

GEOLOGICAL SURVEY OF CANADA



COMMISSION GÉOLOGIQUE DU CANADA

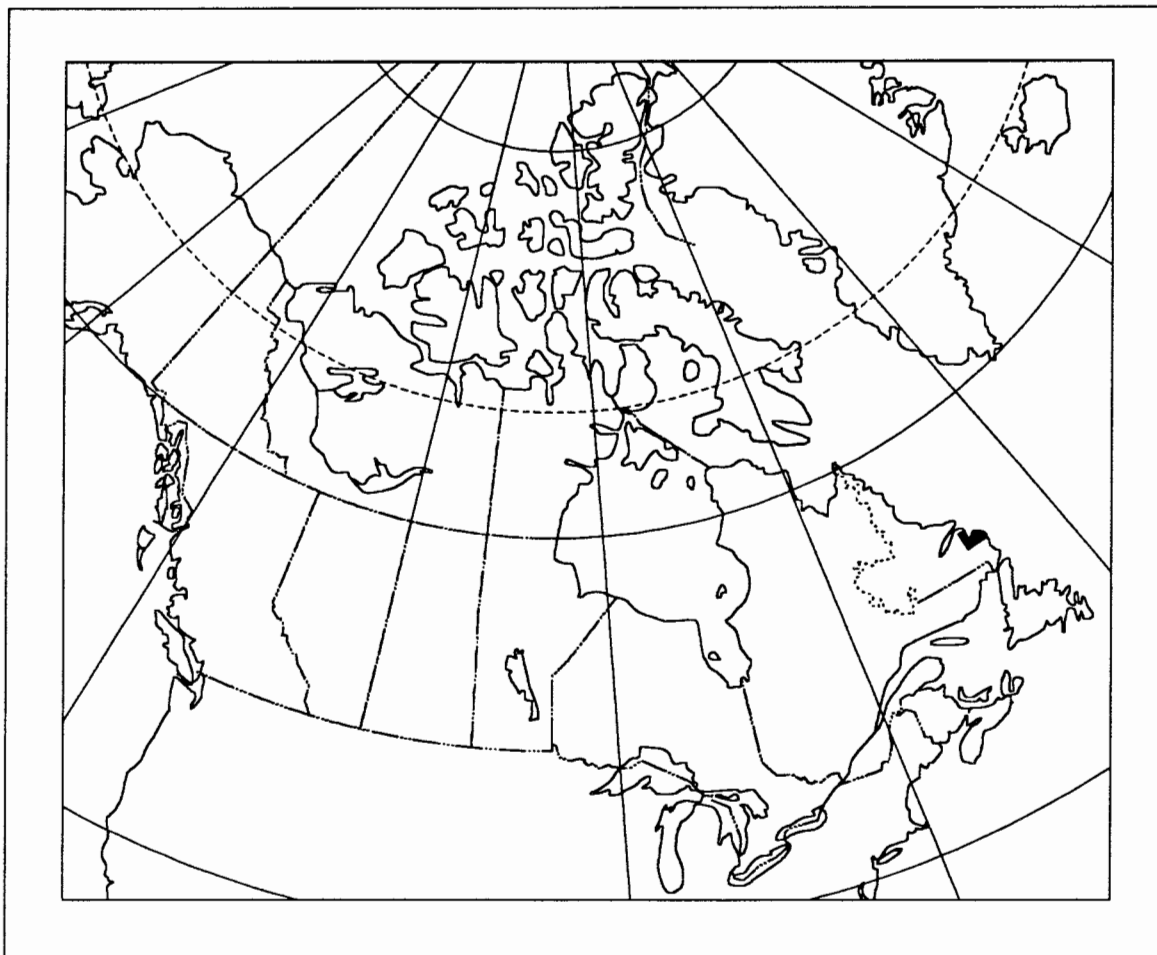
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**GEOLOGICAL SURVEY OF CANADA OPEN FILE 2473  
(NTS 3E and 13H)**

