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**GEOLOGICAL SURVEY OF CANADA
OPEN FILE 8082**

**Geochemical Data for Lake Sediments and Surface Waters,
Abitau Lake Area, Northwest Territories
(NTS 75-B)**

M.W. McCurdy, S.J. Pehrsson, H. Falck, S.J.A. Day, and J.E. Campbell

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2016

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Table of Contents

INTRODUCTION	3
REGIONAL SETTING	5
Location and Physiography.....	5
SAMPLE COLLECTION.....	5
Lake Sediments (<i>Gyttja</i>)	5
Surface Lake Waters	7
SAMPLE PREPARATION	7
Lake Sediments (<i>Gyttja</i>)	7
Surface Lake Waters	7
ANALYTICAL PROCEDURES.....	8
Lake Sediments (<i>Gyttja</i>)	8
Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Other Analyses.....	8
Surface Lake Waters	9
Trace and Major Elements	9
Anions	10
Alkalinity	11
Total organic carbon	11
QUALITY CONTROL FOR GEOCHEMICAL RESULTS (LAKE SEDIMENTS).....	12
Accuracy	13
Precision.....	14
Analysis of Variance (ANOVA).....	14
ACKNOWLEDGEMENTS.....	15
REFERENCES	15

List of Figures

Figure 1 Lake sediment and surface water sampling sites in 2015 on the Abitau Lake map sheet, southeast Northwest Territories, NTS 75-B, showing regional distribution of eskers from Storrar et al. (2013).	4
Figure 2 About 1 kg of wet lake sediment (gyttja) is collected by dropping the sampler (above) from a platform attached to the landing skid opposite the pilot. Ideally, the entire length of the weighted sampler penetrates the lake bottom sediments to a depth below the sediment-water interface so that the upper layers of gyttja are forced out the vent located towards the tail of the sampler, reducing the possibility of contamination of the sample by recent (Anthropocene) sources.....	6
Figure 3 A Bell 206B (Jet Ranger) helicopter on floats (inset) on approach to a typical lake (1 to 5 km ²) for sampling. Pre-labelled synthetic cloth bags and high-density polyethylene (HDPE) bottles are used to collect (approximately) a 1-kg sample of lake sediment and a 250-ml sample of surface lake water.	6

List of Tables

Table 1 Specifications for YSI Professional Plus hand-held multi-parameter meter (for parameters measured in-situ).	7
Table 2 Variables in lake sediments determined by Inductively Coupled Plasma –Mass Spectroscopy (ICP-MS). Analytical methods other than ICP-MS are in brackets; ‘GRAV’ is an abbreviation of ‘Gravimetric’.	8
Table 3 Worksheets in Appendix 1 (lake sediment data) with a brief description of the contents of each worksheet.	9
Table 4 Major and trace elements determined in filtered, acidified surface lake waters in addition to detection limits, units of measurement and methods of analysis.....	10
Table 5 Methods used to detect anions, alkalinity, conductivity, pH and Total Organic Carbon (TOC) in filtered, un-acidified waters (FU).....	11
Table 6 Worksheets in Appendix 2 (water data) with a brief description of the contents of each worksheet.	12
Table 7 Worksheets in Appendix 3 (quality control) with a brief description of contents.	12

Geochemical Data for Lake Sediments and Surface Waters, Abitau Lake Area, Northwest Territories (NTS 75-B)

INTRODUCTION

A regional lake sediment and surface water geochemical survey was carried out in the Abitau Lake map sheet (NTS 75-B), southeast Northwest Territories, in 2015 (Fig. 1). This report consists of field observations and analytical data from 407 sites for 65 elements in lake sediments by a partial method of analysis (aqua regia digestion), and 65 elements in waters. Analytical results and field observations form part of a national geochemical database (Adcock et al., 2013) used for resource assessment, mineral exploration, geological mapping, and environmental studies. Sample collection, preparation procedures and analytical methods are 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 (McCurdy et al., 2014).

Funds for the collection and analysis of lake sediments and waters were made available under the Geo-mapping for Energy and Minerals (GEM) 2 Program of Natural Resources Canada (NRCan). GEM is delivered at the federal level by NRCan.

The GEM program is laying the foundation for sustainable economic development in the North through provision of modern public geoscience that will set the stage for long-term decision making related to investment in responsible resource development and northern community infrastructure and engagement, ultimately enabling northern communities to make informed decisions about their land, economy and society.

