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**GEOLOGICAL SURVEY OF CANADA  
OPEN FILE 8179**

**MANITOBA GEOLOGICAL SURVEY  
OPEN FILE OF2016-1**

**Geochemical data for lake sediments in the Superior  
Province of Manitoba  
(NTS 53-E, 53-L and 53-M)**

**M.W. McCurdy, C.O. Böhm, S.D. Anderson, M.S. Gauthier, and S.D. Amor**

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**M.W. McCurdy<sup>1</sup>, C.O. Böhm<sup>2</sup>, S.D. Anderson<sup>2</sup>, M.S. Gauthier<sup>2</sup>, and S.D. Amor<sup>3</sup>**

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**2017**

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# Geochemical Data for Lake Sediments in the Superior Province of Manitoba (NTS 53-E, 53-L and 53-M)

## INTRODUCTION

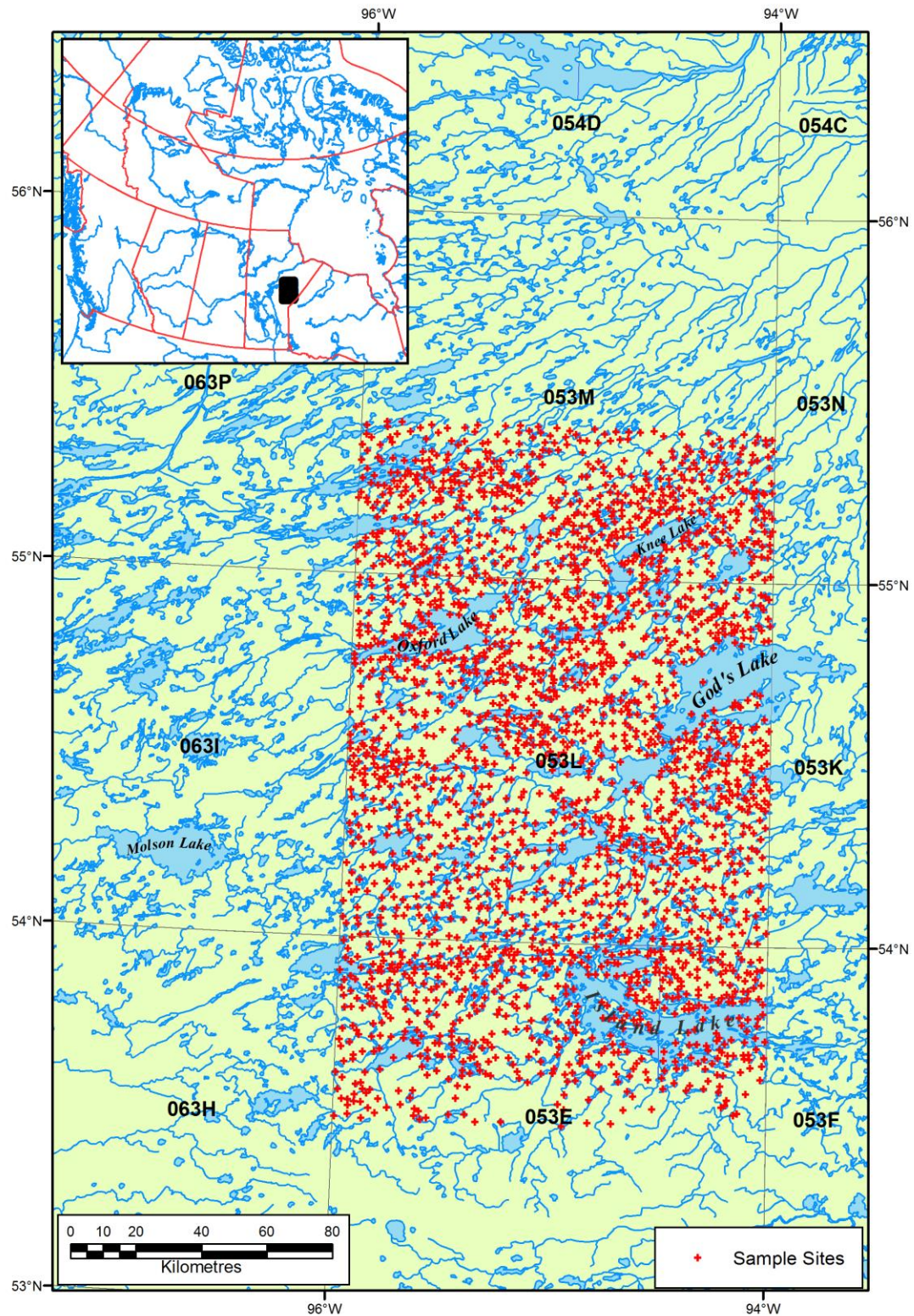
New analytical data for 65 elements in lake sediments collected in 1986 from 2,485 sites in eastern-central Manitoba (Figure 1) are published in this joint Geological Survey of Canada (GSC) and Manitoba Geological Survey (MGS) open file release. Field observations, loss-on-ignition (LOI) in sediments and uranium, fluoride and pH in lake waters originally reported in GSC Open File 1358 (Geological Survey of Canada, 1987) are included with this report. Analytical results and field observations from this project form part of a [national geochemical database](#) (Adcock, 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).

These new data complement data collected by the MGS under the auspices of the Operation Superior Project – a 5-year program of multimedia geochemical surveys in the northwest Superior province that covered the most extensive greenstone belts in NTS areas 53K, 53L, 53M and 63I. This helicopter-supported project involved the collection of rock, till, b-horizon soil, humus and vegetation samples for multi-element geochemical and indicator mineral analysis (*see* Fedikow et al., 2002a, b, and references therein).

Funds for the re-analysis of lake sediments were provided by the MGS. Additional funds 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 Natural Resources Canada (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. Main mandates of the MGS are to provide timely and relevant geoscience data to the accrued knowledge of Manitoba's geology and mineral resources. Furthermore, MGS programming promotes sustainable resource development, informed land-management planning, Aboriginal engagement and mineral education to the public.





