



**GEOLOGICAL SURVEY OF CANADA
OPEN FILE 7232**

**Regional Lake Sediment Geochemical Data, Nonacho Basin –
East Arm of Great Slave Lake Region, Northwest Territories
(NTS 75-C and 75-F)**

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Regional Lake Sediment Geochemical Data, Nonacho Basin – East Arm of Great Slave Lake Region, Northwest Territories (NTS 75-C and 75-F)

Introduction

New analytical data for 60 elements from the reanalysis of lake sediment samples collected in 1975 from 2,000 sites in the Nonacho Basin – East Arm of Great Slave Lake area, Northwest Territories are presented in this Geological Survey of Canada (GSC) open file release. Field observations and analytical data originally reported in GSC Open Files 324 and 325 (1976) are included with this report.

The area outlined in Figure 1 was sampled in 1975 under a Federal Uranium Reconnaissance Program. The Uranium Reconnaissance Program was designed to provide industry with high quality reconnaissance exploration data and to provide the Federal Government with nationally systematic data for undertaking uranium resource appraisals. Funds for the reanalysis of archived samples were made available under the Geo-Mapping for Energy and Minerals (GEM) Program at Natural Resources Canada (NRCan).

The GEM Program is a 5-year investment by the Government of Canada in geoscience information to better define the potential for new energy and mineral resources in Canada. GEM is delivered at the federal level by the Geological Survey of Canada and the Polar Continental Shelf Project (PCSP), Earth Sciences Sector (ESS), Natural Resources Canada (NRCan). The major focus is on large areas of Canada's North where insufficient public geoscience information exists to attract and guide effective private sector investment.

In the final phase of GEM, a project called Operation GEM was initiated to improve geoscience knowledge in the least understood parts of Canada's north. In the spirit of the Intergovernmental Geoscience Accord, co-planning was conducted with our territorial counterparts to identify areas which are perceived to have high potential based on reconnaissance information and current deposit models. In light of the consultation process, geographic areas were prioritized for investigation from the Minerals perspective, and topics were selected for Energy research.

The South Rae Domain Project Area was part of the Operation GEM Project. The goal of this multi-disciplinary activity was to determine if major metal-bearing faults and rock types of northern Saskatchewan extend into southern Northwest Territories in order to stimulate exploration activity for nickel-copper Platinum Group elements (PGEs), rare earth elements (REEs) and uranium. Reconnaissance bedrock mapping and compilation of existing maps combined with integration of data from newly acquired geophysical and geochemical surveys and remote predictive mapping were applied to provide knowledge of an area almost devoid of previous mineral exploration.

Reanalysis of existing samples provides data for additional elements and takes advantage of lower detection limits for many elements, at approximately 5% of the cost of collecting new samples. Analytical results and field observations from this project form part of a national geochemical database used 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.

