

# Geochemical Data for Environmental Studies of Mineral Deposits at Nabesna, Kennecott, Orange Hill, Bond Creek, Bremner, and Gold Hill, Wrangell-St. Elias National Park and Preserve, Alaska

by

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## TABLE OF CONTENTS

| INTRODU         | JCTION                                                                                            | 4  |
|-----------------|---------------------------------------------------------------------------------------------------|----|
| GENERA          | L GEOLOGY, DEPOSIT SETTING, AND MINING HISTORY                                                    | 4  |
| Nabesna         |                                                                                                   | 4  |
| KENNECO         | ΓT                                                                                                | 5  |
| ORANGE H        | IILL AND BOND CREEK                                                                               | 6  |
| <b>B</b> REMNER |                                                                                                   | 6  |
| GOLD HIL        | L                                                                                                 | 7  |
| METHOD          | S OF STUDY                                                                                        | 7  |
| SAMPLE M        | Iedia                                                                                             | 7  |
| SAMPLE C        | OLLECTION AND PREPARATION                                                                         | 8  |
|                 | ediments                                                                                          |    |
|                 | leavy Mineral Concentrates                                                                        |    |
|                 | Cocks                                                                                             |    |
|                 | Vater                                                                                             |    |
|                 | eached Samples                                                                                    |    |
|                 | Precipitates                                                                                      |    |
|                 | Villows                                                                                           |    |
|                 | AL TECHNIQUES                                                                                     |    |
|                 | nductively Coupled Plasma-Atomic Emission Spectrometryon Chromatography                           |    |
|                 | on Chromatography                                                                                 |    |
|                 | tomic Absorption Spectrophotometrytomic Absorption Spectrophotometry                              |    |
|                 | tomic Fluorescence                                                                                |    |
|                 | errous Iron by Colorimetry                                                                        |    |
|                 | lkalinity by Titration                                                                            |    |
|                 | emiquantitative Emission Spectrography                                                            |    |
|                 | Which Technique Should I Use?                                                                     |    |
|                 | TION OF DATA FILES                                                                                |    |
| Eiri b Min      | MBERS                                                                                             | 12 |
|                 | ICAL ANALYSES                                                                                     |    |
|                 | ONS OF TABLES AND FIELDS WITHIN TABLES.                                                           |    |
|                 | Tables containing sample and site descriptive information                                         |    |
|                 | Tables containing geochemical analyses                                                            |    |
|                 |                                                                                                   |    |
| ACKNOV          | VLEDGMENTS                                                                                        | 17 |
| REFEREN         | NCES CITED                                                                                        | 17 |
|                 |                                                                                                   |    |
|                 | LIST OF TABLES                                                                                    |    |
| Table 1.        | Elements determined and analytical methods used for all sample media collected                    | 20 |
| Table 2.        | Limits of determination for sediment and rock samples analyzed by 40-element inductively          |    |
|                 | coupled plasma-atomic emission spectrometry                                                       | 22 |
| Table 3.        | Limits of determination for selected elements in sediment and rock samples                        |    |
| Table 4.        | Limits of determination for acidified water samples analyzed for selected elements by inductively |    |
|                 | coupled plasma-atomic emission spectrometry.                                                      | 24 |
| Table 5.        | Limits of determination for anions in water samples analyzed by ion chromatography                | 25 |
| Table 6.        | Limits of determination for water samples analyzed by 64-element inductively coupled plasma-      |    |
|                 | mass spectrometry (MW)                                                                            | 26 |
| Table 7.        | Limits of determination for nonmagnetic heavy-mineral concentrate samples analyzed by             |    |
|                 | semiquantitative emission spectrography.                                                          | 27 |

## LIST OF FIGURES

|           |                                                                                          | FILENAME    |
|-----------|------------------------------------------------------------------------------------------|-------------|
| Figure 1. | Locations of areas studied within Wrangell-St. Elias National Park and Preserve, Alaska. | FIGURE1.PDF |
| Figure 2. | Site locations for samples collected in the Nabesna mine area, Alaska.                   | FIGURE2.PDF |
| Figure 3. | Detailed site locations for samples collected around the Nabesna mill, Alaska            | FIGURE3.PDF |
| Figure 4. | Site locations for samples collected in the Kennecott mine and mill areas, Alaska.       | FIGURE4.PDF |
| Figure 5. | Site locations for samples collected in the Gold Hill area, Alaska.                      | FIGURE5.PDF |
| Figure 6. | Site locations for samples collected in the Orange Hill and Bond Creek areas, Alaska.    | FIGURE6.PDF |
| Figure 7. | Site locations for samples collected in Bremner area, Alaska.                            | FIGURE7.PDF |

#### INTRODUCTION

Environmental geochemical investigations were carried out between 1994 and 1997 in Wrangell-St. Elias National Park and Preserve (WRST), Alaska. Mineralized areas studied include the historic Nabesna gold mine/mill and surrounding areas; the historic Kennecott copper mill area and nearby Bonanza, Erie, Glacier, and Jumbo mines; the historic mill and gold mines in the Bremner district; the active gold placer mines at Gold Hill; and the unmined copper-molybdenum deposits at Orange Hill and Bond Creek. The purpose of the study was to determine the extent of possible environmental hazards associated with these mineralized areas and to establish background and baseline levels for selected elements. Thus, concentrations of a large suite of trace elements were determined to assess metal loadings in the various sample media collected. This report presents the methodology, analytical results, and sample descriptions for water, leachate, sediment, heavy-mineral concentrate, rock, and vegetation (willow) samples collected during these geochemical investigations. An interpretive U.S. Geological Survey Professional Paper incorporating these geochemical data will follow.

Analytical data for samples collected in 1994 at Nabesna and Kennecott were previously published in a U.S. Geological Survey open-file report (Eppinger and others, 1995a and 1995b). An interpretive report on the 1994 data was also published (Eppinger and others, 1997). The present report supercedes the older analytical data release, bringing all the data from the entire study together in this single report. Also, sample locations are more accurate in the present report. The 1994 sample locations in the earlier report were determined by digitizing from 1:63,360-scale maps. These sites were re-occupied in 1996 and locations were refined using highly accurate military Global Positioning System (GPS) equipment (Rockwell PLGR+96 units) and more detailed field maps provided by WRST.

The Nabesna gold mine is located in the south-central part of the Nabesna 1° X 3° quadrangle, along the northern edge of WRST (fig. 1). Topography varies from relatively subdued near the confluence of Cabin and Jack Creeks (about 2800 ft/854 m in elevation), 4 miles (6.4 km) below the Nabesna mill, to the steep slopes and cliffs of White Mountain (about 6100 ft/1860 m) immediately to the west of the mill. The Nabesna and nearby Rambler mines are located on the eastern flank of White Mountain. The area is accessible to four-wheel drive vehicles by an unimproved mine road at the end of the Nabesna road, which branches off the Glenn Highway (Tok Cut-Off) at Slana.

The Kennecott mill and its associated copper deposits are located in the central part of the McCarthy 1° X 3° quadrangle, surrounded by WRST. The Kennecott mill complex lies at the base of Bonanza Peak (6983 ft/2128 m), along the margin of the Kennicott Glacier, at about 2000 ft (610 m) in elevation, while the mines which supplied the mill are located several thousand feet higher on the steep slopes of Bonanza Peak. Access to the Kennecott mill and associated mines is via foot or hired van from McCarthy. McCarthy is located at the end of the McCarthy road, which branches off the Edgerton Highway at Chitina.

The Orange Hill deposit is located in the south-central part of the Nabesna 1° X 3° quadrangle, about 13 miles (21 km) southeast of the Nabesna mine, near the foot of the Nabesna Glacier. The Bond Creek deposit is about 4 mi (6.4 km) east of Orange Hill. Topography is subdued near the valley bottom at the base of Orange Hill (about 3000 ft/900 m in elevation), but climbs steeply into rugged mountains rising over 8000 ft (2440 m) immediately east of Orange Hill and throughout the Bond Creek area. The area is accessible only by bush plane to a remote airstrip at Orange Hill.

The Bremner mining district is located in the southwestern part of the McCarthy  $1^{\circ}$  X  $3^{\circ}$  quadrangle. Topography varies from about 3500 ft (1070 m) in the valley of Golconda Creek, to over 6000 ft (1830 m) in the surrounding mountains. The area is accessible only by bush plane to a remote airstrip at Bremner.

Gold Hill is located in the Chisana mining district in the southeastern part of the Nabesna 1° X 3° quadrangle, about 6 mi (9.6 km) northeast of the remote rural community of Chisana. Topography in this relatively hilly area varies from about 4000 ft (1220 m) near the base of Gold Hill, to 5940 ft (1811 m) at the summit of Gold Hill. The area is accessible by foot from Chisana, or by bush plane to a remote airstrip at Gold Hill.

## GENERAL GEOLOGY, DEPOSIT SETTING, AND MINING HISTORY

Deposits at Nabesna, Kennecott, Gold Hill, Orange Hill, and Bond Creek are found within the allochthonous Wrangellia terrane (Jones and others, 1977), one of the accretionary terranes that constitute the geology of southern Alaska. Wrangellia originated at low paleo-latitudes in the proto-Pacific region and probably was sutured to southern Alaska in the Late Cretaceous (Plafker and Berg, 1994). The Bremner district and surrounding Chugach Mountains are found within the Chugach terrane, part of a large subduction-related accretionary complex. Accretion of the Chugach terrane to southern Alaska began in the latest Triassic, but occurred mainly in latest Cretaceous to earliest Tertiary time. Subsequently, the Chugach terrane was intruded by numerous Tertiary granitoid plutons (Plafker and others, 1994).

## NABESNA

Rocks exposed near the Nabesna mine, are predominantly Upper Triassic limestone; lesser underlying Nikolai Greenstone, an amygdaloidal subaerial basalt of Middle to Late Triassic age; and lesser overlying calcareous argillite and siltstone of the Upper Jurassic and Lower Cretaceous Nutzotin Mountain Sequence (Moffit, 1943; Wayland, 1943; Lowe and others, 1982; Newberry, 1986). The limestone, referred to as Chitistone Limestone by Wayland (1943), consists of about 366 m of massive limestone, overlain by about 244 m of thin-bedded limestone (Wayland, 1943). It is intruded by Early Cretaceous stocks and dikes of granodiorite and quartz diorite (Wayland, 1943; Lowe and others, 1982; Newberry, 1986; Weglarz, 1991)

that have K-Ar hornblende and biotite ages respectively of 109 and 114 Ma (Nokleberg and others, 1994). The Triassic to Cretaceous rocks are overlain unconformably by andesitic and basaltic lavas of the Tertiary to Quaternary Wrangell Lava (Lowe and others, 1982).