During the summer 2015, the South Rae project of GEM 2 program was initiated to upgrade geoscience knowledge in one of the least studied parts of the Canadian Shield. Southeast Northwest Territories suffers from a coarse and reconnaissance-level of geological coverage; difficult access; widespread surficial deposits that hinder observations of bedrock; and unsettled land claims. Recognizing that GEM data provides a necessary catalyst for exploration in such *terra incognita* and with the goal of completing the map of the North by mapping the most remote regions, the Geomapping Frontiers project of GEM 1 began a data mining (Harris et al., 2013; Pehrsson et al., 2014; Davis et al., 2015) and acquisition project (Kiss and Coyle, 2012; McCurdy et al., 2015), including field reconnaissance (Pehrsson et al., 2014). That exercise demonstrated that the region has unrecognized mineral potential and a dramatically more complex geology than had previously been appreciated.

Following this preliminary project and of publication of critical new supporting geoscience datasets (Campbell and Eagles., 2014; Pehrsson et al., 2015; Davis et al., 2015), the South Rae project of GEM 2 was framed as a joint Geological Survey of Canada and Northwest Territories Geological Survey project to address a number of bedrock and surficial scientific questions relevant to the mineral exploration industry (Pehrsson et al., 2015). The area of study (NTS map sheets 75-A, 75-B, 75-G, 75-H) has not seen field investigation since the GSC's helicopter reconnaissance of 1955-1958 and with the extent of Quaternary sediment cover across the map area geochemical surveys in lake sediments and surface waters are an important tool for expanding understanding of map unit distribution and identifying geochemical anomalies related to mineralization.

