



**GEOLOGICAL SURVEY OF CANADA
OPEN FILE 6986**

**Regional Lake Sediment and Water Geochemical Data, Nueltin
Lake Area, Nunavut (NTS 65A, 65B and 65C)**

M.W. McCurdy, R.J. McNeil, S.J.A. Day, and S.J. Pehrsson

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Publications in this series have not been edited; they are released as submitted by the author.

Regional Lake Sediment and Water Geochemical Data, Nueltin Lake Area, Nunavut (NTS 65A, 65B and 65C)

Introduction

New analytical data for 60 elements from the reanalysis of lake sediment samples collected from 2,526 sites in the Nueltin Lake area, Nunavut, in 1976 are presented in this Geological Survey of Canada (GSC) open file release. Field observations and analytical data originally reported in GSC Open Files 413, 414 NS 415 (1977) are included in a separate digital file with this report, as well as data for uranium and fluoride in corresponding lake waters.

The area outlined in Figure 1 was sampled in 1976 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 archive 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 Earth Science Sector (ESS) of NRCan and the Polar Continental Shelf Project (PCSP). 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.

The GEM Minerals component (MGM) of the GEM Program is designed to raise the level of geoscience knowledge of Canada's North, with emphasis on the acquisition and rapid release of data for mineral exploration and land-use planning. Supported by geochemical and geophysical information, multidisciplinary teams (federal, territorial/provincial, university-based collaborators and students) are targeting areas with high potential for base and precious metals, diamonds and rare metals.

The Nueltin Lake Project is part of the Chesterfield Gold Project of ESS's GEM Program. This 3-year project will improve understanding of the location and character, bounding structures, age and architecture of gold deposits, and tectonostratigraphic controls on gold-bearing units within the Chesterfield-Hearne boundary zone and Hearne domain. It aims to test present models of overlapping Paleoproterozoic/Archean gold localization through targeted structure/stratigraphic/deposit studies in areas of known potential, and to expand the understanding of gold potential in the Hearne through more regional studies.

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.

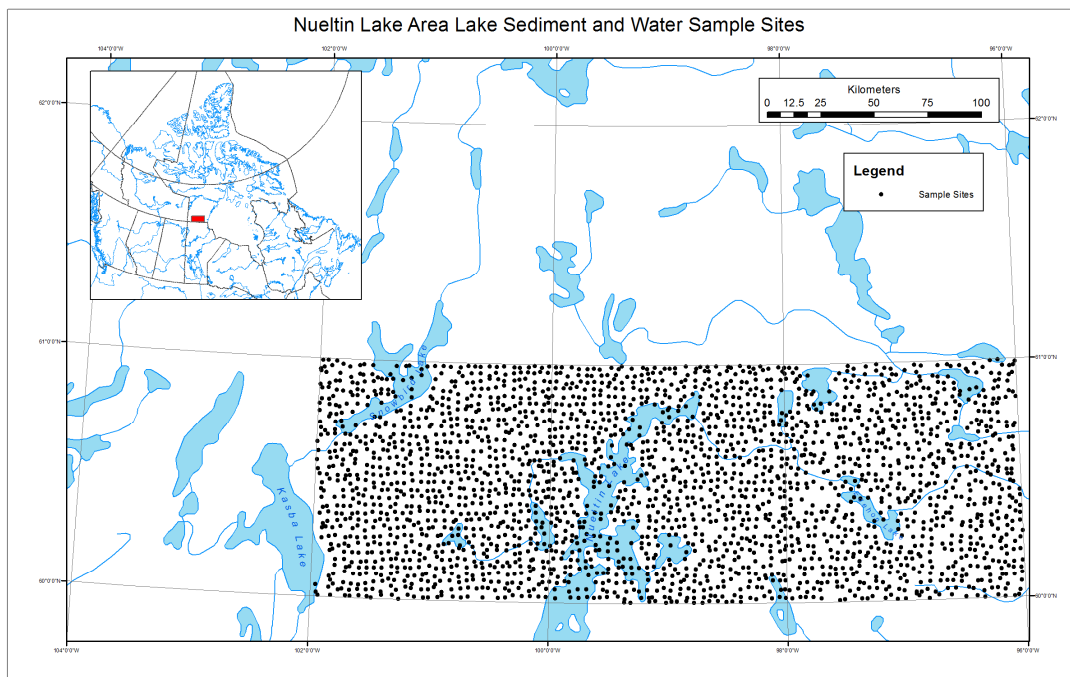


Fig. 1 Map showing location of lake sediment and water samples collected in the Nueltin Lake area.