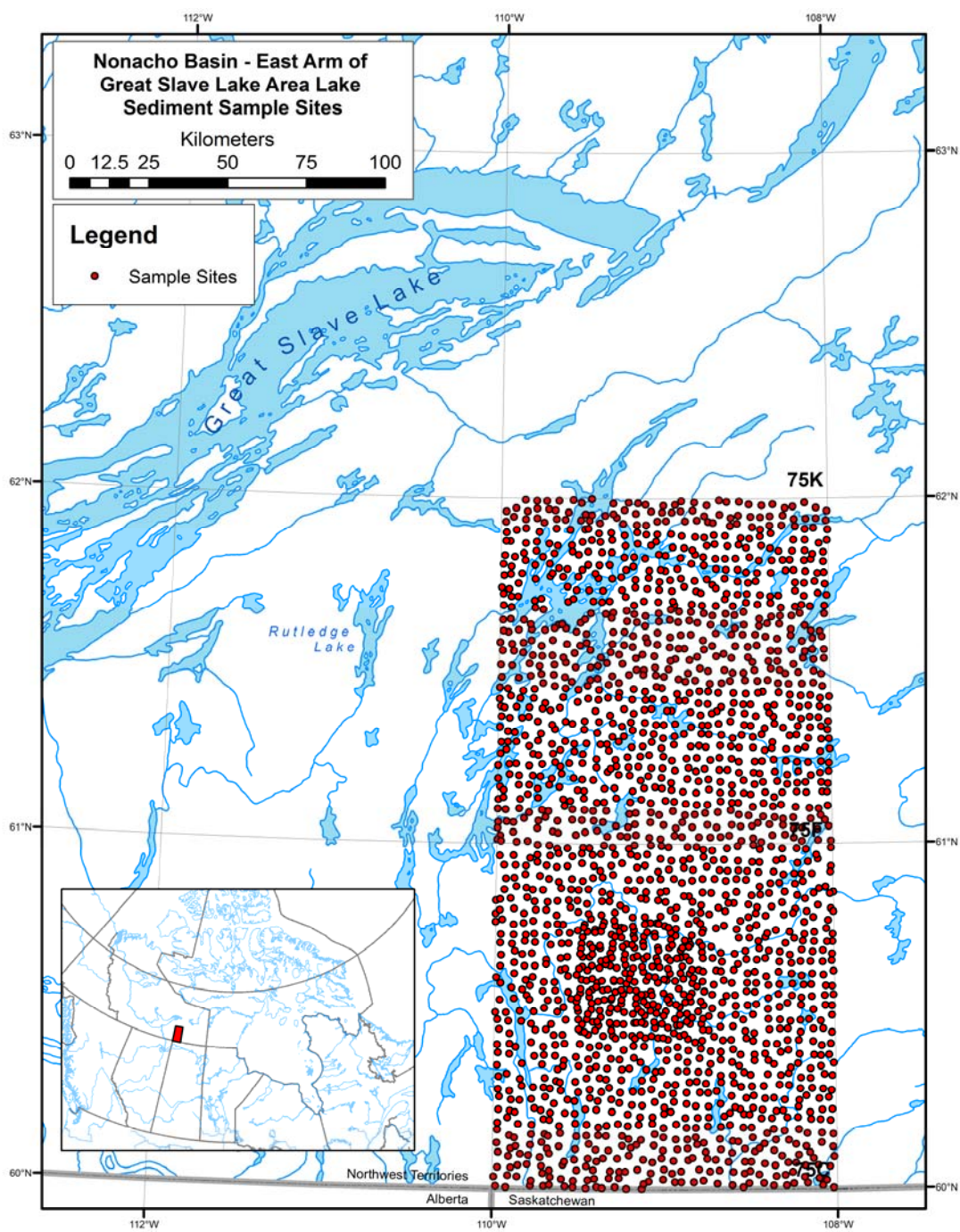


Figure 1 Map showing locations of lake sediment samples collected in the Nonacho Basin – East Arm of Great Slave Lake area.

Methods

Description of Survey and Sample Management

The original lake sediment samples were collected during the summer of 1975. Data for 12 elements in sediments were released in 1976 in two GSC open files (324 and 325). The 2,000 sample sites from which sediments were reanalyzed for this report, shown in Figure 1, are distributed throughout the 24,117 km² area at an average density of one sample per 12.1 km².

A bottom-valved, hollow-pipe sampler was used to collect approximately one kilogram of wet lake sediment. Field observations for each site were recorded on standard forms used by the GSC (Garrett, 1974; see Figure 2). At GSC laboratories in Ottawa, field-dried samples were air-dried and sieved through a minus 80 mesh (177 µm) screen before milling in ceramic-lined puck mills. Typically, one kilogram of the organic gel, the preferred collection material, yielded about 50 g of material for analysis. For quality control purposes, the original samples were arranged in groups (consecutively-numbered blocks) of twenty. Each group of twenty contained field duplicate samples; that is, two samples from a single site. The group also contained an analytical duplicate sample pair (a single site sample split and assigned two non-consecutive sample numbers). Finally, each set included a control reference sample. Before publication, thorough inspections of the field and analytical data were made to check for any missing information and/or analytical errors. A more detailed description of collection and quality control methods used by the GSC for lake sediment samples can be found in Cook and McConnell (2001). Samples selected for reanalysis were retrieved from the GSC archive facility in Ottawa and shipped to commercial laboratories for reanalysis. Within these reanalysis suites, the above-described pattern of distribution of quality control samples was maintained, with the exception that new control reference standards replaced the original ones inserted in 1975.

