



# **GEOLOGICAL SURVEY OF CANADA**

# **OPEN FILE 6272**



Regional Stream Sediment and Water Geochemical Data, Lansing Range area, east central Yukon (NTS 105N)

S.J.A. Day, M.W. McCurdy, P.W.B. Friske, R.J. McNeil, E.H.W. Hornbrook, J.J. Lynch, C.C. Durham, H. Gross, A.C. Galletta

2009



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Yukon Geological Survey Open File 2009-27

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# **Regional Stream Sediment and Water Geochemical Data, Lansing Range area, east central Yukon (NTS 105N)**

#### Introduction

Geological Survey of Canada (GSC) Open File 6272 contains analytical data for stream silt sediment, stream water and field data -originally published as Open File 2363, as well as Inductively Coupled Plasma Mass Spectrometry (ICP-MS) re-analysis data of archived stream silt sediment samples. This publication includes site-specific field observation and analytical data for stream sediments and waters from 783 sites. Silt sediments were analysed for 49 elements plus loss-on-ignition and corresponding waters were analysed for uranium, fluoride and pH.

National Topographic System (NTS) mapsheet 105N was sampled in 1990 as part of the Canada-Yukon Economic Programming Agreement (1989-1990). The reconnaissance survey was undertaken by the Geological Survey of Canada in conjunction with the Department of Indian Affairs and Northern Development. Funding for the ICP-MS re-analysis was provided by the Yukon Geological Survey and supported by the GSC's Environment and Health Program.

Regional geochemical surveys have been carried out since 1976 in Yukon under the National Geochemical Reconnaissance (NGR) program. One centre-lake sediment and water survey and 27 stream silt sediment and water surveys (Fig. 1) have been published or are in publication. Approximately 102,100 km<sup>2</sup> of Yukon have been covered at an average density of 1 sample or site per 13 km<sup>2</sup>.

Analytical results and field observations contribute to building a national geochemical database for resource assessment, mineral exploration, geological mapping, and environmental studies. Sample collection, preparation procedures and analytical methods are strictly specified and carefully monitored to ensure consistent and reliable results regardless of the area, the year of collection or the analytical laboratory undertaking the analyses (Friske and Hornbrook, 1991).

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#### Yukon Geological Survey

Maps & Publications - Geoscience Information and Sales Box 2703 (K102) Whitehorse, Yukon, Canada Y1A 2C6 Phone: (867) 667-5200 Fax: (867) 667-5150 E-Mail: geosales@gov.yk.ca Website: http://www.geology.gov.yk.ca/ Street Address: 102-300 Main Street, Whitehorse, Yukon, Canada



Figure 1: NGR coverage in Yukon, NWT and western Nunavut

#### Methods

#### **Description of Survey and Sample Management**

Helicopter-supported sample collection was carried out during the summer of 1990. Stream sediment and water samples were collected at an average density of one sample per 13 square kilometres throughout the 11,300 square kilometres of the south-central Yukon survey.

Sample site duplicate samples, or field duplicates samples, were routinely collected in each analytical block of twenty samples and were assigned Replicate Status (Rep Stat) values of 10 and 20. Routine samples were assigned a Rep Stat of 0. At the time of collection, one random sample position within each block of twenty samples is left blank for a control reference sample to be inserted prior to analyses.

Field observations were recorded on standard forms used by the Geological Survey of Canada (Garrett, 1974) (see Figure 2, p11).

The sample site positions were marked on appropriate 1:250 000 scale NTS maps in the field. These maps were digitized at the Geological Survey in Ottawa to obtain the sample site UTM (NAD27) coordinates. Using GSC Open File 3939 (Lepard, 2008) these same coordinates were converted to NAD83 decimal degrees.

In Ottawa, field dried samples were air-dried and sieved through a minus 80 mesh (177 micron) screen, and ball-milled before analysis. At this time, control reference and blind duplicate samples were inserted into each block of twenty sediment samples. For the water samples, only control reference samples were inserted into the block. There were no blind duplicate water samples.

The sample site coordinates were checked as follows: a sample location map was produced on a Versatec plotter using the digitized coordinates; the original sample location map produced in the field was then overlain on the Versatec map; the two sets of points were checked for coincidence.

Thorough inspections of the field and analytical data were made to check for any missing information and/or gross errors.

Quality control and monitoring of the geochemical data was undertaken by a standard method used by the Exploration Geochemistry Subdivision (1990) and ICP-MS reanalysis QA/QC measures were carried out by Applied Geochemistry section staff (2008) at the Geological Survey of Canada.

#### Analytical Procedures

A summary of analytical methods and detection limits is provided in Table 1.

