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**GEOLOGICAL SURVEY OF CANADA  
OPEN FILE 8017**

**Regional Lake Sediment and Water Geochemical Data,  
Labrador, Newfoundland and Labrador (NTS 13-L, 14-C,  
14-E, 14-F, 14-L, 23-J, 23-O, 24-A, 24-H and 24-I)**

**M.W. McCurdy**

**2016**

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**2016**

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Analytical Duplicate Data	Analytical data used to estimate precision
Field Duplicate Data	Field duplicate data used for ANOVA estimates

# **Regional Lake Sediment and Water Geochemical Data, Labrador, Newfoundland and Labrador (13-L, 14-C, 14-E, 14-F, 14-L, 23-J, 23-O, 24-A, 24-H, 24-I)**

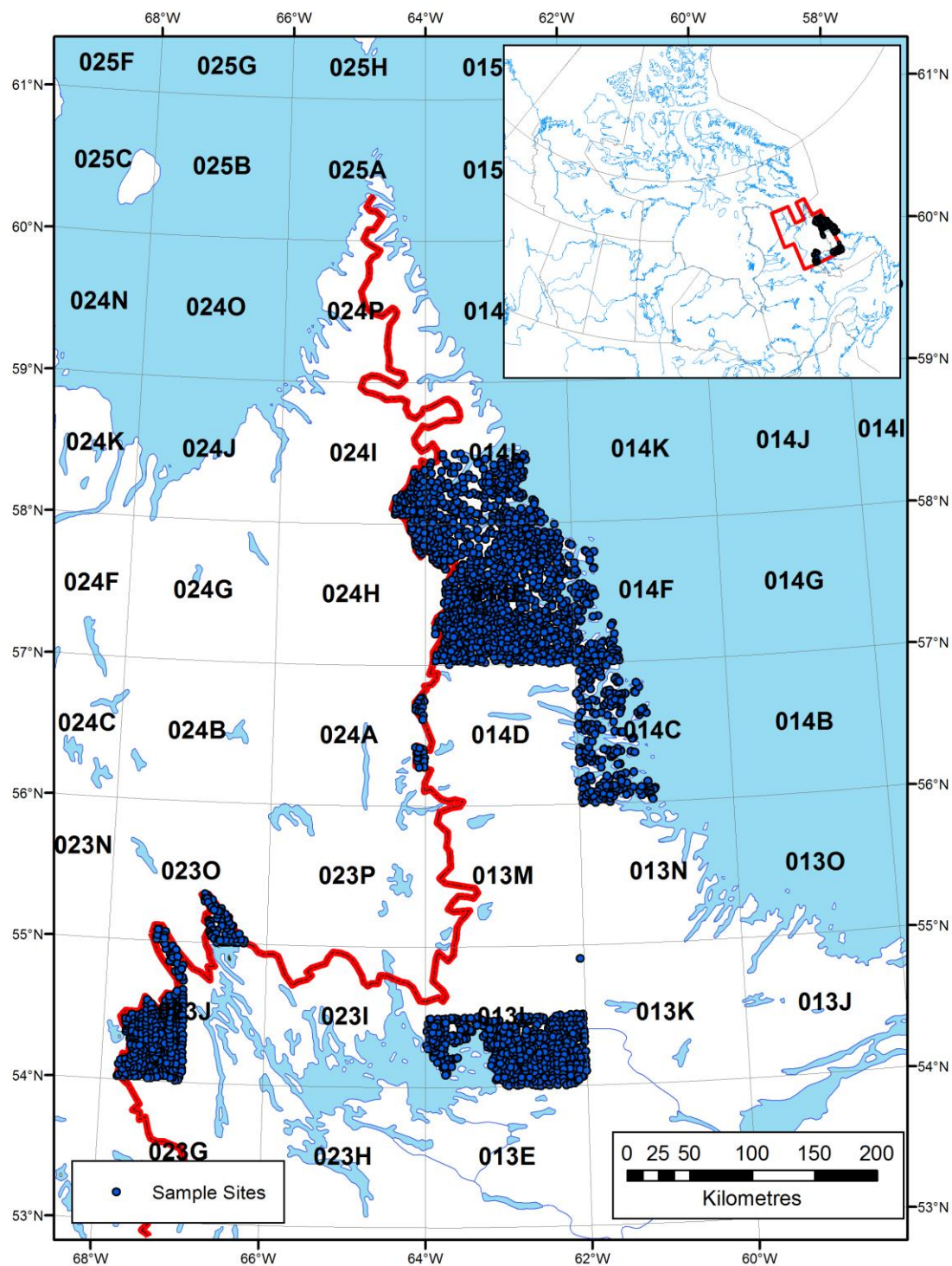
## **INTRODUCTION**

New analytical data for 65 elements from the reanalysis of 2,075 lake sediment samples collected between 1982 and 1985 throughout northern Labrador (Fig. 1) are published in this Geological Survey of Canada (GSC) open file release. Field observations, loss-on-ignition (LOI) in sediments and uranium, fluoride and pH in lake waters originally reported in GSC Open Files 904 (Geological Survey of Canada, 1983), 998 (Geological Survey of Canada, 1984), 1209 (Geological Survey of Canada, 1986) and 1210 (Geological Survey of Canada, 1986) are included with this report.

Funds for the reanalysis of the archived samples have been made available under the second phase of the Geo-Mapping for Energy and Minerals (GEM-2) Program of Natural Resources Canada. The GEM-2 Program provides modern public geoscience that will set the stage for long-term decision making related to investment in responsible resource development. Geoscience knowledge produced by GEM supports evidence-based exploration for new energy and mineral resources and enables northern communities to make informed decisions about their land, economy and society. Building upon the success of its first five-years, GEM has been renewed until 2020 to continue producing new, publically available, regional-scale geoscience knowledge in Canada's North. This report is part of a GEM 2 Hudson-Ungava Project - Core Zone Surficial Activity, focused on northeast Quebec and west central Labrador (cf., McClenaghan et al., 2015). This project is being carried out in collaboration with the Ministère de l'Énergie et des Ressources Naturelles du Québec (MERNQ) and the Geological Survey of Newfoundland and Labrador (GSNL).