**CANADA - NEWFOUNDLAND COOPERATION AGREEMENT  
ON MINERAL DEVELOPMENT (1990-1994)**

**REGIONAL LAKE SEDIMENT AND WATER  
GEOCHEMICAL RECONNAISSANCE DATA  
SOUTHEASTERN LABRADOR**

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Friske, P.W.B., McCurdy, M.W., Gross, H., Day, S.J., Balma, R.G., Lynch, J.J., Durham, C.C. (1994):  
National Geochemical Reconnaissance Lake Sediment and Water Data, Southeastern Labrador  
(NTS 3E and 13H), Geological Survey of Canada Open File 2473

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**24 February 1994**

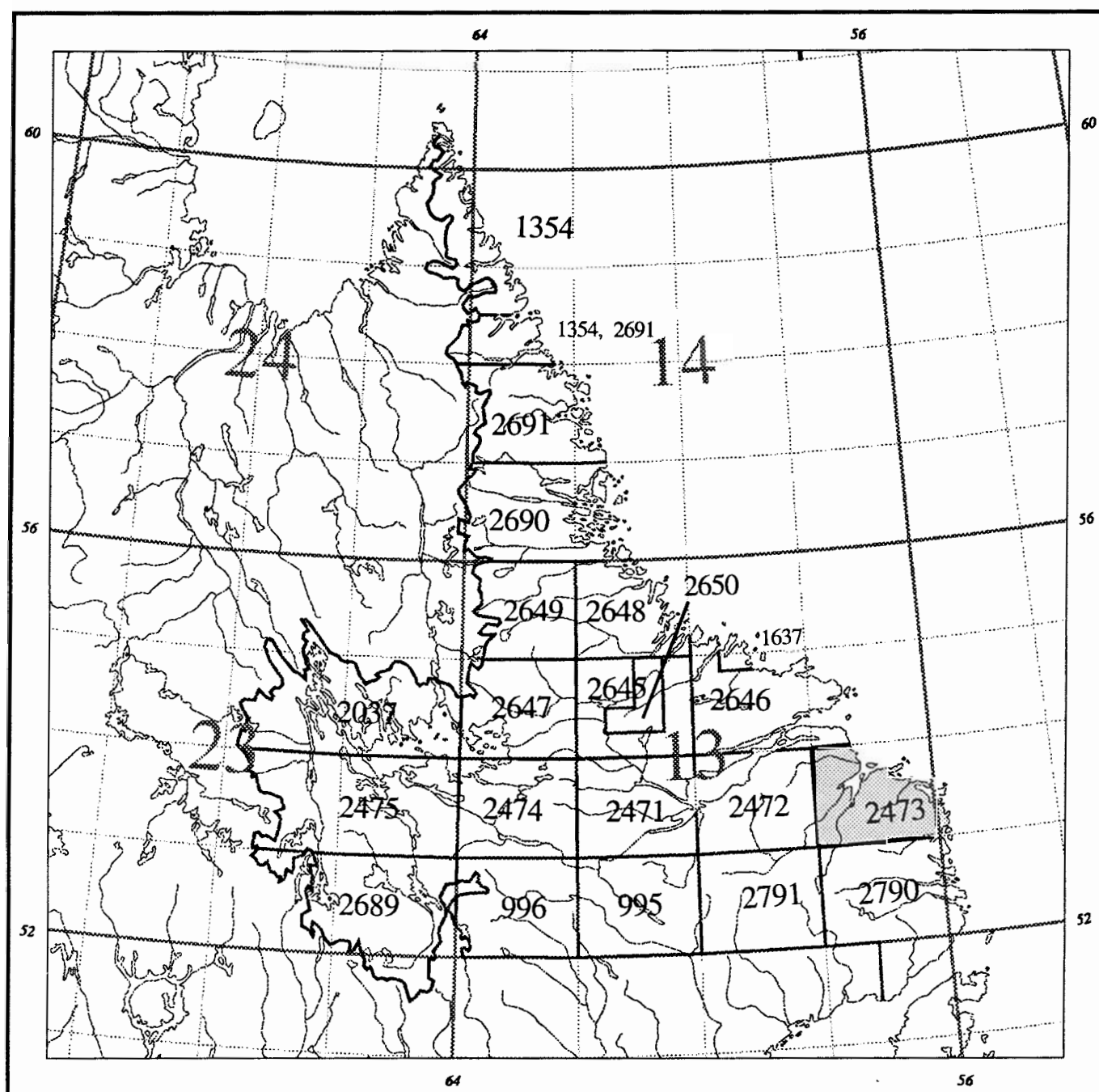


Natural Resources  
Canada

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Canada

Canada

**NATIONAL GEOCHEMICAL RECONNAISSANCE**  
**LAKE SEDIMENT AND WATER GEOCHEMICAL DATA**  
**NEWFOUNDLAND 1994**  
**GEOLOGICAL SURVEY OF CANADA OPEN FILE 2473**  
**NTS 3E AND 13H**