Methods

Description of Survey and Sample Management

The original lake sediment samples were collected during the summer of 1976. Data for 12 elements in sediments and U and F in lake waters were released in 1977 in three GSC open files (413, 414 and 415). The 2 526 sample sites from which sediments were reanalyzed for this report, shown in Figure 1, are distributed throughout the 36 742 km² area at an average density of one sample per 14.6 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. After milling, control reference and blind duplicate samples were inserted into each block of twenty sediment samples. For quality control purposes, the original samples were arranged in groups (consecutively-numbered blocks) of twenty. Each group of twenty contained site 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 group 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 1976.

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Analytical Procedures (2011)

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 (2:2:2 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 spectroscopy on a Perkin-Elmer Elan instrument. Analyses were carried out at Acme Analytical Laboratories, Limited, Vancouver, British Columbia.

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

Element	Detection Limit	Units of Measurement	Analytical Method	Element	Detection Limit	Units of Measurement	Analytical Method
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 on a high resolution germanium detector. Typical counting times were 500 seconds. The sample weights were reported. Analyses were carried out at Becquerel Labs, Mississauga, Ontario.

Table 2. Variables determined by INA Reanalysis of Archive 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

Variable	Detection Limit	Units of Measurement	Variable	Detection Limit	Units of Measurement
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 (1976)

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 brought up 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 diethyldithio-carbamate. 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 diethyldithio-carbamate. 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 10^{12} 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.

Water analyses (1976)

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

After determining fluoride, samples in 225 ml bottles were acidified with 3 ml concentrated HNO₃. Two weeks after acidification, a 5 microlitre aliquot of the sample was then removed for the determination of uranium by fission track analysis. The two-week waiting period was to ensure that all precipitated uranium had returned to solution. Sample aliquots were placed on a polycarbonate tape and dried. The tape was then irradiated in a nuclear reactor for one hour. The tape was etched with 25% NaOH solution and the fission tracks were counted with an optical counter fitted to a microscope. Each tape contained its own calibration standards, blanks and sample duplicates.

Table 3. Summary of original elements determined and methods used in 1976.

Element	Detection Limit	Units of Measurement	Analytical Method
Ag	0.2	ppm	AAS ¹
As	1.0	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
U (waters)	0.01	ppb	FT ⁶
F (waters)	10	ppb	ISE ⁷

- ¹ Atomic Absorption Spectrophotometry
- ² Colorimetric
- ³ Cold vapour Atomic Absorption Spectrometry
- ⁴ Gravimetric methods
- ⁵ Neutron activation – delayed neutron counting
- ⁶ Fission Track
- ⁷ Ion-selective Electrode

Analytical results are presented in Appendix 1 in an Excel® spreadsheet file included with this report: **GSC OF 6986 DATA.xls**. There are five worksheets in this file:

<u>Worksheet</u>	<u>Contents</u>
Field Data	Site-specific field observations including geographic coordinates
Original 1976 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

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 ('blind') duplicates (BD), standard reference materials (SRM), and control reference materials (CRM) samples within the sample suite submitted to the labs. A 'triplicate' or 'triple' is a sample grouping consisting of an analytical or 'blind' duplicate split from one of the field duplicate (FD) pairs, permitting the local sampling variability and analytical variability to be separately estimated. Table 4 provides information on the number of each quality control sample within each sample suite, based on the year of collection. Analytical data for control reference standards, analytical and field duplicates, and blanks are included with this report in **Appendix 2 GSC OF 6986 QUALITY CONTROL.xls**.