The figure shows a detailed field card form. At the top, there are fields for PROJECT NO., AREA, PHOTO, COLLECTOR, and DATE. Below these is a large table with columns for various parameters. The first row of the table is labeled 'RELIEF' and includes sub-columns for LOW, MED, and HIGH. The second row is labeled 'COMPOSITION' and includes sub-columns for SAND, FINE, and ORG. The third row is labeled 'CONTAMINATION' and includes sub-columns for WORK, CAMP, FUEL, and GOIN. The fourth row is labeled 'COLOURS' and includes sub-columns for YEL, GRN, GRY, BRN, BLK, and HVY. The fifth row is labeled 'SUSP.' and includes sub-columns for LIGHT and DARK. The sixth row is labeled 'ELEMENTS' and includes sub-columns for 1 through 80. The card is titled 'GEOCHEMICAL LAKE SEDIMENT SAMPLE CARD (REV.74)' and 'GEOLOGICAL SURVEY OF CANADA'. The card is labeled 'LAKE SEDIMENTS' on the right side.

Figure 2 Field card used to capture site-specific field observation data in 1975.

Quality assurance/quality control measures for the reanalysis of the data by inductively coupled plasma emission spectroscopy/mass spectrometry (ICP-ES/MS) and instrumental neutron activation analysis (INAA) are described in a later section.

Analytical Procedures (2012)

Inductively Coupled Plasma Emission Spectroscopy/Mass Spectrometry (ICP-ES/MS)

For the determination of 53 elements listed in Table 1, a one-gram sample was leached with 6 ml of a mixture of HCl, HNO₃, and distilled, deionized water (1:1:1 volume to volume ratio) 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 spectrometry on a Perkin-Elmer Elan instrument. Analyses were carried out at Acme Analytical Laboratories, Limited, Vancouver, British Columbia.

Table 1 Elements determined by ICP-ES/MS reanalysis of archived lake sediment samples

Element	Detection Limit	Units of Measurement	Analytical Method	Element	Detection Limit	Units of Measurement	Analytical Method
Ag	2	ppb ¹	ICP-MS	Na	0.001	pct	ICP-MS
Al	0.01	pct ²	ICP-MS	Nb	0.02	ppm	ICP-MS
As	0.1	ppm ³	ICP-MS	Ni	0.1	ppm	ICP-MS
Au	0.2	ppb	ICP-MS	P	0.001	pct	ICP-MS
B	20	ppm	ICP-MS	Pb	0.01	ppm	ICP-MS
Ba	0.5	ppm	ICP-MS	Pd	10	ppb	ICP-MS
Be	0.1	ppm	ICP-MS	Pt	2	ppb	ICP-MS
Bi	0.02	ppm	ICP-MS	Rb	0.1	ppm	ICP-MS
Ca	0.01	pct	ICP-ES	Re	1	ppb	ICP-MS
Cd	0.01	ppm	ICP-MS	S	0.01	pct	ICP-MS
Ce	0.1	ppm	ICP-MS	Sb	0.02	ppm	ICP-MS
Co	0.1	ppm	ICP-MS	Sc	0.1	ppm	ICP-MS
Cr	0.5	ppm	ICP-MS	Se	0.1	ppm	ICP-MS
Cs	0.02	ppm	ICP-MS	Sn	0.1	ppm	ICP-MS
Cu	0.01	ppm	ICP-MS	Sr	0.5	ppm	ICP-MS
Fe	0.01	pct	ICP-ES	Ta	0.05	ppm	ICP-MS
Ga	0.2	ppm	ICP-MS	Te	0.02	ppm	ICP-MS
Ge	0.1	ppm	ICP-MS	Th	0.1	ppm	ICP-MS
Hf	0.02	ppm	ICP-MS	Ti	0.001	pct	ICP-MS
Hg	5	ppb	ICP-MS	Tl	0.02	ppm	ICP-MS
In	0.02	ppm	ICP-MS	U	0.1	ppm	ICP-MS
K	0.01	pct	ICP-ES	V	2	ppm	ICP-MS
La	0.5	ppm	ICP-MS	W	0.1	ppm	ICP-MS
Li	0.1	ppm	ICP-MS	Y	0.01	ppm	ICP-MS
Mg	0.01	pct	ICP-ES	Zn	0.1	ppm	ICP-MS
Mn	1	ppm	ICP-ES	Zr	0.1	ppm	ICP-MS
Mo	0.01	ppm	ICP-MS				

- ¹ parts per billion, µg/kg
- ² percent
- ³ parts per million, mg/kg

Instrumental Neutron Activation Analysis (INAA)

Weighed and encapsulated samples were packaged for irradiation along with internal standards and international reference materials. Samples and standards were irradiated together with neutron flux monitors in a two-megawatt pool-type reactor. After a seven day decay period, samples were measured for the elements shown in Table 2 on a high resolution germanium detector. Typical counting times were 500 seconds. The sample weights are reported in **Appendix 1 GSC OF 7232 DATA.xls (Worksheet 'Reanalysis INAA Data')**. Analyses were carried out at Becquerel Labs, Mississauga, Ontario.