Very complex mafic and alkaline magmatic, as well as magmatic-hydrothermal histories, have thus far complicated exploration programs for Ni-Cu-PGE, and polymetallic iron oxide copper-gold (IOCG), albitite-hosted uranium and affiliated deposits within iron oxide and alkali alteration hydrothermal systems in the Labrador Trough and associated eastern Core Zone. To address this issue, the GSC is developing new methods for predicting areas with potential to host IOCG and affiliated deposit types, establishing unique geochemical and mineralization signatures for alteration systems associated with these deposit types. More recent published lake sediment geochemical data are available for both the Labrador Trough and Core Zone, extending from central Labrador into northeast Québec (McConnell and Finch, 2012; Maurice and Labbé, 2009); however the use of different digestions and analytical methods for the two provincial data sets makes the compilation of these data into one dataset for the purpose of producing and interpreting geochemical maps difficult (Amor, 2015). Additionally, key elements (Te, Bi, PGE and Au) needed to outline alteration zones and facilitate Au, IOCG and Ni-Cu-PGE exploration, were never determined or had lower detection limits that are too high to be useful.

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** Lake sediments collected from sites in Labrador between 1982 and 1985 (blue points) were re-analysed and results are published in this report. Inset shows the area of study covering parts of northern Labrador and northern Quebec.

## METHODS

### Description of Surveys and Sample Management

The original lake sediment and water surveys were carried out during the summers of 1982, 1983 and 1985. Data from 2,075 lake sites at an average density of 1 sample per 13 km<sup>2</sup> for 15 to 17 elements in sediments and three variables (U, F, pH) in surface lake waters were released between 1983 and 1986 in GSC Open Files 904 (Geological Survey of Canada, 1983), 998 (Geological Survey of Canada, 1984), 1209 (Geological Survey of Canada, 1986) and 1210 (Geological Survey of Canada, 1986). All samples were subsequently reanalyzed by Instrumental Neutron Activation and these data were released in GSC open files 2037 (Hornbrook and Friske, 1989), 2647 (Friske et al., 1993a), 2690 (Friske et al., 1993b), and 2691 (Friske et al., 1993c). McConnell and Finch (2012) published new inductively-coupled plasma-emission spectrographic (ICP-ES) data for samples collected between 1977 and 1984 in Labrador, including the samples in this report.