**National Topographic System reference and index to adjoining  
geochemical reconnaissance surveys**

Open File 2473 represents a contribution to the Canada – Newfoundland Cooperation Agreement on Mineral Development (1990–1994), a subsidiary agreement under the Economic and Regional Development Agreement. This project was managed by the Geological Survey of Canada.

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# GSC OPEN FILE 2473

## REGIONAL LAKE SEDIMENT AND WATER GEOCHEMICAL DATA, LABRADOR NTS 3E and 13H

### INTRODUCTION

Open File 2473 contains data for gold and 25 other elements obtained by re-analyzing lake sediments collected in 1977 from 758 sites in eastern Labrador. Original analytical data selected from Open File 512 (published in 1978) for 13 elements plus loss-on-ignition in sediments, and uranium, pH and fluoride values in concomitant waters, are also included in this open file.

The original reconnaissance survey was carried out by the Geological Survey of Canada (GSC) in conjunction with the Newfoundland Department of Mines and Energy under the terms of the Canada - Newfoundland Agreement on a Uranium Reconnaissance Program (1976-1982). Fisheries and Environment Canada provided funds for the determination of mercury. Analyses of archive samples for Open File 2473 were undertaken under the Canada - Newfoundland Cooperation Agreement on Mineral Development (1990-1994).

Analytical results and field observations are used to build a national geochemical data base 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 or the analytical laboratory.

Regional geochemical surveys have been carried out by the GSC in Labrador since 1977. A total of 21 open files are available that provide complete coverage of Labrador. Areas surveyed, with associated open file numbers, are shown in Figure 1. Figure 2 illustrates geochemical coverage across Canada. Data from all open files are available on 3.5 or 5.25 inch diskettes, or in the original published form.

### CREDITS

P.W.B. Friske coordinated the activities of contract and GSC staff under the reanalysis program.

E.H.W. Hornbrook directed the original survey.

Contracts were let to the following companies for sample collection, preparation, original analyses and reanalyses and were managed by Geological Survey of Canada staff as follows:

Collection (1977):     Marshall Macklin Monaghan  
                                 Toronto, Ontario  
                                 *E. Hornbrook, Y.T. Maurice*  
                                 *(GSC)*

Preparation (1977):     Golder Associates  
                                 Ottawa, Ontario  
                                 *J.J. Lynch (GSC)*

Analysis (1977):        Chemex Labs  
                                 Vancouver, British Columbia

Barringer Research Limited  
Toronto, Ontario

Atomic Energy of Canada Ltd.  
Ottawa, Ontario

*J.J. Lynch (GSC)*

Preparation (1992):     Bondar-Clegg & Company  
                                 Ottawa, Ontario  
                                 *J.J. Lynch (GSC)*

Analysis (1992)        Becquerel Laboratories  
                                 Mississauga, Ontario  
                                 *J.J. Lynch (GSC)*

M. McCurdy edited open files and coordinated open file production.

H. Gross and S.W. Adcock provided computer processing support.

C.C. Durham, S.J. Day, and R.G. Balma provided technical assistance.

