USGS
science for a changing world

Stream-Sediment Samples Reanalyzed for Major, Rare Earth, and Trace Elements from Seven 1:250,000-Scale Quadrangles, South-Central Alaska, 2007–09

Data Series 532

U.S. Department of the Interior
U.S. Geological Survey

This page intentionally left blank.

Stream-Sediment Samples Reanalyzed for Major, Rare Earth, and Trace Elements from Seven 1:250,000-Scale Quadrangles, South-Central Alaska, 2007–09

By Bruce M. Gamble, Elizabeth A. Bailey, Jeanine M. Schmidt, Nora B. Shew, Keith A. Labay, Matthew Granitto, Richard M. O'Leary, and David E. Detra

Data Series 532

U.S. Department of the Interior
U.S. Geological Survey

U.S. Department of the Interior
KEN SALAZAR, Secretary

U.S. Geological Survey
Marcia K. McNutt, Director

U.S. Geological Survey, Reston, Virginia: 2010

Suggested citation:
Gamble, B.M., Bailey, E.A., Schmidt, J.M., Shew, N.B., Labay, K.A., Granitto, Matthew, O'Leary, R.M., and Detra, D.E., 2010, Stream-sediment samples reanalyzed for major, rare earth, and trace elements from seven 1:250,000-scale quadrangles, south-central Alaska, 2007–09: U.S. Geological Survey Data Series 532, 4 p.

Contents

Plate

Tables

Conversion Factors, Datum, and Abbreviations and Acronyms

Conversion Factors

Inch/Pound to SI

Multiply	By	To obtain
	Length	
inch (in.)	2.54	centimeter (cm)
foot (ft)	0.3048	meter (m)
ounce, fluid (fl. oz)	0.02957	liter (L)
mile (mi)	1.609	kilometer (km)

SI to Inch/Pound

Multiply	By	To obtain
	Length	
milliliter	0.0338	ounce, fluid (fl. oz)
grams	0.0353	ounce, dry (U.S.)

Temperature in degrees Celsius (°C) may be converted to degrees Fahrenheit (°F) as follows:

$$°F=(1.8×°C)+32.$$

Temperature in degrees Fahrenheit (°F) may be converted to degrees Celsius (°C) as follows:

$$°C=(°F–32)/1.8.$$

Datum

Horizontal coordinate information is referenced to the North American Datum of 1927 (NAD 27).

Abbreviations and Acronyms

AES	atomic emission spectrometry
AMRAP	Alaska Mineral Resource Assessment Program
HGAAS	hybride generation-atomic absorption spectrometry
ICP	inductively coupled plasma
KI	potassium iodide
LOD	limit of detection
MS	mass spectometry
NGDB	National Geochemical Database
REE	rare earth element
RSD	relative standard deviation
USGS	U.S. Geological Survey

Stream-Sediment Samples Reanalyzed for Major, Rare Earth, and Trace Elements from Seven 1:250,000-Scale Quadrangles, Alaska, 2007–09

By Bruce M. Gamble, Elizabeth A. Bailey, Nora B. Shew, Keith A. Labay, Jeanine M. Schmidt, Richard M. O'Leary, and David E. Detra

Abstract

During the 1960s through the 1980s, the U.S. Geological Survey conducted reconnaissance geochemical surveys of drainage basins throughout most of the Iliamna, Lake Clark, Lime Hills, and Talkeetna 1:250,000-scale quadrangles and parts of the McGrath, Seldovia, and Tyonek 1:250,000-scale quadrangles in Alaska. These geochemical surveys provide data necessary to assess the potential for undiscovered mineral resources and provide data that may be used to determine regional-scale element baselines. This report provides new data for 1,075 of the previously collected stream-sediment samples. The new analyses include a broader spectrum of elements and provide data that are more precise than the original analyses. All samples were analyzed for arsenic by hydride generation atomic absorption spectrometry, for gold, palladium, and platinum by inductively coupled plasma-mass spectrometry after lead button fire assay separation, and for a suite of 55 major, rare earth, and trace elements by inductively coupled plasma-atomic emission spectrometry and inductively coupled plasma-mass spectrometry after sodium peroxide sinter at 450 degrees Celsius.

Introduction

During the 1960s through the 1980s, the U.S. Geological Survey (USGS) conducted reconnaissance geochemical surveys of drainage basins throughout most of the Iliamna, Lake Clark, Lime Hills, and Talkeetna 1:250,000-scale quadrangles and parts of the McGrath, Seldovia, and Tyonek 1:250,000-scale quadrangles in Alaska as part of the Alaska Mineral Resource Assessment Program (AMRAP). More than 17,000 stream-sediment samples were collected in these seven quadrangles in conjunction with the AMRAP studies (pl. 1).