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; Fig. 2). At GSC laboratories in Ottawa, field-dried samples were air-dried and sieved through a minus 80 mesh (177 µm) screen before ball-milling in ceramic-lined puck mills. Typically, one kilogram of the wet organic gel, the preferred collection material, yielded about 50 g of material for analysis. After milling, control reference and analytical duplicate samples were inserted into each block of twenty sediment samples. For quality control purposes, the original samples were consecutively numbered and arranged in groups ('blocks') of twenty. Each block of twenty contained one site duplicate sample pair; that is, two samples from a single site. The block also contained an analytical duplicate sample pair (a single site sample split and assigned two non-consecutive sample numbers). Finally, one of several 'in-house' check standard samples (Horowitz, 1991) was inserted into each block. 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 archival 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 for previous analyses.

The image shows a field card from the Geological Survey of Canada, titled "GEOCHEMICAL LAKE SEDIMENT SAMPLE CARD (REV.74)". The card is designed for data entry and is divided into several sections. At the top, there are fields for "PROJECT NO.", "AREA", "PHOTO", "COLLECTOR", and "DATE". Below these, there is a large table with 40 columns, numbered 1 to 40. The table is organized into sections for different types of data: "RELIEF" (LOW, MED, HIGH), "COMPOSITION" (SAND, FINE, ORG, GEL), "CONTAMINATION" (WORK, CAMP, FUEL, GDSN, TAN, YEL, GRN, GRY, BRN, BLK, HVY, LGHT), "COLOURS", "SUSP.", "LAKES AREA", "DEPTH", and "REMARKS". The card is also labeled "GEOLOGICAL SURVEY OF CANADA" and "LAKE SEDIMENTS".

Figure 2 Field card used to capture site-specific field observation data in 1982, 1983 and 1985.

## Analytical Procedures (2015)

### *Modified Aqua Regia – ICP-MS*

For the determination of 65 elements listed in Table 1, a 0.5 g sample was weighed into a test tube for sample digestion. A modified aqua regia solution of equal parts concentrated ACS grade HCl and HNO<sub>3</sub> and demineralized water was added to each sample (6 ml/g) to leach in a hot-water bath (~95°C) for one hour. After cooling, the solution was made up to a final volume with 5% HCl. Sample weight to solution volume ratio was 0.5 g per 10 ml. The solution is aspirated into a Perkin Elmer Elan 6000 or 9000 ICP mass spectrometer (description taken from Acme document “Group 1F-MS version 2.0”, dated May 6th, 2009). The analytical results are presented in **Appendix 1 GSC OF 8017 FIELD & ANALYTICAL DATA.xlsx**. Analyses were carried out at Bureau Veritas (formerly ‘Acme’) Analytical Laboratories, Limited, Vancouver, British Columbia.

Element	Lower Detection Limit	Element	Lower Detection Limit
Ag	2 ppb <sup>1</sup>	Na	0.001 %
Al	0.01 % <sup>2</sup>	Nb	0.02 ppm
As	0.1 ppm <sup>3</sup>	Nd	0.02 ppm
Au	0.2 ppb	Ni	0.1 ppm
B	20 ppm	P	0.001 %
Ba	0.5 ppm	Pb	0.01 ppm
Be	0.1 ppm	Pd	10 ppb
Bi	0.02 ppm	Pr	0.02 ppm
Ca	0.01 %	Pt	2 ppb
Cd	0.01 ppm	Rb	0.1 ppm
Ce	0.1 ppm	Re	1 ppb
Co	0.1 ppm	S	0.02 %
Cr	0.5 ppm	Sb	0.02 ppm
Cs	0.02 ppm	Sc	0.1 ppm
Cu	0.01 ppm	Se	0.1 ppm
Dy	0.02 ppm	Sm	0.02 ppm
Er	0.02 ppm	Sn	0.1 ppm
Eu	0.02 ppm	Sr	0.5 ppm
Fe	0.01 %	Ta	0.05 ppm
Ga	0.1 ppm	Tb	0.02 ppm
Gd	0.02 ppm	Te	0.02 ppm
Ge	0.1 ppm	Th	0.1 ppm
Hf	0.02 ppm	Ti	0.001 %
Hg	5 ppb	Tl	0.02 ppm
Ho	0.02 ppm	Tm	0.02 ppm
In	0.02 ppm	U	0.05 ppm
K	0.01 %	V	2 ppm
La	0.5 ppm	W	0.05 ppm
Li	0.1 ppm	Y	0.01 ppm
Lu	0.02 ppm	Yb	0.02 ppm
Mg	0.01 %	Zn	0.1 ppm
Mn	1 ppm	Zr	0.1 ppm
Mo	0.01 ppm		