Table 2 Variables determined by INA reanalysis of archived lake sediment samples

Variable	Detection Limit	Units of Measurement	Variable	Detection Limit	Units of Measurement
As	0.5	ppm ¹	Ni	10	ppm
Au	2	ppb ²	Rb	5	ppm
Ba	40	ppm	Sb	0.1	ppm
Br	0.5	ppm	Sc	0.2	ppm
Cd	5	ppm	Sm	0.1	ppm
Ce	5	ppm	Sn	100	ppm
Co	5	ppm	Ta	0.5	ppm
Cr	20	ppm	Tb	0.5	ppm
Cs	0.5	ppm	Te	10	ppm
Eu	1	ppm	Th	0.2	ppm
Fe	0.2	pct ³	Ti	500	ppm
Hf	1	ppm	U	0.2	ppm
Ir	50	ppb	W	1	ppm
La	2	ppm	Wt	0.1	g ⁴
Lu	0.2	ppm	Yb	2	ppm
Mo	1	ppm	Zn	100	ppm
Na	0.02	pct	Zr	200	ppm

¹ parts per million, mg/kg

² parts per billion, µg/kg

³ percent

⁴ grams

Analytical Procedures (1975)

Atomic Absorption Spectroscopy (AAS) and Other Analyses

For the determination of Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe and Cd, a 1 g sample was reacted with 3 ml concentrated HNO₃ in a test tube overnight at room temperature. After digestion, the test tube was immersed in a hot water bath at room temperature and heated to 90° C and held at this temperature for 30 minutes with periodic shaking. One ml of concentrated HCl was added and heating continued for another 90 minutes. The sample solution was then diluted to 20 ml with metal-free water and mixed. Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe and Cd were determined by AAS using an air-acetylene flame. Background corrections were made for Pb, Ni, Co, Ag and Cd.

Molybdenum was determined by AAS using a nitrous oxide acetylene flame. A 0.5 g sample was reacted with 1.5 ml concentrated HNO₃ 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 30 minutes with periodic shaking. 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 g sample was reacted with 20 ml concentrated HNO₃ 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% weight per volume (w/v) SnSO₄ in 1 M H₂SO₄. 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.

Arsenic was determined by a colorimetric method using silver diethyldithiocarbamate. Sample material was digested by heating a 1 g sample with 20 ml of 6M HCl at 90°C for 1.5 hours. Arsenic in the reaction solution was converted to arsine, which was evolved and then complexed with silver diethyldithiocarbamate. The intensity of the colour of the complex was determined with a spectrophotometer. Colorimetric measurements were made at 520 nm.

Analyses for LOI and the 12 elements described above and listed in Table 3 were carried out at Chemex Laboratories, Vancouver, British Columbia.

Uranium was determined using a neutron activation method with delayed neutron counting. A detailed description of this 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 samples were pneumatically transferred from an automatic loader to a 'Slowpoke' reactor, where each sample was irradiated for 60 seconds in an operating flux of 10¹² neutrons/cm²/sec. After irradiation, the samples were transferred to a counting facility where, after a ten second delay, each sample was counted for 60 seconds with six BF₃ detector tubes embedded in paraffin wax. Following counting, samples were ejected into a shielded storage container. Analysis of uranium in lake sediments was carried out at Atomic Energy of Canada, Limited (AECL), Ottawa, Ontario.

Table 3 Summary of original elements determined and methods used in 1975

Element	Detection Limit	Units of Measurement	Analytical Method
Ag	0.2	ppm	AAS ¹
As	1	ppm	COL ²
Co	2	ppm	AAS
Cu	2	ppm	AAS
Fe	0.02	pct	AAS
Hg	10	ppb	CV-AAS ³
LOI	1.0	pct	GRAV ⁴
Mn	5	ppm	AAS
Mo	2	ppm	AAS
Ni	2	ppm	AAS
Pb	2	ppm	AAS
U	0.5	ppm	NADNC ⁵
Zn	2	ppm	AAS

- 1 Atomic Absorption Spectroscopy
- 2 Colorimetric
- 3 Cold vapour Atomic Absorption Spectroscopy
- 4 Gravimetric methods
- 5 Neutron activation – delayed neutron counting

Analytical results are presented in Appendix 1 in an Excel® spreadsheet file included with this report: **Appendix 1 GSC OF 7232 DATA.xls**. There are four worksheets in this file:

<u>Worksheet</u>	<u>Contents</u>
Field Data	Site-specific field observations including geographic coordinates
Original 1975 Data*	AAS and specific methods analytical data for silt and water samples
Reanalysis ICP Data*	ICP-MS/ES analytical data for silt samples
Reanalysis INAA Data*	INAA analytical data for silt samples

* ‘NA’ in data field indicates data “Not Available” because of insufficient sample material or missing sample.