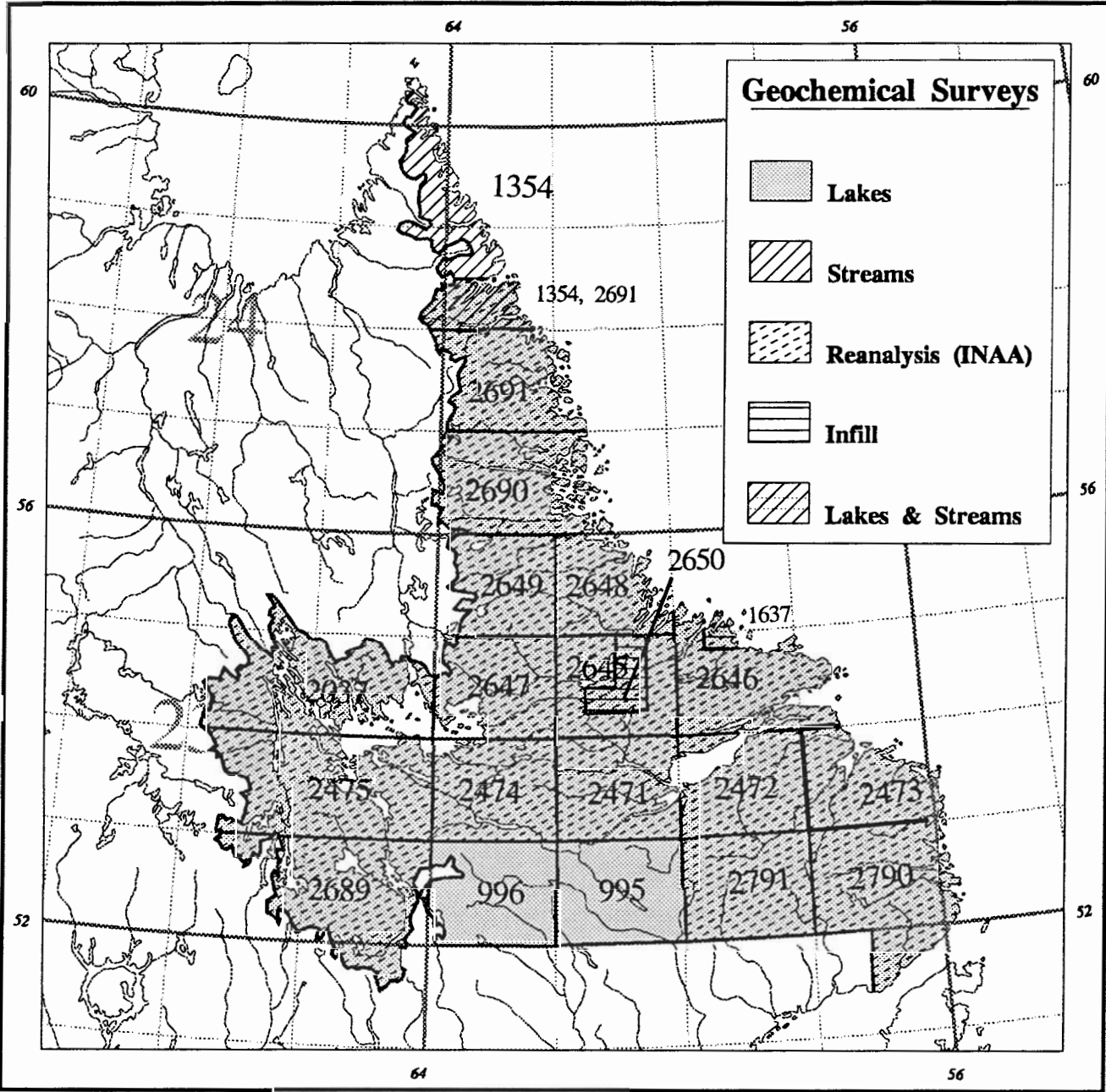
### DESCRIPTION OF SURVEY AND SAMPLE MANAGEMENT

Helicopter-supported sample collection was carried out during the summer of 1977. Lake sediment and water samples were collected at an average density of one sample per 15.8 km<sup>2</sup> throughout the 11 950 square kilometres covered by the survey.