The intrusion of granodiorite into the carbonate sequence recrystallized some limestone and formed gold-bearing, ironrich skarn bodies. These skarns were exploited at the Nabesna and Rambler mines. Principal ore minerals were pyrite, pyrrhotite, magnetite, and chalcopyrite, with minor galena, sphalerite, arsenopyrite, and stibnite. Gangue minerals included garnet, wollastonite, vesuvianite, epidote, actinolite, hornblende, chlorite, scapolite, apatite, serpentine, and quartz (Wayland, 1943; Newberry, 1986; Weglarz, 1991). The main gold ores were pyrite veins along crosscutting fractures in limestone and sulfide-silica-rich bodies (Nokleberg and others, 1994). Minor gold was produced from massive magnetite and pyrrhotite bodies (Wayland, 1943). Total gold production is listed in Theodore and others (1991) as 0.08 million metric tons at 25 g/t Au. Wayland (1943) also reports unquantified, but small, production of silver and copper. As noted by Newberry and others (1997), classification of the deposits, based on production records, has been problematic. The Nabesna skarn deposit has been classified as an iron skarn with byproduct gold (Theodore and others, 1991, table 3; Nokleberg and others, 1994), and as a gold-rich copper skarn (Newberry and others, 1997). Similarly, the Rambler deposit has been classified as an iron skarn (Nokleberg and others, 1994) and as a gold skarn (Newberry and others, 1997).

Gold was first panned at the foot of White Mountain in 1899 (Wayland, 1943). The earliest claims were located in the Nabesna mine area between 1903 and 1905, and sporadic work continued into the 1920's. In 1929, the Nabesna Mining Corporation was formed and mine development was accelerated (Hunt, 1996). By 1935, the operation was running year-round and a stamp mill was processing 60 tons of ore per day. In the 1930s both mercury amalgamation and cyanide treatment were attempted to recover gold (Holdsworth, 1937; Wayland, 1943). By 1940, the deposits at the Nabesna mine were exhausted. However, the discovery in 1941 of a nearby gold-bearing pyrrhotite skarn body (probably the Rambler orebody) prompted small-scale exploration and mining (Moffit, 1944). Sporadic exploration and drilling continued at Nabesna into the 1980's. Presently, the mill and associated buildings are standing but in disrepair; a small amount of stockpiled ore remains near the old tram; mine-waste rock remains near the mill and on slopes below caved adits high on White Mountain; and a thin (less than 1 m thick) veneer of fine, pyrite-rich and iron-oxide-rich mill tailings is present over a several-thousand-square-meter area below the mill.

#### KENNECOTT

Strata-bound copper deposits in the Kennecott area are found in the lower part of the Chitistone Limestone, near the disconformable contact with the underlying Nikolai Greenstone. Basalt flows of the Nikolai Greenstone are mainly tholeiitic, have a high background copper content of around 150 parts per million (ppm), and are more than 2,740 m thick in the Kennecott region (MacKevett and others, 1997). The Chitistone Limestone grades upward into the Nizina Limestone. The carbonate rocks represent a deepening-upward succession; the lower part of the Chitistone accumulated in an intertidal-supratidal, locally sabkha setting; whereas the Nizina was deposited in a moderately deep water marine environment (MacKevett and others, 1997). In the McCarthy quadrangle, the maximum thickness of the Chitistone Limestone is about 600 m and the maximum thickness of the Nizina Limestone is about 500 m (MacKevett, 1978).

The Kennecott deposits were mined for their spectacularly high-grade copper ore, which locally exceeded 70 percent. MacKevett and others (1997) suggest that the Kennecott deposits formed through the following sequence: (1) copper-enriched Nikolai Greenstone was extruded during the Middle or Late Triassic; (2) carbonate sediments (Chitistone Limestone) were deposited in a Late Triassic marine embayment on the Nikolai Greenstone; (3) sabkha-facies deposits, rich in sulfates and organic matter, formed locally in the embayment and restricted circulation, leading to evaporation that resulted in brine development; (4) karst features developed in exposed parts of the lower Chitistone Limestone following marine regression; (5) the Chitistone was buried by as much as 3,050 m of marine limestone, black shale, and other sedimentary rocks of the Nizina Limestone and the McCarthy, Nizina Mountain, and Root Glacier Formations; (6) the section was folded, faulted, and uplifted during the Late Jurassic to Early Cretaceous; (7) uplift and folding provided hydrologic head that caused brine circulation and leaching of copper from the Nikolai Greenstone; and (8) large orebodies formed in fissures and breccias in the lower Chitistone Limestone when migrating copper-rich brines mixed with reduced fluids derived from gypsum-organic matter mixtures in the sabkha horizons. The copper ore was mainly chalcocite and djurleite, with lesser chalcopyrite, bornite, covellite, digenite, anilite, luzonite, idaite, malachite, azurite, chalcanthite, and orpiment (Bateman and McLaughlin, 1920; MacKevett and others, 1997). More than 535,000 metric tons of copper and several million ounces of silver were produced from 1911 to 1938, the major period of mining activity (MacKevett and others, 1997). Average grade of the ore was about 13 percent.

Early Russian explorers reported implements of copper used by Copper River Indians at the mouth of the Copper River (Douglass, 1964). Prospecting in the region in the late 1890's probably was stimulated by the Klondike gold rush. The first Kennecott-type deposit was found by prospectors in 1900, who located extensive copper-stained outcrops crowning the Bonanza deposit, high above the Kennicott Glacier (Douglass, 1964; Hunt, 1996). The other principal deposits were located within the next few years. The first ore was shipped in 1911, but the peak of mining activity was from 1915 to 1929. Copper sulfide ore was gravity-concentrated and shipped directly for processing in Washington. Copper was stripped from carbonate ore (malachite and azurite) at the mill using a Kennecott-developed ammonia leach process, and then it was shipped for further refining. The mines ceased production in 1938 because of low reserves, low copper prices, and labor problems (MacKevett and

others, 1997). Presently, the mill area remains intact, and carbonate-rich mine-waste rock remains below caved adits high above the mill on Bonanza Ridge.

#### ORANGE HILL AND BOND CREEK

Rocks in the vicinity of the Orange Hill and Bond Creek porphyry copper-molybdenum deposits are composed predominantly of a variety of intrusive rocks of the Cretaceous Nabesna batholith, a quartz diorite to granodiorite pluton dated at 105 Ma (Newberry and others, 1997). Less extensive are Pennsylvanian to Permian volcanic and volcaniclastic rocks; and lesser Triassic Nikolai Greenstone. Volumetrically minor thin bodies of Permian limestone are present locally (Richter, 1973). In both areas, quartz diorite and granodiorite of the Nabesna pluton are intruded by slightly younger hornblende-plagioclase porphyry (Nokleberg and others, 1994; Richter, 1973). Limonite staining and hydrothermally altered rocks are extensive; alteration mineral suites include biotite-quartz, quartz-sericite, and late-stage chlorite-sericite-epidote (Richter, 1973; Nokleberg and others, 1987; Nokleberg and others, 1994). Hydrothermally altered rock occupies an area of 1 X 3 km at Orange Hill and 2 X 3 km at Bond Creek (Richter and others, 1975).

Mineralization consists of pyrite, chalcopyrite, and minor molybdenite in potassic and sericitic quartz veins and as disseminations, commonly found along altered granite porphyry dikes (Richter and others, 1975; Nokleberg and others, 1994). Magnetite and anhydrite are locally abundant. Associated with the porphyry deposits at Orange Hill are minor, gold-poor skarns, found near limestone contacts. The skarn deposits consist of red, brown, and green garnet; vesuvianite; pyroxene; epidote; diopside; wollastonite; hematite; magnetite; and the sulfides pyrite, chalcopyrite, bornite, covellite, massive pyrrhotite, sphalerite, and minor molybdenite (Van Alstine and Black, 1946; Linn, 1973; Nokleberg and others, 1994; Newberry and others, 1997). The Orange Hill deposit contains 100 to 320 million tonnes grading 0.30 to 0.35 percent copper and 0.02 to 0.03 percent molybdenum. The Bond Creek deposit contains an estimated 500 million tonnes grading 0.15 to 0.40 percent copper and 0.02 percent molybdenum (Young and others, 1997).

Neither Orange Hill nor Bond Creek have been mined, although both have been explored in the past and patented mining claims are still held at Orange Hill. Early reports of ground staking at Orange Hill were for gold, although only negligible quantities were found (Moffit and Knopf, 1910; Moffit, 1943). By the early 1930s, several copper lode claims were held at Orange Hill and a few thousand feet of drill core had been obtained (Stewart, 1931; Van Alstine and Black, 1946). Sporadic exploration in the 1960s and 1970s included mapping and drilling at Orange Hill (Linn, 1973) and Bond Creek (Gillespie, 1970). Exploration activity ceased around 1980, when the areas were included in the newly established National Park and Preserve. In 1996, only a few prospects, scattered drill core, and a cabin were evident at Orange Hill. Evidence of exploration activity at Bond Creek in 1997 consisted of 3 small wooden drilling platforms and associated debris, all subsequently removed by the NPS in 1998.

#### **BREMNER**

The most recent geologic maps of the Bremner area are the 1:250,000-scale reconnaissance maps of the McCarthy quadrangle by MacKevett (1978) and Bering Glacier quadrangle by Plafker (unpublished mapping, 1998), the sources of the following summary. Mountainous outcrop in the region around the Bremner gold deposits is predominantly the Cretaceous Valdez Group, and intervening valleys are filled with unconsolidated Quaternary alluvial, colluvial, and glacial deposits. The flysch deposits of the Valdez Group are at least several thousand meters thick and are composed interlayered metagraywacke and argillite, and minor schist, slate, and phyllite. Rocks of the Valdez Group in the Bremner area are variably metamorphosed to chlorite- or biotite-grade greenschist facies and have northwest-striking foliation and lineaments. The Valdez Group represents turbidite deposits that formed extensive deep-sea fans. Hypabyssal, hornblende-rich, early to middle Tertiary granodiorite and tonalite plutons, and dacite dikes and sills cut the Valdez Group. Large Tertiary intrusions cutting rocks of the Valdez Group are found about 8 mi (13 km) northeast and 5 mi (8 km) south of the Bremner deposits. During our study of the Bremner deposits, individual and swarms of granodiorite and dacite dikes were observed, all generally less than 2 m thick. We also noted pervasive thin quartz and quartz-calcite veins and veinlets (metamorphic sweat veins?) cutting the Valdez Group metasediments.