**Figure 1** Lake sediment sample sites in 1986, east-central Manitoba, NTS 53-E, 53-L and 53-M

## REGIONAL SETTING

### Location and Physiography

The survey falls within the Hayes River Upland ecoregion lying northeast of Lake Winnipeg and extends east into Ontario. The mean annual temperature is approximately  $-4^{\circ}\text{C}$ ; the mean annual summer temperature is  $11.5^{\circ}\text{C}$  and the winter mean is  $-20^{\circ}\text{C}$ . The mean annual precipitation range is 400–600 mm. This ecoregion is classified as having a sub-humid high boreal climate and is part of the boreal forest and tundra transition extending from Labrador to Alaska. The characteristic vegetation consists mainly of black spruce, jack pine and birch with a predominating shrub layer of ericaceous shrubs, willow and alder. Ground cover consists of mosses and lichens, low ericaceous shrubs and some herbs. Bedrock exposures have fewer trees and are covered with lichens. Archean granites and gneisses form locally steeply-sloping uplands overlain by discontinuous ridged to hummocky till in the southern section and loamy, calcareous till in the northern section. Grey Luvisols and Dystric and Eutric Brunisols are the most widely distributed soil types. Permafrost is discontinuous and found mainly in organic deposits throughout the ecoregion. Many small, rock-bound lakes and medium to large lakes, linked by the primary drainage ways of the Nelson, Stupart, Hayes, and God's rivers are a prominent feature of this ecoregion. Characteristic wildlife includes wolf, lynx, otter, marten, beaver, moose, black bear, woodland caribou, snowshoe hare, red squirrel, short-tailed weasel, red-backed vole and least chipmunk. Spruce grouse, sharp-tailed grouse, willow ptarmigan, common nighthawk, raven, grey jay, bald eagle, hawk owl, and numerous passerine and waterfowl species are among the bird species common within this region. Land uses include water-oriented recreation, tourism, trapping, and hunting (Canadian Council on Ecological Areas, 2014).

### Bedrock geology

The survey area is underlain by metamorphosed Archean plutonic, volcanic and sedimentary rocks in the northwestern Superior province of the Canadian Shield (Figure 2). The bedrock geology in the northern portion of the survey area, including Oxford Lake, Knee Lake and God's Lake, was compiled at 1:50 000 scale under the auspices of the Greenstones Project, which had the objective of improving the geological database to support exploration activity in the region (Gilbert, 1985; Hubregtse, 1985). The bedrock geology of the Island Lake area was compiled at a scale of 1:250 000 by Ermanovics et al. (1987). Concurrent or subsequent to these regional compilations, bedrock mapping at 1:20 000 scale has been completed in numerous locations throughout the survey area (a comprehensive listing of reports and maps from this work can be accessed online through the Bibliography of Manitoba Geology, Manitoba Growth, Enterprise and Trade, Mineral Resources Library).

In the regional tectonic scheme of Percival et al. (2006), the survey area spans the northern margin of the North Caribou superterrane – the largest Mesoarchean tectonic domain in the Superior province. This superterrane is characterized by widespread isotopic evidence for development of sialic crust by 3.0 Ga and includes a locally-preserved cover of quartzite, iron formation and ultramafic–mafic volcanic rocks thought to represent early (ca. 3.0–2.9 Ga) platform or rift sequences. The north margin of the superterrane includes a series of mostly juvenile domains of continental margin and oceanic affinity, which from north to south include the Oxford-Stull, Munro Lake and Island Lake domains (Figure 2). These are interpreted to have been tectonically imbricated and accreted to the north margin of the Mesoarchean core of the superterrane during the ca. 2.72–2.71 Ga Northern Superior orogeny (Percival et al., 2006).

Each of these domains consists mainly of granitoid rocks and orthogneiss, with narrow belts of supracrustal rocks. In Manitoba, uranium-lead ages indicate that magmatism occurred over a 300 m.y. time interval, with major pulses of volcanism and plutonism at ca. 2.9–2.88, 2.85–2.82 and 2.75–2.7 Ga (Corkery et al., 2000; Lin et al., 2006; Parks et al., 2006). The largest supracrustal belts in the survey area are the Oxford Lake–Knee Lake, God's Lake, Munro Lake, Beaverhill Lake, Island Lake and Bigstone Lake belts. The internal stratigraphy of these belts is highly complex. However, they typically include one or more, older (2.9–2.8 Ga) assemblages dominated by pillowed tholeiitic basalt, overlain by younger (ca. 2.75–2.7 Ga), more diverse volcanic rocks of tholeiitic to calcalkaline arc affinity. Alkaline volcanic rocks and associated subvolcanic intrusions locally constitute a significant component of these younger assemblages, most notably at Knee Lake and Oxford Lake, where syenite plutons (e.g., the Cinder Lake alkaline intrusive complex),

carbonatite and lamprophyre dikes, and shoshonitic volcanic rocks were emplaced during the latest stages of volcanism (e.g., Anderson, 2016a).

The early volcanic assemblages are locally overlain, either conformably or across a pronounced angular unconformity, by clastic sedimentary assemblages that commonly include both marine and subaerial facies (e.g., Lin et al., 2013; Anderson, 2016b). Detrital zircons in these assemblages are representative of underlying volcanic assemblages (Corkery et al., 2000; Lin et al., 2013). Coarse subaerial facies occur in discrete, fault-bounded basins and detrital zircon U-Pb ages constrain sedimentation to be younger than 2.71 Ga, roughly coeval with the Northern Superior orogeny (Percival et al., 2006). Early thrust faulting was followed by folding and development of crustal-scale shear zones that dominate the map pattern in most belts.

