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Regional Lake Sediment and Water Geochemical Data, Northern Saskatchewan, (NTS 64-L, 64-P, 74-N, 74-O and 74-P)

M.W. McCurdy, S.J. Pehrsson, J.E. Campbell, S.W. Adcock

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Canada

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doi:10.4095/295977

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Recommended citation
McCurdy, M.W., Pehrsson, S.J., Campbell, J.E., and Adcock, S.W., 2015. Regional Lake Sediment and Water Geochemical Data, Northern Saskatchewan, (NTS 64-L, 64-P, 74-N, 74-O and 74-P); Geological Survey of Canada, Open File 7746, 1 zip file. doi:10.4095/295977

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

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Regional Lake Sediment and Water Geochemical Data, Northern Saskatchewan, (NTS 64-L, 64-P, 74-N, 74-O and 74-P)

INTRODUCTION

New analytical data for 86 variables are being made available from the reanalysis of 5,698 lake sediment samples collected in 1977 and 1993 north of the Athabasca Basin, Saskatchewan (Fig. 1), and are published in this Geological Survey of Canada (GSC) open file release. Field observations and analytical data for 12 or 13 elements in lake sediments and U, F and pH in surface lake waters originally reported in GSC Open Files 508 (Hornbrook et al., 1978), 2857 (Friske et al., 1994a) and 2858 (Friske et al., 1994b) are included with this report.

Areas of NTS 64-L and 64-M in northeast Saskatchewan were sampled in 1973 under a Federal Uranium Reconnaissance Program. Areas in the north-central and northwestern part of the province were sampled in 1993 under the Canada - Saskatchewan Partnership Agreement on Mineral Development (1990-1995).

Funds for the reanalysis of archived samples have been made available under the second phase of the Geo-Mapping for Energy and Minerals (GEM) Program of Natural Resources Canada (NRCan). The GEM II program is a new 7-year investment by the Government of Canada in geoscience knowledge and information to better define the potential for new energy and mineral resources in northern 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.

In the final phase of GEM I, 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 is one of the regions identified as a priority and is now a major focus of GEM II research. This large area of the broader Rae craton has had a highly heterogeneous history of geologic study, ranging from systematic modern 1:250,000 or 1:100,000 scale mapping in northern Saskatchewan, targeted 1:100,000 scale modern bedrock and remote predictive mapping in southeastern-most Northwest Territories, to a single phase of 1950's helicopter bedrock reconnaissance at 1:750,000-1:500,000 scale. An initial modern bedrock reconnaissance across the most poorly known part of the /area was conducted in 2012 as part of the Operation GEM project of GEM I.

The broader South Rae domain is known to host deposits and occurrences of nickel-copper platinum group elements (PGEs), rare earth elements (REEs), gold and uranium in its better mapped portions in Saskatchewan and the Northwest Territories. One goal of this new GEM II multi-disciplinary activity will be to establish if major metal-bearing faults and rock types of northern Saskatchewan extend into southern Northwest Territories. Regional to reconnaissance bedrock and Quaternary mapping, compilation of existing maps, integration of data from newly acquired geophysical and geochemical surveys and remote predictive mapping will be combined to provide knowledge for areas almost devoid of previous mineral exploration. In support of these efforts, archival lake sediment samples from northern Saskatchewan were reanalysed to provide a comprehensive geochemical suite of elements for mineral exploration and resource evaluation, as well as a background set of metadata that will be used for predictive mapping of rock units based on geochemical associations.

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.