While gold can be panned from most streams in the area, paying placers were limited to (in order of increasing importance) parts of the Bremner River, the Little Bremner River, and Golconda Creek (Moffit, 1914). The Golconda Creek basin is where most placer and nearly all lode mining took place. Alluvial gravels along Golconda Creek were generally less than 8 ft thick and were interpreted by Moffit as re-worked bench gravels. The fluvial re-working of the bench gravels reconcentrated the gold already disseminated within the bench deposits, making the deposits profitable (Moffit, 1914). Little lode prospecting had been done along Golconda Creek prior to Moffit's (1914) study, although he noted the presence of numerous dikes and locally-mineralized calcite-bearing quartz veins. Moffit (1914) believed the placer gold was derived from the quartz veins; subsequent discovery of gold-bearing quartz veins in the basin supports his theory. At the Lucky Girl mine (mislabeled as the Yellowband mine on the USGS McCarthy A-7 quadrangle) we found a series of three adits following a northwest-trending, near vertical, pyrite-rich, banded, white quartz vein cutting slate. Brecciated portions of the vein are up to 2 m thick, vuggy, and iron-oxide-rich. Ore at the adjacent mill site consisted of white quartz cobbles with sericite, chlorite, iron oxide as clots and pseudomorphs after pyrite, calcite; and lesser galena, arsenopyrite, sphalerite, and free gold. At the Yellowband mine, located about 4 km south of the Lucky Girl mine, we found a gold-bearing, pyritic, iron-stained quartz vein containing sparse sericite and chlorite, and following a granodiorite dike swarm. Biotite in the granodiorite dikes has been altered to chlorite. Vein quartz from the Sheriff mine, located about 3.2 km southeast of the Lucky Girl mine, contained iron-oxide clots, pyrite,

chlorite, sericite, free gold, calcite, and probably arsenopyrite. While production records from the Bremner district are scant, MacKevett (1976) lists placer gold production at between 2000 and 3000 oz (56,700 and 85,000 g) and lode gold production at about 750 oz (21,300 g).

The gold placers along the Little Bremner River were discovered in 1901 by two parties of prospectors and placer claims were staked on Golconda Creek in that year (Moffit, 1914). Initial placer mining was by pick and hammer, but by 1911, hydraulic giants were in use, with associated ditch lines and flumes (Moffit, 1914). By the early 1930s, both lode mine development and placer prospecting were underway at Bremner, aided by construction of an airstrip in 1931 (Stewart, 1933). Placer activity was apparently much-reduced from that reported by Moffit (1914), consisting of prospecting at the mouth of Standard Creek and on Golconda Creek over a 2.5 mi stretch both above and below Standard Creek (Stewart, 1933). Lodes at the Lucky Girl, Yellowband, and Sheriff were apparently being exploited, and a small mill utilizing mercury amalgamation was constructed at the Lucky Girl mine. Mining activities at Bremner ceased during World War II and never resumed (Hunt, 1996). Presently, the mill and mine buildings have collapsed, but several cabins remain standing. Mill tailings and mine waste piles are evident at the lode mines. However, only scattered hand-stacked piles of boulders remain along Golconda Creek as evidence of the earlier frenzied placer activities.

#### GOLD HILL

The following description of the geology of the Gold Hill area is extracted from Richter and Jones (1973). Bedrock in the Gold Hill area is predominantly marine and subaerial volcanic and volcaniclastic rocks of the Lower Cretaceous Chisana Formation. The 10,000-ft thick Chisana Formation consists of andesitic volcanic flows; volcanic conglomerate; tuffs; fragmental volcanic units (probably lahars and avalanche deposits); volcanic sandstone and siltstone; and, in the lower part of the section, thin lenses of marine argillite, graywacke, pebble conglomerate and tuffaceous mudstone. Jurassic to Cretaceous argillite and mudstone deposits are found locally. North of Gold Hill are extensive deposits of rhythmically interbedded Jurassic deposits of graywacke, siltstone, and argillite. Small bodies and dikes of Tertiary to Cretaceous andesite porphyry and porphyritic diorite are scattered throughout the placered area. A large intrusion of pyroxene-diorite monzonite is exposed just west of Gold Hill. Quaternary to Tertiary unconsolidated gravels and isolated patches of Wrangell Lava are present locally. Most of the bedrock around Gold Hill is concealed by Quaternary glacial gravel and sand deposits, fluvioglacial (?) deposits, solifluction deposits, and alluvial deposits.

The productive placer gravels are Tertiary, lying near or on volcanic and sedimentary rocks of the Chisana Formation (Richter and Matson, 1972). All placer mines in the district are found in an area 5 mi (8 km) in diameter, centered on Gold Hill, and include Bonanza Creek and its tributaries, Gold Run Creek, and Big Eldorado Creek (Capps, 1915; Moffit, 1943). Other heavy minerals found in the placer deposits include native copper, native silver, galena, cinnabar, and molybdenite (Nokleberg and others, 1987). Total gold production from the placers is about 1.4 million grams (Nokleberg and others, 1987). Scattered about Gold Hill are pyritic, iron-oxide stained outcrops of andesite (?) and a few prospects that explore gold- and silver-bearing base metal lodes. Several areas of iron-oxide-stained, sulfate-bearing, altered bedrock are found near the junction of Bonanza and Chathenda Creeks (Richter and others, 1975). One of the lodes visited in this study, on N.P. Nelson Hill, is a galena-sphalerite-chalcopyrite-calcite-quartz vein breccia containing malachite and limonite. The vuggy vein cuts feldspar-andesite porphyry. Other small gold-bearing veins of similar character are described briefly by Capps (1915), Stewart (1931), Moffit (1943), and Richter and others (1975). There is no reported production from the lode deposits. Capps (1915) believed that the gold-bearing lodes were the source of the placer gold deposits. The placer deposits produced about 50,000 oz (1.4 million g) of gold (Nokleberg and others, 1987).

Placer gold was discovered at Gold Hill in May of 1913, resulting in initial claims on Bonanza Creek, where 200 oz of gold was recovered in two days (Bleakley, 1996). A gold rush ensued. Several thousand stampeders were in the Chisana district by October, 1913, when Chisana City was founded, and all available sites had been claimed by July, 1914. However, the boom was short-lived, the district was in decline by 1915, and by 1920 only eight mines remained, employing less than 20 men (Bleakley, 1996; Hunt, 1996). Small-scale placer mining has been fairly continuous in the district since then and continues into the present on unpatented claims located prior to formation of the National Park and Preserve in 1980. Currently, a few scattered cabins are occupied sporadically during summer months, when small-scale sluicing activities take place.

#### METHODS OF STUDY

### SAMPLE MEDIA

Geochemical sample media include rock, mine waste, mill tailings, stream-sediment, heavy-mineral-concentrate, water, precipitates, and (at Nabesna only) willow samples. Rock samples were collected generally as composite chip samples from outcrop, alluvium, mine, and mill tailings. Several mine waste, mill tailings and mineralized bedrock samples were collected for leaching, using a synthetic meteoric water leach test. The stream-sediment and heavy-mineral-concentrate samples were collected at mined and unmined areas to provide geochemical information about drainage basins proximal and distal to mine areas.

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 mineral deposits 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, but also from seeps, springs, and ponds. Mineral deposits rich in sulfide minerals, solid waste from mine dumps and mill tailings derived from such deposits, and sulfide-rich rocks of non-economic significance are possible sources of acid and metal loading in the environment. The water samples provide data reflecting the effects of mining on the environment and data indicating water quality associated with undeveloped mineralized areas.

Precipitate samples were collected from a few sites at Nabesna and Orange Hill. These were collected as flocs precipitating from metalliferous waters.

Reconnaissance willow samples were collected at and below the Nabesna mill site. The willows were collected to test whether the plants had taken up metals related to the mineral extraction activities.

#### SAMPLE COLLECTION AND PREPARATION

A count of the various types of samples collected from each area studied is listed in the table below. In the table, "site dupes" and "anal. dupes" refer to sample quality control site duplicates (collected in the field) and analytical duplicates (split from sample in the laboratory), respectively. Sample sites maps are provided in figure 2 (Nabesna mine area), figure 3 (Nabesna mill area), figure 4 (Kennecott), figure 5 (Gold Hill), figure 6 (Orange Hill and Bond Creek), and figure 7 (Bremner).

|             | Total | Nabesna | Kennecott | Gold Hill | Orange<br>Hill | Bond<br>Creek | Bremner |
|-------------|-------|---------|-----------|-----------|----------------|---------------|---------|
| Water       | 101   | 51      | 11        | 15        | 7              | 8             | 9       |
| Site dupes  | 10    | 4       | 2         | 1         | 1              | 1             | 1       |
| Anal. dupes | 1     | 1       |           |           |                |               |         |
| Sediment    | 70    | 22      | 11        | 15        | 6              | 8             | 8       |
| Site dupes  | 9     | 3       | 2         | 1         | 1              | 1             | 1       |
| Anal. dupes | 1     |         |           |           |                |               | 1       |
| Leach       | 18    | 12      |           |           | 2              | 1             | 3       |
| Anal. dupes | 3     | 1       |           |           | 1              |               | 1       |
| Rock        | 56    | 24      | 10        | 7         | 3              | 7             | 5       |
| Anal. dupes | 2     | 1       |           |           |                | 1             |         |
| Concentrate | 51    | 11      | 7         | 14        | 5              | 5             | 9       |
| Anal. dupes | 1     |         |           |           | 1              |               |         |
| Precipitate | 7     | 2       |           |           | 5              |               |         |
| Willow      | 32    | 32      |           |           |                |               |         |

#### **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 (1-kg) sample of minus-10 mesh sediment was collected in a cloth bag and air-dried. Sediment samples were predominantly from stream alluvium, but a few were collected from ponds and springs. Sediments were also collected from just below mill tailings and mine waste piles. These were composited in a similar manner as described above

In the laboratory, 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 disgarded. The fine sediment fractions from samples collected during the initial year of study (1994) were further sieved into two fractions: minus-80 mesh to plus-200 mesh (0.074 mm), and minus-200 mesh. The two sieve fractions were chosen to assess variation of metal content with sediment particle size. This variation was found to be minimal, so in the subsequent years of sample collection, the minus-80 mesh sample was analyzed in toto. 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. Sediment sieve size and sources are listed in the sediment data file.