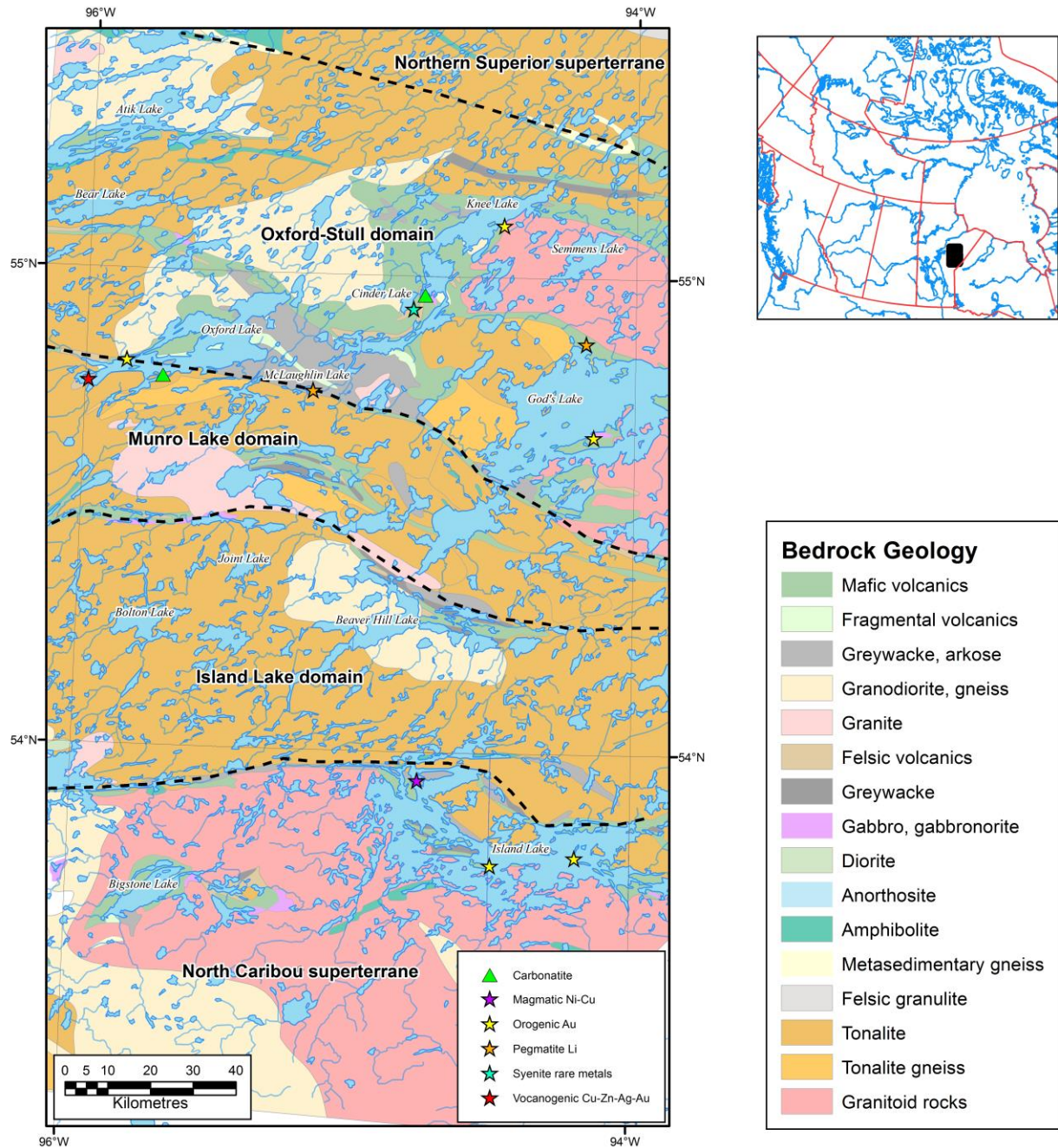
The survey area contains numerous mineral deposits and occurrences (Figure 2). Magmatic nickel-copper mineralization is locally associated with differentiated sills, small plugs or komatiitic flows within older ultramafic–mafic volcanic assemblages. The most significant occurrence of this type is the Nickel Island deposit, hosted by ca. 2.85 Ga (Parks et al., 2006) spinifex-textured komatiite flows and associated intrusions in the northwest portion of the Island Lake belt. Volcanogenic massive sulphide (Cu-Zn-Ag-Au) mineralization is associated with coherent and fragmental felsic volcanic rocks within older assemblages at Oxford Lake and Knee Lake; however, the Hyers Island deposit at the western end of Oxford Lake is the only significant deposit of this type in the survey area.

Gold mineralization is associated with quartz-carbonate vein systems in major and minor shear zones in several locations in the survey area. Important occurrences include: the Rusty Zone deposit at the western end of Oxford Lake; the Knee Lake mine in the northern basin of Knee Lake; the God's Lake mine on Elk Island in Gods Lake; and the Island Lake mine and High Rock Island deposit in the south-central portion of Island Lake (Richardson and Ostry, 1996).

Rare-metal mineralization is associated with pegmatite dikes and alkaline intrusions in the Knee Lake and Gods Lake areas. Spodumene (Li) pegmatite occurs at McLaughlin Lake, southwest of Knee Lake (Lenton, 1985) and west of God's River, in the northwest portion of God's Lake, and syenitic phases of the Cinder Lake alkaline intrusive complex (located west of Knee Lake) are strongly enriched in rare-earth elements (Chakhmouradian et al., 2008). Carbonatite has been documented in the south-central portion of Oxford Lake (Reimer, 2014) and a carbonatite association has been inferred for the Cinder Lake complex on the basis of textural and geochemical criteria (Chakhmouradian et al., 2008; Kressall et al., 2010), suggesting potential for carbonatite-hosted rare-metal mineralization in these locations (Anderson, 2016a). The widespread occurrence of kimberlite indicator minerals in surficial sediments (Fedikow et al., 2002a), most notably in the Knee Lake area, also indicates potential for conventional kimberlite-hosted diamond deposits or unconventional deposits hosted by lamprophyre or derived sedimentary rocks (Anderson, 2016a).

Post-tectonic gabbro and diabase dikes of the 1.88 Ga Molson (Heaman et al., 1986) and 1.27 Ga Mackenzie (LeCheminant and Heaman, 1989) swarms are the youngest Precambrian rocks in the survey area.