#### Inductively Coupled Plasma Mass Spectrometry (ICP-MS)

For the determination of 35 elements listed in Table 1, a one-gram sample was leached with 6 ml of a mixture of HCl,  $HNO_3$ , and distilled, deionized water (2:2:2 v/v) at 95°C for one hour. The sample solution was diluted to 20 ml and analysed by inductively coupled plasma emission spectroscopy on a Jarell-Ash instrument and inductively coupled plasma mass spectroscopy on a Perkin-Elmer Elan instrument.

#### Instrumental Neutron Activation Analysis (INAA)

Weighed and encapsulated samples are packaged for irradiation along with internal standards and international reference materials. Samples and standards are irradiated together with neutron flux monitors in a two-megawatt pool type reactor. After a seven day decay period, samples are measured on a high resolution germanium detector. Computer control is achieved with a Microvax II computer. Typical counting times are 500 seconds. Elements determined by INAA include: Au, Ag, As, Ba, Br, Ca, Co, Cr, Cs, Fe, Hf, Hg, Ir, Mo, Na, Ni, Rb, Sb, Sc, Se, Sn, Sr, Ta, Th, U, W, Zn, La, Ce, Nd, Sm, Eu, Tb, Yb and Lu. The sample weight is also reported. Data for Ag, Ca, Hg, Ir, Mo, Ni, Se, Sn, Sr and Zn are not published because of inadequate detection limits and/or precision.

#### Atomic Absorption Spectroscopy (AAS) and Other Analyses

For the determination of Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe, Cd and As, a 1 gram sample is reacted with 3 ml concentrated  $HNO_3$  in a test tube overnight at room temperature. After digestion, the test tube is immersed in a hot water bath at room temperature and brought up to 90°C and held at this temperature for 30 minutes with periodic shaking. One ml of concentrated HCl is added and heating continues for another 90 minutes. The sample solution is then diluted to 20 ml with metal-free water and mixed. Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe and Cd are determined by atomic absorption spectroscopy using an air-acetylene flame. Background corrections are made for Pb, Ni, Co, Ag, and Cd.

Molybdenum and vanadium are determined by atomic absorption spectroscopy using a nitrous oxide acetylene flame. A 0.5 gram sample is reacted with 1.5 ml concentrated HNO<sub>3</sub> at 90°C for 30 minutes. At this point, 0.5 ml concentrated HCl is added and the digestion continued at 90°C for an additional 90 minutes. After cooling, 8 ml of 1250 ppm Al solution are added and the sample solution diluted to 10 ml before aspiration.

Mercury is determined by the Hatch and Ott procedure with some modifications. The method is described by Jonasson et al. (1973). A 0.5 gram sample is reacted with 20 ml concentrated HNO<sub>3</sub> and 1 ml concertrated HCl in a test tube for 10 minutes at room temperature prior to two hours of digestion with mixing at 90°C in a hot water bath. After digestion, the sample solutions are cooled and diluted to 100 ml with metal-free water. The Hg present is reduced to the elemental state by the addition of 10 ml 10% w/v SnSO<sub>4</sub> in M H<sub>2</sub>SO<sub>4</sub>. The Hg vapour is then flushed by a stream of air into an absorption cell mounted in the light path of an atomic absorption spectrophotometer. Absorption measurements are made at 253.7 nm.

Loss-on-ignition is determined using a 500 mg sample. The sample, weighed into a 30 ml beaker, is placed in a cold muffle furnace and brought up to 500 °C over a period of two to three hours. The sample is held at this temperature for four hours, then allowed to cool to room temperature for weighing.

Fluorine is determined as described by Ficklin (1970). A 250 mg sample is sintered with 1 gram of a flux consisting of two parts by weight sodium carbonate and one part by weight potassium nitrate. The residue is then leached with water. The sodium carbonate is neutralized with 10 ml 10% (w/v) citric acid and the resulting solution diluted to 100 ml with water. The pH of the solution should range from 5.5 to 6.5. The fluoride content of the test solution is measured using a fluoride ion electrode. Standard solutions contain sodium carbonate and citric acid in the same quantities as the sample solution.

Tin in stream sediments is determined by heating a 200 mg sample with  $NH_4I$ : the sublimed  $SnI_4$  is dissolved in acid and the tin determined by atomic absorption spectrometry after solvent extraction of the tin into methyl isobutyl ketone containing trioctylphosphine oxide (TOPO). The method is described by Welsch and Chao (1976).

Water Analyses

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Fluoride in water samples is determined using a fluoride electrode. Prior to measurement an aliquot of the sample is mixed with an equal volume of TISAB II buffer solution (total ionic strength adjustment buffer). The TISAB II buffer solution is prepared as follows: to 50 ml metal-free water add 57 ml glacial acetic acid, 58 g NaCl and 4 g CDTA (cyclohexylene dinitrilo tetraacetic acid). Stir to dissolve and cool to room temperature. Using a pH meter, adjust the pH between 5.0 and 5.5 by slowly adding 5 M NaOH solution. Cool and dilute to one litre in a volumetric flask. Detection limit = 20 ppb.