## **Heavy Mineral Concentrates**

Most panned concentrate samples were collected from the same active alluvium as sediment samples. Some samples were also collected from mill tailings and mine waste. Alluvial samples were collected 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, 1996a). 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.

#### Rocks

Mineralized and unmineralized rock samples were collected from outcrop, mine waste, mill tailings, and alluvium. Rock samples were typically composite chip samples collected at the sites. Single grab samples were collected where compositing was not possible. Rock descriptions were recorded in field notes and later entered into the rock data file.

In the laboratory, rock samples were coarsely crushed to pea-sized pieces and then split with a Jones splitter, generally following the procedure of Taylor and Theodorados (1996b). For each sample, an approximate 6.5 oz (185 g) portion was pulverized and saved for chemical analysis and subsequent archival of any remaining material. Clean quartz rock and sand was crushed and pulverized, respectively, between samples to reduce the risk of cross-contamination between samples.

#### Water

A 1-liter polypropylene bottle was used for collecting water for 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 spring. When possible, we collected below riffles--natural zones of mixing. 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 pre-rinsed 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 plastic sheets to cover work areas.

A variety of water samples was collected from the areas studied, and the kinds of water samples collected evolved over the years as the study progressed. During the first year (1994) 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 cool on ice or permafrost 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.

Additional water samples collected in 1996 and 1997 include water for ferrous iron content, water for mercury content, and (in 1997 only) water for measuring alkalinity in the laboratory. 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. The mercury water samples were filtered as described above; collected in acid-rinsed glass bottles with Teflon lids; and preserved with potassium dichromate/nitric acid. The waters for laboratory-determined total alkalinity analysis were collected at sites where water pH was > 4.5, as unfiltered, unacidified samples, and were kept cool as described above. The 1997 laboratory alkalinity analyses provide a means to assess quality of companion alkalinity analyses collected on site throughout the study, as described below. In general, the agreement between the two alkalinity methods was found to be good.

Other water data collected and recorded on-site include temperature, pH, conductivity, dissolved oxygen content, an estimate of the water flow rate, total acidity, 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 acidity measurements were collected in 1997 only, using a field-portable Hach titration kit, and titrating to the phenolphthalein endpoint. Only waters with pH values below 7.0 were tested for acidity, which is a measure of total base-

neutralizing capacity of the water, reported in ppm as  $CaCO_3$ . On-site total alkalinity measurements were collected from 1994 through 1997 at each water sample site where pH was > 4.5, using a field-portable CHEMetrics titration kit. Alkalinity is a measure of total acid-neutralizing capacity of water and is reported in ppm as  $CaCO_3$ .

#### **Leached Samples**

Samples were collected from mine waste, mill tailings, and outcrop for a synthetic meteoric water leachability test. Samples were collected by scraping material from the upper 1 inch (2.5 cm) of the surface layer and filling a large canvas bag with approximately 2 lb (1 kg) of material. Samples were composited by collecting several increments from several places at the sample site. For better sample representativity, an attempt was made to collect 20 to 30 increments at each site. Four deviations from this sample collection methodology were made at Nabesna (sites 6NA031-6NA034), where mill tailings core samples rather than surficial samples were composited. Unsieved leach samples were saved for further preparation in the laboratory.

Laboratory preparation for the samples, described briefly below, followed a modification of the EPA Synthetic Precipitation Leaching Procedure 1312 (Environmental Protection Agency, 1994). This method is used to determine mobility of inorganic analytes present in samples of soils, wastes, and waste waters. The composited, solid, dry sample was passed through a 9.5 mm (0.375 mesh) sieve. Water used for leaching was organic-free, deionized water, acidified with sulfuric acid/nitric acid (60/40 weight percent mixture) to a pH of  $4.20 \pm 0.05$ , the pH recommended in the procedure for waste materials. Using a ratio of 20:1 water to sample, 1 liter of acidified, deionized water (temperature,  $23^{\circ}$  C) was added to 50 g of sample in a rinsed polypropylene bottle. The mixture was rotated at  $30 \pm 2$  rpm for  $18 \pm 2$  hours. Blank samples using the acidified, deionized water were included for each batch of samples leached. Following rotation, measurements for pH, conductivity, temperature, oxygen content, and alkalinity were collected on the leachates. Our modification to the EPA 1312 procedure was done at the filtering stage. Rather than filtering the entire 1-liter leachate through a stainless steel positive pressure filtration unit, we filtered only the amount of sample required for analysis, using sterile 0.45-micron disposable filters. Following filtration, leachates were acidified with ultra-pure, concentrated nitric acid. Prior to leaching, splits of the solid phases of the samples were saved for analysis, permitting comparison of metal contents in the total sample (hereafter referred to as leach solid phase) with those in the leachable portion (hereafter referred to as leachate).

#### **Precipitates**

A few precipitate samples were collected at Nabesna and Orange Hill. These samples are flocs precipitating out of metalliferous waters onto the underlying substrates. The samples were composited from wet areas on alluvium or mill tailings and placed in sealed plastic bags. In the laboratory the precipitates were dried, hand-ground with a mortar and pestle, and then split for chemical and mineralogical analysis by X-ray diffraction. Clean quartz sand was hand ground between samples to clean the mortar and pestle, thereby reducing the risk of cross-contamination between samples.

## Willows

All willow samples were collected from Nabesna on August 13, 1997. Samples of two willow species, *Salix Alaxensis* and *Salix Glauca*, were collected by compositing leaves from new-growth (1997) stems. At each sample site, samples from approximately five willow bushes of a single species within a 30 ft (9 m) radius were composited and placed in a tightly woven, breathable, 5.5 in by 10.5 in Hubco bag. If both willow species were present at a given site, *Salix Glauca* was the species collected. Samples were collected at numerous points on the Nabesna mill tailings, on a traverse about 3000 ft (910 m) downstream of the mill tailings, and on a traverse about 4400 ft (1340 m) lateral to the mill tailings.

In the laboratory, each willow sample was placed in a plastic colander and washed, first under tap water, then under deionized water (Peacock, 1996). Next, the samples were dried in a drying oven, and weighed. Then the samples were ashed, and the ash was weighed and saved for analysis. The weight percent ash was calculated from the sample and ash weights (Peacock, 1996). Analyses of willow samples are reported on a dry-weight basis.

## **ANALYTICAL TECHNIQUES**

A large number of chemical elements were determined, using a variety of quantitative and semi-quantitative analytical techniques. Table 1 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 samples. Solid samples were analyzed by both U.S. Geological Survey laboratories and by a contractor, XRAL Laboratories, Inc., as indicated in the data files.

Throughout the study, QA/QC concerns were addressed through the use of internal reference standards, field blanks, sample site duplicates, and analytical duplicates. QA/QC samples comprised 19 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. About 5 percent of the QA/QC samples were reference standards. 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. Field blanks comprised about 6 percent of the total number of water samples. Sediment and water sample site duplicates were collected randomly and comprise about 6 percent of the total number of samples. The site duplicates were collected following the same procedures as those used for normal water and sediment samples. Analytical duplicates for the

various sample media comprise about 2 percent of the total. Only the sample site duplicates 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 atomic absorption spectrophotometric (AAS) analytical method provides the most statistically representative results, due to the larger sample weight analyzed; a 10-gram sample weight is used for the AAS analysis, whereas a 10-milligram sample weight is used in the emission spectrographic technique.

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.

#### Inductively Coupled Plasma-Atomic Emission Spectrometry

Three ICP-AES methods were used in the study for multi-element analyses: (1) a 40-element total digestion method (designated "ET" in table 1 and in the data files) for sediments, rocks, precipitates, leach solid phases, and willows (willow analyses are dry weight); (2) a 10-element, partial-extraction method (designated "EP" in table 1 and in the data files) for sediments, rocks, and leach solid phases; and (3) a trace- and major-element scan for acidified water and leachates (designated "EW" in table 1 and in the data files.

In the first multi-element method, 40-element ICP-AES, samples were digested and analyzed following the procedure of Briggs (1996). Samples 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. Limits of determination for 40-element ICP-AES are shown in table 2.

In the second multi-element method, 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). Samples 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. Mineralized rock samples with high copper content (above 10,000 ppm) required dilution for the 10-element ICP-AES method because of copper interferences on other elements. Thus, the ICP-AES partial extraction (EP) trace-element analyses for mineralized rocks with high copper content (principally those from mines or prospects) are qualitative estimates and should be used cautiously. Limits of determination for 10-element ICP-AES are shown in table 3.

In the third multi-element method, 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). Water samples collected in 1994 were preconcentrated by a factor of 20 to 1 by evaporation at  $100^{\circ}$  C and subsequent dissolution the residue in nitric acid. The residue solutions were then aspirated into the plasma and element concentrations were determined by ICP-AES. The preconcentration step was used when specific conductivities for the water samples were less than 2000 microsiemens/cm; water samples with conductivities greater than 2000 microsiemens/cm were not preconcentrated and were analyzed directly by ICP-AES, using a modification of the method by Lichte and others (1987) (Paul H. Briggs, personal communication, 1995). Beginning in 1995, the preconcentration step was eliminated for water samples because a new instrument with better dynamic range was used. Limits of determination for the multi-element ICP-AES method for water samples are shown in table 4.

A separate ICP-AES method was used to determine tungsten in sediment and rock samples collected in 1994 only, the ion-exchange separation ICP-AES method developed by Doughten and Aruscavage (1996). The samples were decomposed with nitric, hydrofluoric, and hydrochloric acids and then evaporated to dryness. The residue was dissolved in hydrochloric acid and this solution was passed through an ion-exchange column, where the chloride form of tungsten was adsorbed onto the resin. Tungsten was eluted from the resin, dissolved in hydrochloric acid, and determined by ICP-AES. This method is designated IE in table 1 and in the data files. Limits of determination for tungsten in sediment and rock samples are shown in table 3. Tungsten was not determined on samples collected after 1994.

#### Ion Chromatography

The anions Cl $^{\circ}$ , F $^{\circ}$ , NO $_3$ , and sO $_4$ <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 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. This method is designated IC in table 1 and in the data files. Limits of determination for anions in raw water samples are shown in table 5.