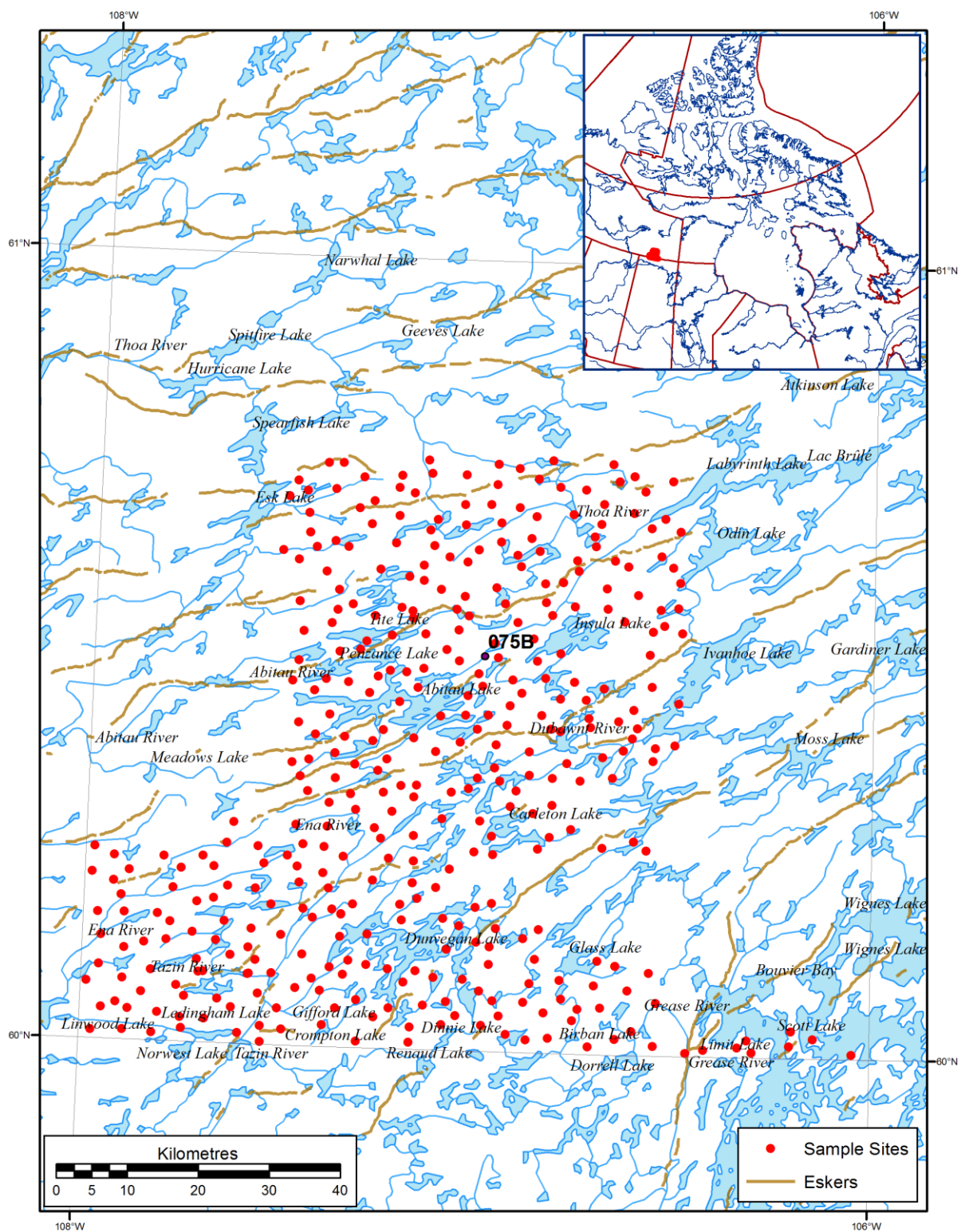


Figure 1 Lake sediment and surface water sampling sites in 2015 on the Abitau Lake map sheet, southeast Northwest Territories, NTS 75-B, showing regional distribution of eskers from Storrar et al. (2013).

REGIONAL SETTING

Location and Physiography

The survey falls within the Selwyn Lake Upland ecoregion that extends northwest from the Churchill River in Manitoba to the East Arm Hills at the eastern end of Great Slave Lake. Most of the region is 500 m or higher above sea level (asl). The mean annual temperature is approximately -5°C; the mean annual summer temperature is 11°C and the winter mean is -21.5°C. The mean annual precipitation ranges 250-400 mm. This ecoregion is classified as having a low subarctic climate and is part of the boreal forest and tundra transition extending from Labrador to Alaska. The characteristic vegetation consists of stunted black spruce, dwarf birch and Labrador tea, with a ground cover of lichen and moss. Poorly drained wetlands are dominated by bog-fen sequences of black spruce, ericaceous shrubs and mosses. Ridged to hummocky massive, crystalline rocks form broad, sloping uplands and lowlands covered with discontinuous sandy tills with significant shallow clay lacustrine deposits at lower elevations. Prominent sinuous esker ridges and lakes are common throughout the region. Dystric Brunisols and Organic Cryosols are the most widely distributed soil types. Permafrost is extensive and discontinuous with low to medium ice content throughout the ecoregion. Characteristic wildlife includes barren ground caribou, black bear, wolverine, marten, timber wolf, arctic fox, mink, snowshoe hare and red-backed vole. Spruce grouse, willow ptarmigan, sandhill crane, waterfowl and shorebirds are among the bird species common within this region. Land uses include recreation, trapping, and hunting (Ecoregions Working Group, 1989).

SAMPLE COLLECTION

Lake Sediments (*Gyttja*)

Sample site targets were digitally preselected and uploaded to a Global Positioning System (GPS) unit, which was used both to navigate to the target sample sites and to record daily helicopter tracks. Field observations were digitally recorded on a tablet using a standard form developed jointly by the GSC and the Northwest Territories Geological Survey. A bottom-valved, hollow-pipe sampler (Fig. 2) was used to collect approximately one kilogram of wet lake sediment.

At GSC laboratories in Ottawa, field-dried samples were air-dried, crushed, and sieved through an 80 mesh (177 µm) screen. Typically, one kilogram of *gyttja*, the preferred collection material, yielded about 50 g of material for analysis. For quality control purposes, the samples were arranged in groups (consecutively-numbered blocks) of twenty. Two lake sediment samples, at a site picked at the discretion of the sampler (generally based on ease of collection) and assigned sequential sample numbers, were collected at one site within each sequence of 20 samples (field duplicates). Each block of twenty also contained an analytical duplicate sample pair (a single site sample split and assigned two non-consecutive sample numbers) and a certified reference material (CRM) sample. A detailed description of quality control methods used by the GSC for lake sediment samples can be found in McCurdy and Garrett (2016).



Figure 2 About 1 kg of wet lake sediment (gyttja) is collected by dropping the sampler (above) from a platform attached to the landing skid opposite the pilot. Ideally, the entire length of the weighted sampler penetrates the lake bottom sediments to a depth below the sediment-water interface so that the upper layers of gyttja are forced out the vent located towards the tail of the sampler, reducing the possibility of contamination of the sample by recent (Anthropocene) sources.