Hydrogen ion activity (pH) is measured with a combination glass-calomel electrode and a pH meter.

Uranium in waters is determined by a laser-induced fluorometric method using a Scintrex UA-3 uranium analyzer. A complexing agent, known commercially as Fluran and composed of sodium pyrophosphate and sodium monophosphate (Hall, 1979) is added to produce the uranyl pyrophosphate species which fluoresces when exposed to the laser. Since organic matter in the sample can cause unpredictable behaviour, a standard addition method is used. Further, the reaction of uranium with Fluran can be delayed or sluggish; for this reason an arbitrary 24 hour time delay between the addition of the Fluran and the actual reading is incorporated into this method. In practice, 500 microlitres of Fluran solution are added to a 5 ml sample and allowed to stand for 24 hours. At the end of this period fluorescence readings are made with the addition of 0.0, 0.2 and 0.4 ppb U. For high samples the additions are 0.0, 2.0 and 4.0 (20 microlitre aliquots of either 55 or 550 ppb U are used). All readings are taken against a sample blank.

#### Presentation and Interpretation of Gold Data

The following discussion reviews the format used to present the gold geochemical data and outlines some important points to consider when interpreting this data. This discussion is included in recognition of the special geochemical behaviour and mode of occurrence of gold in nature and the resultant difficulties in obtaining and analyzing samples which reflect the actual concentration level at a given site.

The correct interpretation of geochemical gold data from regional stream sediment or lake sediment surveys requires an appreciation of the unique chemical and physical characteristics of gold and its mobility in the surficial environment. Key properties of gold that distinguish its geochemical behaviour from most other elements (Harris, 1982) include :

1) Gold occurs most commonly in the native form which is chemically and physically resistant. A significant proportion of the metal is dispersed in a micron-sized particulate form, and the high specific gravity of gold results in a heterogeneous distribution, especially in stream sediment and clastic-rich (low LOI) lake sediment environments. Gold distribution appears to be more homogeneous in organic- rich fluviatile and lake sediments.

2) Gold typically occurs at low concentrations in the ppb range. Whereas gold concentrations of only a few ppm may represent economic deposits, background levels in stream and centre-lake sediments seldom exceed 10 ppb, and commonly are near the detection limit of 2 ppb.

These factors result in a particle sparsity effect wherein very low concentrations of gold are heterogeneously enriched or depleted in the surficial environment. Hence, a major problem facing the geochemist is to obtain a representative sample. In general, areas where concentrations of gold in sediments are low, and/or grain sizes of the gold present relatively high require proportionally larger samples to reduce the uncertainty between subsample analytical values and actual values. Conversely, as actual gold concentrations increase or grain size decreases, the number of gold particles to be shared in random subsamples increases and variability of results decreases (Clifton et al., 1969; Harris, 1982). The limited amount of material collected during the rapid, reconnaissance-style regional surveys and the need to analyze for a broad spectrum of elements, precludes the use of a significantly large sample weight for the gold analyses. Therefore, to obtain representative samples, grain size is reduced by sieving and ball milling of the dried sediments.

The following control methods are currently employed to evaluate and monitor the sampling and analytical

variability which are inherent in the analysis of gold in geochemical media:

- (1) For each block of 20 samples:
  - (a) random insertion of a standard reference sample to control analytical accuracy and long-term precision;
- (b) collection of a field duplicate (two samples from one site) to measure sampling and analytical variance;
- (c) analysis of a second subsample (blind duplicate) from one sample to measure and control short-term precision or analytical variance.

(2) For both stream and lake sediments, routine repeat analyses on a second subsample are performed for all samples having values that are statistically above approximately the  $90^{th}$  percentile of the total data set.

(3) For lake sediments only, a routine repeat analysis on a second subsample is performed on those samples with LOI values below 10%, indicating a large clastic component. Ongoing studies suggest that the gold distribution in these samples is more likely to be variable than in samples with a higher LOI content.

The presentation of gold data, statistical treatment and the value map format are different than for other elements. Gold data listed in the open file may include initial analytical results, values determined from repeat analyses, together with sample weights and corresponding detection limits for all analyzed samples. The gold, statistical parameters and regional symbol-trend plots are determined using only the first analytical value. Gold values less than the detection limit are set to half that limit. On the value map, initial values are followed by a comma and a value obtained by a second analysis, where determined. Sample weights used can be found in the text. Following are possible variations in data presentation on a value map.