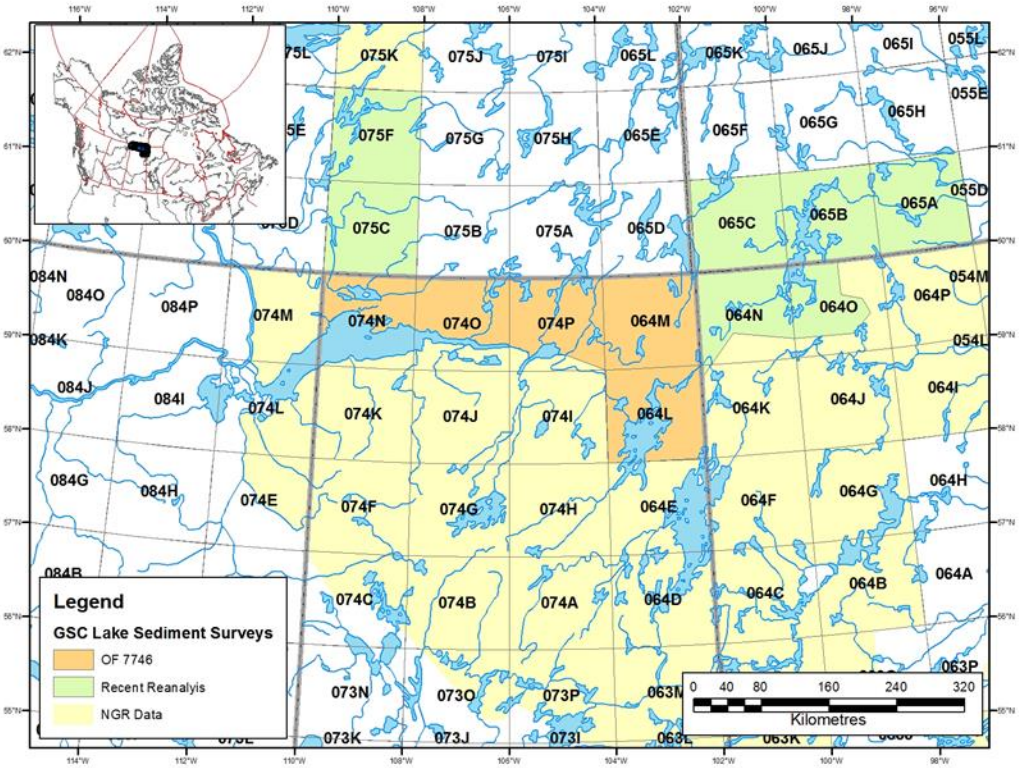

Figure 1 Lake sediments collected in orange areas have been recently re-analysed and results are published in this report. Contiguous green areas have been re-analysed for the GEM-1 program. Lake sediments collected in yellow areas have not been reanalysed.

REGIONAL SETTING

Location and Physiography

The survey areas covers the entire margin of Saskatchewan north of the Athabasca Basin along the border with Northwest Territories between longitudes 110° and 102° and latitudes 59° and 60°, extending in the northeast as far south as Lake Wollaston at 58° latitude (Fig. 1). Stony Rapids, Fond du Lac, Uranium City and Wollaston Lake are the communities located within the region of the geochemical surveys. The areas lie within the Taiga Shield Ecozone (Ecoregions Working Group, 1989) marking the transition between tundra and boreal coniferous forest. Summers are relatively short, with mean temperatures averaging 11°C; winter mean temperatures are about -21.5°C. Annual precipitation ranges between 200-400 mm. Vegetation consists of extensive open stands of low, stunted black spruce, dwarf birch and Labrador tea with a ground cover of moss and lichens. Soils are mainly Dystric Brunisols. Discontinuous permafrost with low ice-content is distributed sporadically throughout the region (Ecoregions Working Group, 1989). Western areas drain west into the Mackenzie River; eastern areas drain into Hudson Bay by the Thelon River to the north and Churchill River to the south.

Regional Quaternary Geology

Although northern Saskatchewan has undergone multiple glaciations, the present-day landscape and glacial deposits are predominantly the result of the Late Wisconsinan glaciation (Schreiner, 1984a, Dyke et al., 2002). The Keewatin Lobe of the Laurentide Ice Sheet advanced south-southwestward from a dispersal centre in Nunavut (Prest et al., 1968; Prest, 1984, McMartin and Henderson, 2004). Although large-scale streamlined landforms, eskers and small-scale erosional indicators record regional ice flow primarily during deglaciation, earlier flow directions are preserved in the striae record and rare cross-cutting landforms (Fig. 2). The main ice flow direction ranges from south-southwest in the east, shifting to southwest over the Athabasca Basin to westward immediately along its northern margin. North of the Basin, the regional ice flow was predominantly to the south-southwest. Towards the western end of Lake Athabasca however, the dominant flow recorded by striations is to the west. This is likely related to thin ice channelled westward through the Athabasca Low along the Fond du Lac River and Lake Athabasca during the advance and late retreat of the ice (Fulton, 1989; Campbell et al., 2006a, 2007a; Hanson, 2014).

As the ice retreated northeastward, deglaciation of the area began approximately 8900 14C yrs BP in the west, and was completely ice free by 8000 14C yrs BP (Schreiner, 1984a; Dyke and Prest, 1987; Dyke et al., 2002, Dyke et al., 2003; Dyke, 2004). Various proglacial lakes ponded against the ice margin as it retreated from the area. The western and central part of the area was inundated by Glacial Lake Athabasca, which extended up the Athabasca Low along the Fond du Lac River at least as far as Black Lake (Schreiner, 1984a; Campbell et al., 2007a). In the east, Glacial Lake Wollaston, the northwest arm of Lake Agassiz and other short-lived ice-marginal lakes inundated the northeastern corner of Saskatchewan (Schreiner, 1984a; Campbell, 2002a, Campbell, 2005a; Smith, 2006; Smith and Kaczowka, 2007). Models for deglaciation and the northern extent of the proglacial lakes in Saskatchewan are presented by Schreiner (1984a), Dyke and Prest (1987), Dyke and Dredge (1989), Dyke et al. (2002), Dyke et al. (2003) and Dyke (2004).