QUALITY CONTROL FOR GEOCHEMICAL RESULTS

Reliability (Trueness, Accuracy and Precision) of analytical data returned from commercial laboratories was determined by incorporating field duplicates (FD pairs) within the sampling protocol, and including analytical duplicates (AD), standard reference materials (SRM), and control reference materials (CRM) samples within the sample suite submitted to the labs. Table 4 provides information on the number of each quality control sample within the sample suite. Analytical data for control reference standards, analytical and field duplicates, and blanks are included with this report in **Appendix 2 GSC OF 7232 QUALITY CONTROL.xls**.

Nonacho Basin –East Arm of Great Slave Lake (75-C, 75-F)			
FD Pairs	AD Pairs	SRM	CRM
165	170	55	110

Table 4 Number of quality control samples and sample pairs included with reanalyzed lake sediment samples. Quality control data (reference standards, field and analytical duplicates) from a survey of NTS map sheet 75-K are included with quality control data for this open file.

Data quality was evaluated in a four-step process using standard reference materials to evaluate trueness, control reference materials to evaluate accuracy, analytical duplicate samples to evaluate analytical precision, and field duplicates to carry out Analysis of Variance (ANOVA) in order to assess fitness-of-purpose (‘Are differences between sample sites real?’) for mapping.

Tables 1 through 6 (Worksheets ‘Trueness’, ‘Accuracy’, ‘AD Precision’, ‘ANOVA (FD)’) in **Appendix 2 GSC OF 7232 QUALITY CONTROL.xls** can be used to estimate the quality of analysis for almost every element found in Tables 1 and 2 of this document. Elements are grouped based on their position in the Periodic Table.

‘Trueness’

‘Trueness’ of analytical data was evaluated by inserting Canadian Certified Reference Lake Sediments LKSD-1 and LKSD-4 at random locations throughout the analytical suite. These two standards were incorporated into Nonacho Basin – East Arm of Great Slave Lake area samples. LKSD-1 is a combination of lake sediments from two lakes located in central Ontario (Brady Lake, 31M and Joe Lake, 31F). Sediment from three lakes, Big Gull Lake (31C) in Ontario and Key Lake and Seahorse Lake (74H) in Saskatchewan, were combined to make up LKSD-4 (Lynch, 1990).

In Tables 1 and 2 of Appendix 2 (‘Trueness’), means and standard deviations (MEAN \pm SD) for control reference standards LKSD-1 and LKSD-4, for which provisional values have been published by Lynch (1990; 1999) are shown. Lower detection limits (LDL), standard deviation (SD) and Relative Standard Deviation (RSD) for elements determined by ‘partial’ and total methods from repeated analyses of reference standards LKSD-1 and LKSD-4 are listed. Relative Standard Deviation (RSD), expressed as a percentage, facilitates comparison of the repeatability of elements measured in different units and varying means (Reimann *et al.*, 2008). RSD is independent of both the magnitude of the data and the units. Accepted values in square brackets are derived from unpublished data ($n \geq 40$) collected from recent projects at the GSC.

For LKSD-1 and LKSD-4, and for elements for which an accepted mean exists, almost all are within one Standard Deviation of an accepted mean. Elements with possible analytical problems, as indicated by a relatively high (>33%) Relative Standard Deviation (RSD), are shown in bold type. However, a relatively high RSD, suggesting poor repeatability, may also be an indication that analytical results are close to the detection limit for that element.

Accuracy

The accuracy of analytical results received from commercial laboratories, in the sense of an absence of bias, or ‘drift’ over time, was monitored by inserting one of two control reference materials at random locations in each block of twenty samples. Stream sediments were collected from Bonanza and Hunker Creeks near Dawson City, YT, and then dried, sieved and homogenized for use as internal control reference standards at the GSC. Results for each element are shown in Tables 3 and 4 of Appendix 2 (‘Accuracy’).