## Inductively Coupled Plasma-Mass Spectrometry

Acidified-filtered and acidified-unfiltered waters, and water from the synthetic meteoric water leach test were analyzed to determine 64 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 1 and in the data files. The method is used to determine over 60 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 64 elements by ICP-MS are shown in table 6. Limits vary because a newer ICP-MS instrument with improved sensitivity was acquired during the life of the study.

## Atomic Absorption Spectrophotometry

Various atomic absorption spectrophotometric (AAS) methods were used for determining selected elements in sediment, rock, and water samples. These methods are described individually below. Determination limits for these techniques are given in table 3.

Concentrations of thallium in the sediment and rock samples were determined by the AAS technique of O'Leary (1996). The samples were digested using hydrofluoric acid, sulfuric acid, hydrochloric acid, and hydrogen peroxide. Thallium was extracted into a solution of 10 % Aliquat 336 and MIBK (methyl isobutyl ketone) in the presence of potassium iodide and ascorbic acid and determined by flame AAS. This method is designated AA in table 1 and in the data files. Sediment and rock samples for gold analysis were digested using a hydrobromic acid-bromine digestion, an MIBK extraction, and then gold was determined on the solutions by flame AAS . However, samples with gold concentrations of less than 0.050 ppm were subsequently analyzed by graphite-furnace AAS, which has a 0.002 ppm lower determination limit for gold (O'Leary and Meier (1996). The gold determinations are designated GF in table 1 and in the data files.

Mercury was measured in sediment, rock, and water samples using the cold-vapor AAS technique of O'Leary and others (1996). The sediment and rock samples were decomposed with nitric acid and sodium dichromate. 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. This method is designated CV in table 1 and in the data files.

Sediment and rock samples were analyzed for arsenic, antimony, and selenium using a continuous-flow hydride generation AAS (Hageman and Welsch, 1996). The samples were digested using concentrated nitric, perchloric, sulfuric, and hydrofluoric acids; 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. A similar procedure was used for arsenic and antimony. This method is designated HY in table 1 and in the data files. Some of the samples were not analyzed for arsenic by the HY technique because of high arsenic concentrations beyond the working range of the method (for these, arsenic was determined by ICP-AES methods), because of elemental interferences in certain mineralized rock samples, or because of insufficient sample remaining following analyses by other methods (the HY method was the last method used).

## Atomic Fluorescence

Water samples analyzed for mercury by cold-vapor atomic absorption were all found to contain concentrations less than the analytical detection limit of 0.1 parts per billion (ppb). Therefore, the water samples were analyzed by atomic fluorescence, which has limits of detection of 0.01 parts per billion (J. Crock, personal communication, 1998). For this method, mercury (II) was reduced as described above for the cold-vapor atomic absorption, and then was released into an atomic fluorescence detector, where the mercury concentration was determined. This method is designated AF in table 1 and in the data files. Limits of determination are shown in table 3.

## 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 3.

## 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 in 1997 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 3.

## Semiquantitative Emission Spectrography

The minus-20-mesh nonmagnetic heavy-mineral-concentrate samples were analyzed by a direct-current arc, semiquantitative emission spectrographic (SES) technique and 37 major, minor, and trace elements were determined (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. The precision of this analytical technique is approximately  $\pm$  one reporting interval at the 83 percent confidence level and  $\pm$  two reporting intervals at the 96 percent confidence level (Motooka and Grimes, 1976). 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 1 and in the data files. Limits of determination for elements determined by SES are listed in table 7.

## Which Technique Should I Use?

In some sample media, the same elements were determined by more than one analytical technique (Table 1). This section is provided as guidance for those instances where multiple values are listed for a given element in a sample. Filtered/acidified and unfiltered acidified water samples and leachate 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. Similarly,  $SO_4^{2^-}$  values determined by ion chromatography are preferred over those determined by ICP-MS. 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 (above 10,000 ppm) in solid samples can cause interferences on other elements (Ag, As, Au, Bi, Cd, Mo, Pb, Sb, and Zn) in the 10-element, partial extraction ICP-AES method. In these instances, the total extraction ICP-AES method is preferred for these elements.

#### **DESCRIPTION OF DATA FILES**

The analytical data files are provided in two formats, as a Microsoft Access 97 database and as separate Lotus .WK1 files. The data in the Access database are arranged as a series of tables and associated queries, and follow Access naming conventions. The tables and brief descriptions follow below. For those not using Microsoft Access 97, the tables are saved as separate .WK1 files with the same name and fields. The following abbreviations are used in the tables: FeOx, iron oxide; MnOx, manganese oxide; XRD, X-ray diffraction; GX, sample collected for geochemical analysis; HS, hand sample collected for reference.

#### FIELD NUMBERS

The field number coding scheme is as follows: The first number indicates the year the sample was collected, i.e., 4 indicates 1994, 6 indicates 1996, and 7 indicates 1997. The next two letters indicate samples from the Nabesna (NA), Kennecott (KE), Orange Hill (OR), Bond Creek (BC), Bremner (BR), or Gold Hill (GH). The next 3 digits indicate the sample site number. The above 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. These suffixes evolved from the initial 1994 season to the latter seasons, as indicated below:

| Media                                    | Suffix                                                | Year        |
|------------------------------------------|-------------------------------------------------------|-------------|
| Rock                                     | R                                                     | all         |
|                                          | (R1, R2, R3, etc., indicate multiple rock samples fro | m the site) |
| Stream sediment                          | S, sieve size minus-80 mesh                           | 1996, 1997  |
|                                          | S1, sieve size minus-200 mesh                         | 1994        |
|                                          | S2, sieve size minus-80 to plus-200 mesh              | 1994        |
| Heavy-mineral concentrate                | C                                                     | all         |
| Sample for leaching                      | varies: L. R, or T                                    | all         |
| Vegetation sample                        | V                                                     | all         |
| Filtered/unacidified water               | W1                                                    | 1994        |
|                                          | FU                                                    | 1996, 1997  |
| Unfiltered/acidified water               | W                                                     | 1994        |
|                                          | RA                                                    | 1996, 1997  |
| Filtered/acidified water                 | W3                                                    | 1994        |
|                                          | FA                                                    | 1996, 1997  |
| Water for mercury analysis               | HG                                                    | 1996, 1997  |
| Water for ferrous iron analysis          | FE                                                    | 1996, 1997  |
| Water for laboratory alkalinity analysis | ALK                                                   | 1996, 1997  |

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 4NA021S1 indicates a 1994 minus-200-mesh stream sediment from Nabesna site 021, while field number 4NA0212DS1 indicates a site duplicate of the same.

#### 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 table 1. Units of measurement are: PPM, parts per million; PPB, parts per billion; and PCT, percent. The analytical methods and associated code letters are:

AA atomic absorption spectrophotometry AF atomic fluorescence for mercury

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

IE ion exchange inductively coupled plasma-atomic emission spectrometry

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 plasmaatomic emission spectrometry. For the geochemical data, 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. An H indicates that the sample was not analyzed because of an elemental interference. A blank entry indicates that the sample was not analyzed for that particular element.

#### **D**ESCRIPTIONS 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 Contents

**TblSampleLog** table of all sample sites and the various sample media collected at each site

Fields:
Site ID site identifier

Field\_No\_Root field number root (= site identifier with applicable QA/QC coding)

Water\_RA unfiltered, acidified water Water\_FA filtered, acidified water Water\_FU filtered, unacidified water

Water\_FE filtered, acidified water for Fe2+ analysis Water\_HG filtered, preserved water for Hg analysis

Water\_ALK unfiltered, unacidified water for laboratory alkalinity analysis

Sediment sediment sample

Concentrate heavy-mineral concentrate sample

Rock rock sample

Leach sample collected for leaching

Precipitate precipitate sample

Vegetation vegetation (willow) sample

Notes additional notes on samples collected

*Table* Contents

**TblSampleSiteInfo** information related to the sample site

Fields:

Site ID site identifier

NPS ID WRST geologist site identifier

Latitude latitude in decimal degrees
Longitude longitude in decimal degrees

LatD latitude, degrees LatM latitude, minutes

LatS latitude, decimal seconds
LonD longitude, degrees
LonM longitude, minutes

LonS longitude, decimal seconds

XYError\_m location error in meters, as determined by GPS instrument

Datum used for coordinate determination

Coordinate\_Source how coordinates were obtained
Date\_Coll date of sample collection at site
Time\_Coll time of sample collection at site
Location brief description of sample location

1X3\_Quadrangle 1° X 3° quadrangle Bedrock\_Geology bedrock geology at the site Additional\_Info additional site-related information

*Table* Contents

**TblWaterSiteInfo** information related to water sample sites

Fields:

Site\_ID site identifier

Field No Root field number root (= site identifier with applicable QA/QC coding)

QA/QC\_Info information on QA/QC samples

Temp\_C water temperature at collection site, degrees Celsius

Estimated\_Flow\_L/MIN estimated water flow in liters per minute

pH water pH at collection site

Conductivity uS/CM water conductivity at collection site, microsiemens/centimeter

Dissolved\_O2\_PPM dissolved oxygen in water at collection site

Alkalinity\_Site\_PPM water alkalinity at collection site, as parts per million CaCO3
Acidity\_PPM water acidity at collection site, as parts per million CaCO3

Table Contents

**TblLeachInfo** information related to samples leached following modification of EPA 1312 method

Fields:

Site\_ID site identifier Field No sample field number

Source source of sample (mine waste, mill tailings, outcrop, etc.)
Leach\_Sample\_Desc descriptive information for sample collected for leaching

XRD\_Leach\_Major
XRD\_Leach\_Minor
XRD\_Leach\_Minor
XRD\_Leach\_Trace

minerals identified by X-ray diffraction, >20 %
minerals identified by X-ray diffraction, 5 % to 20 %
minerals identified by X-ray diffraction, less than 5 %

pH\_Final final pH of leachate at time of collection T\_C\_Final temperature of leachate at time of collection

Conductivity\_uS/CM\_Final conductivity of leachate at time of collection, microsiemens per

centimeter

Alkalinity\_PPM\_Final alkalinity of leachate at time of collection, as parts per million CaCO3

Date\_Leach\_Done date that leach was completed Time\_Leach\_Done time that leach was completed

Tables containing geochemical analyses

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

Site\_ID site identifier; for example 6BC009 (this field is common to ALL tables)

Field\_No\_Root field number root; this is the site identifier with applicable QA/QC suffix; for example 6BC009D sample field number; this is the site identifier with applicable QA/QC suffix and sample media suffix;

for example 6BC009DFA

Lab\_No sample laboratory-assigned number

Job\_No job number for job in which sample was analyzed

Lab analytical laboratory (USGS = U.S. Geological Survey laboratory; XRAL = XRAL, Inc. contract

laboratory)

Sample\_Description description of prepared sample for analysis

QA/QC\_Info quality assurance/quality control sample information

For the following tables, geochemical analyses follow the common fields described above.