Figure 3 A Bell 206B (Jet Ranger) helicopter on floats (inset) on approach to a typical lake (1 to 5 km²) for sampling. Pre-labelled synthetic cloth bags and high-density polyethylene (HDPE) bottles are used to collect (approximately) a 1-kg sample of lake sediment and a 250-ml sample of surface lake water.

Surface Lake Waters

At each site the 6 variables listed in Table 1 were measured in lake waters using a YSI ('Yellow Springs Instrument Company') Professional Plus handheld multi-parameter meter.

Table 1 Specifications for YSI Professional Plus hand-held multi-parameter meter (for parameters measured in-situ).

	Range	Accuracy	Resolution	Units (Recorded)
Dissolved Oxygen (%)	0-500%	0 to 200% ($\pm 2\%$ of reading or 2% air saturation, whichever is greater)	1% or 0.1% air saturation	%
Temperature	-5 to 70°C	$\pm 0.2^\circ\text{C}$	0.1°C	°C
Conductivity	0 to 200 mS/cm	$\pm 0.5\%$ of reading or 0.001 mS/cm, whichever is greater	0 to 500 $\mu\text{S}/\text{cm}=0.001$; 501 to 5000 $\mu\text{S}/\text{cm}=0.01$	μS
pH	0 to 14 units	± 0.2 units	0.01 units	pH units
Oxidation-Reduction Potential (ORP)	-1999 to +1999 mV	± 20 mV in redox standards	0.1 mV	mV
Air Pressure	375 to 825 mmHg	± 1.5 mmHg from 0 to 50°C	0.1 mmHg	kPa

Waters were sampled from within 10-15 cm of the lake surface. One 250-ml HDPE bottle was collected at each site for a filtered, acidified sample ('FA' in appendices) and a filtered, un-acidified sample ('FU' in appendices). Visual observations (colour, clarity) were noted. In addition to one sample from each 'Routine' site, at sites picked by the sample crew for duplicate lake sediment samples, another water sample was collected, the 'First' and 'Second' field duplicate.

SAMPLE PREPARATION

Lake Sediments (*Gyttja*)

Synthetic cloth bags containing the lake sediment samples were suspended about 1.5 m above ground for up to five days from nylon cord strung between trees. The partially dried samples were then placed into plastic bags which were taped closed with electrical tape and shipped in metal pails directly to the GSC laboratories in Ottawa, where they were unpacked and air-dried at temperatures below 40°C. After drying, samples were disaggregated and sieved through a minus 80-mesh (177 μm) screen (Girard et al, 2004). An aliquot sample of a Certified Reference Material (CRM) and an analytical duplicate sample were inserted into each block of twenty samples. An analytical duplicate sample is a split from a Routine Sample or a Field Duplicate after the samples have been prepared for analysis but before analysis, and analyzed using the same methods as the routine samples (McCurdy and Garrett, 2016).

Surface Lake Waters

Surface lake water samples collected in 250-ml bottles were filtered on the same day the samples were collected. Samples were filtered by pouring lake water from the 250 ml bottle into a 60 ml plastic syringe and filtering into 60-ml HDPE bottles labeled FA (filtered-acidified) and FU (filtered-unacidified) through a 0.45 μm disposable filter unit. Filtered water samples were kept cool and away from light until shipment to GSC laboratories in Ottawa. Upon arriving at the laboratory, one sample (FA) was acidified with 0.5 ml 8M HNO_3 . Samples to monitor quality assurance (acid and sample blanks) were prepared and added to each batch of samples in the field.

Certified reference standards were inserted into each block of 20 water samples in the lab. Analytical duplicate water samples were included in the sample suite.

ANALYTICAL PROCEDURES

Lake Sediments (*Gyttja*)

Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Other Analyses

Samples were analysed at Bureau Veritas Commodities Canada Limited, Vancouver, using a proprietary ‘AQ250 – Ultratrace by ICP Mass Spec.’ package with the optional extended packages for rare earth elements (+REE) and precious metals Pt and Pd (+PGM). For the determination of 65 elements listed in Table 3, a 0.5 gram sample was leached with a modified aqua regia solution (HCl:HNO₃, 1:1). The sample solution was analysed by inductively coupled plasma emission spectroscopy.

