected elements in stream sediment samples analyzed by other methods. [Values are in parts per million; EP, partial extraction 10-element inductively coupled plasma-atomic emission spectrometry; ; GF, graphite-furnace atomic absorption spectrophotometry; CV, cold-vapor atomic absorption spectrophotometry; HY, hydride generation atomic absorption spectrophotometry]

Element	Method	Lower Determination Limit <sup>1</sup>	Upper Determination Limit <sup>1</sup>
Ag, silver	EP	0.08	400
As, arsenic	EP	1	6,000
Au, gold	EP	0.1	1,500
Bi, bismuth	EP	1	6,000
Cd, cadmium	EP	0.05	500
Cu, copper	EP	0.05	500
Mo, molybdenum	EP	0.1	900
Pb, lead	EP	1	6,000
Sb, antimony	EP	1	6,000
Zn, zinc	EP	0.05	500
Au, gold	GF	0.002	10,000
Hg, mercury	CV	0.02	1.8
Se, selenium	HY	0.01	4

<sup>&</sup>lt;sup>1</sup> Limits of determination shown here are nominal and limits may vary in the data files. The variability in limits of determination is due to variable sample weight used, dilution of the sample solution, instrumental interference correction, and slight changes in methodology over time.

## APPENDIX (cont.)

Table A3. Limits of determination for nonmagnetic heavy-mineral concentrate samples

analyzed by semiquantitative emission spectrography (ES).

Element	Lower Determination Limit	Upper Determination Limit	Element	Lower Determination Limit	Upper Determination Limit
Percent			Parts Per Million		
Ca, calcium	0.1	50	Ge, germanium	20	200
Fe, iron	0.1	50	La, lanthanum	100	2,000
Mg, magnesium	0.05	20	Mn, manganese	20	10,000
Na, sodium	0.5	10	Mo, molybdenum	10	5,000
P, phosphorus	0.5	20	Nb, niobium	50	5,000
Ti, titanium	0.005	2	Ni, nickel	10	10,000
P	arts Per Million		Pb, lead	20	50,000
Ag, silver	1	10,000	Pd, palladium	10	2,000
As, arsenic	500	20,000	Pt, platinum	50	2,000
Au, gold	20	1,000	Sb, antimony	200	20,000
B, boron	20	5,000	Sc, scandium	10	200
Ba, barium	50	10,000	Sn, tin	20	2,000
Be, beryllium	2	2,000	Sr, strontium	200	10,000
Bi, bismuth	20	2,000	Th, thorium	200	5,000
Cd, cadmium	50	1,000	V, vanadium	20	20,000
Co, cobalt	20	5,000	W, tungsten	50	20,000
Cr, chromium	20	10,000	Y, yttrium	20	5,000
Cu, copper	10	50,000	Zn, zinc	500	20,000
Ga, gallium	10	10,000	Zr, zirconium	20	2,000

Table A4. Limits of determination for acidified water samples analyzed for 24 elements by

inductively coupled plasma-atomic emission spectrometry (EW).

Element	Lower Determination Limit <sup>1</sup>	Upper Determination Limit	Element	Lower Determination Limit <sup>1</sup>	Upper Determination Limit
Parts Per Million			Parts Per Billion		
Al, aluminum	0.1	1,000	Cr, chromium	1	10,000
Ca, calcium	1	1,000	Co, cobalt	1	10,000
Fe, iron	0.2	1,000	Cu, copper	1	10,000
K, potassium	0.2	1,000	Li, lithium	1	10,000
Mg, magnesium	1	1,000	Mn, manganese	1	10,000
Na, sodium	1	1,000	Mo, molybdenum	80	10,000
P, phosphorus	0.5	1,000	Ni, nickel	1	10,000
Si, silicon	1	1,000	Pb. lead	20	10,000
Parts Per Billion			Sr, strontium	20	10,000
B, boron	10	10,000	Ti, titanium	200	10,000
Ba, barium	1	10,000	V, vanadium	1	10,000
Be, beryllium	1	10,000	Zn, zinc	1	10,000
Cd, cadmium	1	10,000			

<sup>&</sup>lt;sup>1</sup> Limits of determination shown here are nominal and limits may vary in the data files. The variability in limits of determination is due to variable sample weight used, dilution of the sample solution, and instrumental interference correction.

## APPENDIX (cont.)

Table A5. Limits of determination for anions in water samples determined by ion chromatography (IC).

Anion	Lower Determination Limit	Upper Determination Limit <sup>1</sup>		
Parts Per Million				
Cl <sup>-</sup> , chloride	0.1	4		
F <sup>-</sup> , fluoride	0.1	2		
NO <sub>3</sub> , nitrate	0.1	10		
SO <sub>4</sub> <sup>2</sup> , sulfate	0.1	20		

<sup>&</sup>lt;sup>1</sup> Samples containing concentrations greater than the upper limits of determination listed here required dilution.

Table A6. Limits of determination for water samples analyzed for 51 elements by

inductively coupled plasma-mass spectrometry (MW).

Element	Lower Determination Limit <sup>1</sup>	Element	Lower Determination Limit <sup>1</sup>	
Parts per million (ppm)		Parts per billion (ppb)		
Ca, calcium	0.05	Mn, manganese	0.1	
Na, sodium	1	Mo, molybdenum	0.2	
Parts per b	oillion (ppb)	Nb, niobium	0.2	
Ag, silver	4	Nd, neodymium	0.2	
As, arsenic	1	Ni, nickel	0.9	
Au, gold	0.1	Pb, lead	0.3	
Ba, barium	0.1	Pr, praseodymium	0.1	
Be, beryllium	0.1	Rb, rubidium	0.1	
Bi, bismuth	0.5	Re, rhenium	0.1	
Cd, cadmium	0.1	Sb, antimony	0.2	
Ce, cerium	0.1	Sm, samarium	0.2	
Co, cobalt	0.1	Sn, tin	0.2	
Cr, chromium	0.1	Sr, strontium	0.1	
Cs, cesium	0.02	Ta, tantalum	0.1	
Cu, copper	0.5	Tb, terbium	0.1	
Dy, dysprosium	0.4	Te, tellurium	0.7	
Er, erbium	0.3	Th, thorium	1	
Eu, europium	0.1	TI, thallium	0.4	
Ga, gallium	0.3	Tm, thulium	0.1	
Gd, gadolinium	0.2	U, uranium	0.1	
Ge, germanium	0.3	V, vanadium	0.2	
Hf, hafnium	0.1	W, tungsten	0.1	
Ho, holmium	0.1	Y, yttrium	0.8	
In, indium	0.1	Yb, ytterbium	0.7	
La, lanthanum	0.1	Zn, zinc	1	
Li, lithium	2	Zr, zirconium	0.1	

<sup>&</sup>lt;sup>1</sup> Limits of determination shown here are nominal and limits may vary in the data files. The variability in limits of determination is due to variable sample weight used, dilution of the sample solution, and instrumental interference correction. Upper limits of determination are not shown because samples with high concentrations were diluted and re-analyzed.