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REANALYSIS OF HISTORICAL U.S. GEOLOGICAL SURVEY SEDIMENT SAMPLES FOR GEOCHEMICAL DATA FROM THE WESTERN PART OF THE WRANGELLIA TERRANE, ANCHORAGE, GULKANA, HEALY, MT. HAYES, NABESNA, AND TALKEETNA MOUNTAINS QUADRANGLES, ALASKA

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REANALYSIS OF HISTORICAL U.S. GEOLOGICAL SURVEY SEDIMENT SAMPLES FOR GEOCHEMICAL DATA FROM THE WESTERN PART OF THE WRANGELLIA TERRANE, ANCHORAGE, GULKANA, HEALY, MT. HAYES, NABESNA, AND TALKEETNA MOUNTAINS QUADRANGLES, ALASKA

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Melanie B. Werdon[1], Jaime S. Azain[2], and Matthew Granitto[2]

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* 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*, thousands of historical geochemical samples from DGGS, U.S. Geological Survey (USGS), and U.S. Bureau of Mines archives are being reanalyzed by DGGS using modern, quantitative, geochemical-analytical methods. The objective is to update the statewide geochemical database to more clearly identify areas in Alaska with SCM potential.

The USGS is also undertaking SCM-related geologic studies in Alaska through the federally funded *Alaska Critical Minerals* cooperative project. DGGS and USGS share the goal of evaluating Alaska's strategic and critical minerals potential and together created a Letter of Agreement (signed December 2012) and a supplementary Technical Assistance Agreement (#14CMTAA143458) to facilitate the two agencies' cooperative work. Under these agreements, DGGS contracted the USGS in Denver to reanalyze historical USGS sediment samples from Alaska.

For this report, DGGS funded reanalysis of 1,682 historical USGS sediment samples from the statewide Alaska Geochemical Database Version 2.0 (AGDB2; Granitto and others, 2013). Samples were chosen from an area covering the western half of the Wrangellia Terrane in the Anchorage, Gulkana, Healy, Mt. Hayes, Nabesna, and Talkeetna Mountains quadrangles of south-central Alaska (fig. 1). USGS was responsible for sample retrieval from the Denver warehouse through the final quality assurance/quality control (QA/QC) of the geochemical analyses obtained through the USGS contract lab. The new geochemical data are published in this report as a coauthored DGGS report, and will be incorporated into the statewide geochemical databases of both agencies.

DOCUMENTATION OF METHODS

SAMPLE COLLECTION

The 1,682 Alaska sediment samples of interest were collected as part of several projects between 1964 and 2001. Details of initial sample collection and analytical methods for these historical samples have been compiled into a single digital database, AGDB2 (Granitto and others, 2013). The original analyses for most of these samples were previously published, and the citations for the original results are included in the References and Original Data Sources section.

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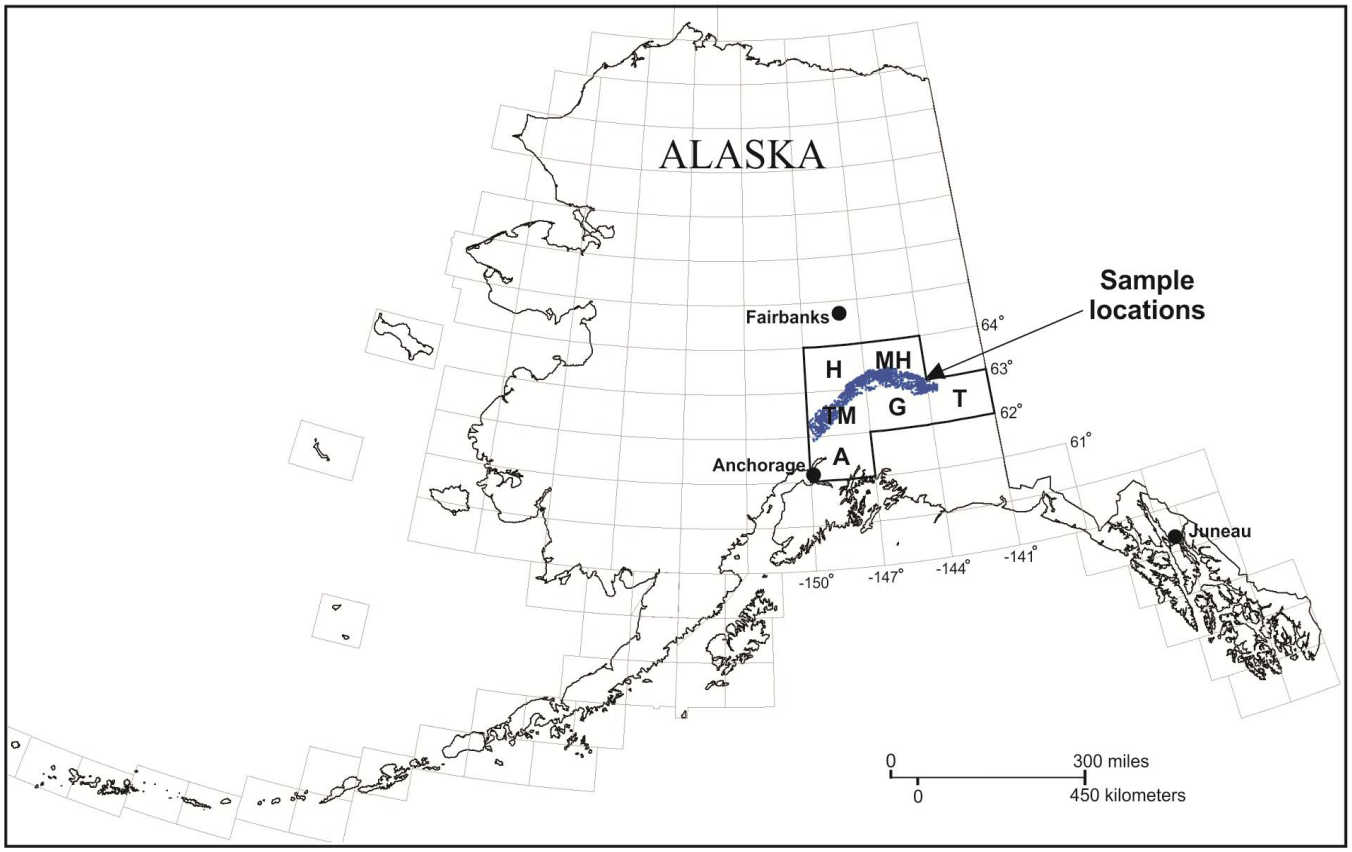