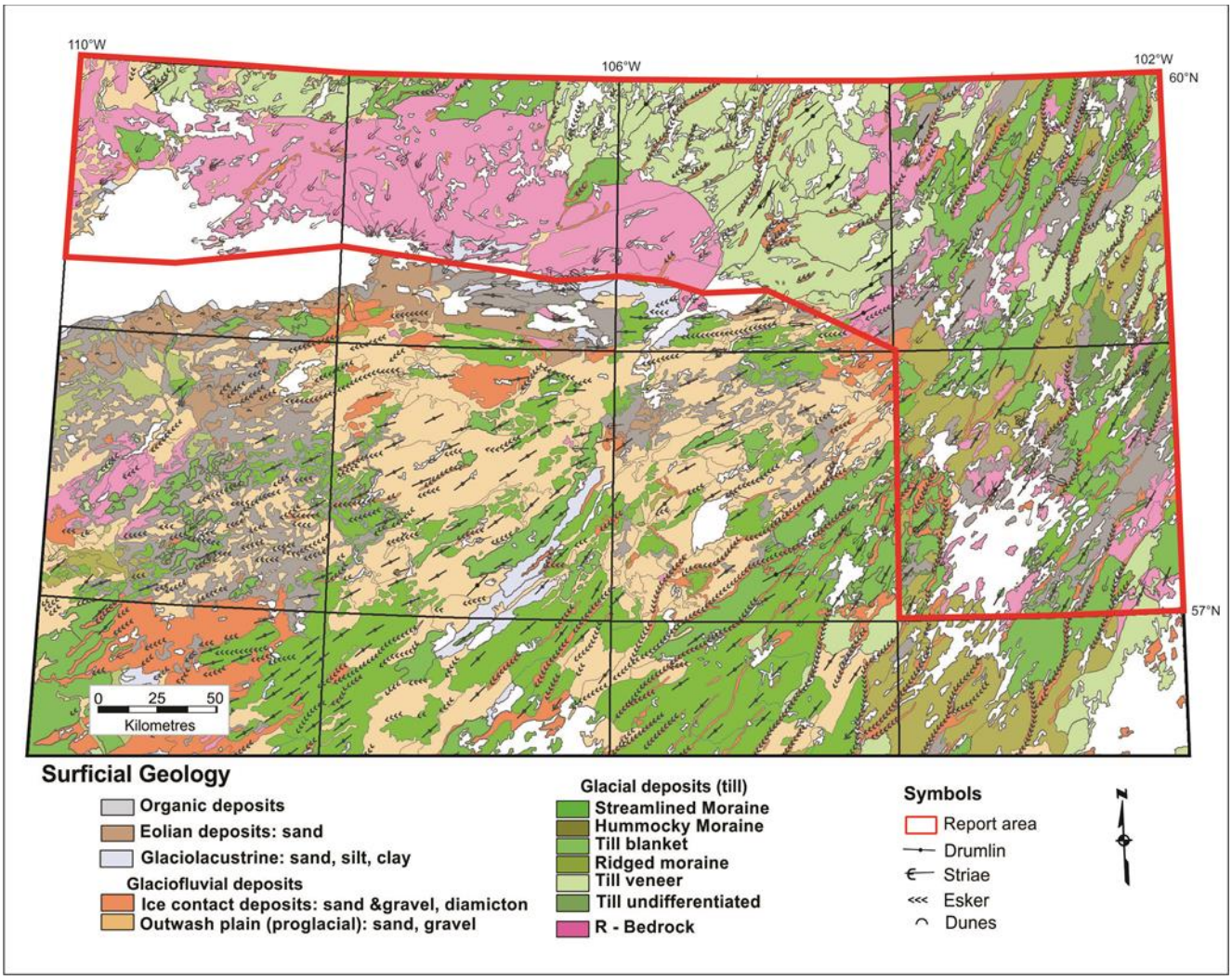

Figure 2 Regional surficial geology of northernmost Saskatchewan (modified from Schreiner, 1984a, b, c, d, e; McNamara, 1984).