**TblWaterDataAnions**anion analyses, filtered/unacidified water samples
cation analyses, filtered/acidified water samples
cation analyses, filtered/acidified water samples
cation analyses, unfiltered/acidified water samples
TblWaterDataFe2+

Fe<sup>2+</sup> analyses, filtered/acidified water samples

**TblWaterDataFe2+** Fe<sup>2+</sup> analyses, filtered/acidified water samples Hg analyses, filtered/preserved water samples

**TblLeachSolidData** solid-phase analyses of splits of samples collected for leaching

**TblLeachDataAnions** anion analyses for samples leached following modification of EPA 1312

method

**TblLeachDataCations** cation analyses for samples leached following modification of EPA 1312

method

The following fields are unique to specific tables:

Table Contents

TblConcentrateData heavy-mineral concentrate descriptive and analytical data

Fields:

Field\_Mineralogy\_Exam on-site hand lens examination of panned concentrate

Sieve\_Size sample sieve size as prepared in laboratory

Character character of sample: single grab or composite of several increments

Source sample collected from outcrop, float, alluvium, etc.

Geochemical analyses follow at this point.

Table Contents

**TblPrecipitateData** precipitate descriptive and analytical data

Fields:

XRD Info minerals in sample identified by X-ray diffraction

Character character of sample: single grab or composite of several increments

Source sample collected from outcrop, float, alluvium, etc.

Geochemical analyses follow at this point.

Table Contents

**TblRockData** rock descriptive and analytical data

Fields:

XRD\_Info minerals in sample identified by X-ray diffraction

Near\_Mine yes/no: Was sample site near or distal from mine or prospect?

Character character of sample: single grab or composite of several increments

Source sample collected from outcrop, float, alluvium, etc.

Geochemical analyses follow at this point.

Table Contents

**TblSedimentData** sediment descriptive and analytical data

Fields:

Sieve\_Size sample sieve size as prepared in laboratory

Character character of sample: single grab or composite of several increments

Source sample collected from outcrop, float, alluvium, etc.

Increm/Reach\_m format: X or X/Y, where X is number of sample increments collected and

Y is length of sampled reach in meters

Geol\_Info\_Alluvium geologic description of alluvium at sample site

Geochemical analyses follow at this point.

Table Contents

TblWaterDataAlkalin laboratory-determined alkalinity for water samples

Fields:

Alkalinity Lab PPM TI total alkalinity, as parts per million CaCO3

Analytical\_Note note from analyst

Table Contents

**TblWillowData** willow descriptive and dry-weight analytical data

Fields:

Species species of willow sample analyzed

Geochemical analyses follow at this point.

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Table 1.--Elements determined and analytical methods used for all sample media collected. [MW, water by inductively coupled plasma-mass spectrometry; EW, water by inductively coupled plasma-atomic emission spectrometry; EP, solid by partial-extraction inductively coupled plasma-atomic emission spectrometry; EP, solid by partial-extraction inductively coupled plasma-atomic emission spectrometry; HY, hydride generation atomic absorption; GF, graphite-furnace atomic absorption; CV, cold-vapor atomic absorption; AF, atomic fluorescence; AA, atomic absorption; IE, specific-ion-electrode atomic absorption; ES, semiquantitative emission spectrography; IC, ion chromatography; CO, colorimetry; 1, water, acidified-filtered and acidified-unfiltered samples; 2, water, unacidified-unfiltered samples; 3, water leaches, acidified-filtered and acidified-unfiltered samples; 4, sediment samples; 5, rock samples; 6, nonmagnetic, heavy-mineral-concentrate samples; 7, precipitate samples; 8, willow samples.

| ELEMENT                         | MW         | EW         | ET                 | EP  | HY      | GF  | CV      | AF | AA | IE | ES | IC | СО                     |
|---------------------------------|------------|------------|--------------------|-----|---------|-----|---------|----|----|----|----|----|------------------------|
| Ag, silver                      | 1,3        | 3          | 4,5,7,8            | 4,5 |         |     |         |    |    |    | 6  |    |                        |
| Al, aluminum                    | 1,3        | 1,3        | 4,5,7,8            | ,   |         |     |         |    |    |    |    |    |                        |
| As, arsenic                     | 1,3        | 3          | 4,5,7,8            | 4,5 | 4,5,8   |     |         |    |    |    | 6  |    |                        |
| Au, gold                        | 1,3        |            | 4,5,7,8            | 4,5 | , - , - | 4,5 |         |    |    |    | 6  |    |                        |
| B, boron                        | , -        | 1,3        | 7-7-7-             | ,-  |         | ,-  |         |    |    |    | 6  |    |                        |
| Ba, barium                      | 1,3        | 1,3        | 4,5,7,8            |     |         |     |         |    |    |    | 6  |    |                        |
| Be, beryllium                   | 1,3        | 1,3        | 4,5,7,8            |     |         |     |         |    |    |    | 6  |    |                        |
| Bi, bismuth                     | 1,3        | .,.        | 4,5,7,8            | 4,5 |         |     |         |    |    |    | 6  |    |                        |
| Ca, calcium                     | 1,3        | 1,3        | 4,5,7,8            | .,0 |         |     |         |    |    |    | 6  |    |                        |
| Cd, cadmium                     | 1,3        | 1,3        | 4,5,7,8            | 4,5 |         |     |         |    |    |    | 6  |    |                        |
| Ce, cerium                      | 1,3        | .,0        | 4,5,7,8            | .,0 |         |     |         |    |    |    |    |    |                        |
| Cl <sup>-</sup> , chloride      | 1,,0       |            | 1,0,1,0            |     |         |     |         |    |    |    |    | 2  |                        |
| Co, cobalt                      | 1,3        | 1,3        | 4,5,7,8            |     |         |     |         |    |    |    | 6  |    |                        |
| Cr, chromium                    | 1,3        | 1,3        | 4,5,7,8            |     |         |     |         |    |    |    | 6  |    |                        |
| Cs, cesium                      | 1,3        | .,0        | 1,0,1,0            |     |         |     |         |    |    |    |    |    |                        |
| Cu, copper                      | 1,3        | 1,3        | 4,5,7,8            | 4,5 |         |     |         |    |    |    | 6  |    |                        |
| Dy, dysprosium                  | 1,3        | .,0        | 1,0,1,0            | .,0 |         |     |         |    |    |    |    |    |                        |
| Er, erbium                      | 1,3        |            |                    |     |         |     |         |    |    |    |    |    |                        |
| Eu, europium                    | 1,3        |            | 4,5,7,8            |     |         |     |         |    |    |    |    |    |                        |
| Fe, iron                        | 1,3        | 1,3        | 4,5,7,8            |     |         |     |         |    |    |    | 6  |    |                        |
| Fe <sup>2+</sup> , ferrous iron | 1,0        | 1,0        | 7,0,7,0            |     |         |     |         |    |    |    | 0  |    | 1                      |
| F, fluoride                     |            |            |                    |     |         |     |         |    |    |    |    | 2  | <u> </u>               |
| Ga, gallium                     | 1,3        |            | 4,5,7,8            |     |         |     |         |    |    |    | 6  |    |                        |
| Gd, gadolinium                  | 1,3        |            | 4,3,7,0            |     |         |     |         |    |    |    | 0  |    |                        |
| Ge, germanium                   | 1,3        |            |                    |     |         |     |         |    |    |    | 6  |    |                        |
| Hf, hafnium                     | 1,3        |            |                    |     |         |     |         |    |    |    | U  |    |                        |
| Hg, mercury                     | 1,5        |            |                    |     |         |     | 1,4,5,8 | 1  |    |    |    |    |                        |
| Ho, holmium                     | 1,3        |            | 4,5,7,8            |     |         |     | 1,4,5,6 | '  |    |    |    |    |                        |
| In, indium                      | 1,3        |            | 4,3,7,0            |     |         |     |         |    |    |    |    |    |                        |
| Ir, iridium                     | 1          |            |                    |     |         |     |         |    |    |    |    |    |                        |
| K, potassium                    | 1,3        | 1,3        | 4,5,7,8            |     |         |     |         |    |    |    |    | İ  | 1                      |
| La, lanthanum                   | 1,3        | 1,0        | 4,5,7,8            |     |         |     |         |    |    |    | 6  |    |                        |
| Li, lithium                     | 1,3        | 1,3        | 4,5,7,8            |     |         |     |         |    |    |    | U  |    |                        |
| Lu, lutetium                    | 1          | 1,3        | 4,5,7,6            |     |         |     |         |    |    |    |    |    |                        |
| Mg, magnesium                   |            | 1.2        | 1570               |     |         |     |         |    |    |    | 6  |    | -                      |
| Mn, manganese                   | 1,3<br>1,3 | 1,3<br>1,3 | 4,5,7,8<br>4,5,7,8 | +   | -       |     |         |    |    |    | 6  | -  | $\vdash$               |
| Mo, molybdenum                  | 1,3        | 1,3        | 4,5,7,8            | 4,5 |         |     |         |    |    |    | 6  | -  | $\vdash$               |
| Na, sodium                      | 1,3        | 1,3        | 4,5,7,8            | 4,3 |         |     |         |    |    |    | 6  | 1  | $\vdash$               |
| Nb, niobium                     | 1,3        | 1,0        | 4,5,7,8            | +   |         |     |         |    |    |    | 6  | -  | $\vdash$               |
| Nd, neodymium                   | 1,3        |            | 4,5,7,8            | +   | -       |     |         |    |    |    | U  | -  | $\vdash$               |
|                                 |            | 1.2        |                    |     |         |     |         |    |    |    | 6  |    |                        |
| Ni, nickel                      | 1,3        | 1,3        | 4,5,7,8            |     |         |     |         |    |    |    | 6  | 2  | -                      |
| NO <sub>3</sub> , nitrate       |            | 4.0        | 4.5.7.0            |     |         |     |         |    |    |    | 0  | 2  |                        |
| Ph. load                        | 1 2        | 1,3        | 4,5,7,8            | 1 5 | -       |     |         |    |    |    | 6  | -  | $\vdash$               |
| Pb, lead                        | 1,3        | 1,3        | 4,5,7,8            | 4,5 |         |     |         |    |    |    | 6  | -  | $\vdash \vdash \vdash$ |
| Pd, palladium                   | 1          |            | 1                  | +   |         |     |         |    |    |    | 6  | -  | $\vdash \vdash \vdash$ |
| Pr, praseodymium                | 1,3        |            |                    | +   | ļ       |     | -       |    |    |    | 0  |    | $\vdash$               |
| Pt, platinum                    | 1          |            |                    | 1   | ļ       |     |         |    |    |    | 6  |    | $\vdash$               |
| Rb, rubidium                    | 1,3        |            |                    | 1   | ļ       |     |         |    |    |    |    |    | $\vdash$               |
| Re, rhenium                     | 1,3        |            |                    | 1   |         |     |         |    |    |    |    | 1  | $\sqcup$               |
| Rh, rhodium                     | 1          |            |                    |     | 20      |     |         |    |    |    |    |    |                        |

