GEOLOGICAL SURVEY OF CANADA OPEN FILE 2472 (NTS 13G) CANADA-NEWFOUNDLAND COOPERATION AGREEMENT ON MINERAL DEVELOPMENT (1990-1994)

REGIONAL LAKE SEDIMENT AND WATER GEOCHEMICAL RECONNAISSANCE DATA EASTERN LABRADOR



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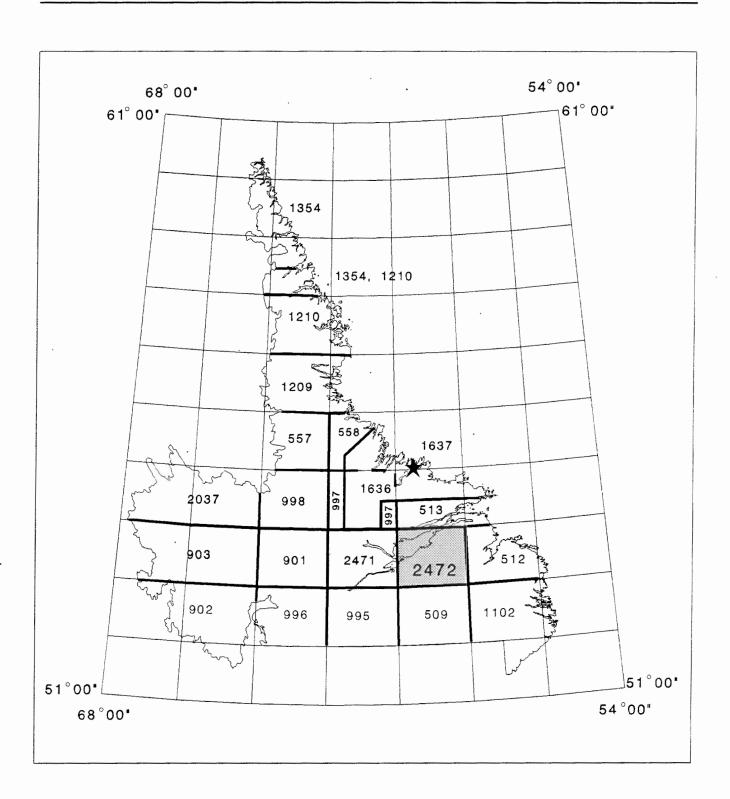
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NATIONAL GEOCHEMICAL RECONNAISSANCE LAKE SEDIMENT AND WATER GEOCHEMICAL DATA NEWFOUNDLAND 1992 GEOLOGICAL SURVEY OF CANADA OPEN FILE 2472 NTS 13G



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

Open File 2472 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 2471 REGIONAL LAKE SEDIMENT AND WATER GEOCHEMICAL DATA, LABRADOR NTS 13G

INTRODUCTION

Open File 2472 contains data for gold and 25 other elements obtained by re-analyzing lake sediments collected in 1977 from 832 sites in eastern Labrador. Original analytical data selected from Open File 511 (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 surveys were 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. Fisheries and Environment Canada provided funds for the determination of mercury. Analyses of archive samples for Open File 2472 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 have been published, covering all of Labrador. Areas surveyed, with associated open file numbers, are shown in Fig. 1. Fig. 2 shows cross-Canada coverage. Data from all open files are available on 3.5 or 5.25 inch diskettes and in the original published form.

CREDITS

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

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

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. Maurice (GSC)

Preparation (1977): -Golder Associates

Ottawa, Ontario

-J.J. Lynch (GSC)

Analysis (1977): -Chemex Labs

Vancouver, British Columbia

-Barringer Research Toronto, Ontario -Atomic Energy of Canada

Ottawa, Ontario

-J.J. Lynch (GSC)

Preparation (1992): -Bondar-Clegg & Company

Ottawa, Ontario

-J.J. Lynch (GSC)

Analysis (1992): -Bondar-Clegg and Company

Ottawa, 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, H. Karam, S.J. Day, and S. Carberry 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 15.2 km² throughout the 12 665 square kilometres of the central Labrador 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 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.

