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**ANALYSES OF HISTORIC U.S. BUREAU OF MINES SAMPLES FOR GEOCHEMICAL  
TRACE-ELEMENT AND RARE-EARTH-ELEMENT DATA FROM KOOK LAKE,  
SITKA QUADRANGLE, ALASKA**

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# ANALYSES OF HISTORIC U.S. BUREAU OF MINES SAMPLES FOR GEOCHEMICAL TRACE-ELEMENT AND RARE-EARTH-ELEMENT DATA FROM KOOK LAKE, SITKA QUADRANGLE, ALASKA

by

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## INTRODUCTION

The State of Alaska's *Strategic and Critical Minerals (SCM) Assessment* project, a state-funded Capital Improvement Project (CIP), is designed to evaluate Alaska's statewide potential for SCM resources. The *SCM Assessment* project is being implemented by the Alaska Division of Geological & Geophysical Surveys (DGGs), and involves obtaining new airborne-geophysical, geological, and geochemical data. For the geochemical part of the *SCM Assessment* project, thousands of historic geochemical samples from the DGGs, U.S. Geological Survey, and U.S. Bureau of Mines (USBM) archives are being re-analyzed by DGGs using modern, quantitative, geochemical-analytical methods. The objective is to update the State of Alaska's statewide digital geochemical database in order to more clearly identify areas with SCM potential.

For this report, DGGs obtained 26 new geochemical analyses on historic USBM samples, including 4 rock, 9 stream sediment, 3 soil, and 10 heavy mineral concentrate (pan concentrate) samples. These samples were originally collected by the USBM to investigate rare-earth-element occurrences associated with the Kook Lake intrusive complex, Sitka Quadrangle, Alaska (Barker and Lamal, 1988). Historic USBM sample materials were retrieved by DGGs from the DGGs Geologic Materials Center (GMC), where the USBM samples were transferred as part of the federally funded *Minerals Data and Information Rescue in Alaska (MDIRA)* program in the late 1990s and early 2000s.

The text and analytical data and tables associated with this report are being released in digital format as PDF files and .csv files. Additional details about the sampling project can be found in the metadata file associated with the digital version of this report, which is available from the DGGs website (doi:[10.14509/27300](https://doi.org/10.14509/27300)) at no charge.

## DOCUMENTATION OF METHODS

### SAMPLE COLLECTION

**Historic sample-collection and analytical procedures** are documented in the original publication for USBM samples (Barker and Lamal, 1988).

### ASSIGNING LOCATION COORDINATES

**Location data** for each sample were derived by DGGS staff by scanning station-location map figures from Barker and Lamal (1988), georegistering the map figures in ArcGIS v. 10.1, creating a point layer of station locations, and extracting latitude–longitude coordinates. Location data for each sample are presented in latitude and longitude coordinates in decimal degrees with NAD27 datum and Clarke 1866 spheroid.

### SAMPLE PREPARATION

**Sample pulps** were retrieved from the GMC, examined, and split by DGGS staff into aliquots needed for the analyses, with leftover portions of the pulps saved and stored at the GMC. Splits of pulps were submitted to ALS Minerals Laboratories (ALS) for analysis. Where pulps were unavailable, coarse-reject material was prepped by ALS using the following method:

- a. **Method PUL-31—Pulverize split to 85 percent <75 µm.** Coarse-reject samples were pulverized at ALS using “flying disk” or “ring and puck” style, low chrome, steel grinding mills. A sample split or total sample up to 250 g is pulverized, with 85 percent of the resulting material passing through a screen of 75 microns.

## ANALYTICAL METHODS

**Data validation:** Analytical results obtained from samples submitted to geochemical laboratories must pass through two levels of data validation. The first level of quality control checks occurs at ALS, which is accredited to ISO/IEC 17025-2005 standards. The laboratory’s quality control protocol is to insert a reagent blank and a reference sample material with every batch of 20 samples to measure the analytical accuracy. Duplicate samples are analyzed at the end of the sample set to measure analytical variance as well as sample variance. Data passing the quality control criteria are sent to DGGS.

The second level of data validation is performed by DGGS. All samples submitted to ALS are accompanied by a variety of DGGS in-house, ore-geochemical reference samples (blinds) submitted at the rate of 1, or occasionally more than 1, per sample batch. Upon receipt of analyses from the lab, reference standards are checked by DGGS against their accepted values. If present, any batches with reference standards with elemental values falling outside 2 standard deviations of their accepted values were submitted to the lab to be reanalyzed.

**Samples were analyzed** for ore-related trace elements and the full suite of rare-earth elements by one or more of the methods listed below. For each sample, data tables either contain assay values, or they contain coded-value place holders (that is, null = not analyzed; -1 = the element’s assay result is less than the lower detection limit for the method; -2 = the element’s assay result is greater than the upper detection limit for the method). Detection limits for each of the reported elemental values obtained by the various methods are documented in the metadata file.