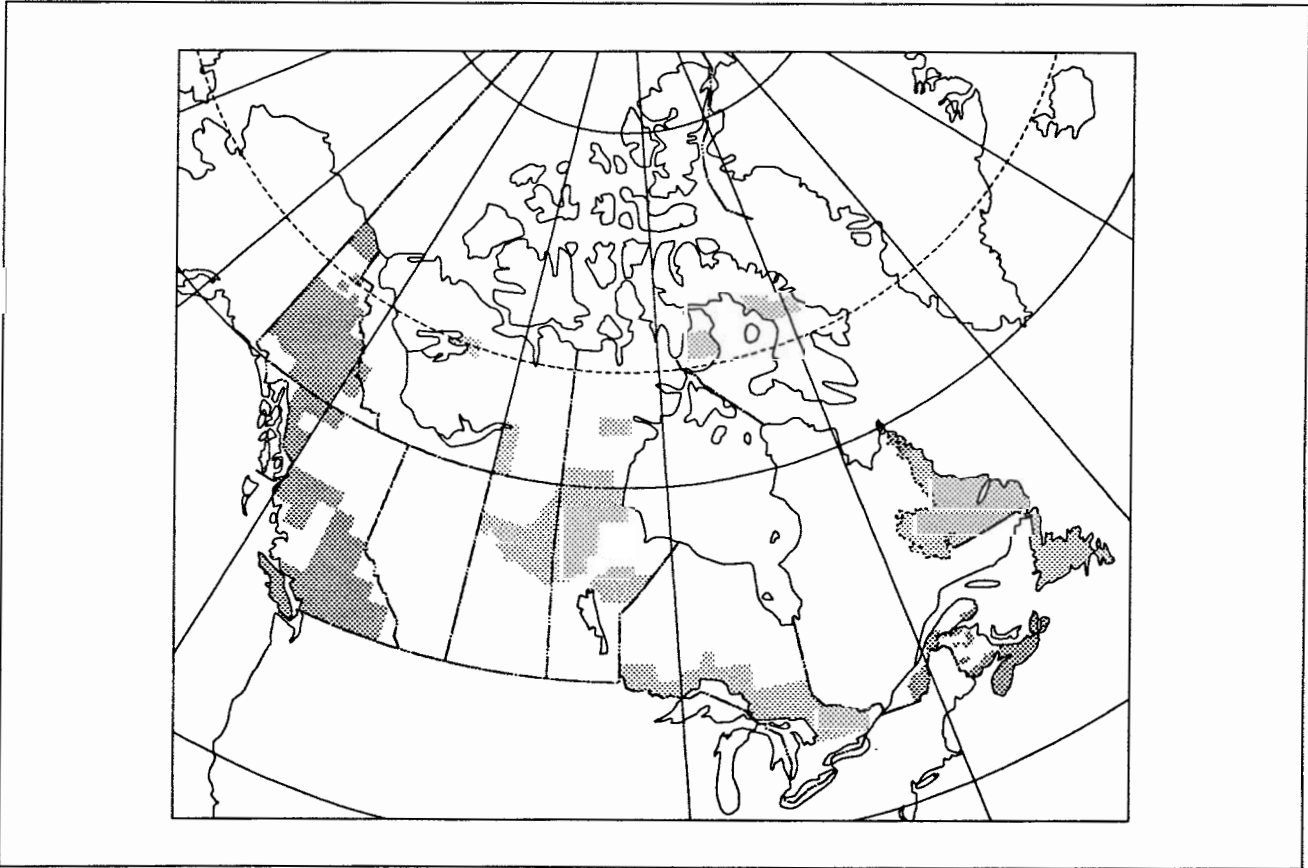
Sample site duplicate samples were routinely collected in each analytical block of twenty samples. Field observations were recorded on standard forms used by the Geological Survey of Canada (Garrett, 1974).

Site positions were marked on 1:250 000 scale NTS maps in the field and later digitized at the Geological Survey in Ottawa to obtain Universal Transverse Mercator (UTM) coordinates. The dominant rock types in the lake catchment basins were identified on appropriate geological maps used as the bedrock geological base on NGR maps.

In Ottawa, field dried samples were air-dried, crushed and ball-milled. The minus 80 mesh (177 micron) fraction was obtained and used for subsequent



**Fig. 1.** Areas of Labrador covered by geochemical surveys, showing current GSC open file numbers.



**Fig. 2.** Drainage surveys to National Geochemical Reconnaissance standards.

analyses. 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. Additional lake sediment material required for INAA analyses was taken from archive storage. Particle reduction was accomplished using a ceramic puck mill.

Analytical data from labs were monitored for reliability with standard methods used by the Applied Geochemistry Subdivision at the Geological Survey of Canada.