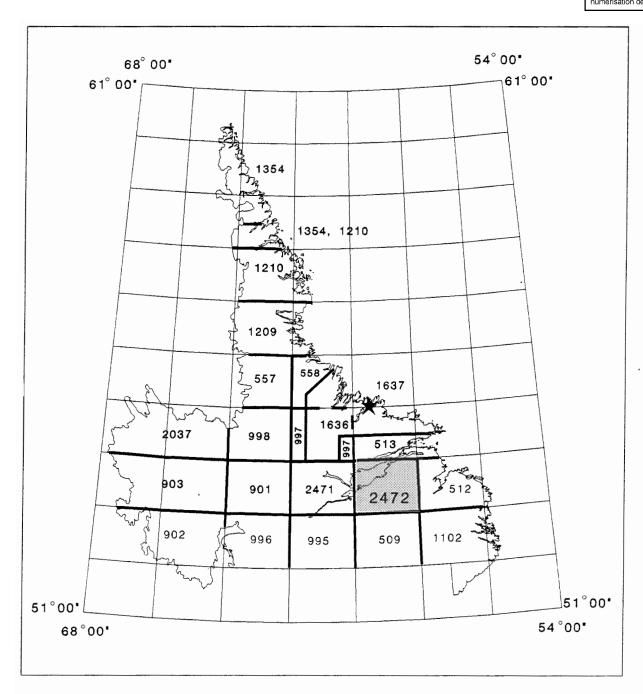


Fig. 1. Areas of Labrador covered by geochemical surveys, showing current GSC open file numbers.

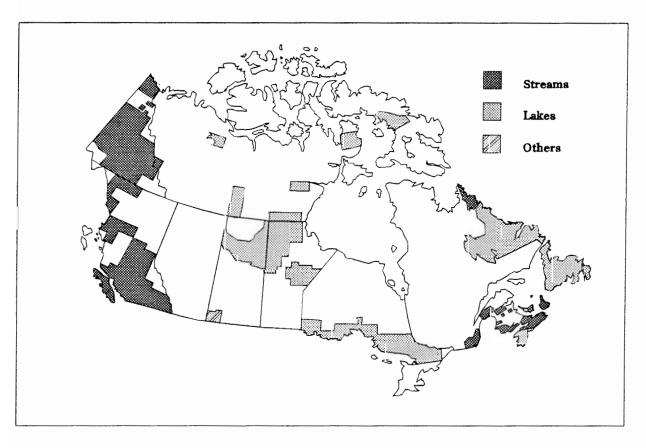


Fig. 2 Drainage surveys to National Geochemical Reconnaissance standards.

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 twomegawatt 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, Sc, 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 HNO₃ 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° 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 $\rm HNO_3$ at 90° C for 30 minutes. At this point 0.5 ml concentrated HCl was added and the digestion continued at 90° 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 $_3$ 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° 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 $_4$ in M H $_2$ SO $_4$. 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° 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 original method is provided by Boulanger et al. (1975). In brief, a 1 gram sample was weighed in a 7 dram polyethylene vial, capped and sealed. The irradiation was provided by the Slowpoke 10¹² with an operating flux of reactor neutrons/cm²/second. The samples were pneumatically transferred from an automatic loader to the reactor, where each sample was irradiated for 60 seconds. After irradiation, the samples were again transferred pneumatically to the counting facility where, after a 10 second delay, the sample was counted for 60 seconds with six BF3 detector tubes embedded in paraffin. Following counting, the samples were automatically ejected into a shielded storage container. Calibration was carried out twice a day as a minimum, using natural materials of known uranium concentration.

Water Analyses

Uranium was determined by fission track analysis. 225 ml of water was acidified with 3 ml concentrated HNO₃. 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 1 hour at a flux of 10¹³ neutrons/sq cm/sec. 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 before acidification of the sample for the uranium determination.

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) solution. The modification consisted of adding 60 ml of 8M KOH solution th the buffer. This permitted the reanalysis of fluoride in acidified water samples when required. When this analysis was required, acidified standard solutions were used for calibration.