Table 1 Lower detection limits for elements determined using aqua regia digestion/ICP-ES/MS analytical procedures.



## Analytical Procedures (1982, 1983 and 1985)

### *Analytical Procedures for Loss-on-Ignition (1982, 1983, 1985)*

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, and then allowed to cool to room temperature for weighing.

### *Analytical Procedures for Surface Lake Waters (1982, 1983, 1985)*

pH in lake water samples was determined using a glass-calomel combination electrode and pH meter. Fluoride in lake water samples was determined using an Orion fluoride electrode and a Model 404 Orion specific ion meter. Prior to measurement an 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.

Element	Sample Media	Detection Limit	Units of Measurement	Analytical Method
LOI	Lake sediment	1.0	pct	GRAV <sup>1</sup>
F	Lake water	10	ppb	ISE <sup>2</sup>
pH	Lake water			

<sup>1</sup> Gravimetric methods

<sup>2</sup> Ion selective electrode

Table 2 Data for loss-on-ignition in lake sediments and pH and the concentration of F (fluoride) determined in surface lake waters in 1982, 1983 and 1985 are provided in this report.

## FORMAT OF ANALYTICAL DATA FILES

Analytical results are presented in Appendix 1 in an Excel® spreadsheet file included with this report: **Appendix 1 GSC OF 8017 FIELD & ANALYTICAL DATA.xlsx**. There are four worksheets in this file (Table 3).

Worksheet	Contents
Field Data (1982, 1983, 1985)	Original site-specific field observations including geographic coordinates for lake sediment samples collected in 1982, 1983 and 1985
Reanalysis ICP Data (2015)	New ICP-MS analytical data for lake sediment samples originally collected and analyzed in 1982, 1983 and 1985
LOI (1982, 1983, 1985)	Original loss-on-ignition analytical data for lake sediment samples collected in 1982, 1983 and 1985
Water Data (1982, 1983, 1985)	Original F and pH data for lake surface waters collected in 1982, 1983 and 1985

Table 3 Worksheets in Appendix 1 with a brief description of the contents of each worksheet.

## QUALITY CONTROL FOR GEOCHEMICAL RESULTS

Reliability (accuracy and precision) of analytical data returned from commercial laboratories was determined by incorporating field duplicate (FD) pairs within the sampling protocol, and including analytical

(‘blind’) duplicates (AD), and control reference materials (CRMs) in the sample suite submitted to the labs. Analytical data for CRMs, analytical and field duplicates are included with this report in **Appendix 2 GSC OF 8017 QUALITY CONTROL.xlsx**.

Data quality was estimated using control reference materials to evaluate accuracy and analytical duplicate samples to evaluate analytical precision. Field duplicate data were used to carry out an Analysis of Variance (ANOVA) in order to compare the estimated sampling and analytical variability for mapping purposes.

Tables A1 through A6 in Appendix 2 (Quality Control) can be used to estimate the quality of analysis for elements listed in Table 1. Elements are grouped based on their position in the Periodic Table. Data used for calculations are included in separate worksheets (Table 4).