In preparation for future mineral resource assessments, a percentage of these samples (about 6 percent) were selected for reanalysis because recently developed analytical methods can detect additional elements of interest, such as platinum (Pt), palladium (Pd), and rare earth elements (REE), and elements that have lower detection limits than the methods used when the samples were originally analyzed.

Methods of Study

Sample Selection

Stream-sediment samples selected for reanalysis primStream-sediment samples selected for reanalysis primarily were collected for the USGS AMRAP studies during the 1960s–1980s. The sample details and previous analytical data can be found in the following reports: Detterman and Reed (1965); Reed and Detterman (1966); Reed (1967); Reed and Elliott (1970); King and others (1978); O'Leary and others (1978); Bailey and others (1986); Erlich and others (1988); and Motooka and others (1990). Data are digitally stored in the USGS National Geochemical Database (NGDB) (S.M. Smith, U.S. Geological Survey, oral commun., 2008). Samples for reanalysis were selected randomly from 10×10 km grid cells that were overlaid on the study area to ensure a relatively even sample distribution. When available, two samples were selected from each cell.

Sample materials remaining from the original analyses are stored in the USGS sample archive in Building 810 at the Denver Federal Center in Denver, Colo. A total of 1,075 samples (available for download as a Microsoft© Excel spreadsheet in the appendix of this report) were selected and retrieved from the archive for reanalysis for this study. Archived material sufficient for reanalysis was not available for all samples initially selected. When possible, new selections were made in an attempt to have two samples from each cell.

The original sample material collected consisted of fine-grained active alluvium that was collected primarily from first- or second-order streams as shown on USGS 1:63,360-scale maps. Each sample was composited by collecting sediment increments from several places at the sample site, generally along a 30-ft stretch of the channel. Most samples were wet sieved on site into a 14–16 in. gold pan through a 10-mesh (2-mm) screen to remove pebbles.

Sample Preparation

When the samples originally were collected, they were either air-dried or oven-dried at less than 50 ˚C and then sieved with an 80-mesh (less than 180-µm) screen. Sediment that passed through the sieve was ground in a vertical pulverizer with ceramic plates to 100-mesh (less than 150-µm) (Peacock and others, 2002). Samples were prepared by USGS laboratories in Anchorage, Alaska, and Denver, Colorado.

Sample Analyses

The selected stream-sediment samples were analyzed for 55 major, rare earth, and trace elements by inductively coupled plasma-atomic emission spectrometry-mass spectrometry (ICP-AES-MS) (U.S. Geological Survey, 2010a, at http://minerals.cr.usgs.gov/projects/analytical_chem/references.html#m22) using a modification of Meier and Slowik (2002). Samples were decomposed using a sodium peroxide sinter at 450 °C. The resultant cake was leached with water and acidified with nitric acid. After an addition of tartaric acid, aliquots of the digested samples were aspirated into the ICP-AES and the ICP-MS. The concentrations of the optimal elements from the ICP-AES and ICP-MS were determined. Data were considered acceptable if recovery for all 55 elements was ±15 percent at five times the lower reporting limit and the calculated relative standard deviation (RSD) of duplicate sample analysis was no greater than 15 percent. Lower and upper reporting limits for this method are shown in table 1.

Table 1. Lower and upper reporting limits for 55 elements determined by ICP-AES and ICP-MS (sodium peroxide sinter decomposition).

[ICP, inductively coupled plasma; AES, atomic emission spectrometry; MS, mass spectrometry; ppm, parts per million]