Analytical data for internal standards can be used to create ‘x-charts’, by plotting the ‘Sequence’ (first column) against the elemental value. Trends caused by instrumental drift or obvious deviations can usually be detected in these diagrams (Reimann *et al.*, 2008).

Precision

Precision is considered in terms of the closeness of agreement between analytical duplicate samples analyzed by the same method, i.e. independent test results obtained using the same equipment within short intervals of time on duplicate project samples. In order to provide an estimate of precision for each element, the squared difference between two analytical duplicates was calculated for $N = 170$ duplicate pairs. The sum of these values was divided by the number of samples ($(2*N) = 340$) to estimate a measure of variability (variance). Standard Deviation was then obtained by calculating the square root of this variance (Garrett, 1969). The resulting numerical estimates of precision are shown in Table 5 in Appendix 2 (‘AD Precision’) represented by the Relative Standard Deviation, where the Standard Deviation is divided by the overall mean of the samples and multiplied by 100 to obtain a percentage (Reimann *et al.*, 2008). Elements (or analytes) are grouped based on their position in the Periodic Table. Included with the element or analyte and method of analysis are the Lower Detection Limit (LDL), the percentage of data below the Lower Detection Limit (% Below LDL), the Range and the Mean. This information provides context for the estimate of Precision in the last column of Table 5.

Elements with precisions poorer than 15% in Table 5, Appendix 2, tend towards generally low concentrations in samples, as indicated by the Range, the Mean and the percentage of data below the detection limit. Such is the case for elements such as Pd, Hf, Ta, W, Re, Pt, In, Ge, Sb and Te by partial

methods, and Be, Pb and Sn by total methods. Results for Au by a partial method are affected by the particulate nature of gold ('nugget effect') and should be considered accordingly.

Analysis of Variance (ANOVA)

Precision and accuracy are 'external' criteria against which geochemical survey data are evaluated. In order to establish that these data are 'fit for purpose,' an Analysis of Variance (ANOVA) is required. Results from two types of ANOVA are shown in this report. Appendix 2, Table 6 ('ANOVA (FD)') shows the results from an ANOVA undertaken on field duplicate pairs collected throughout the Nonacho Basin area survey.

Field Duplicates

A one-way random effects model ANOVA was undertaken on each element in a set of 165 field duplicate pairs, representing one field duplicate pair within each block of 20 sequential sample numbers, to estimate, as a percentage, how much of the total variability is due to sampling and analysis of a lake ('within') and how much can be attributed to regional variability across the survey area ('between'). Results are shown in Table 6 of Appendix 2 ('ANOVA (FD)'). Data were not log-transformed because in all but one case (Au), the range of observations did not exceed 1.5 orders of magnitude. The sampling variability was estimated from field duplicates using the 'anova2' function in the 'rgr' package running under the R system, a random effects ANOVA model estimating whether the combined sampling and analytical variability between duplicate pairs is significantly smaller than the variability between lakes (Garrett, 2011).

FORMAT OF DATA FILES

Quality control data are presented in Appendix 2, in an Excel® spreadsheet file included with this report: **Appendix 2 GSC OF 7232 QUALITY CONTROL.xls**. There are seven worksheets in this file:

<u>Worksheet</u>	<u>Contents</u>
Trueness	Compares accepted values for two Canadian Certified Reference Standards with results from analysis of Nonacho Basin samples
Accuracy	Estimates repeatability using results from analyses of two internal standards
AD Precision	Provides an estimate of precision using analytical duplicate pairs
ANOVA (FD)	Simple pair ANOVA estimates proportion of total variability due to each of sampling and analysis
Control Reference Data	Analytical data used to estimate 'trueness' and accuracy
Analytical Duplicate Data	Analytical data used to estimate precision
Field Duplicate Data	Field duplicate data

As noted earlier, quality control data (reference standards, field and analytical duplicates) from a survey of NTS map sheet 75-K are included with quality control data for this open file.

ACKNOWLEDGEMENTS

E.H. Hornbrook directed the original survey carried out in 1976, coordinating the activities of contract and Geological Survey of Canada staff. Contracts for sample preparation and analysis were managed by J.J. Lynch, GSC.

Quality control of data was carried out with the assistance of Robert Garrett, GSC *Emeritus* Research Scientist. Wendy Spirito of the GSC reviewed this open file and provided useful comments and suggestions.

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