| Du ruthonium                           | 1   |     |         |     |                                                  |  | 1 |     |     |   |   |  |
|----------------------------------------|-----|-----|---------|-----|--------------------------------------------------|--|---|-----|-----|---|---|--|
| Ru, ruthenium                          | •   |     |         | +   | <del>                                     </del> |  |   |     |     |   | - |  |
| Sb, antimony                           | 1,3 | 3   |         | 4,5 | 4,5                                              |  |   |     |     | 6 |   |  |
| Sc, scandium                           | 1,3 |     | 4,5,7,8 |     |                                                  |  |   |     |     | 6 |   |  |
| Se, selenium                           | 1,3 |     |         |     | 4,5                                              |  |   |     |     |   |   |  |
| Si, silicon                            | 1,3 | 1,3 |         |     |                                                  |  |   |     |     |   |   |  |
| Sm, samarium                           | 1,3 |     |         |     |                                                  |  |   |     |     |   |   |  |
| Sn, tin                                | 1,3 | 3   | 4,5,7,8 |     |                                                  |  |   |     |     | 6 |   |  |
| SO <sub>4</sub> <sup>2</sup> , sulfate |     |     |         |     |                                                  |  |   |     |     |   | 2 |  |
| Sr, strontium                          | 1,3 | 1,3 | 4,5,7,8 |     |                                                  |  |   |     |     | 6 |   |  |
| Ta, tantalum                           | 1,3 |     | 4,5,7,8 |     |                                                  |  |   |     |     |   |   |  |
| Tb, terbium                            | 1,3 |     |         |     |                                                  |  |   |     |     |   |   |  |
| Te, tellurium                          | 1,3 |     |         |     |                                                  |  |   |     |     |   |   |  |
| Th, thorium                            | 1,3 |     | 4,5,7,8 |     |                                                  |  |   |     |     | 6 |   |  |
| Ti, titanium                           | 1,3 | 1,3 | 4,5,7,8 |     |                                                  |  |   |     |     | 6 |   |  |
| TI, thallium                           | 1,3 |     |         |     |                                                  |  |   | 4,5 |     |   |   |  |
| Tm, thulium                            | 1,3 |     |         |     |                                                  |  |   |     |     |   |   |  |
| U, uranium                             | 1,3 |     | 4,5,7,8 |     |                                                  |  |   |     |     |   |   |  |
| V, vanadium                            | 1,3 | 1,3 | 4,5,7,8 |     |                                                  |  |   |     |     | 6 |   |  |
| W, tungsten                            | 1,3 |     |         |     |                                                  |  |   |     | 4,5 | 6 |   |  |
| Y, yttrium                             | 1,3 |     | 4,5,7,8 |     |                                                  |  |   |     |     | 6 |   |  |
| Yb, ytterbium                          | 1,3 |     | 4,5,7,8 |     |                                                  |  |   |     |     |   |   |  |
| Zn, zinc                               | 1,3 | 1,3 | 4,5,7,8 | 4,5 |                                                  |  |   |     |     | 6 |   |  |
| Zr, zirconium                          | 1,3 |     |         |     |                                                  |  |   |     |     | 6 |   |  |

Table 2.--Limits of determination for sediment, rock, precipitate, leach solid phase, and willow samples analyzed by 40-element inductively coupled plasma-atomic emission spectrometry, total extraction (ET). [Element names are shown in Table 1]

|         | Solid F                      | Precipitates, Leach | Willows <sup>1</sup>         |                              |  |  |  |
|---------|------------------------------|---------------------|------------------------------|------------------------------|--|--|--|
| Element | Lower Determination<br>Limit | Limit               | Lower Determination<br>Limit | Upper Determination<br>Limit |  |  |  |
|         |                              | Percent             |                              |                              |  |  |  |
| Al      | 0.005                        | 50                  | 0.0009                       | 50                           |  |  |  |
| Са      | 0.005                        | 50                  | 0.0009                       | 50                           |  |  |  |
| Fe      | 0.02                         | 25                  | 0.0009                       | 25                           |  |  |  |
| K       | 0.01                         | 50                  | 0.0018                       | 50                           |  |  |  |
| Mg      | 0.005                        | 5                   | 0.0009                       | 5                            |  |  |  |
| Na      | 0.006                        | 50                  | 0.0009                       | 50                           |  |  |  |
| Р       | 0.005                        | 50                  | 0.0009                       | 50                           |  |  |  |
| Ti      | 0.005                        | 25                  | 0.0009                       | 25                           |  |  |  |
|         |                              | Parts Per Milli     |                              |                              |  |  |  |
| Ag      | 2                            | 10,000              | 0.3                          | 10,000                       |  |  |  |
| As      | 10                           | 50,000              | 2                            | 50,000                       |  |  |  |
| Au      | 8                            | 50,000              | 2                            | 50,000                       |  |  |  |
| Ва      | 1                            | 35,000              | 0.2                          | 35,000                       |  |  |  |
| Be      | 1                            | 5,000               | 0.2                          | 5,000                        |  |  |  |
| Bi      | 10                           | 50,000              | 2                            | 50,000                       |  |  |  |
| Cd      | 2                            | 25,000              | 0.3                          | 25,000                       |  |  |  |
| Се      | 5                            | 50,000              | 0.7                          | 50,000                       |  |  |  |
| Со      | 2                            | 25,000              | 0.2                          | 25,000                       |  |  |  |
| Cr      | 2                            | 50,000              | 0.2                          | 50,000                       |  |  |  |
| Cu      | 2                            | 15,000              | 0.2                          | 15,000                       |  |  |  |
| Eu      | 2                            | 5,000               | 0.3                          | 5,000                        |  |  |  |
| Ga      | 4                            | 50,000              | 0.7                          | 50,000                       |  |  |  |
| Но      | 4                            | 5,000               | 0.7                          | 5,000                        |  |  |  |
| La      | 2                            | 50,000              | 0.3                          | 50,000                       |  |  |  |
| Li      | 2                            | 50,000              | 0.3                          | 50,000                       |  |  |  |
| Mn      | 4                            | 50,000              | 0.7                          | 50,000                       |  |  |  |
| Мо      | 2                            | 50,000              | 0.3                          | 50,000                       |  |  |  |
| Nb      | 4                            | 50,000              | 0.7                          | 50,000                       |  |  |  |
| Nd      | 9                            | 50,000              | 0.7                          | 50,000                       |  |  |  |
| Ni      | 3                            | 50,000              | 0.3                          | 50,000                       |  |  |  |
| Pb      | 4                            | 50,000              | 0.7                          | 50,000                       |  |  |  |
| Sc      | 2                            | 50,000              | 0.3                          | 50,000                       |  |  |  |
| Sn      | 5                            | 50,000              | 0.8                          | 50,000                       |  |  |  |
| Sr      | 2                            | 15,000              | 0.3                          | 15,000                       |  |  |  |
| Та      | 40                           | 50,000              | 7                            | 50,000                       |  |  |  |
| Th      | 6                            | 50,000              | 0.7                          | 50,000                       |  |  |  |
| U       | 100                          | 100,000             | 20                           | 100,000                      |  |  |  |
| ٧       | 2                            | 30,000              | 0.3                          | 30,000                       |  |  |  |
| Υ       | 2                            | 25,000              | 0.3                          | 25,000                       |  |  |  |
| Yb      | 1                            | 5,000               | 0.2                          | 5,000                        |  |  |  |
| Zn      | 2                            | 15,000              | 0.3                          | 15,000                       |  |  |  |

<sup>&</sup>lt;sup>1</sup> Lower determination limits for willow samples are lower than those for other media because the willow samples were weighed and ashed prior to analysis—essentially a preconcentration step. The values were then converted back to a whole-sample basis, resulting in lower determination limits.

Table 3.--Limits of determination for selected elements in sediment, rock, leach solid phase, willow, and water samples. [Element names are shown in Table 1; values for solids are in parts per million; values for water samples are as indicated; EP, partial extraction 10-element inductively coupled plasma-atomic emission spectrometry; ; GF, graphite-furnace atomic absorption spectrophotometry; CV, cold-vapor atomic absorption spectrophotometry; AF, atomic fluorescence, HY, hydride generation atomic absorption spectrophotometry; AA, atomic absorption spectrophotometry; IE, ion exchange separation inductively coupled plasma-atomic emission spectrometry; CO, colorimetry; ppm. [parts per million; ppb, parts per billion]

|                  |        | Sediments, Rocks, Lea<br>Willows, values in parts | s per million                          | Water, values                             | s as indicated                         |
|------------------|--------|---------------------------------------------------|----------------------------------------|-------------------------------------------|----------------------------------------|
| Element          | Method | Lower Determination<br>Limit <sup>1</sup>         | Upper Determination Limit <sup>1</sup> | Lower Determination<br>Limit <sup>1</sup> | Upper Determination Limit <sup>1</sup> |
| Ag               | ΕP     | 0.08                                              | 400                                    |                                           |                                        |
| As               | EP     | 1                                                 | 6,000                                  |                                           |                                        |
| Au               | EP     | 0.1                                               | 1,500                                  |                                           |                                        |
| Bi               | EP     | 1                                                 | 6,000                                  |                                           |                                        |
| Cd               | EP     | 0.05                                              | 500                                    |                                           |                                        |
| Cu               | EP     | 0.05                                              | 500                                    |                                           |                                        |
| Мо               | EP     | 0.1                                               | 900                                    |                                           |                                        |
| Pb               | EP     | 1                                                 | 6,000                                  |                                           |                                        |
| Sb               | EP     | 1                                                 | 6,000                                  |                                           |                                        |
| Zn               | EP     | 0.05                                              | 500                                    |                                           |                                        |
| Au               | GF     | 0.002                                             | 0.05                                   |                                           |                                        |
| Hg               | CV     | 0.02                                              | 1.8                                    | 0.1 ppb                                   | none <sup>2</sup>                      |
| Hg               | AF     |                                                   |                                        | 0.01 ppb                                  | none <sup>2</sup>                      |
| As               | HY     | 0.6                                               | 20                                     |                                           |                                        |
| Sb               | HY     | 0.6                                               | 20                                     |                                           |                                        |
| Se               | HY     | 0.2                                               | 4                                      |                                           |                                        |
| TI               | AA     | 0.1                                               | 10                                     |                                           |                                        |
| W                | IE     | 1                                                 | 200                                    |                                           |                                        |
| Fe <sup>2+</sup> | CO     |                                                   |                                        | 0.01 ppm                                  | none <sup>2</sup>                      |

<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.