Worksheet	Contents
<b>Table A1 Accuracy – LKSD-1</b>	Compares accepted values for Certified Reference Material LKSD-1 with results from reanalysis of Labrador samples
<b>Table A2 Accuracy – LKSD-2</b>	Compares accepted values for Certified Reference Material LKSD-2 with results from reanalysis of Labrador samples
<b>Table A3 Accuracy – LKSD-3</b>	Compares accepted values for Certified Reference Material LKSD-3 with results from reanalysis of Labrador samples
<b>Table A4 Accuracy – LKSD-4</b>	Compares accepted values for Certified Reference Material LKSD-4 with results from reanalysis of Labrador samples
<b>Table A5 Precision</b>	Provides an estimate of precision using analytical duplicate pairs
<b>Table A6 ANOVA</b>	Simple pair ANOVA estimates the proportion of total variability due to each of sampling and analysis
<b>Certified Reference Data</b>	Analytical data used to estimate ‘trueness’ and accuracy
<b>Analytical Duplicate Data</b>	Analytical data used to estimate precision
<b>Field Duplicate Data</b>	Field duplicate data used to calculate Analysis of Variance

Table 4 Worksheets in Appendix 2 (quality control) with a brief description of contents.

## Accuracy

Accuracy of analytical data was evaluated by inserting Canadian Certified Reference Lake Sediments LKSD-1, LKSD-2, LKSD-3 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, NTS 31M and Joe Lake, NTS 31F). LKSD-2 was prepared using lake sediment from Calabogie Lake in central Ontario and unused portions of sample material collected in NTS map sheets 86K and 86L (East Arm of Great Bear Lake in Northwest Territories). LKSD-3 consists of a mixture of lake sediments from Calabogie Lake and unused portions of sample material from different surveys in central Ontario (NTS 31M, 41P, 42A), eastern Quebec (NTS 31N, 32C, 32D) and northeastern Saskatchewan (64L, 64M). 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 A1 through A4 of Appendix 2 (‘Accuracy – LKSD-1, -2, -3, -4’), accepted means and standard deviations (MEAN  $\pm$  SD) for control reference standards LKSD-1, LKSD-2, LKSD-3 and LKSD-4 analyzed using a strong acid (concentrated HNO<sub>3</sub>-concentrated HCl) digestion, published by Lynch (1990, 1999) and Hechler (2013), are shown. Accepted values in square brackets are derived from published and unpublished data (n > 30) collected from recent projects at the GSC. Lower detection limits (LDL) for each element estimated by Bureau Veritas are listed. A per cent Relative Standard Deviation (RSD %) is calculated for each element with values above detection limits.

Several elements have concentrations below detection in one or more CRM, including Pd (-1, -2, -3, -4), Ta (-1, -2, -3, -4), Pt (-1, -2, -3), B (-1, -2, -3, -4), and Ge (-1), and therefore no statistics are calculated. A relatively high RSD, suggesting poor analytical repeatability, can result when concentrations in a CRM are close to the detection limit for that element (Thompson, 1983). Such elements include Be (LKSD-1, -4), Hf (LKSD-1, -4), W (LKSD-3), Re (LKSD-1, -2, -3, -4), Pt (LKSD-4), Ge (LKSD-2, -3, -4), Te (LKSD-1, -2, -3, -4), and Se (LKSD-2, -3). Low detectable concentrations and subsequent relatively high RSD values (>20%) in some CRMs can be caused by elements being present within discrete, often refractory, minerals, including spinels, beryl, tourmalines, chromite, zircon, monazite, niobates, tungstates, topaz, tantalite and cassiterite (Crock and Lamothe, 2011). For Au, RSD % will be relatively high (>20%) due to the difficulty of creating homogeneous standard materials (Harris, 1982). Elements with a relatively high (>20%) RSD are shown in bold type.

## 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 up to N = 121 duplicate pairs. Duplicate pairs with one or both values below detection were removed before calculations were made. The sum of the remaining values was divided by the number of samples to estimate a measure of variability (variance). A Standard Deviation was then obtained by calculating the square root of this variance (Garrett, 1969). The resulting numerical estimate of precision for variables is listed in Table A5 in Appendix 2 (Precision) as a per cent Relative Standard Deviation (the Standard Deviation was 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 and method of analysis are the Lower Detection Limit (LDL), the percentage of duplicate sample pairs below the Lower Detection Limit (% Below LDL), the number of duplicate pairs removed from the calculations because one or both values are below detection ('Duplicate Pairs Removed') the Range of the remaining sample pairs and the Mean of the data used for each calculation of precision. This information provides context for the estimate of Precision in the last column of Table A5.