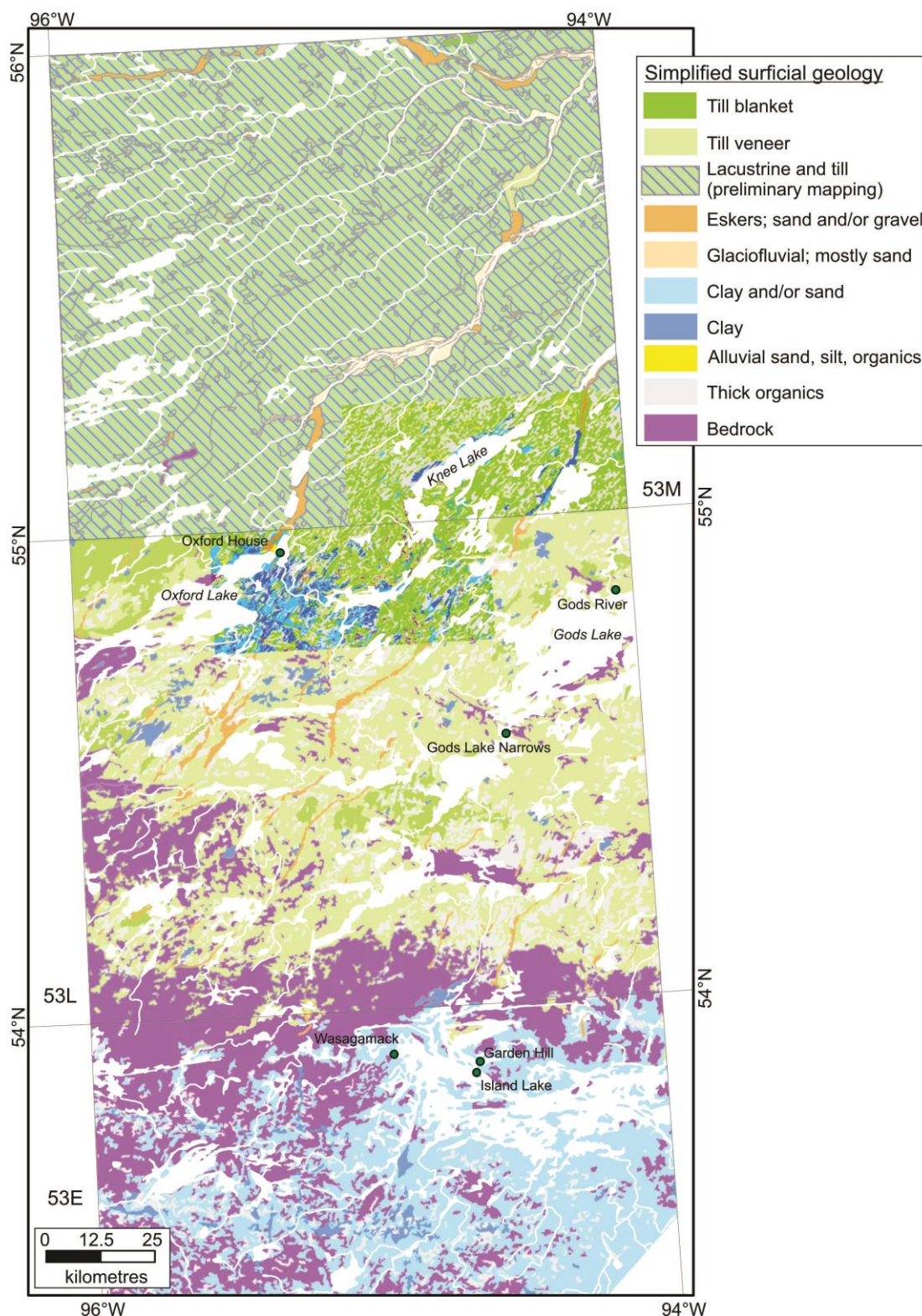


**Figure 2** Regional geological setting of the survey area within the Manitoba segment of the northwestern Superior province, showing major geological domains (modified from Manitoba Mineral Resources, 2013).

### Surficial geology

The survey area is mantled by organic and glaciolacustrine deposits of variable thickness, which overlie till and/or bedrock. Till is thickest in the north, and disappears in the south (Figure 3).





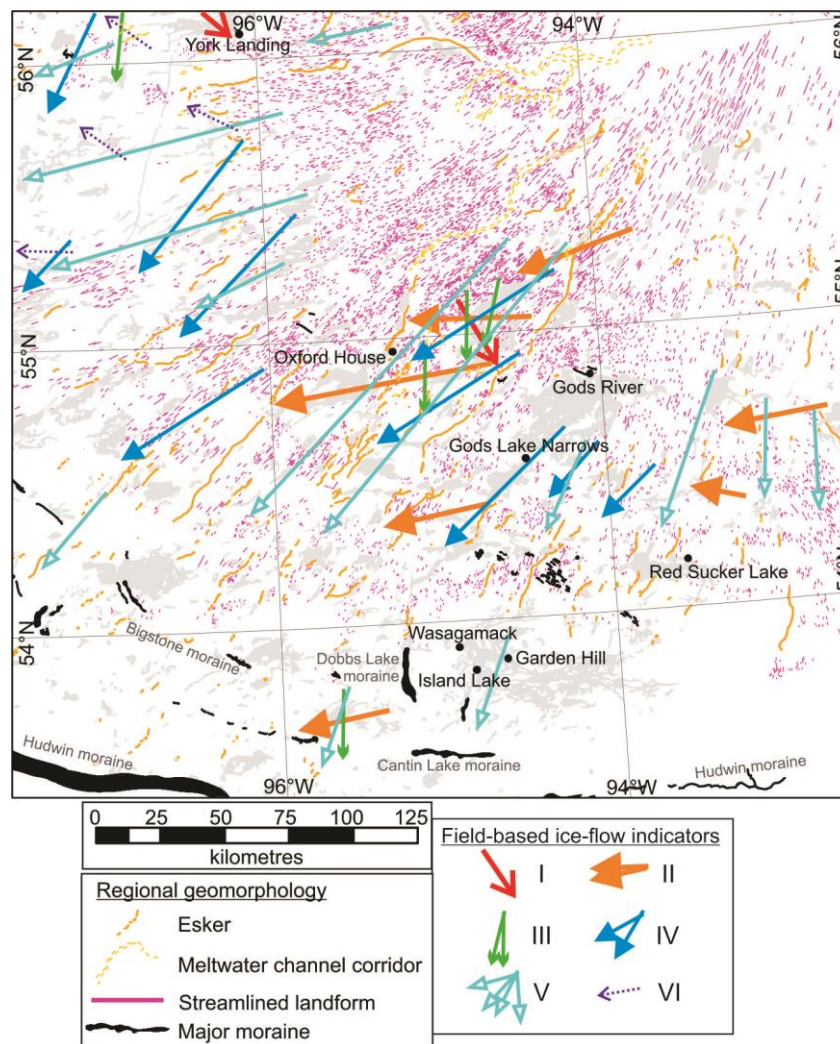
**Figure 3** Simplified surficial geology of the lake sediment survey area, compiled from a number of maps (see below) at different scales and survey levels (Klassen and Netterville, 1979; Clarke, 1988; 1989; Klassen and Netterville, 1985; Trommelen, 2015a-e)