Table 1 provides a summary of analytical data and methods.

TABLE 1. Summary of Analytical Data and Methods

ELEMENT		DETE	ECTION L	METHOD
SEDII	MENTS:			
Zn	Zinc	2	ppm	AAS
Cu	Copper	2	ppm	AAS
Pb	Lead	2	ppm	AAS
Ni	Nickel	2 2 2 2	ppm	AAS
Co	Cobalt	2	ppm	AAS
Ag	Silver	0.2	ppm	AAS
Mn	Manganese	5	ppm	AAS
As	Arsenic	1	ppm	COL
Мо	Molybdenum	2	ppm	AAS
Fe	iron	0.02	pct	AAS
Hg	Mercury	10	ppb	CV-AAS
U	Uranium	0.2	ppm	NADNC
LOI	Loss-on-ignition	1	pct	GRAV
F	Fluorine	40	ppm	ISE
As	Arsenic	0.5	ppm	INAA
Au	Gold	2	ppb	INAA
Ва	Barium	50	ppm	INAA
Br	Bromine	0.5	ppm	INAA
Ce	Cerium	5	ppm	INAA
Co	Cobalt	5 .	ppm	INAA
Cr	Chromium	20	ppm	INAA
Cs	Cesium	0.5	ppm	INAA
Eu	Europium	1	ppm	INAA
Fe	Iron	0.2	pct	INAA
Hf	Hafnium	1	ppm	INAA
La	Lanthanum	2	ppm	INAA
Lu	Lutetium	0.2	ppm	INAA
Мо	Molybdenum	1	ppm	INAA
Na	Sodium	0.02	pct	INAA
Ni	Nickel	10	ppm	INAA
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
W	Tungsten	1	ppm	INAA
Yb	Ytterbium	0.01	ppm	-
1	Wt Weight		gm	INAA
	WATERS:		m m le	loc
F-W	Fluoride	20	ppb	ISE
рН	Hydrogen ion			GCM
U-W	activity Uranium	0.01	ppb	GCM FT

AAS - atomic absorption spectrometry

FT - fission track analysis

GCM glass Calomel electrode and pH meter
 gravimetry

GRAV

- cold vapour atomic absorption spectrometry CV-AAS INAA - Instrumental Neutron Activation Analysis

ISE - ion selective electrode

NADNC - neutron activation, delayed neutron counting

COL - colorimetrically

COMPARISON OF DATA PRODUCED BY TWO METHODS

The data listing in II-1 to II-69 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.

- The original data for Ni, Co, As, Mo, and Fe were obtained by AAS using a <u>partial extraction</u> (HNO₃ and HCl). The data for these elements obtained on re-analysis are by INAA, which produces 'total' data. Hence, the original data will likely be somewhat lower than the INAA data.
- The data for U and Ba (the latter in stream sediments only) were derived by 'total' methods both originally and on re-analysis.
- 3. The sample preparation for the original analyses differed from the preparation employed for the re-analysis. Originally, a portion of the collected sample was prepared. Prior to re-analysis 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 re-analysis. Disagreement between original and re-analyzed data for some elements might be attributed to heterogeneity of the two different splits used for the two analysis.

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 2472 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:

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, 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.

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 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.

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FIELD DATA LEGEND

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

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 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.
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- 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.
- Friske, P.W.B. and Hornbrook, E.H.W. (1991) Canada's National Geochemical Reconnaissance programme; in Transactions of the Institution of Mining and Metallurgy, Section B; Volume 100, p. 47-56
- **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	13G
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	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: Arkose = arkosic arenite	ARKS GRGS GRNG AMPB GRDR QZMZ ANRS UMFC
LAKE AREA	The area of the water body sampled: Pond	pond .25-1 1-5 >5
LAKE DEPTH	Distance in feet from the surface of the lake to the bottom	0 - 99
TERRAIN RELIEF	Relief of lake catchment basin: Low	Lo 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	Tn Yl Gn Gy Br Bk

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FIELD RECORD	DEFINITION	TEXT CODE
SUSP MATL	Suspended matter in water: None	- Hvy Lgt
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 gm