Loss-on-ignition was determined at Bureau Veritas using a one-gram sample. Each sample, in a Leco® crucible, was placed into a 100° C muffle furnace and brought up to 500° C for one hour. The oven was then cooled to 100° C and the crucibles transferred to a desiccator followed by cooling to room temperature. The crucibles were re-weighed to determine the loss-on-ignition.

Table 2 Variables in lake sediments determined by Inductively Coupled Plasma –Mass Spectroscopy (ICP-MS). Analytical methods other than ICP-MS are in brackets; ‘GRAV’ is an abbreviation of ‘Gravimetric’.

Element	Detection Limit	Units of Measurement	Element	Detection Limit	Units of Measurement
Ag	2	ppb	Mo	0.01	ppm
Al	0.01	pct	Na	0.001	pct
As	0.1	ppm	Nb	0.02	ppm
Au	0.2	ppb	Nd	0.02	ppm
B	20	ppm	Ni	0.1	ppm
Ba	0.5	ppm	P	0.001	pct
Be	0.1	ppm	Pb	0.01	ppm
Bi	0.02	ppm	Pd	10	ppb
Ca	0.01	pct	Pt	2	ppb
Cd	0.01	ppm	Pr	0.02	ppm
Ce	0.1	ppm	Rb	0.1	ppm
Co	0.1	ppm	Re	1	ppb
Cr	0.5	ppm	S	0.02	pct
Cs	0.02	ppm	Sb	0.02	ppm
Cu	0.01	ppm	Sc	0.1	ppm
Dy	0.02	ppm	Se	0.1	ppm
Er	0.02	ppm	Sm	0.02	ppm
Eu	0.02	ppm	Sn	0.1	ppm
Fe	0.01	pct	Sr	0.5	ppm
Ga	0.1	ppm	Ta	0.05	ppm
Gd	0.02	ppm	Tb	0.02	ppm
Ge	0.1	ppm	Te	0.02	ppm

Element	Detection Limit	Units of Measurement	Element	Detection Limit	Units of Measurement
Hf	0.02	ppm	Th	0.1	ppm
Hg	5	ppb	Ti	0.001	pct
Ho	0.02	ppm	Tl	0.02	ppm
In	0.02	ppm	Tm	0.02	ppm
K	0.01	pct	U	0.1	ppm
La	0.5	ppm	V	2	ppm
Li	0.1	ppm	W	0.1	ppm
LOI (GRAV)	0.1	pct	Y	0.01	ppm
Lu	0.02	ppm	Yb	0.02	ppm
Mg	0.01	pct	Zn	0.1	ppm
Mn	1	ppm	Zr	0.1	ppm

Analytical results are presented in an Excel® workbook included with this report: **Appendix 1 GSC OF 8082 FIELD OBSERVATIONS & SEDIMENT DATA.xlsx**. The two worksheets in this file are described below in Table 4.

Table 3 Worksheets in Appendix 1 (lake sediment data) with a brief description of the contents of each worksheet.

Worksheet	Contents
Field Observations	Site-specific field observations and location coordinates
ICP-MS Data	ICP-MS analytical data for lake sediment samples

Surface Lake Waters

Trace and Major Elements

Filtered and acidified (FA) surface lake water samples were analyzed for trace metal and major elements at GSC laboratories in Ottawa. A complete list of elements and stated detection limits are given in Table 4.

Trace metal analysis was performed using a Thermo X Series 2 quadrupole inductively coupled plasma mass spectrometer (ICP-MS) with Xt cones, PlasmaScreen fitted, standard concentric nebulizer and Peltier cooled conical impact bead spray chamber (3°C) using Rh and Ir as internal standards. Most isotopes measured and corrections for spectral interferences are detailed in Hall et al. (1995, 1996). Data for hafnium and zirconium are not published because these elements are not sufficiently stabilized in waters by the addition of nitric acid. Data for indium, selenium, silver, tantalum and thulium are not published because of inadequate detection limits and/or precision.

Table 4 Major and trace elements determined in filtered, acidified surface lake waters in addition to detection limits, units of measurement and methods of analysis.