Figure 1. Map showing the location of reanalyzed sediment samples in the Healy (H), Mt. Hayes (MH), Talkeetna Mountains (TM), Gulkana (G), Tanacross (T), and Anchorage (A) quadrangles, Alaska.

Location data for each sample are presented in latitude and longitude coordinates in decimal degrees with NAD27 datum and Clarke 1866 spheroid; resolution is variable, and ranges from 5-digit GPS precision to others with less precision. Original locations were collected with NAD27 datum and Clarke 1866 spheroid prior to 2000, but some of the samples (after 2000) were collected in other projections. DGGS converted the coordinates for these samples to NAD27 datum and Clarke 1866 spheroid using appropriate transformations in ArcGIS; original location information is documented in Granitto and others (2013).

SAMPLE PREPARATION

Historical sediment samples were retrieved from the National Geochemical Sample Archive (NGSA) in Denver, Colorado, by USGS staff, then weighed, ground to -200 mesh and, where sufficient material was available, split into aliquots needed for analyses. Leftover material was re-archived at the NGSA. Sample splits were submitted to the USGS contract lab (SGS Minerals Services, Lakefield, Canada) for analysis.

ANALYTICAL METHODS

Where sufficient sample material was available, sample splits were analyzed for gold (Au), palladium (Pd), platinum (Pt), tellurium (Te), and 55 major and trace elements. Samples previously analyzed for Au, Pd, and Pt by fire assay were not reanalyzed as part of this project because the historical analyses are the same as modern analytical methods.

Data Validation: Analytical results obtained from samples submitted to the contract laboratory (SGS) must pass through two levels of data validation. The first level of quality control checks occurs at SGS Minerals Services.

SGS is accredited to ISO/IEC 17025:2005 standards and conforms to requirements of CAN-P-4E and CAN-P-1579 (Standards Council of Canada, 2014). 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 the USGS and imported into the Laboratory Information Management System.

The second level of data validation is performed at the USGS. All samples submitted to SGS are accompanied by a set of USGS in-house reference samples (blinds) submitted at the rate of 10 percent. Using a program in the Laboratory Information Management System, the data for the reference samples are evaluated by comparing the "obtained" analytical value to the "expected" value for each element. The values must fall within the range of acceptance, which varies between ±5 percent to ±20 percent depending on the analytical method. Analytical results for samples associated with the accepted quality control data are then released to the sample submitter. Samples associated with rejected quality control data are reanalyzed by SGS. In this study, there weren't any samples associated with rejected quality control data, therefore no samples were reanalyzed.

The following list describes each analytical method, sample weight (in grams), detection limits, and acceptable analytical performance criteria:

a. **Method 20—Gold (Au), Platinum (Pt), and Palladium (Pd).** Gold, Pt, and Pd content were determined in geologic materials by Inductively Coupled Plasma–Mass Spectrometry (ICP-MS) after collection by fire assay. An assay ton (30 grams) was weighed into a crucible with 150 grams of flux and mixed. One mg of silver nitrate was added and covered with borax, and then placed in the furnace for 45 minutes 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 the lead was removed. The resulting doré 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 are 1 ppb for Au and Pd, and 0.5 ppb for Pt. The upper limit for all elements is 10,000 ppb. Data were deemed acceptable if recovery of gold, platinum, and palladium was ±20 percent at five times the lower limit of detection (LOD) and the calculated Relative Standard Deviation (RSD) of duplicate samples was no greater than 20 percent.