Drift cover is variable in thickness, distribution and type of material (Fig. 2). The region immediately north of Lake Athabasca and the Fond du lac River (NTS 74-N, -O and -P (West)) is characterised by exposed bedrock uplands and ridges. Thicker sediments, locally overlain by thin organic deposits occur in linear depressions and valleys between the bedrock ridges. Drift cover increases northward and eastward. Here, the till is the dominant surficial material occurring primarily as a veneer less than 1m thick, and to a lesser degree, as stagnant ice moraine. Several NE-SW trending esker systems cross the area resulting in discontinuous discreet linear sand and gravel deposits. Minor glaciolacustrine deposits derived from reworked glacial sediments, are restricted to the proglacial lake basins (Schreiner, 1984a; Campbell, 2006, 2007; Campbell et al., 2006a, 2007a; Hanson, 2010, 2012a, 2012b; Hanson and Knox, 2009, 2010, 2012).

In the east (NTS 64-L and -M) bedrock exposure is much less common, generally less than 15 % (e.g. Campbell, 2001b, 2002b; Smith et al., 2006b; Smith and Kaczowka, 2008a, 2008b). The more subdued landscape reflects both glacial landforms as well as bedrock topography. Much of the eastern region landscape has been modified by meltwater erosion. The dominant surficial sediments are till, sand and gravel, boulder fields and organic terrain. Till is commonly present as veneers, blankets, stagnant ice and streamlined moraines. Sand and gravel

deposits are more abundant than in western part, primarily associated with well-developed regional subglacial (esker systems) and proglacial (outwash) meltwater drainage systems. Detailed discussions and maps of the surficial geology for selected areas within the report area are provided by Schreiner (1984a,b,c,d,e), Campbell (2001a, 2001b, 2002a, 2002b, 2006, 2007), Campbell et al. (2006a, 2006b, 2007a, 2007b) Hanson, (2010, 2012a, 2012b) Hanson and Knox (2009, 2010, 2012), Smith (2006,), Smith and Kaczowka (2007, 2008a, 2008b) and Smith et al. (2006a, 2006b). Reconnaissance scale (Campbell, 2005b) and select regional scale (Harper et al., 2002; Campbell, 2004, 2009; Smith et al., 2009) till geochemical, indicator minerals, gold grains and textural datasets are available.

Bedrock Geology

The bedrock of Northern Saskatchewan is underlain by the Rae and Hearne Provinces; two large Archean cratons that were variably reworked during major Paleoproterozoic orogenic events. The boundary between the two is the high-grade and ductile- to brittle-ductile Snowbird tectonic zone (Hoffman, 1990).

The Rae Province in the study area comprises a number of lithotectonic domains, metamorphosed under upper amphibolite- to granulite-facies conditions, variably and repeatedly, between 2.6 and 1.8 Ga (Ashton et al., 2013). Mesoarchean to Neoarchean granitoids and gneisses predominate (Pehrsson et al., 2014), with local discrete intrusions of 2.6 Ga (Nolan block, Ashton et al., 2007b), ca. 2.3 Ga (Beaverlodge; Hartlaub et al., 2007) and 1.95-1.89 Ga (Talston magmatic zone, McDonough et al., 2000). Archean rocks are locally overlain, and deformed together with, high grade metamorphosed Paleoproterozoic supracrustal belts (Iannelli et al., 1995; Ashton et al., 2007a). These in turn are overlain by two, variably deformed, but little metamorphosed, continental redbed sequences (Thluicho group, ca. 1.9 Ga, Yeo, 2005; Martin group, ca. 1.82 Ga., Morelli et al., 2009).

The Hearne craton in the study area (van Breemen et al., 2007; Pehrsson et al., 2014) dominantly comprises amphibolite-facies Neoarchean mafic-felsic volcanic and sedimentary rocks of the 2.72-2.8 Ga Ennadai group and ca. 2.7 Ga intermediate-felsic orthogneisses and granitoids. The Archean rocks are unconformably overlain by, and deformed together with, ca 2.0-1.9 Ga lower grade metasandstone-carbonate-argillite rocks of the Hurwitz group. Local 1.83 Ga granites intrude the older basement and are broadly coeval with regional deformation and metamorphism related to the Trans-Hudson orogeny.

Unconformably overlying all aforementioned units and both cratons are rocks of the Paleo-Mesoproterozoic Athabasca Group, an unmetamorphosed, flat-lying, dominantly quartz sandstone succession (Ramaekers et al., 2007). The latest magmatic events in the study area are intrusion of diabase dyke swarms in the Paleo- to Mesoproterozoic (Pehrsson et al., 2014 and references therein).