Element	Detection Limit	Units of Measurement	Analytical Method	Element	Detection Limit	Units of Measurement	Analytical Method
Al	0.005	ppm	ICP-ES	Mn	0.1	ppb	ICP-MS
As	0.1	ppb	ICP-MS	Mo	0.05	ppb	ICP-MS
B	0.5	ppb	ICP-ES	Na	0.05	ppm	ICP-ES
Ba	0.2	ppb	ICP-MS	Nb	0.01	ppb	ICP-MS
Be	0.005	ppb	ICP-MS	Nd	0.005	ppb	ICP-MS
Bi	0.02	ppb	ICP-MS	Ni	0.2	ppb	ICP-MS
Br	0.05	ppm	ICP-ES	P	0.05	ppm	ICP-ES
Ca	0.02	ppm	ICP-ES	Pb	0.01	ppb	ICP-MS
Cd	0.02	ppb	ICP-MS	Pr	0.005	ppb	ICP-MS
Ce	0.01	ppb	ICP-MS	Rb	0.05	ppb	ICP-MS
Cl	0.1	ppm	ICP-ES	Re	0.005	ppb	ICP-MS
Co	0.05	ppb	ICP-MS	S	0.05	ppm	ICP-ES
Cr	0.1	ppb	ICP-ES	Sb	0.01	ppb	ICP-MS
Cs	0.01	ppb	ICP-MS	Si	0.02	ppm	ICP-ES
Cu	0.1	ppb	ICP-MS	Sm	0.005	ppb	ICP-MS
Dy	0.005	ppb	ICP-MS	Sn	0.01	ppb	ICP-MS
Er	0.005	ppb	ICP-MS	Sr	0.5	ppb	ICP-MS
Eu	0.005	ppb	ICP-MS	Tb	0.005	ppb	ICP-MS
Fe	0.005	ppm	ICP-ES	Te	0.02	ppb	ICP-MS
Ga	0.01	ppb	ICP-MS	Th	0.02	ppb	ICP-MS
Gd	0.005	ppb	ICP-ES	Ti	0.5	ppb	ICP-MS
Ge	0.02	ppb	ICP-MS	Tl	0.005	ppb	ICP-MS
Ho	0.005	ppb	ICP-MS	U	0.005	ppb	ICP-MS
K	0.05	ppm	ICP-ES	V	0.1	ppb	ICP-MS
La	0.1	ppb	ICP-MS	W	0.02	ppb	ICP-MS
Li	0.02	ppb	ICP-MS	Y	0.01	ppb	ICP-MS
Lu	0.005	ppb	ICP-MS	Yb	0.005	ppb	ICP-MS
Mg	0.005	ppm	ICP-ES	Zn	0.5	ppb	ICP-MS

Major element analysis was performed using an axial Spectro Arcos, inductively coupled plasma optical emission spectrometer (ICP-ES) using a 1% CsNO₃ buffer (1:5 ratio) as a matrix modifier with a Burgener Teflon Mira Mist Nebulizer (uptake rate 1 mL/min) and a cyclonic spray chamber. The argon flow-rates are: Coolant 14.5 L/min-1, Auxiliary 0.9 L/min-1, and Nebulizer 0.8 L/min-1. The RF power is 1500 watts. Inter-element correction factors were applied as required to correct for various spectral interferences. Data for scandium are not published because of inadequate detection limits and/or precision.

Anions

Anion analysis of filtered, un-acidified (FU) waters was done at GSC laboratories in Ottawa by a Dionex ICS 2100 Ion Chromatograph fitted with an AS-AP auto-sampler. Bromide, chloride, fluoride, nitrate, phosphate and sulphate were separated using a three-step gradient elution (12 to 52 mm potassium hydroxide eluant) with an AS-18 column. The anion concentrations were quantified using conductivity in comparison with known

concentration calibration standards and Dionex Chromeleon software. Variables, lower detection limits, units of measurement and methods are listed in Table 5 for anions, dissolved organic carbon and alkalinity.

Table 5 Methods used to detect anions, alkalinity, conductivity, pH and Total Organic Carbon (TOC) in filtered, un-acidified waters (FU).