Regional Quaternary geology maps for this area are available in Klassen and Netterville (1979) and Clarke (1988, 1989), at a scale of 1:250 000, and Klassen and Netterville (1985) at a scale of 1:500 000. Detailed descriptions of sediments and the surficial geology of the Kree Lake area (1:50 000) are provided in Trommelen (2015a-e). The Island

Lake – Red Sucker lake area is mapped at 1:100 000 scale (Nielsen 1980). This mapping is not digital, and hence has not been included in Figure 3.

### *Ice flow history*

The survey area contains evidence of at least five different ice-flow phases (Figure 4). The old, rare, ice-flow phase trending to the southeast (between 150 and 160° azimuth, phase I), and the more widespread old phase trending to the west (between 255 and 270°, phase II) are likely correlative to the older Sundance and Amery glaciations. Late Wisconsinan ice-flow phases include a rare but widespread southward-trending ice-flow phase (between 180 and 194°, and toward 200°, phase III), followed by a major southwest-trending phase (between 230 and 248°, phase IV). There are also major and minor young phases related to late deglaciation between 175 and 260°, phase V and phase VI (Trommelen et al., 2013; Trommelen, unpublished data).



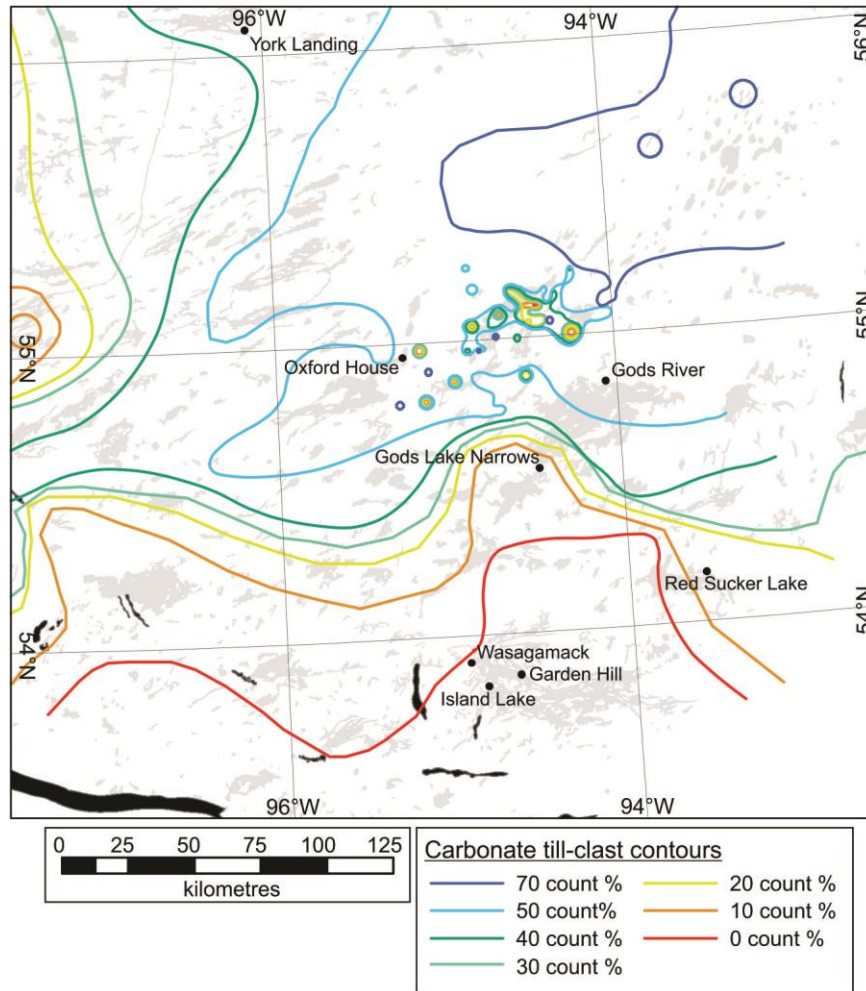
**Figure 4** Summary of ice-flow history for the Superior Province (modified from Trommelen et al., 2013).

The major and minor young deglacial phases are associated with the deglacial Hayes lobe, and recorded by streamlined landforms (Figure 4). This deglacial ice lobe was 300 by >400 km, and flowed southwest from the Hudson Bay area. This far-travelled till is correlated to the Hayes Lobe Circa 8700  $^{14}\text{C}$  BP: a large portion of this lobe was thought to have stagnated over the study area, resulting in preservation of the streamlined landforms (Klassen, 1983).



## Drift Prospecting

The regional till contains significant carbonate detritus derived from the carbonate platform in Hudson Bay, to the northeast. There are at least four different populations of sandy-silt till in the survey area, which are separated on the proportion of calcareous clasts. These are noncalcareous (<1 count %), weakly-calcareous (1–5 count %), calcareous (5–40 count %) and highly calcareous (>40 count %). While the till-types can be mapped spatially (Figure 5), detailed study in the Knee Lake and Oxford House area shows that the surface till is a heterogeneous patchy mosaic of variable carbonate concentration (Trommelen and McMartin, 2014). Hence, the data in Figure 5 are presented to serve as a guide, but detailed work is required for a specific exploration project.



**Figure 5** Hand-contoured spatial map of the distribution of carbonate-bearing clasts in till for the survey area (modified from Trommelen and McMartin, 2014).