References for detailed discussions and maps of the bedrock geology for selected areas within the report area are provided in GSC Open File 5744 (Pehrsson et al., 2013).

METHODS

Description of 1977 and 1993 Surveys and Sample Management

The original lake sediment and water surveys were carried out during the summers of 1977 and 1993. Data from 2,016 lake sites at an average density of 1 sample per 6.25 km^2 (Hornbrook et al., 1978) for 12 elements in sediments and three variables (U, F, pH) in surface lake waters were released in 1978 in GSC Open File 508. Data from 2,803 sample sites over a total area of 34,944 km^2 (1 sample per 12.4 km^2) for 35 elements in sediments and U, F and pH in surface lake waters were released in 1994 in GSC Open Files 2857 (Friske et al., 1994a) and 2858 (Friske et al., 1994b).

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 and

Fig. 3 of this report). 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 consecutively numbered and arranged in groups of twenty known as blocks. Each block of twenty contained site duplicate samples; 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, each block 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 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 in 1977 and 1993.

Figure 3 Field card used to capture site-specific field observation data in 1977 and 1993.

Quality assurance/quality control measures for the reanalysis of the data by inductively coupled plasma emission spectroscopy/mass spectrometry (ICP-ES/MS) using HCl/HNO_3 and lithium metaborate digestions are described in a later section.

Analytical Procedures (2014)

Lithium Metaborate/Tetraborate Fusion – ICP-MS

For the determination of 48 elements or oxides listed in Table 1, a 0.2 g sample was dissolved by mixing it with 1.5 g of lithium metaborate ($LiBO_2$) and lithium tetraborate (Li_2B4O_7) flux in a graphite crucible and fused in an oven at 980°C for 30 minutes. The bead was dissolved into a solution of dilute (5%) nitric acid and mixed continuously until dissolved (~30 minutes). Analysis for major oxides and some trace elements was by ICP-

emission spectrometry. The same sample solution was analysed for rare-earth elements and refractory elements by ICP-mass spectrometry.

Element/Oxide	Lower Detection Limit	Element/Oxide	Lower Detection Limit
Al_2O_3	0.01%[1]	TiO_2	0.01%
CaO	0.01%	Total C (LECO)	0.02%
Cr_2O_3	0.002%	Total S (LECO)	0.02%
Fe_2O_3	0.04%	Ba	5ppm[2]
K_2O	0.01%	Nb	5 ppm
LOI (1000°C)	0.1%	Ni	20 ppm
MgO	0.01%	Sc	1 ppm
MnO	0.01%	Sr	2 ppm
Na_2O	0.01%	Y	3 ppm
P_2O_5	0.01%	Zr	5 ppm
SiO_2	0.01%		

[1] percent
[2] parts per million, mg/kg

Table 1 Lower detection limits for elements and oxides determined by 'total' analytical methods.

Total Carbon and Sulphur

For the determination of total C and total S, a 0.1 g sample was combusted with an induction flux (accelerator) in a ceramic sample holder at >1650°C in an induction furnace coupled to a LECO (Lincoln Electric Holdings, Inc.) infrared detector. Carbon and sulphur were measured by adsorption in an infrared spectrometric cell. Results are total and attributed to the presence of carbon and sulphur in all forms.

For the determination of Loss-on-ignition (LOI), a 2 g sample was combusted at 1000° C and the weight difference was used to calculate the percentage of weight lost during combustion. Total digestion analytical results are presented in **Appendix 1 GSC OF 7746 FIELD & ANALYTICAL DATA**.xlsx.

Modified Aqua Regia - ICP-MS

For the determination of 65 elements listed in Table 2, a 0.5 g sample is weighed into a test tube for sample digestion. A modified aqua regia solution of equal parts concentrated ACS grade HCl and HNO_3 and demineralized water is added to each sample (6 ml/g) to leach in a hot-water bath (~95°C) for one hour. After cooling, the solution is made up to a final volume with 5% HCl. Sample weight to solution volume ratio is 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 7746 FIELD & ANALYTICAL DATA.xlsx.** Analyses were carried out at Acme Analytical Laboratories, Limited, Vancouver, British Columbia.

Element	Lower Detection Limit	Element	Lower Detection Limit
Ag	2 ppb[1]	Na	0.001 %
Al	0.01 %[2]	Nb	0.02 ppm
As	0.1 ppm[3]	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		

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

Table 2 Lower detection limits for elements determined by 'partial' aqua regia digestion/ICP-ES/MS analytical procedures.