Variables	Lower Detection Limit	Units of Measurement	Analytical Method
Br^-	0.02	ppm	Ion Chromatography (ICS-2100)
Cl^-	0.01	ppm	Ion Chromatography (ICS-2100)
F^-	0.01	ppm	Ion Chromatography (ICS-2100)
NO_3^-	0.02	ppm	Ion Chromatography (ICS-2100)
PO_4^{3-}	0.02	ppm	Ion Chromatography (ICS-2100)
SO_4^{2-}	0.02	ppm	Ion Chromatography (ICS-2100)
Alkalinity	1	ppm	Titration
Conductivity	10	$\mu\text{S}/\text{cm}$	meter
pH	0.1	unit	meter
TOC	1	ppm	Combustion

Alkalinity

Alkalinity measurements were done at GSC laboratories in Ottawa using a Man-Tech PC-Titrate™ system with a Titra-Sip™ Module on the FU samples. The total alkalinity was measured by potentiometric titration with 0.02N sulphuric acid. The software determined the volume of acid required to reach the bicarbonate equivalence point. Alkalinity results are reported as equivalents of CaCO_3 in ppm.

Total organic carbon

Total Organic Carbon (TOC) analysis was completed at GSC laboratories in Ottawa with a Shimadzu TOC-L analyser using a 680°C combustion catalytic oxidation method combined with NDIR detection. This is reported as DOC on a 0.45- μm Durapore®-filtered (FU) sample. In environmental and water samples where the inorganic carbon (IC) concentration may be high, the (total or dissolved) organic carbon is measured by a non-purgeable organic carbon (NPOC) method. This method is the same as the TOC combustion measurement method with the addition of acidification and sparging to remove the inorganic carbon (IC) in the sample prior to TOC analysis.

Analytical results are presented in an Excel® workbook included with this report: **Appendix 2 GSC OF 8082 WATER DATA.xlsx**. There are 5 worksheets in this file described below in Table 6.

Table 6 Worksheets in Appendix 2 (water data) with a brief description of the contents of each worksheet.

Worksheet	Contents
Field Observations	Field observations and data collected on-site with YSI-Pro plus multi-parameter meter
ICP-MS	Concentrations of 52 trace elements in filtered, acidified surface lake water samples using ICP-MS
ICP-ES	Concentrations of 11 major elements in filtered, acidified surface lake water samples determined using ICP-ES
Anions	Concentrations of 6 anions (ion chromatography) in filtered, un-acidified water samples
Other	Alkalinity, Conductivity, pH and Total Organic Carbon data for filtered, un-acidified surface lake waters

QUALITY CONTROL FOR GEOCHEMICAL RESULTS (LAKE SEDIMENTS)

Reliability (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 (AD), 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 3 GSC OF 8082 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 A3-1 through A3-6 in Appendix 3 (Quality Control) can be used to estimate the quality of analysis for elements listed in Tables 2 and 3 above. Elements are grouped based on their position in the Periodic Table. Data used for calculations are included in separate worksheets (Table 7).

Table 7 Worksheets in Appendix 3 (quality control) with a brief description of contents.

Worksheet	Contents
A3-1 Accuracy – LKSD-1	Compares accepted values for Certified Reference Material LKSD-1 with results from samples inserted into Abitau Lake sample suite
A3-2 Accuracy – LKSD-2	Compares accepted values for Certified Reference Material LKSD-2 with results from samples inserted into Abitau Lake sample suite
A3-3 Accuracy – LKSD-3	Compares accepted values for Certified Reference Material LKSD-3 with results from samples inserted into Abitau Lake sample suite
A3-4 Accuracy – LKSD-4	Compares accepted values for Certified Reference Material LKSD-4 with results from samples inserted into Abitau Lake sample suite
A3-5 Precision	Provides an estimate of precision using analytical duplicate pairs
A3-6 ANOVA	Simple pair ANOVA estimates proportion of total variability due to each of sampling and analysis
Control Reference Data	CRM analytical data used to calculate estimates of accuracy in Tables A3-1 to A3-4

Worksheet	Contents
Analytical Duplicate Data	Analytical duplicate pair data used to estimate precision listed in Table A3-5
Field Duplicate Data	Field duplicate pair data used to calculate ANOVA statistics shown in Table A3-6