This heterogeneous till sheet was likely formed during ice-flow phases II and IV, when the large component of allochthonous calcareous detritus (carbonate-bearing clasts, carbonate-hosted fossils, shell fragments) was transported far west and/or southwest from the carbonate platform in Hudson Bay (Trommelen 2015a; Trommelen and Ross 2014). Patches of weak to noncalcareous till were likely formed during early ice-flow phases in the Late Wisconsinan or the penultimate glaciation, when ice flowed south and southeast across the area. These sites were protected (not diluted or reworked) from the ice-flow phases that transported substantial calcareous subglacial detritus to the area. Weak to noncalcareous till is also present south of Red Sucker Lake (Nielsen 1980). While data from this area are sparse, it suggests that calcareous detritus has never been transported further south than this.

Within the calcareous till of the Knee Lake area, till-matrix geochemistry does not depict any spatial patterns or obvious dispersal trains of metals from known mineralized source outcrops (Trommelen 2015a). Weakly calcareous and noncalcareous tills are ‘more prospective’ and may be ‘truer’ indications of local mineral potential. As such, detailed attention must be paid to the distinction between total calcium (instrumental neutron activation analysis), total carbonate (Chittick or calcium-magnesium method) and/or calcium oxide (near-total digestion and inductively coupled plasma–emission spectrometry) concentrations. Additionally, calcareous till samples with moderate to elevated metal concentrations may be more prospective than non-calcareous till samples containing similar metal concentrations. To compensate for this masking effect, the two populations should not be statistically treated as a single dataset.

In the Knee Lake area, all till types occur within streamlined landforms, as well as till blankets or veneers over bedrock (Trommelen and Ross 2014). This diverse geomorphology indicates that the process of drumlinization within the deglacial Hayes Lobe (300 by >400 km) was by subglacial erosion, modification or cannibalization of pre-existing inherited sediment. As such, in the Knee Lake area, the orientation of these widespread streamlined landforms should NOT be used as an indicator of ice-flow transport direction for drift exploration.

## **SAMPLE COLLECTION**

### **Lake Sediments (*Gyttja*)**

The original lake sediment and water survey was carried out during the summer of 1986 over an area of 39,000 km<sup>2</sup> at an average density of 1 sample per 11.4 km<sup>2</sup>. Data from 3,431 lake sites for 17 elements in sediments and six variables (U, F, pH, Ca, Mg, total alkalinity) in surface lake waters were released in 1987 in GSC Open File 1358 (Geological Survey of Canada, 1987). This current open file release presents new analytical data for a subset of 2,485 lake sites derived from archived samples stored in the GSC Collections Facility located at Tunney’s Pasture in Ottawa.

Sediment samples were collected from the centre of lake basins, using a helicopter on floats (Figure 6). A bottom-valved, hollow-pipe sampler (Figure 7) 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; Figure 8). At the 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 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). Before publication, thorough inspections of the field and analytical data were made to check for any missing information and/or analytical errors.

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. A detailed description of current quality control methods used by the GSC for lake sediment samples can be found in McCurdy and Garrett (2016).



**Figure 6** A Bell 206B (Jet Ranger) helicopter on floats (inset) on approach to a typical lake (1 to 5 km<sup>2</sup>) 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.



**Figure 7** 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 8** Field card used to record site-specific field observation data in 1986.

## ANALYSIS

### Analytical Procedures (2016)

#### *Modified Aqua Regia - Inductively Coupled Plasma Mass-Spectrometry (ICP-MS)*

Samples were analysed at Bureau Veritas Commodities Canada Ltd., Vancouver, using a proprietary ‘AQ250 – Ultratrace by ICP Mass Spec.’ package with the extended packages for rare earth elements (+REE) and precious metals Pt and Pd (+PGM). For the determination of 65 elements listed in Table 1, a 0.5 gram sample was leached with a modified aqua regia solution (HCl:HNO<sub>3</sub>, 1:1). The sample solution was analysed by ICP-MS.

**Table 1** Variables in lake sediments determined by Inductively Coupled Plasma – Mass Spectroscopy (ICP-MS).

Element	Detection Limit	Units of Measurement	Element	Detection Limit	Units of Measurement
Ag	2	ppb	Na	0.001	pct
Al	0.01	pct	Nb	0.02	ppm
As	0.1	ppm	Nd	0.02	ppm
Au	0.2	ppb	Ni	0.1	ppm
B	20	ppm	P	0.001	pct
Ba	0.5	ppm	Pb	0.01	ppm
Be	0.1	ppm	Pd	10	ppb
Bi	0.02	ppm	Pt	2	ppb
Ca	0.01	pct	Pr	0.02	ppm
Cd	0.01	ppm	Rb	0.1	ppm
Ce	0.1	ppm	Re	1	ppb
Co	0.1	ppm	S	0.02	pct
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

Element	Detection Limit	Units of Measurement	Element	Detection Limit	Units of Measurement
Er	0.02	ppm	Sn	0.1	ppm
Eu	0.02	ppm	Sr	0.5	ppm
Fe	0.01	pct	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	pct
Hg	5	ppb	Tl	0.02	ppm
Ho	0.02	ppm	Tm	0.02	ppm
In	0.02	ppm	U	0.1	ppm
K	0.01	pct	V	2	ppm
La	0.5	ppm	W	0.1	ppm
Li	0.1	ppm	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
Mo	0.01	ppm			

### Analytical Procedures (1986)

#### *Analytical Procedures for Loss-on-Ignition (1986)*

Loss-on-ignition (Table 2) 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 (1986)*

pH (Table 2) in lake water samples was determined using a glass-calomel combination electrode and pH meter. Fluoride (Table 2) 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.

**Table 2** Lake sediment and water variables determined in 1986 and included in this report (Appendix 1).

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

Analytical results are presented in an Excel® workbook included with this report: **Appendix 1 FIELD OBSERVATIONS & ANALYTICAL DATA.xlsx**. The four worksheets in this file are described below in Table 3.

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

Worksheet	Contents
<b>Field Observations (1986)</b>	Site-specific field observations and location coordinates
<b>Reanalysis ICP Data (2016)</b>	ICP-MS analytical data for lake sediment samples
<b>Analytical Data (1986)</b>	Data for F and Loss-on-Ignition in lake sediments and pH of lake waters
<b>Filtered Data (40 km)</b>	Values for each element ranked as percentile with respect to all values ('neighbours') within a circle of 40 km radius centred on each site

## 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 2 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 A2-1 through A2-5 in Appendix 2 (Quality Control) can be used to estimate the quality of analysis for elements listed in Tables 1 and 2 above. Elements are grouped based on their position in the Periodic Table. Data used for calculations are included in separate worksheets described in Table 4.