Analytical Procedures (1997 and 1993)

Instrumental Neutron Activation Analysis (1993 only)

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 3 on a high resolution germanium detector. Typical counting times were 500 seconds. The sample weights

are reported in **Appendix 1 GSC OF 7746 FIELD & ANALYTICAL DATA.xlsx**. Analyses were carried out at Becquerel Labs, Mississauga, Ontario.

Variable	Lower Detection Limit	Variable	Lower Detection Limit
As	0.5 ppm[1]	**Mo**	1 ppm
Au	2 ppb[2]	**Na**	0.02 pct
Ba	50 ppm	**Ni**	10 ppm
Br	0.5 ppm	**Rb**	5 ppm
Ce	5 ppm	**Sb**	0.1 ppm
Co	5 ppm	**Sc**	0.2 ppm
Cr	20 ppm	**Sm**	0.1 ppm
Cs	0.5 ppm	**Ta**	0.5 ppm
Eu	1 ppm	**Tb**	0.5 ppm
Fe	0.2 pct[3]	**Th**	0.2 ppm
Hf	1 ppm	U	0.2 ppm
La	2 ppm	W	1 ppm
Lu	0.2 ppm	Yb	2 ppm

[1] parts per million (mg/kg)
[2] parts per billion, (μg/kg)
[3] percent

Table 3 Variables determined by Instrumental Neutron Activation analysis of lake sediment samples.

Atomic Absorption Spectroscopy (AAS) and Other Analyses

For the determination of Zn, Cu, Pb, Ni, Co, Ag, Mn, Fe and Cd (1993), a 1 g sample was reacted with 3 ml concentrated HNO_3 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 and vanadium (1993) were determined by AAS using a nitrous oxide acetylene flame. A 0.5 g sample was reacted with 1.5 ml concentrated HNO_3 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 HNO_3 and 1 ml concentrated HCl in a test tube for 10 minutes at room temperature prior to two hours of digestion with mixing at 90° C in a hot water bath. After digestion, the sample solutions were cooled and diluted to 100 ml with metal-free water. The Hg present was reduced to the elemental state by the addition of 10 ml 10% weight per volume (w/v) $SnSO_4$ in 1 M H_2SO_4. The Hg vapour was then flushed by a stream of air into an absorption cell mounted in the light path of an atomic absorption spectrophotometer. Absorption measurements were made at 253.7 nm.

Loss-on-ignition was determined using a 500 mg sample. The sample, weighed into a 30 ml beaker, was placed in a cold muffle furnace and brought up to 500° C over a period of two to three hours. The sample was held at this temperature for four hours, and then allowed to cool to room temperature for weighing.

Arsenic (1977) 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 diethyldithio-carbamate. The intensity of the colour of the complex was determined with a spectrophotometer. Colorimetric measurements were made at 520 nm.

Uranium (1977) 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/cm^2/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.

Fluorine (1993) was determined in lake sediments as described by Ficklin (1970). A 250 mg sample was sintered with 1 g of a flux consisting of two parts by weight sodium carbonate and one part by weight potassium nitrate. The residue was then leached with water. The sodium carbonate was neutralized with 10 mL 10% (w/v) citric acid and the resulting solution was diluted to 100 mL with water. The fluoride in the resulting solution was then measured using a fluoride ion electrode. Standard solutions contained sodium carbonate and citric acid in the same quantities as the sample solution.

Element	**Sample Media**	**Detection Limit**	**Units of Measurement**	**Analytical Method**
Ag	Lake sediment	0.2	ppm	AAS[1]
As (1977)	Lake sediment	1.0	ppm	COL[2]
Cd (1993)	Lake sediment	0.2	ppm	
Co	Lake sediment	2	ppm	AAS
Cu	Lake sediment	2	ppm	AAS
F (1993)	Lake sediment	40	ppm	
Fe	Lake sediment	0.02	pct	AAS
Hg	Lake sediment	10	ppb	CV-AAS[3]
LOI	Lake sediment	1.0	pct	GRAV[4]
Mn	Lake sediment	5	ppm	AAS
Mo	Lake sediment	2	ppm	AAS
Ni	Lake sediment	2	ppm	AAS
Pb	Lake sediment	2	ppm	AAS
U (1977)	Lake sediment	0.5	ppm	NADNC[5]
V (1993)	Lake sediment	5	ppm	
Zn	Lake sediment	2	ppm	AAS
U (1977)	Lake water	0.01	ppb	FT[6]
U (1993)	Lake water	0.05	ppb	LIF[7]
F (1993)	Lake water	10	ppb	ISE[8]
pH	Lake water			

[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] Laser-induced fluorescence
[8] Ion selective electrode

Table 4 Summary of original elements determined and methods used in 1977 and 1993.