- a. **Method ME-MS81—38-element, fusion, ICP-MS.** A prepared sample (0.200 g) is added to lithium metaborate flux (0.90 g), mixed well and fused in a furnace at 1,000°C. The resulting melt is then cooled and dissolved in 100 mL of 4% HNO<sub>3</sub> / 2% HCl<sub>3</sub> solution. This solution is then analyzed by inductively coupled plasma–mass spectrometry (ICP-MS). Note: Some base-metal oxides and sulfides may not be completely decomposed by the lithium borate fusion. Results for Ag, Co, Cu, Mo, Ni, Pb, and Zn will not likely be quantitative by this method.
- b. **Method ME-ICP61—33-element, 4-acid, ICP-AES.** A prepared sample (0.25 g) is digested with perchloric, nitric, hydrofluoric, and hydrochloric acids. The residue is topped up with dilute hydrochloric acid and the resulting solution is analyzed by inductively coupled plasma–atomic emission spectrometry (ICP-AES). Results are corrected for spectral inter-element interferences. Note: Four-acid digestions are able to dissolve most minerals, however, although the term “near total” is used, depending on the sample matrix, not all elements may be quantitatively extracted.
- c. **Method ME-MS61—48-element, 4-acid, ICP-AES or ICP-MS.** A prepared sample (0.25 g) is digested with perchloric, nitric, hydrofluoric, and hydrochloric acids. The residue is topped up with dilute hydrochloric acid and analyzed by inductively coupled plasma–atomic emission spectrometry (ICP-AES). Following this analysis, the results are reviewed for high concentrations of bismuth, mercury, molybdenum, silver, and tungsten and diluted accordingly. Samples meeting this criterion are then analyzed by inductively coupled plasma–mass spectrometry (ICP-MS). Results are corrected for spectral inter-element interferences. Note: Four-acid digestions are able to dissolve most minerals, however, although the term “near total” is used, depending on the sample matrix, not all elements may be quantitatively extracted. Tin and zirconium assays by this method are not quantitative.
- d. **Method PGM-ICP23—Pt, Pd, Au, 30-gram, FA-ICP-AES.** Gold (Au), platinum (Pt), and palladium (Pd) values were obtained from a prepared, 30 g sample, fused with a mixture of lead oxide, sodium carbonate, borax, and silica, inquarted with 6 mg of gold-free silver, and then cupelled to yield a precious-metal bead. The bead is digested for 2 minutes at high power by microwave in dilute nitric acid. The solution is cooled and hydrochloric acid is added. The solution is digested for an additional 2 minutes at half power by microwave. The digested solution is then cooled, diluted to 4 mL with 2% hydrochloric acid, homogenized, and then analyzed for gold, platinum, and palladium by inductively coupled plasma–atomic emission spectrometry (ICP-AES).
- e. **Method ME-OG62—Ore-grade elements, 4-acid, ICP-AES\* (exceptions discussed under this method).** Samples with elements (where, ME=Ag, Au, Cu, Pb, or Zn) above the detection limits in the analytical methods described above were re-analyzed using method ME-OG62. Assays for the evaluation of ores and high-grade materials are optimized for accuracy and precision at high concentrations. Ultra-high-concentration samples (greater than 15–20%) may require the use of methods such as titrimetric and gravimetric analysis, in order to achieve maximum accuracy. A prepared sample is digested with nitric, perchloric, hydrofluoric, and hydrochloric acids, and then evaporated to incipient dryness. Hydrochloric acid and de-ionized water are added for further digestion, and the sample is heated for an additional allotted time. The sample is cooled to room temperature and transferred to a volumetric flask (100 mL). The resulting solution is diluted to volume with de-ionized water, homogenized and the solution is analyzed by inductively coupled plasma–atomic emission spectroscopy (ICP-AES) or by atomic absorption spectrometry (AAS). \*Note: ICP-AES is the default finish technique for ME-OG62. However, under some conditions and at the discretion of the laboratory an AAS finish may be substituted. No analyses were determined by AAS for this report.
- f. **Method Au-GRAV21—Au, 30-g, FA.** Samples with gold (Au) above the detection limits in analytical method ME-OG62 described above were re-analyzed using method Au-GRAV21. A prepared, 30 g sample is fused with a mixture of lead oxide, sodium carbonate, borax, silica, and other reagents in order to produce a lead button. The lead button containing the precious metals is cupelled to remove the lead. The remaining gold and silver bead is parted in dilute nitric acid, annealed, and weighed as gold.

- g. **Method Ag-GRAV21—Ag, 30-g, FA ICP, GRAV finish.** Samples with silver (Ag) above the detection limits in analytical method ME-OG62 described above were re-analyzed using method Ag-GRAV21. A prepared, 30 g sample is fused with a mixture of lead oxide, sodium carbonate, borax, silica, and other reagents in order to produce a lead button. The lead button containing the precious metals is cupelled to remove the lead. The remaining gold and silver bead is parted in dilute nitric acid, annealed, and weighed as gold. Silver is then determined by the difference in weights.

## REFERENCES

Barker, J.C., and Lamal, Kathryn, 1988, Investigation of rare earth element occurrences near Kook Lake: U.S. Bureau of Mines Field Report, 19 p. <http://www.dggs.alaska.gov/pubs/id/23103>.