Element	Reporting limit (in percent, unless otherwise noted)		Element	Reporting limit (in percent, unless otherwise noted)		Element	Reporting limit (in percent, unless otherwise noted)	
	Lower	Upper		Lower	Upper		Lower	Upper
Aluminum, Al	0.01	25	Copper, Cu	5 ppm	1	Praesodynium, Pr	0.05 ppm	0.10
Calcium, Ca	0.01	35	Dysprosium, Dy	0.05 ppm	0.10	Rubidium, Rb	0.2 ppm	1
Iron, Fe	0.01	30	Erbium, Er	0.05 ppm	0.10	Antimony, Sb	0.1 ppm	500 ppm
Potassium, K	0.01	25	Europium, Eu	0.05 ppm	0.10	Scandium, Sc	5 ppm	5
Magnesium, Mg	0.01	30	Gadolinium, Gd	0.05 ppm	0.10	Samarium, Sm	0.1 ppm	0.10
Manganese, Mn	10 ppm	10	Gallium, Ga	1 ppm	0.10	Tin, Sn	1 ppm	1
Phosphorous, P	0.01	0.25	Germanium, Ge	1 ppm	0.10	Strontium, Sr	0.1 ppm	0.10
Titanium, Ti	0.01	25	Hafnium, Hf	1 ppm	1	Tantalum, Ta	0.5 ppm	1
Silver, Ag	1 ppm	0.10	Holmium, Ho	0.05 ppm	0.10	Thallium, Tl	0.5 ppm	0.10
Arsenic, As	30 ppm	10	Indium, In	0.2 ppm	0.10	Thorium, Th	0.1 ppm	0.10
Barium, Ba	0.5 ppm	1	Lanthanum, La	0.1 ppm	1	Thulium, Tm	0.05 ppm	0.10
Beryllium, Be	5 ppm	0.25	Lead, Pb	5 ppm	1	Tungsten, W	1 ppm	1
Bismuth, Bi	0.1 ppm	0.10	Lithium, Li	10 ppm	5	Terbium, Tb	0.05 ppm	0.10
Cadmium, Cd	0.2 ppm	1	Lutetium, Lu	0.05 ppm	0.10	Uranium, U	0.05 ppm	0.10
Cerium, Ce	0.1 ppm	1	Molybdenum, Mo	2 ppm	1	Vanadium, V	5 ppm	1
Cesium, Cs	0.1 ppm	1	Neodymium, Nd	0.1 ppm	1	Ytterbium, Yb	0.1 ppm	0.10
Chromium, Cr	10 ppm	10	Nickel, Ni	5 ppm	1	Yttrium, Y	0.5 ppm	1
Cobalt, Co	0.5 ppm	1	Niobium, Nb	1 ppm	1	Zinc, Zn	5 ppm	1
						Zirconium, Zr	0.5 ppm	1

Gold (Au), Pt, and Pd concentrations were measured in the samples by ICP-MS after separation by lead button fire assay using a modification of Meier and others (1996). An assay ton (30 g) was weighed into a crucible with 150 g of flux and mixed. One mg of silver nitrate was added and covered with borax, then placed in the furnace for 45 min at 1,080 °C. The melt was poured into a cast iron mold, cooled, and hammered to free the lead button from the slag. The lead button was placed on a cupel and heated at 950 °C until all lead was removed. The resulting dore bead was dissolved in a mixture of nitric acid and hydrochloric acid and heated in a water bath. The final solution was adjusted to 10 mL and introduced into the ICP-MS. The lower reporting limits were 1 part per billion (ppb) for Au, 1 ppb for Pt, and 0.5 ppb for Pd. The upper reporting limit for all three elements is 10,000 ppb. Data were considered acceptable if recovery of Au, Pt, and Pd was ± 20 percent at five times the lower reporting limit and the calculated percent RSD of duplicate samples was no greater than 20 percent.

Arsenic concentrations were determined by hydride generation-atomic absorption spectrometry (HGAAS) (U.S. Geological Survey, 2010b, at http://minerals.cr.usgs.gov/projects/analytical_chem/references.html#M9), a method modified from Hageman and others (2002). An aliquot of sample (0.1 g) was weighed into a zirconium crucible. Approximately 0.75 g of sodium peroxide was added and mixed. The mixture was heated in a muffle furnace set at 750 °C for 4 min. The sample was cooled, and then 15 mL of water and 5 mL of concentrated HCl were added. A 1 mL aliquot was shaken with 0.25 mL of an ascorbic acid/potassium iodide (KI) solution, then diluted to 10 mL with 20 percent HCl and allowed to stand overnight. The reporting range was 0.6 to 20 ppm. Data were considered acceptable if recovery of As was ± 20 percent at five times the limit of detection (LOD) and the calculated percent RSD of duplicate samples was no greater than 20 percent.

Data for these 1,075 stream-sediment samples are in the appendix and available for download as a Microsoft© Excel spreadsheet at http://pubs.usgs.gov/ds532/. All new analyses were completed by laboratories under contract with the USGS.

References Cited

Bailey, E.A., Sutley, S.J., Arbogast, B.F., Willson, W.R., and Church, S.E., 1986, Analytical results and sample locality map of stream-sediment and heavy-mineral-concentrate samples from the Mt. Katmai Quadrangle, and portions of the Naknek, Afognak, and Iliamna quadrangles, Alaska: U.S. Geological Survey Open-File Report 86-586, 126 p., 1 pl.

Detterman, R.L., and Reed, B.L., 1965, Results of stream-sediment sampling in the Iliamna quadrangle, Alaska: U.S. Geological Survey Open-File Report 65-41, 2 p., 1 oversize table.

Erlich, Olga, Motooka, J.M., VanTrump, George, Jr., and Church, S.E., 1988, Analytical data and sample locality map for aqua-regia leachates of stream sediments analyzed by ICP from the Mt. Katmai Quadrangle, and portions of the Naknek, Afognak, and Iliamna quadrangles, Alaska: U.S. Geological Survey Open-File Report 88-422, 72 p., 1 map.