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

Worksheet	Contents
<b>A2-1 Accuracy – LKSD-2</b>	Compares accepted values for Certified Reference Material LKSD-2 with results from samples inserted into Manitoba sample suite
<b>A2-2 Accuracy – LKSD-3</b>	Compares accepted values for Certified Reference Material LKSD-3 with results from samples inserted into Manitoba sample suite
<b>A2-3 Accuracy – LKSD-4</b>	Compares accepted values for Certified Reference Material LKSD-4 with results from samples inserted into Manitoba sample suite
<b>A2-4 Precision</b>	Provides an estimate of precision using analytical duplicate pairs
<b>A2-5 ANOVA</b>	Simple pair ANOVA estimates proportion of total variability due to each of sampling and analysis
<b>Certified Reference Data</b>	CRM analytical data used to calculate estimates of accuracy in Tables A2-1 to A2-3
<b>Analytical Duplicate Data</b>	Analytical duplicate pair data used to estimate precision listed in Table A2-4
<b>Field Duplicate Data</b>	Field duplicate pair data used to calculate ANOVA statistics shown in Table A2-5

### Accuracy

Accuracy of analytical data was evaluated by inserting Canadian Certified Reference Lake Sediments 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-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 A2-1 through A2-3 of Appendix 2 accepted means and standard deviations (MEAN  $\pm$  SD) for control reference standards 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 means and standard deviations reported by Lynch (1990, 1999) were derived from results received from different laboratories using a concentrated HNO<sub>3</sub>-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 the re-analysis of lake sediments from east-central Manitoba reported here. Lower detection limits (LDL) for each element estimated by Bureau Veritas are listed. A percent Relative Standard Deviation (RSD %) is calculated for each element with values above the detection limit. 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 the 'rgr' package running under the R system (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 (LKSD-2, -3, -4), Ta (LKSD-2, -3, -4), Pt (LKSD-2, -3) and B (LKSD-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, -3, -4), Hf (LKSD-2, -4), W (LKSD-3), Re (LKSD-2, -3, -4), Ge (LKSD-2, -4), Te (LKSD-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. The estimation of the analytical precision follows the procedure of Youden (1951) for up to 144 duplicates where both results were above the respective detection limits. The resulting numerical estimate of precision for variables is listed in Table A2-4 (Precision) in **Appendix 2 QUALITY CONTROL.xlsx** as a percent 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 A2-4.

Elements with precisions poorer than 20% in Table A2-4 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, W, Re and Hg. Three elements, Ca, Pb and U, have mean values that are above the lower detection limits and concentrations in all analytical duplicate pairs are detectable, however, analytical precision is unexpectedly poorer than 20% for these elements. The mean content of organic material determined by loss-on-ignition in re-analysed Manitoba lake sediment samples, determined in 1986 and included with this report, is 46.2 %, a value that is relatively high compared with surveys at higher latitudes. As geochemical relationships between elements in lake sediments are affected by the amount of organic material (Garrett et al., 1990; Amor, 2015) it may be the case that analytical precision is influenced as well in organic-rich sediments. Results for Au may be influenced by the particulate nature of gold ('nugget effect') and should be considered accordingly (Harris, 1982). For the elements Ta and Ge, less than two pairs of analytical duplicates have both values above detection and no statistics are listed.

### 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 as well as 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.

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 A2-5 in **Appendix 2 QUALITY CONTROL.xlsx** shows results from an ANOVA undertaken on up to 146 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).

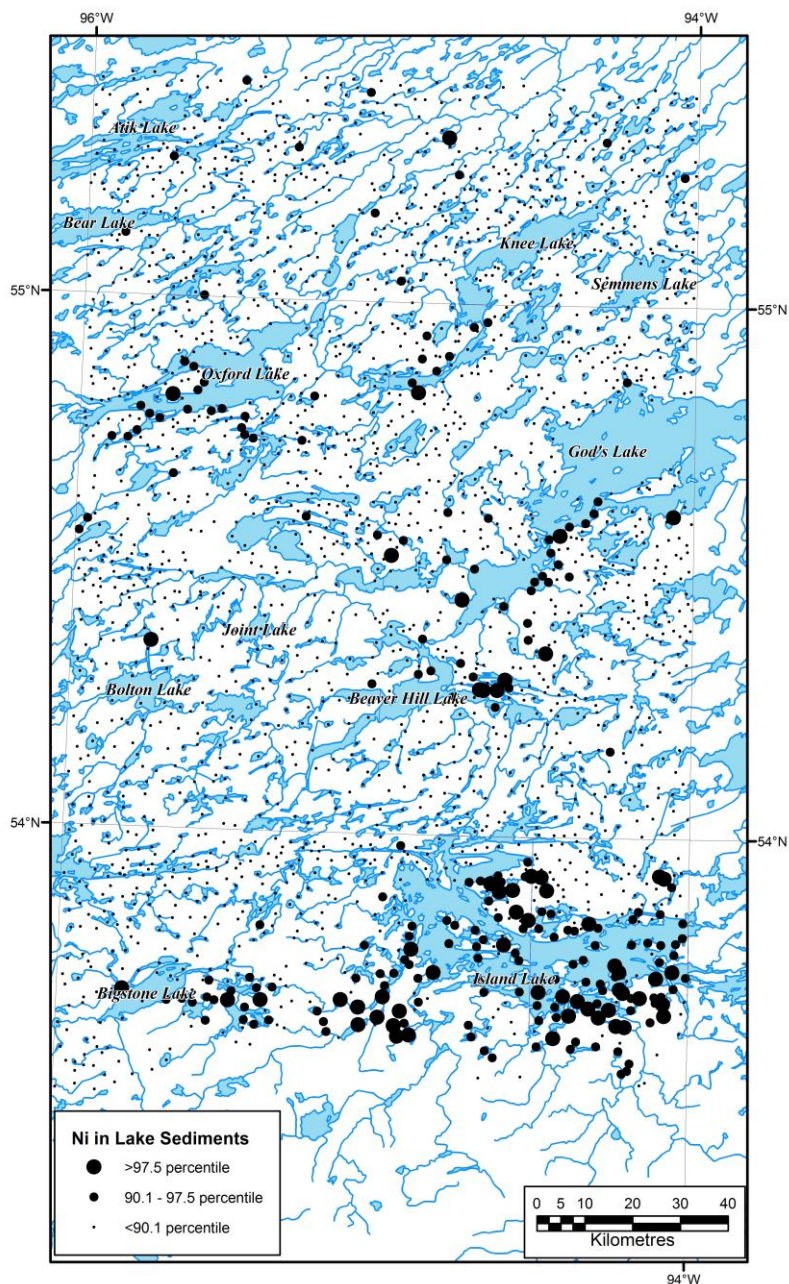
The Analysis of Variance (ANOVA) of field duplicates partitions variability into two components, 'Between Sites' and 'At Sites' in Table A2-5. The variance ratio, F, is calculated in 'anova2' to gauge whether the variance 'within' is significantly smaller than the variation 'between'. As a 'rule of thumb' 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 A2-5 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 W, Pt, Au, In, and Te. Because the number of pairs of samples available for statistical calculations is <30 for Pt, In, B and Te, results for these elements should be treated with caution. For the elements Pd, Ta and Ge, 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.