Nueltin Lake (65A, 65B, 65C)				
N = 2,526				
FD Pairs	BD Pairs	SRM	CRM	'Triples'
148	153	34	120	17

Table 4. Number of quality control samples or sample pairs, or 'triples' included with reanalyzed lake sediment samples.

Data quality was evaluated using standard reference materials to evaluate trueness, control reference materials to evaluate accuracy and analytical duplicate samples to evaluate analytical precision. Field duplicates and 'triples' were used to carry out Analyses of Variance (ANOVA) in order to assess fitness-of-purpose ('Are differences between sample sites real?') for mapping and to compare the estimated analytical, site and regional variability.

Tables 1 through 7 (Worksheets 'Trueness', 'Accuracy', 'BD Precision', 'ANOVA (FD)', 'ANOVA (Triples)') in **Appendix 2 GSC OF 6986 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 with 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. 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), 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. Control reference materials consist of stream sediments from two creeks near Dawson City, YT, collected, dried, sieved and homogenized for use as internal standards at the GSC. Results for each element are shown in Tables 3 and 4 of Appendix 2 ('Accuracy').

Analytical data for internal standards Bonanza and Hunker Creek are included with this report and 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 $N = 11$ duplicate pairs. The sum of these values was divided by the number of samples ($(2*N) = 22$) 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 ('BD 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 results from an ANOVA undertaken on field duplicate pairs collected throughout the Nueltin Lake area survey, and Table 7 in Appendix 2, illustrates how triplicates can be used to estimate analytical variability as well as sampling variability both within lakes and between lakes.

Field Duplicates

A one-way random effects model ANOVA was undertaken on each element in a set of 148 field duplicate pairs, representing one field duplicate 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 (‘within’) of a lake and how much can be attributed to regional variability across the survey area (‘between’). Results are shown in Table 6 (‘ANOVA (FD)’)) of Appendix 2. 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, 2011a).

Triplicates

The data for triplicates, where the analytical duplicate is split from one of the field duplicate pair, are used to separately estimate the percentage of variation between samples collected within few metres of each other (field duplicates), between samples collected on different lakes, and due to chemical analysis. Seventeen triplicate data sets are used to make the estimates shown in Appendix 2, Table 7 (‘ANOVA (Triples)’)). The separate analytical and sampling variability’s were estimated from these sets using the ‘gx.triples.aov’ function found in the ‘rgr’ package running under the R system, a random effects ANOVA model (Garrett, 2011a). This permits the local sampling variability without the influence of analytical variability to be compared to the field survey variability. Furthermore, if local and analytical variability are high, the ANOVA identifies the source of the variability so that changes to decrease local or analytical variability by modifying sampling or analytical procedures can be implemented (Garrett, 2011b).

Table 7 in Appendix 2 (‘ANOVA (Triples)’)) shows results for all elements listed in Tables 1 and 2 (above). Variability is partitioned into three components, ‘Between Lakes’, ‘Within Lakes’, and ‘Analytical’ in the table. Elements are grouped based on their position with the Periodic Table.

The ANOVA indicates that the sampling variability is significantly lower than the field survey variability, at the $p < 0.05$ level (>95% confidence level) for elements not in bold print in Table 6. From this it is inferred that maps of the distribution of these elements will display the true spatial variability of those elements. For elements that fail either of the ANOVA tests, sophisticated methods of data manipulation are not recommended (Reimann et al., 2008; Garrett, 1969).

FORMAT OF DATA FILES

Quality control data are presented in Appendix 2, in an Excel® spreadsheet file included with this report: **GSC OF 6986 QUALITY CONTROL.xls**. There are nine worksheets in this file:

<u>Worksheet</u>	<u>Contents</u>
Trueness	Compares accepted values for two international reference standards with results from analysis of Nueltin Lake samples

<u>Worksheet</u>	<u>Contents</u>
Accuracy	Estimates repeatability using results from analyses of two internal standards
BD 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
ANOVA (Triples)	Provides an estimate of analytical variation between sites, at sites and between analyses
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
Triples Data	Triplicate sets used to estimate sampling and analytical variation

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