Elements with precisions poorer than 20% ( $\pm 10\%$ ), but with more than 25% (30 or more) of pairs having both values above detection in Table A5, Appendix 2, tend towards concentrations at or just above detection in samples, as indicated by the Range, the Mean and the percentage of data below the detection limit. Such is the case for the elements Hf, Re, In, Bi, Ge, Sb and Te. As noted above, low detectable concentrations and subsequent relatively high RSD values (>20%) in some CRMs can be caused by elements being present within discrete, often refractory, minerals, including spinels, beryl, tourmalines, chromite, zircon, monazite, niobates, tungstates, topaz, tantalite and cassiterite (Crock and Lamothe, 2011). Results for Au are affected by the difficulty of homogenizing this element (Harris, 1982) and should be considered accordingly. Although a precision was estimated for the elements Pt, In, Ge and Te, less than 30 sample pairs were available for the calculation and the user should consider these values less reliable than those using 30 or more sample pairs in the calculation (Walpole, 1982). For the elements Pd, Ta and B, 100% of one or both of the duplicate sample pairs were below detection and no precision was estimated.

## 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. Table A6 in **Appendix 2 GSC OF 8017 QUALITY CONTROL.xlsx** shows results from an ANOVA undertaken on up to 125 field duplicate pairs originally collected during the Labrador surveys. Duplicate pairs of which one or both values of

an element are below detection were removed from the calculations. Calculations were only carried out if the number of duplicate pairs with both values above detection exceeds 1.

Field duplicates are used to estimate the combined variation due to sampling and analysis between samples collected within a few metres of each other. Field duplicate samples were collected to provide means of estimating variability introduced by field sampling procedures and by sediment heterogeneity. The combined analytical and sampling variability was estimated from these sample pairs using Analysis of Variance (ANOVA). Using the 'anova2' function found in the 'rgr' package running under the R system, a random effects model Analysis of Variance (ANOVA) determines the combined sampling and analytical variability between sets of duplicate field samples (Garrett, 2016). This combined variability is more important than analytical variability alone because if the combined sampling and analytical variability is not significantly smaller than the field survey variability, it cannot be stated that there are statistically significant spatial patterns in the data, and thus the data are likely not suitable for mapping (Garrett, 2016), nor are sophisticated methods of data manipulation recommended (Reimann et al., 2008; Garrett, 1969).

The Analysis of Variance (ANOVA) of field duplicates partitions variability into two components, 'Between Sites' and 'Within Sites' in Appendix 2, Worksheet 'ANOVA', Table A6. The variance ratio, F, is calculated in 'anova2' to gauge whether the variance 'within' is significantly smaller than the variation 'between'. As a 'rule of thumb' this ratio should exceed 4.0 for sampling and analytical errors to be significantly smaller at the 95% confidence level. The p-value is a measure of the exact level of confidence in the results. Generally an acceptable p-value is less than 0.05 (>95th percentile), i.e. there is a <5% probability the observed F ratio could have occurred due to chance alone. It should be noted that in cases where an element is evenly distributed throughout all samples, 'F' and 'p-values' may fall below levels of confidence.

The ANOVA indicates that the sampling and analytical variability is significantly lower than the field survey variability, at the  $p < 0.05$  level (>95% confidence level) for all but Pt, Au, In, Ge, and Te in Table A6. From this it is inferred that maps of the distribution for all but the elements listed above will display the true spatial variability of those elements. Results for elements with less than 30 field duplicate pairs available for ANOVA calculations (Pt, In, Ge and Te) should be considered less reliable than those for which more than 30 pairs are available. For the elements Pd, Ta and B, 100% of one or both of the field duplicate sample pairs were below detection and ANOVA was not calculated.

## ACKNOWLEDGEMENTS

Geological Survey of Canada staff directed the original survey carried out in 1982, 1983 and 1985, coordinating the activities of contract and Geological Survey of Canada staff. Contracts for sample preparation and analysis were managed by J.J. Lynch, GSC. Roger Paulen of the GSC reviewed this open file and provided many useful comments and suggestions.

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