Analytical Procedures for Lake Waters (1977 and 1993)

pH in lake water samples (1977 and 1993) was determined using a glass-calomel combination electrode and pH meter.

Fluoride in lake water samples (1977) was determined using an Orion fluoride electrode and a Model 401 Orion specific ion meter. Prior to measurement an aliquot of the sample was mixed with an equal volume of a modified TISAB 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.

Fluoride in lake water samples (1993) was determined using a fluoride electrode. Prior to measurement, an aliquot of the sample was mixed with an equal volume of TISAB II buffer solution (total ionic strength adjustment buffer). The TISAB II buffer solution was prepared as follows: to 50 mL metal-free water add 57 mL glacial acetic acid, 58 g NaCl and 4 g CDTA (cyclohexylene dinitrilo tetraacetic acid). Stir to dissolve and cool to room temperature. Using a pH meter, adjust the pH between 5.0 and 5.5 by slowly adding 5 M NaOH solution. Cool and dilute to one litre in avolumetric flask.

In 1977, after determining fluoride, samples in 225 ml bottles were acidified with 3 ml concentrated HNO_3. 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.

Uranium in waters (1993) was determined by a laser-induced fluorometric method using a Scintrex UA-3 uranium analyser. A complexing agent, known commercially as Fluran and composed of sodium pyrophosphate and sodium monophosphate (Hall, 1979) was added to produce the uranyl pyrophosphate species which fluoresces when exposed to the laser. Since organic matter in the sample can cause unpredictable behaviour, a standard addition method was used. Further, the reaction of uranium with Fluran can be delayed or sluggish; for this reason an arbitrary 24 hour time delay between the addition of the Fluran and the actual reading was incorporated into this method. In practice, 500 microlitres of Fluran solution were added to a 5 mL sample and allowed to stand for 24 hours. At the end of this period fluorescence readings were made with the addition of 0.0, 0.2 and 0.4 ppb U. For U-rich samples the additions are 0.0, 2.0 and 4.0 (20 microlitre aliquots of either 55 or 550 ppb U were used). All readings were taken against a sample blank.

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 7746 FIELD & ANALYTICAL DATA.xlsx**. There are five worksheets in this file:

Worksheet	Contents
Field Data	Site-specific field observations including geographic coordinates
Reanalysis ICP Data*	ICP-MS/ES analytical data for lake sediment samples
AAS Data (1977 & 1993)*	AAS and specific methods analytical data for lake sediment and water samples
INAA Data (1993 Only)*	INAA analytical data for lake sediment samples
Water Data (1977 & 1993)	U, F and pH data for lake surface waters

* 'NA' in data field indicates data "Not Available' because of insufficient sample material or missing sample.

QUALITY CONTROL FOR GEOCHEMICAL RESULTS

Reliability (Accuracy and Precision) of analytical data returned from commercial laboratories was estimated by incorporating field duplicate pairs within the sampling protocol, preparing analytical ('blind') duplicate samples and inserting certified reference materials (CRM) within the sample suite submitted to the labs. Analytical data for certified reference standards, analytical and field duplicates are included with this report in **Appendix 2 GSC OF 7746 QUALITY CONTROL.xlsx**.

Certified reference material data were used to estimate accuracy and analytical duplicate samples to estimate analytical precision. Field duplicate pair data were used to calculate Analyses of Variance (ANOVA) in order to assess fitness-of-purpose ('Are differences between sample sites real?') for mapping.

Tables 1 through 6 (Worksheets 'Accuracy' (x4), 'Precision', and 'ANOVA) in Appendix 2 can be used to estimate the quality of analysis for almost every element found in Tables 1 and 2 of this report. Elements are grouped based on their position in the Periodic Table.

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'), means and standard deviations (MEAN ± SD) for control reference standards LKSD-1, LKSD-2, LKSD-3 and LKSD-4, for which provisional values have been published by Lynch (1990, 1999) and Hechler (2013) 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 the four certified reference standards are listed. Relative Standard Deviation (RSD), expressed as a percentage, facilitates comparison of variables measured in different units and varying means (Reimann et al., 2008). RSD is independent of both the magnitude of the data and the units. Estimated values for LKSD-1 in square brackets are derived from unpublished data ($n \geq 40$) collected from recent projects at the GSC.