Accuracy

Accuracy of analytical data was evaluated by inserting Canadian Certified Reference Lake Sediments LKSD-1, LKSD-2, LKSD-3 or LKSD-4 at random locations within each block of 20 samples throughout the analytical suite (McCurdy and Garrett, 2016). 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 A3-1 through A3-4 of Appendix 3 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 HNO₃-concentrated HCl) digestion, published by Lynch (1990, 1999) and Hechler (2013), are shown. Accepted means and standard deviations reported by Lynch (1990, 1999) were derived from results received from different laboratories using a concentrated HNO₃-concentrated HCl partial extraction with variations in fuming times, acid ratios and sample weights. It is assumed that the Standard Deviation reflects these variations. No allowance was made when comparing re-analysis results with the values reported by Hechler (2013). Accepted values in square brackets are derived from published and unpublished data (n > 30) collected from recent projects at the GSC, from analytical results using the same digestion method used for Abitau Lake map sheet (NTS 75-B) lake sediments reported here. 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. A relatively low RSD (<20%) is an indication of good repeatability, whereas a relatively high RSD highlights elements with possible analytical quality problems. If no obvious reason can be determined, such as contamination, for the poor RSD, the laboratory that analyzed the samples is contacted and attempts are made to resolve the problem. Individual CRM analyses falling outside the range of two standard deviations are identified by plotting the values using 'crm.plot' in 'rgr' (Garrett, 2016). In cases when obvious contamination, misplacement of CRM samples or wrongly identified CRM samples are not the cause of the high RSD, the block of 20 samples within which the CRM is inserted is re-analyzed.

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, -4), B (-1, -2, -3, -4), and Ge (-2, -3, -4), 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-2, -4), Hf (LKSD-1, -2, -4), W (LKSD-1, -3, -4), Re (LKSD-1, -2, -3, -4), Ge (LKSD-1), Te (LKSD-1, -2, -3), and Se (LKSD-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. The estimation of the analytical precision follows the procedure of Youden (1951) for up to 24 duplicates where both results were above the respective detection limits. The resulting numerical estimate of precision for variables is listed in Table A3-5 (Precision) in **Appendix 3 GSC OF 8082 QUALITY CONTROL.xlsx** 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 samples pairs below the Lower Detection Limit (Total % 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 estimates of 'Precision (RSD %)' in Table A3-5.

Elements with precisions poorer than 20% in Table A3-5 tend towards generally low concentrations in samples, as indicated by the number of duplicate pairs removed, Range, the Mean and the percentage of data below the detection limit. Such is the case for elements Be, Re, Au, Bi, As and Te using an aqua regia ('partial') digestion. Results for Au can also be affected by the particulate nature of gold ('nugget effect') and should be considered accordingly (Harris, 1982). For the elements Pd, Ta, Pt, In and B, less than two pairs of analytical duplicates have one or both of the field duplicate sample pairs are below detection and no ANOVA results are reported.

Analysis of Variance (ANOVA)

Field duplicate data were used to test the hypothesis that the combined sampling and analytical variability, s_{sa}^2 , was equal to the 'regional' variability, s_r^2 , across the areal extent of the field duplicates, i.e. $H_0: s_{sa}^2 = s_r^2$, using a one-way Analysis of Variance (ANOVA) (Garrett, 1983). It is desirable that this test fails and the sampling and analytical variability not be equal to the regional variability, but smaller. Otherwise there is as much average variability at the sample sites as there is across the survey area, and if that is the case spatial variation across the survey area cannot be reliably identified. The ANOVA procedure allows the variance components to be estimated, and thus the percentage of the variability in the field duplicate pair data that can be ascribed to sampling and analytical variability and regional variability; ideally the latter percentage should be greater than the former, and statistical significance of the underlying F-test can be used as annotation in an abbreviated table of ANOVA results, that focuses on the key issues.

Using the 'anova2' function found in the 'rgr' package running under the R system, a random effects model Analysis of Variance (ANOVA) estimates the combined sampling and analytical variability between sets of duplicate field samples (Garrett, 2016). Table A3-6 in **Appendix 3 GSC OF 8082 QUALITY CONTROL.xlsx** shows results from an ANOVA undertaken on up to 24 field duplicate pairs collected for the original 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. Data were logarithmically transformed (base 10) to meet homogeneity of variance considerations (i.e. severe heteroscedasticity) and to account for ranges of observations exceeding 1.5 orders of magnitude (Garrett, 2016).

The Analysis of Variance (ANOVA) of field duplicates partitions variability into two components, 'Between Sites' and 'Within Sites' in Table A2-4. 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 whether the observed F-ratio could have occurred by chance alone. 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 statistics in Table A3-6 indicate 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 elements except Re, Au, Ge, and As. For the elements Pd, Ta, Pt, In, B and Te, 100% of one or both of the field duplicate sample pairs are below detection and no ANOVA results are reported. From this it is inferred that maps of the distribution for all other elements will display the true spatial variability of those elements.

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