In summary, geochemical follow-up investigations for gold should be based on a careful consideration of all geological and geochemical information, and especially a careful appraisal of gold geochemical data and its variability. In some instances, prospective follow-up areas may be indirectly identified by pathfinder element associations in favourable geology, although an analogous gold response due to natural variability may be lacking. Once an anomalous area has been identified, field investigations should by designed to include detailed geochemical follow-up surveys and collection of large representative samples. Subsequent repeat subsample analyses will increase the reliability of results and permit a better understanding of natural variability which can then by used to improve sampling methods and interpretation.

Element	Analytical Method	Detection Limit	Units of Measurement
		Silt	
		Sii	
Al	ICP	0.01	%
As	ICP	0.1	ppm
As	INA	0.5	ppm
Au (1)	INA	2	ppb
Au (1) weight	INA	0.01	g

TABLE 1. Summary of Analytical Data and Methods

Au (2)	INA	2	ppb
Au (2) weight	INA	0.01	g
Ba	ICP	0.5	ppm
Ba	INA	50	ppm
Bi	AAS	0.1	ppm
Bi	ICP	0.02	ppm
Br	INA	0.5	ppm
Ca	ICP	0.01	%
Cd	AAS	0.2	ppm
Cd	ICP	0.01	ppm
Ce	INA	3	ppm
Со	AAS	2	ppm
Co	ICP	0.1	ppm
Co	INA	1	ppm
Cr	ICP	0.5	ppm
Cr	INA	5	ppm
Cs	INA	1	ppm
Cu	AAS	2	ppm
Cu	ICP	0.01	ppm
Eu	INA	0.2	ppm
F	ISE	20	ppm
Fe	AAS	0.02	%
Fe	ICP	0.01	%
Fe	INA	0.01	%
Ga	ICP	0.1	ppm
Hf	INA	1	ppm
Hg	CV-AAS	10	ppb
Hg	ICP	5	ppb
К	ICP	0.01	%
La	ICP	0.5	ppm
La	INA	0.5	ppm
LOI	GRAV	1.0	%
Lu	INA	0.05	ppm
Mg	ICP	0.01	%
Mn	AAS	5	ppm
Mn	ICP	1	ppm
Mo	AAS	2	ppm
Mo	ICP	0.01	ppm
Na	ICP	0.001	%
Na	INA	0.01	%
Nd	INA	5	ppm
Ni	AAS	2	ppm

Ni	ICP	0.1	ppm
Р	ICP	0.001	%
Pb	AAS	2	ppm
Pb	ICP	0.01	ppm
Rb	INA	5	ppm
S	ICP	0.02	%
Sb	ICP	0.02	ppm
Sb	INA	0.1	ppm
Sc	ICP	0.1	ppm
Sc	INA	0.1	ppm
Se	AAS	0.1	ppm
Se	ICP	0.1	ppm
Sm	INA	0.1	ppm
Sn	AAS	1	ppm
Sr	ICP	0.5	ppm
Та	INA	0.5	ppm
Tb	INA	0.5	ppm
Te	ICP	0.02	ppm
Th	ICP	0.1	ppm
Th	INA	0.2	ppm
Ti	ICP	0.001	%
Tl	ICP	0.02	ppm
U	ICP	0.1	ppm
U	INA	0.5	ppm
V	AAS	5	ppm
V	ICP	2	ppm
W	ICP	0.1	ppm
W	INA	1	ppm
Yb	INA	0.2	ppm
Zn	AAS	2	ppm
Zn	ICP	0.1	ppm

Waters			
F	ISE	20	ppb
pН	GCM	0.1	
U	LIF	0.05	ppb

AAS - atomic absoprtion spectrometry CV-AAS - cold vapour (flameless) atomic absorption

#### **CREDITS (1990)**

E.H.W. Hornbrook directed the survey.

P.W.B. Friske coordinated the operational activities of contract and Geological Survey of Canada staff.

Contracts were let to the following companies for sample preparation and analysis and were managed by the following staff of the Regional Geochemical Studies section:

Collection:	Northway Map Technology Don Mills, Ontario E.H.W. Hornbrook P.W.B. Friske
Preparation:	Bondar-Clegg and Company Ottawa, Ontario J.J. Lynch
Analysis:	Bondar-Clegg and Company Ottawa, Ontario
	Activation Laboratories Ltd. Ancaster, Ontario
	Chemex Labs North Vancouver, British Columbia J.J. Lynch

M. McCurdy edited open files and coordinated production.

H. Gross and A.C. Galletta provided computer processing support.

Pat Doyle, C.C. Durham and Helena Karam provided technical assistance.

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S. Day edited and compiled this open file.

S.W. Adcock of the GSC reviewed this open file and provided many useful comments and suggestions.

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Figure 2: Card used to capture site-specific field observation data (Garrett, 1974)