Analytical data for elements, oxides and other variables for which an accepted mean exists are almost all are within one Standard Deviation of that mean. Elements with possible analytical problems, as indicated by a relatively high (>20%) RSD, are shown in bold type. However, a relatively high RSD, suggesting poor repeatability, may also be an indication that concentrations in a CRM are close to the detection limit for that element

(Thompson, 1983). Such elements include In, Hf, Te, Be, Ge and Re by aqua regia digestion. Au, Pt, Re and Hg data are affected by the inherent inhomogeneity of these elements and the small (0.5 g) sample size. Relatively poor analytical precision (relatively high RSD %) for elements such as Na, Se, Zr and Th by partial extraction and Nb by total extraction may be the result of these elements being present in resistate, difficult to digest minerals (spinels, beryl, tourmaline, zircon, monazite, tungstates, topaz, niobates, etc.).

Precision

Precision is considered in terms of the closeness of agreement between analytical duplicate samples analysed 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 = 289 duplicate pairs. The sum of these values was divided by the number of samples ((2*N) = 578) 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 estimate of precision for variables is listed in Table A5 in Appendix 2 (Precision) as a per cent Relative Standard Deviation (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 A5.

Elements with precisions poorer than 20% (but more than 15% of the values above detection) in Table A5, 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 Be and Sb by partial methods. Results for Au and Hg from an aqua regia ‘partial’ digestion are affected by the difficulty of homogenizing these elements and should be considered accordingly. Tungsten (W) may be present within the molecular structure of a tungstate, a mineral resistant to digestion by aqua regia.

Analysis of Variance (ANOVA)

Field duplicate samples are collected at sample sites to provide a means of comparing the combined variability of geochemical data introduced by field sampling procedures, sediment heterogeneity and analysis with regional variability. When a sufficient number of field duplicate pairs, at least 30, have been analysed, a one-way random effects model Analysis of Variance can be carried out. Functions ‘anova1’ or ‘anova2’ in the ‘rgr’ package running under the R system (Garrett, 2013) are used to estimate the combined sampling and analytical variability (‘within’ sites) and regional variability (‘between’ sites) in sets of duplicate field samples. This combined variability is more important than analytical variability alone because if the combined sampling and analytical component is not significantly smaller than the field survey component, it cannot be stated that there are statistically significant spatial patterns in the data, and thus the data are misleading, or at worst, meaningless (Garrett, 2013; Reimann et al., 2008; Thompson & Howarth, 1973).

The variance ratio, F, is calculated in ‘anova’ 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. Results of total and partial analysis of 288 field duplicate pairs from the reanalysis of northern Saskatchewan lake sediments are shown in Table A6 (Appendix 2, ‘Anova’). For most elements the component of analytical variability introduced by sampling and laboratory

procedures is significantly less than the regional variability and these data are suitable for mapping and statistical purposes. Two elements, Pd and Ta, cannot be estimated because all values are below detection.

A logarithmic transformation of the geochemical data is carried out in recognition that the data are drawn from constant sum compositions (Filzmoser et al., 2009), and in order to minimize the effects of heteroscedasticity, i.e. where the variance of the duplicate pairs varies as a function of the means of the pairs across the range of the data. Homogeneity of variance, lack of heteroscedasticity, is a fundamental assumption of the ANOVA procedure.

FORMAT OF QUALITY CONTROL DATA FILES

Quality control data are presented in **Appendix 2 GSC OF 7746 QUALITY CONTROL.xlsx**, an Excel® spreadsheet file included with this report. There are nine worksheets in this file:

Worksheet	Contents
Table A1 Accuracy – LKSD-1	Compares accepted values for Certified Reference Material LKSD-1 with results from analysis of northeastern Saskatchewan samples
Table A2 Accuracy – LKSD-2	Compares accepted values for Certified Reference Material LKSD-2 with results from analysis of northeastern Saskatchewan samples
Table A3 Accuracy – LKSD-3	Compares accepted values for Certified Reference Material LKSD-3 with results from analysis of northeastern Saskatchewan samples
Table A4 Accuracy – LKSD-4	Compares accepted values for Certified Reference Material LKSD-4 with results from analysis of northeastern Saskatchewan samples
Table A5 Precision	Provides an estimate of precision using analytical duplicate pairs
Table A6 ANOVA	Simple pair ANOVA estimates the 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 used to calculate Analysis of Variance

ACKNOWLEDGEMENTS

E.H. Hornbrook and W.B. Coker directed the original survey carried out in 1977 and P.W.B. Friske and C.C. Durham directed the 1993 surveys, coordinating the activities of contract and Geological Survey of Canada staff. Contracts for sample preparation and analysis were managed by J.J. Lynch, GSC, in 1977 and 1993.

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 many useful comments and suggestions.

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