## ANALYTICAL PROCEDURES

### 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: Ag, As, Au, Ba, Br, Cd, Ce, Co, Cr, Cs, Eu, Fe, Hf, Ir, La, Lu, Mo, Na, Ni, Rb, Sb, Se, Sm, Sn, Ta, Tb, Te, Th, U, W, Yb, Zn, and Zr. The sample weights are also reported. Data for Ag, Cd, Ir, Se, Sn, Te, Zn, and Zr 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, and Fe, a 1 g sample was reacted with 6 ml of a mixture of 4M HNO<sub>3</sub> and M HCl in a test tube overnight at room temperature. After digestion, the test tube was immersed in a hot water bath at room temperature and brought up to 90 degrees C and held at this temperature for 2 hours with periodic shaking. The sample solution was then diluted to 20 ml with metal-free water and mixed. Zn, Cu, Pb, Ni, Co, Ag, Mn and Fe were determined by atomic absorption spectroscopy using an air-acetylene flame. Background corrections were made for Pb, Ni, Co, and Ag.

Arsenic was determined colorimetrically using silver diethyldithiocarbamate. Decomposition was accomplished by heating a 1 g sample with 20 ml of 6M HCl at 90° C for 1.5 hours. Colorimetric measurements were made at 520 nm.

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

were added and the sample solution diluted to 10 ml before aspiration.

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

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

Fluorine was determined as described by Ficklin (1970). A 250 mg sample was sintered with 1 gram of a flux consisting of two parts by weight sodium carbonate and one part by weight potassium nitrate. The residue was then leached with water. The sodium carbonate was neutralized with 10 ml 10% (w/v) citric acid and the resulting solution was 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 was measured using a fluoride ion electrode. Standard solutions contained sodium carbonate and citric acid in the same quantities as the sample solution.

Uranium was determined using a neutron activation method with delayed neutron counting. A detailed description of the method is provided by Boulanger *et al.* (1975). In brief, a 1 g sample was weighed into a 7 dram polyethylene vial, capped, and sealed. The irradiation was provided by a Slowpoke reactor with an operating flux of 10<sup>12</sup> neutrons/cm<sup>2</sup>/second. The samples were pneumatically transferred from an automatic loader to the reactor, where each sample was irradiated for 60 seconds. After irradiation, the sample was again transferred pneumatically to the counting facility where, after a 10 second delay, the sample was counted for 60 seconds with six BF<sub>3</sub> detector tubes imbedded in paraffin. Following counting, the samples were automatically ejected into a shielded storage container. Calibration was carried out twice a day at a minimum using natural materials of known uranium concentration.

### Water Analyses

Uranium in waters was determined by fission track analysis. The 225 ml of sample remaining following analysis for fluoride and pH was acidified with 3 ml concentrated HNO<sub>3</sub>. After a two week waiting period to ensure total dissolution of any precipitated uranium,



a 5 microlitre aliquot of the sample was removed, placed on a polycarbonate tape, and dried. The tape was irradiated in a nuclear reactor at McMaster University for one hour at a flux of  $10^{13}$  neutrons/cm<sup>2</sup>/second. The irradiated tape was etched with 25% NaOH solution and the fission tracks were counted with an optical counter fitted to a microscope. The number of tracks was proportional to the uranium concentration. Each tape contained its own calibration standards, blanks, and sample duplicates.

Hydrogen ion activity (pH) was measured with a Beckman combination electrode and a Model 401 Orion specific ion meter.

Fluoride in lake water samples was determined using an Orion fluoride electrode and a Model 401 Orion specific ion meter. Prior to measurement, an aliquot of the sample was mixed with an equal volume of a modified TISAB (total ionic strength adjustment buffer). The modification consisted of adding 60 ml of 8M KOH solution to the buffer. This permitted the reanalysis of fluoride in acidified water samples when required. When this analysis was necessary, acidified standard solutions were used for calibration.

Table 1 provides a summary of analytical data and methods.

## COMPARISON OF DATA PRODUCED BY TWO METHODS

The data listing in II-1 to II-63 allows users to make a comparison of data generated by two different analytical methods for a number of elements. Before attempting such a comparison some caution should be exercised.