Hageman, P.L., Brown, Z.A., and Welsch, Eric, 2002, Arsenic and selenium by flow injection or continuous flow-hydride generation-atomic absorption spectrophotometry, *in* Taggart, J.E., Jr., ed., Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey: U.S. Geological Survey Open-File Report 2002-223-L, version 5.0, chap. L, p. L1–L7, accessed July 23, 2010, at http://pubs.usgs.gov/of/2002/ofr-02-0223/L06AS24Se_M.pdf.

King, H.D., Crim, W.D., Cooley, E.F., McDanal, S.K., O'Leary, R.M., and Cohen, D.K., 1978, Spectrographic and chemical analyses of geochemical samples from the Lake Clark Quadrangle, Alaska: U.S. Geological Survey Open-File Report 78-788, 59 p., 1 map.

O'Leary, R.M., Cooley, E.F., Day, G.W., McDougal, C.M., Curtin, G.C., and Nelson, S.W., 1978, Spectrographic and chemical analyses of geochemical samples from Talkeetna Quadrangle, Alaska: U.S. Geological Survey Open-File Report 78-143, 141 p., 1 plate.

Meier, A.L., Carlson, R.R., Lichte, F.E., and Bullock, J.H., 1996, Platinum group elements by nickel sulfide fire assay separation and inductively coupled plasma-mass spectrometry, in Arbogast., B.F., ed., Analytical methods manual for the Mineral Resource Surveys Program: U.S. Geological Survey Open-File Report 96-525, p. 158–165.

Meier, A.L., and Slowik, T., 2002, Rare earth elements by inductively coupled plasma-mass spectrometry, *in* Taggart, J.E., Jr., ed., Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey: U.S. Geological Survey Open-File Report 2002-223-K, version 5.0, chapt. K, p. K1-K8, accessed July 23, 2010, at http://pubs.usgs.gov/of/2002/ofr-02-0223/K19NewREE_M.pdf.

Motooka, J.M., Allen, M.S., Malcolm, M.J., and Slaughter, K.E., 1990, Analytical results and sample locality map for stream-sediment samples collected from the eastern part of the Lime Hills Quadrangle, Alaska: U.S. Geological Survey Open-File Report 90-70, 105 p., 1 map.

Peacock, T.R., Taylor, C.D., and Theodorakos, P.M., 2002, Stream-sediment sample preparation, *in* Taggart, J.E., Jr., ed., Analytical methods for chemical analysis of geologic and other materials, U.S. Geological Survey: U.S. Geological Survey Open-File Report 2002-223-A2, version 5.0, chap. A, p. A2-1-A2-4, accessed July 23, 2010, at http://pubs.usgs.gov/of/2002/ofr-02-0223/A2SedPrep_M.pdf.

Reed, B.L., 1967, Results of stream sediment sampling and bedrock analyses in the eastern part of the Iliamna quadrangle, and at Kasna Creek, Lake Clark quadrangle, Alaska: U.S. Geological Survey Open-File Report 67-185, 18 p., 1 map.

Reed, B.L., and Detterman, R.L., 1966, Results of stream sediment sampling in the Iliamna quadrangle, Alaska: U.S. Geological Survey Open-File Report 66-108, 4 p.

Reed, B.L., and Elliott, R.L., 1970, Reconnaissance geologic map, analyses of bedrock and stream-sediment samples, and an aeromagnetic map of parts of the southern Alaska Range: U.S. Geological Survey Open-File Report 70-271, 145 p., 4 sheets, scale 1:125,000.147 p., 4 plates.

U.S. Geological Survey, 2010a, References, USGS In-House Methods, Method 22—55 Element ICP-AES-MS sodium peroxide sinter: accessed July 23, 2010, at http://minerals.cr.usgs.gov/projects/analytical_chem/references.html#m22.

U.S. Geological Survey, 2010b, References, USGS In-House Methods, Method 9 & 10—Arsenic and Antimony: accessed July 23, 2010, at http://minerals.cr.usgs.gov/projects/analytical_chem/references.html#M9.

Appendix A. Major, Rare Earth, and Trace Element Data for 1,075 Stream-Sediment Samples Previously Collected by the U.S. Geological Survey in the Iliamna, Lake Clark, Lime Hills, McGrath, Seldovia, Talkeetna, and Tyonek 1:250,000-Scale Quadrangles, Alaska, 2007–09.

The appendix is a Microsoft© Excel spreadsheet and can be accessed and downloaded at http://pubs.water.usgs.gov/ds352/.

Publishing support provided by the U.S. Geological Survey
Publishing Network, Tacoma Publishing Service Center

For more information concerning the research in this report, contact the

Director, Alaska Science Center
U.S. Geological Survey
4210 University Drive
Anchorage, Alaska 99508
http://alaska.usgs.gov