b. **Method 22—55-Element ICP-AES-MS sodium peroxide sinter.** Fifty-five major (except Si and Na), rare-earth, and trace elements were determined in geologic materials by inductively coupled plasma–atomic emission spectrometry (ICP-AES) and ICP-MS. The 0.10 g sample was decomposed using a sodium-peroxide sinter at 450°C. The resultant cake was leached with water for a minimum of 4 hours, and acidified with nitric acid. After an addition of tartaric acid, aliquots of the digested sample 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. Calibration on the ICP-AES was performed by standardizing with digested rock reference materials and a series of multi-element solution standards. The ICP-MS was calibrated with aqueous standards, and internal standards were used to compensate for matrix effects and internal drifts. Reporting limits for the 55 elements are presented in the tables accompanying this report. Data were deemed acceptable if recovery for all 55 elements was ±15 percent at five times the lower LOD and the calculated RSD of duplicate samples was no greater than 15 percent.

c. **Method 13—Tellurium (Te)**. Tellurium content was determined by weighing 0.25 g of sample into a Teflon tube, adding a mixture of nitric, hydrofluoric, and perchloric acids and heating the sample. After the solution cooled, hydrochloric and nitric acids were added, and the sample was heated again, and then cooled. The samples were diluted and analyzed using hydride-generation atomic absorption spectrometry with an auto-analyzer and automated data collection system from Labtronics. The lower reporting limit for Te is 0.1 ppm and the upper detection limit is 1,000 ppm. Data for Te were deemed acceptable if recovery of that element was ±20 percent at five times the lower LOD and the calculated RSD of duplicate samples was no greater than 20 percent.

d. **Method 15—XRF Major Element Analysis.** For this study, Method 15 was applied to niobium (Nb), thorium (Th), and zirconium (Zr), when results were higher than the upper detection limit by Method 22. This occurred in only one sample: Lab No. C-382999/Field No. 68ASB1103). Niobium, Th, and Zr were determined on sediment sample pulps by wavelength dispersive X-ray fluorescence spectrometry (WDXRF). The sample was fused with 50/50 lithium metaborate–lithium tetraborate flux and the resultant glass disk was introduced into a wavelength dispersive X-ray spectrometer. The disk was irradiated by a Rhodium X-ray tube. X-ray photons emitted by the elements in the sample were counted and concentrations determined using previously prepared calibration curves. Calibration curves for each element (in typical major-element analyses) were derived from a variety (about 40) of international reference materials (National Institute of Standards and Technology, USA [NIST], U.S. Geological Survey [USGS], Canada Center for Mineral and Energy Technology [CANMET], National Institute for Metallurgy, South Africa [NIM]) and a number of synthetic standards to extend the range for certain elements. The standards cover a wide range of geologic materials, biased toward igneous rock types. Unusual rock compositions may require a special calibration. The lower detection limit for Nb, Th, and Zr is 0.01 percent, and the upper detection limit is 100 percent. Data for Nb, Th, and Zr were deemed acceptable if recovery of the elements was ± 5 percent at the lower LOD and the calculated RSD of duplicate samples was no greater than 5 percent. The SGS lab reports major-element oxides in percentages, so Nb, Th, and Zr values were converted from oxide to elemental concentrations.

RESULTS

This report includes the following components: A text report (PDF file), an analytical-data table (.xlsx file), a detection-limits table (.csv file), a metadata file (.htm) documenting additional details about the reanalysis project, and a ReadMe file (.htm), which provides an overview of files associated with this report. All files associated with this report are available from the DGGS website (doi:10.14509/27287) at no charge.

In the analytical-data table, field names (column headers) show the element and the units in which they are reported. Where a numerical suffix is shown, this element was analyzed by more than one method. In the detection-limits table and the metadata file, documentation is provided to explain each field name, as well as additional details such as lab name and method codes, analytical-method types and documentation, and the upper and lower detection limits for each of the elements and methods. For each element, for each sample, the analytical-data table either contains assay values, or it contains 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; -3 = composition of this sample makes detection impossible by this method; interference problems).

One sample (Lab No. C-382999/Field No. 68ASB1103) contains unusually high values of Nb, REEs, Y, and Zr, as well as elevated levels of Sn, Ta, Th, U, and W. The original data publication (Bailey and others, 1999) was checked to make sure this sample wasn't a pan-concentrate sample. The field number for this sample is part of a series of field numbers associated with sediment samples. The sample was collected from a stream about 1.5 km upstream from the Valdez Creek placer gold mine. Approximately 1.5 km upstream from this sample, the stream cuts through an undated, Tertiary(?) granitic intrusion (unit Tegr; Wilson and others, 1998). Because of the unusually high values, in summer 2015 DGGS plans to sample upstream and downstream of this anomaly to validate or refute the high elemental values, and to investigate the intrusion (unit Tegr) to determine if it could be a potential source for the anomaly.

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