1. The original data for Ni, Co, Mo, and Fe were obtained by AAS using a partial extraction (HNO<sub>3</sub> and HCl). Original data for As were obtained using a hot 6M HCl attack with a colorimetric finish (silver diethyldithiocarbamate. The data for these elements obtained on reanalysis are by INAA, which produces 'total' data. Hence, the original data will likely be somewhat lower than the INAA data.
2. The data for U were derived by 'total' methods, both originally and on reanalysis.
3. The sample preparation for the original analyses differed from the preparation employed for the reanalysis. Originally, a portion of the collected sample was prepared. Prior to reanalysis all of the remaining original sample was prepared and bottled. As a result, most of the original data were obtained from a different split of the unprepared sample than that which was used for reanalysis. Disagreement between original and reanalyzed data for some elements might be attributed to heterogeneity of the two different splits used for the two analyses.

## PRESENTATION AND INTERPRETATION OF GOLD DATA

The following general 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.

Samples that have gold values that are statistically above approximately the 90th percentile, or those with LOI values below 10%, are normally analyzed again in accordance with standard NGR procedures. **There will be no repeat data published in Open File 2473 however, as insufficient material remained after the initial neutron activation analyses.** The correct interpretation of gold geochemical 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. In organic-rich fluvial and lake sediments, gold distribution appears to be more homogeneous.
- 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, in areas where concentrations of gold in sediments are low, and/or grain sizes of the gold present relatively high, proportionally larger samples are required 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.

Table 1. Summary of Analytical Data and Methods

ELEMENT		DETECTION LEVEL		METHOD
<u>SEDIMENTS:</u>				
Ag	Silver	0.2	ppm	AAS
As	Arsenic	1	ppm	COL
As	Arsenic	0.5	ppm	INAA
Au	Gold	2	ppb	INAA
Wt	Sample Weight	0.01	g	-
Ba	Barium	50	ppm	INAA
Br	Bromine	0.5	ppm	INAA
Ce	Cerium	5	ppm	INAA
Co	Cobalt	2	ppm	AAS
Co	Cobalt	5	ppm	INAA
Cr	Chromium	20	ppm	INAA
Cs	Cesium	0.5	ppm	INAA
Cu	Copper	2	ppm	AAS
Eu	Europium	1	ppm	INAA
F	Fluorine	40	ppm	ISE
Fe	Iron	0.02	pct	AAS
Fe	Iron	0.2	pct	INAA
Hf	Hafnium	1	ppm	INAA
Hg	Mercury	10	ppb	AAS
La	Lanthanum	2	ppm	INAA
LOI	Loss-on-ignition	1	pct	GRAV
Lu	Lutetium	0.2	ppm	INAA
Mn	Manganese	5	ppm	AAS
Mo	Molybdenum	2	ppm	AAS
Mo	Molybdenum	1	ppm	INAA
Na	Sodium	0.02	pct	INAA
Ni	Nickel	2	ppm	AAS
Ni	Nickel	10	ppm	INAA
Pb	Lead	2	ppm	AAS
Rb	Rubidium	5	ppm	INAA
Sb	Antimony	0.1	ppm	INAA
Sc	Scandium	0.2	ppm	INAA
Sm	Samarium	0.1	ppm	INAA
Ta	Tantalum	0.5	ppm	INAA
Tb	Terbium	0.5	ppm	INAA
Th	Thorium	0.2	ppm	INAA
U	Uranium	0.2	ppm	INAA
U	Uranium	0.2	ppm	NADNC
W	Tungsten	1	ppm	INAA
Yb	Ytterbium	1	ppm	INAA
Zn	Zinc	2	ppm	AAS
<u>WATERS:</u>				
F-W	Fluoride	20	ppb	ISE
pH	Hydrogen ion activity	-	-	GCM
U-W	Uranium	0.01	ppb	FT

- AAS

COL

FT

GCM

GRAV

INAA

ISE

LIF

NADNC
- atomic absorption spectrometry

- colorimetry

- fission track analysis

- glass Calomel electrode and pH meter

- gravimetry

- Instrumental Neutron Activation Analysis

- ion selective electrode

- laser-induced fluorescence

- neutron activation, delayed neutron counting



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.