<sup>&</sup>lt;sup>2</sup> Samples with high concentrations were diluted and re-analyzed.

Table 4.--Limits of determination for acidified water and leachate samples analyzed for 24 elements by inductively coupled plasma-atomic emission spectrometry (EW). [Element names are shown in Table 1; see text for discussion of preconcentration]

|                   | No Precor | ncentration       | Precond                    | entrated                   |  |  |  |  |  |  |  |
|-------------------|-----------|-------------------|----------------------------|----------------------------|--|--|--|--|--|--|--|
| Element           |           |                   | <b>Lower Determination</b> | <b>Upper Determination</b> |  |  |  |  |  |  |  |
|                   | Limit     | Limit             | Limit                      | Limit                      |  |  |  |  |  |  |  |
| Parts Per Million |           |                   |                            |                            |  |  |  |  |  |  |  |
| Al                | 0.1       | 1,000             | 0.025                      | 1,000                      |  |  |  |  |  |  |  |
| Ca                | 1         | 1,000             | 0.05                       | 1,000                      |  |  |  |  |  |  |  |
| Fe                | 0.1       | 1,000             | 0.025                      | 1,000                      |  |  |  |  |  |  |  |
| K                 | 1         | 1,000             | 0.05                       | 1,000                      |  |  |  |  |  |  |  |
| Mg                | 1         | 1,000             | 0.05                       | 1,000                      |  |  |  |  |  |  |  |
| Na                | 1         | 1,000             | 0.05                       | 1,000                      |  |  |  |  |  |  |  |
| P                 | 0.05      | 1,000             | 0.025                      | 1,000                      |  |  |  |  |  |  |  |
| Si                | 1         | 1,000             | 0.05                       | 1,000                      |  |  |  |  |  |  |  |
|                   |           | Parts Per Billion |                            |                            |  |  |  |  |  |  |  |
| Ва                | 5         | 10,000            | 1                          | 10,000                     |  |  |  |  |  |  |  |
| Ве                | 10        | 10,000            | 1                          | 10,000                     |  |  |  |  |  |  |  |
| В                 | 10        | 10,000            | 2.5                        | 10,000                     |  |  |  |  |  |  |  |
| Cd                | 10        | 10,000            | 1                          | 10,000                     |  |  |  |  |  |  |  |
| Cr                | 10        | 10,000            | 2                          | 10,000                     |  |  |  |  |  |  |  |
| Со                | 10        | 10,000            | 2                          | 10,000                     |  |  |  |  |  |  |  |
| Cu                | 10        | 10,000            | 4                          | 10,000                     |  |  |  |  |  |  |  |
| Pb                | 50        | 10,000            | 5                          | 10,000                     |  |  |  |  |  |  |  |
| Li                | 10        | 10,000            | 5                          | 10,000                     |  |  |  |  |  |  |  |
| Mn                | 10        | 10,000            | 2                          | 10,000                     |  |  |  |  |  |  |  |
| Мо                | 20        | 10,000            | 4                          | 10,000                     |  |  |  |  |  |  |  |
| Ni                | 10        | 10,000            | 4                          | 10,000                     |  |  |  |  |  |  |  |
| Sr                | 1         | 10,000            | 1                          | 10,000                     |  |  |  |  |  |  |  |
| Ti                | 50        | 10,000            | 10                         | 10,000                     |  |  |  |  |  |  |  |
| V                 | 10        | 10,000            | 2                          | 10,000                     |  |  |  |  |  |  |  |
| Zn                | 10        | 10,000            | 2                          | 10,000                     |  |  |  |  |  |  |  |

Table 5.--Limits of determination for anions in water samples determined by ion chromotography. [Element names are shown in Table 1]

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

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

Table 6.--Limits of determination for water samples analyzed by 64-element inductively coupled plasma-mass spectrometry (MW). [Element names are shown in Table 1]

| Element | Lower Determination<br>Limit <sup>1</sup> | Element                 | Lower Determination<br>Limit <sup>1</sup> |  |  |  |
|---------|-------------------------------------------|-------------------------|-------------------------------------------|--|--|--|
| Parts   | s per million (ppm)                       | Parts per billion (ppb) |                                           |  |  |  |
| Ca      | 0.05                                      | Li                      | 0.10                                      |  |  |  |
| Mg      | 0.01                                      | Mn                      | 0.01                                      |  |  |  |
| Na      | 0.01                                      | Мо                      | 0.02                                      |  |  |  |
| Si      | 0.25                                      | Nb                      | 0.02                                      |  |  |  |
| Part    | ts per billion (ppb)                      | Nd                      | 0.01                                      |  |  |  |
| Ag      | 0.01                                      | Ni                      | 0.10                                      |  |  |  |
| Al      | 0.01                                      | Pb                      | 0.05                                      |  |  |  |
| As      | 0.20                                      | Pr                      | 0.01                                      |  |  |  |
| Au      | 0.01                                      | Rb                      | 0.01                                      |  |  |  |
| Ва      | 0.02                                      | Re                      | 0.02                                      |  |  |  |
| Be      | 0.05                                      | Sb                      | 0.02                                      |  |  |  |
| Bi      | 0.01                                      | Sc                      | 0.10                                      |  |  |  |
| Cd      | 0.02                                      | Se                      | 0.20                                      |  |  |  |
| Ce      | 0.01                                      | Sm                      | 0.01                                      |  |  |  |
| Со      | 0.02                                      | Sn                      | 0.05                                      |  |  |  |
| Cr      | 1.00                                      | Sr                      | 0.02                                      |  |  |  |
| Cs      | 0.01                                      | Та                      | 0.02                                      |  |  |  |
| Cu      | 0.50                                      | Tb                      | 0.005                                     |  |  |  |
| Dy      | 0.005                                     | Те                      | 0.10                                      |  |  |  |
| Er      | 0.005                                     | Th                      | 0.01                                      |  |  |  |
| Eu      | 0.005                                     | Ti                      | 0.10                                      |  |  |  |
| Fe      | 10.00                                     | TI                      | 0.05                                      |  |  |  |
| Ga      | 0.02                                      | Tm                      | 0.005                                     |  |  |  |
| Gd      | 0.005                                     | U                       | 0.01                                      |  |  |  |
| Ge      | 0.02                                      | ٧                       | 0.10                                      |  |  |  |
| Hf      | 0.05                                      | W                       | 0.02                                      |  |  |  |
| Но      | 0.005                                     | Υ                       | 0.01                                      |  |  |  |
| In      | 0.01                                      | Yb                      | 0.01                                      |  |  |  |
| K       | 0.30                                      | Zn                      | 0.50                                      |  |  |  |
| La      | 0.01                                      | Zr                      | 0.05                                      |  |  |  |

<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, slight changes in methodology over time, and acquisition of a newer, more sensitive instrument during the life of this study. Upper limits of determination are not shown because samples with high concentrations were diluted and re-analyzed.

Table 7.--Limits of determination for nonmagnetic heavy-mineral concentrate samples analyzed by semiquantitative emission spectrography. [Element names are shown in Table 1]

| Element | Lower Determination<br>Limit | Upper Determination<br>Limit | Element           | Lower Determination<br>Limit | Upper Determination<br>Limit |  |  |
|---------|------------------------------|------------------------------|-------------------|------------------------------|------------------------------|--|--|
| Percent |                              |                              | Parts Per Million |                              |                              |  |  |
| Ca      | 0.1                          | 50                           | Ge                | 20                           | 200                          |  |  |
| Fe      | 0.1                          | 50                           | La                | 100                          | 2,000                        |  |  |
| Mg      | 0.05                         | 20                           | Mn                | 20                           | 10,000                       |  |  |
| Na      | 0.5                          | 10                           | Мо                | 10                           | 5,000                        |  |  |
| Р       | 0.5                          | 20                           | Nb                | 50                           | 5,000                        |  |  |
| Ti      | 0.005                        | 2                            | Ni                | 10                           | 10,000                       |  |  |
|         | Parts Per Milli              | on                           | Pb                | 20                           | 50,000                       |  |  |
| Ag      | 1                            | 10,000                       | Pd                | 10                           | 2,000                        |  |  |
| As      | 500                          | 20,000                       | Pt                | 50                           | 2,000                        |  |  |
| Au      | 20                           | 1,000                        | Sb                | 200                          | 20,000                       |  |  |
| В       | 20                           | 5,000                        | Sc                | 10                           | 200                          |  |  |
| Ва      | 50                           | 10,000                       | Sn                | 20                           | 2,000                        |  |  |
| Ве      | 2                            | 2,000                        | Sr                | 200                          | 10,000                       |  |  |
| Bi      | 20                           | 2,000                        | Th                | 200                          | 5,000                        |  |  |
| Cd      | 50                           | 1,000                        | ٧                 | 20                           | 20,000                       |  |  |
| Со      | 20                           | 5,000                        | W                 | 50                           | 20,000                       |  |  |
| Cr      | 20                           | 10,000                       | Υ                 | 20                           | 5,000                        |  |  |
| Cu      | 10                           | 50,000                       | Zn                | 500                          | 20,000                       |  |  |
| Ga      | 10                           | 10,000                       | Zr                | 20                           | 2,000                        |  |  |