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 a analogous gold response due to natural variability may be lacking. Once an anomalous area has been identified, field investigations should be 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 be used to improve sampling methods and interpretation.

## FIELD DATA LEGEND

Table 2 describes the field and map information appearing on the following pages preceding the analytical data for each sample site.

## REFERENCES

- Boulanger, A., Evans, D.J.R. and Raby, B.F. (1975)** Uranium analysis by neutron activation delayed neutron counting; Proceedings of the 7th Annual Symposium of Canadian Mineral Analysts, Thunder Bay, Ontario, September 22-23, 1975.
- Clifton, H.E., Hunter, R.E., Swanson, F.J., and Phillips, R.L. (1969)** Sample size and meaningful gold analysis; U.S. Geological Survey Professional Paper 625-C.
- Friske, P.W.B. and Hornbrook, E.H.W. (1991)** Canada's National Geochemical Reconnaissance Program; in Transactions of the Institution of Mining and Metallurgy, Section B; Volume 100, p. 47-56.
- Ficklin, W.H. (1970)** A rapid method for the determination of fluoride in rocks and soils, using an ion selective electrode; U.S. Geol. Surv. Paper 700C, pp. 186-188.
- Garrett, R.G. (1974)** Field data acquisition methods for applied geochemical surveys at the Geological Survey of Canada; Geol. Surv. Can. Paper 74-52.
- Harris, J.F. (1982)** Sampling and analytical requirements for effective use of geochemistry in exploration for gold; in Levinson, A.A., Editor, Precious Metals in the Northern Cordillera, proceedings of a symposium sponsored by the Association of Exploration Geochemists and the Cordilleran Section of the Geological Association of Canada, pp. 53-67.
- Jonasson, I.R., Lynch, J.J. and Trip, L.J. (1973)** Field and laboratory methods used by the Geological Survey of Canada in geochemical surveys; No. 12, Mercury in Ores, Rocks, Soils, Sediments and Water, Geol. Surv. Can. Paper 73-21.

TABLE 2. Field Observations Legend

FIELD RECORD	DEFINITION	TEXT CODE
MAP SHEET	National Topographic System (NTS): lettered quadrangle (1:250 000 or 1:50 000 scale) Part of sample number	3E, 13H
SAMPLE ID	Remainder of sample number: Year ..... Field crew ..... Sample sequence number .....	77 1 or 3 001-999
REP STAT	Replicate status; the relationship of the sample to others within the analytical block of 20: Routine regional sample ..... First of field duplicate ..... Second of field duplicate .....	00 10 20
UTM	Universal Transverse Mercator (UTM) Coordinate System; digitized sample location coordinates.	
ZN	Zone (7 to 22)	
EASTING	UTM Easting in metres	
NORTHING	UTM Northing in metres	
ROCK UNIT	Major rock type of catchment area: garnet gneiss ..... granite gneiss ..... amphibolite ..... granodiorite ..... anorthosite ..... ultramafic .....	GRGS GRNG AMPB GRDR ANRS UMFC
LAKE AREA	The area of the water body sampled: Pond ..... 1/4 to 1 square kilometre ..... 1 to 5 square kilometres ..... greater than 5 square kilometres .....	pond .25-1 1-5 >5
LAKE DEPTH	Distance in metres from the surface of the lake to the bottom .....	0 - 99
TERRAIN RELIEF	Relief of lake catchment basin: Low ..... Medium ..... High .....	Low Med Hi
SAMPLE CONT.	Contamination; human or natural: None ..... Work ..... Camp ..... Fuel ..... Gossan .....	- Wo Ca Fu Go
SAMPLE COLOUR	Sediment sample colour; up to two colours may be selected: Tan ..... Yellow ..... Green ..... Grey ..... Brown ..... Black .....	Tan Yellow Green Grey Brown Black

FIELD RECORD	DEFINITION	TEXT CODE
SUSP MATL	Suspended matter in water: None ..... Heavy ..... Light .....	- Heavy Light
Miscellaneous	Refers to missing data in any field ..... no sample material for analysis ..... parts per million ..... parts per billion ..... percent ..... weight (of sample) ..... gram .....	* ns ppm ppb pct Wt g