### DISPLAYING GEOCHEMICAL DATA

Methods of graphically presenting geochemical data for individual sites over large areas with diverse bedrock and surficial geology generally rely upon procedures that accentuate the highest values within the entire data set, using percentile classes based on an element's frequency distribution or setting a threshold equal to the arithmetic mean plus two

standard deviations<sup>a</sup>. However, a strong geochemical response over bedrock lithologies such as black shales (e.g. U, Mo, V, Cu) or ultramafic rocks (e.g. Ni, Cr) can subdue or mask more subtle but no less significant anomalies over other surfaces, drawing attention away from local maxima with lower background values (Figure 9).



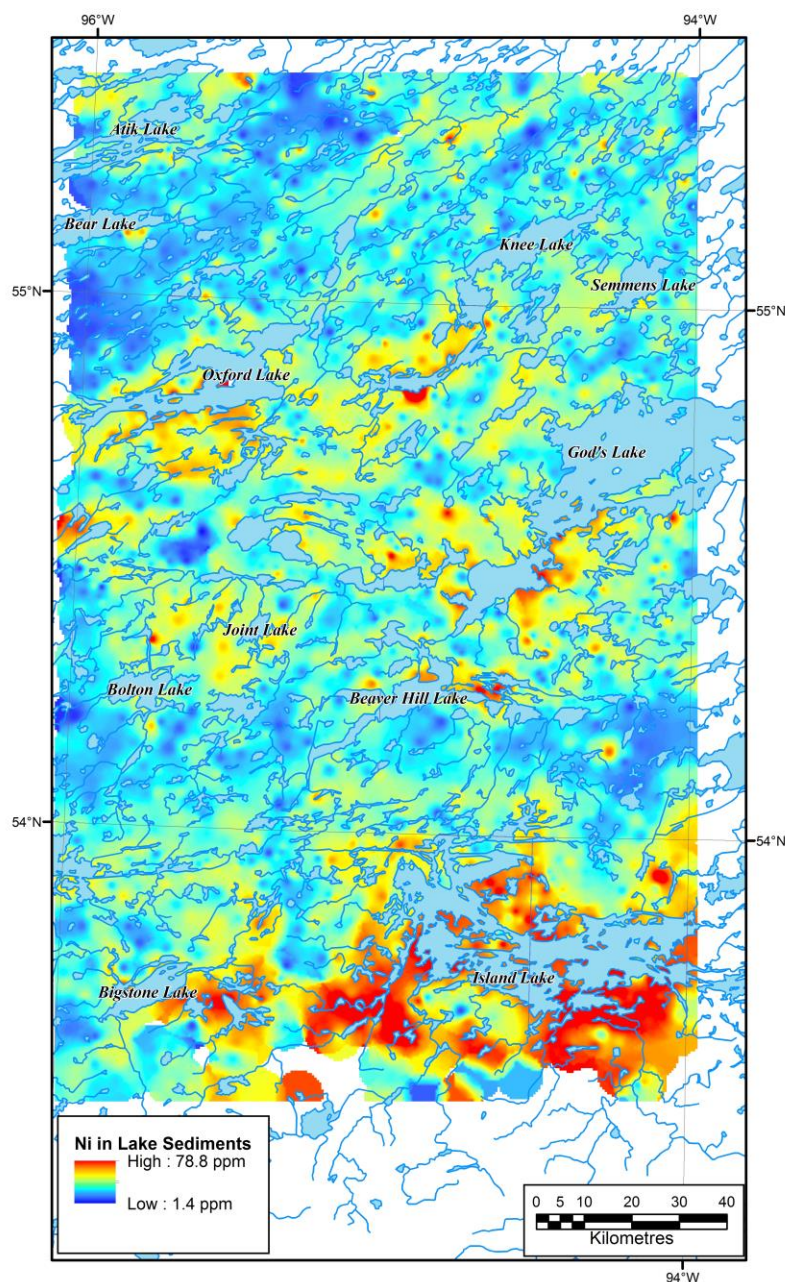
**Figure 9** The position of each Ni value in the entire data set is expressed as a percent of the data distribution and divided into three classes, with the highest 2.5 percent of the values assigned the largest spot. In this example, the highest values occur over exposed mafic volcanic and amphibolitic rocks in the south (Figure 2). Ni values over similar rocks overlain by till or lacustrine materials are subdued or masked (Figure 3).

Another common approach uses some form of moving average or median with an interpolation technique (kriging, inverse distance weighting, etc.) to produce surface maps from the raw data (Figure 10). A two-dimensional ‘window’ is moved across the map and all points falling within the window are used to estimate a value at the centre of the

<sup>a</sup> This was an early approach to the identification of anomalous geochemical values that is still occasionally seen in reports. See Garrett (1989) and Amor (2013) for explanations why this method should be avoided.



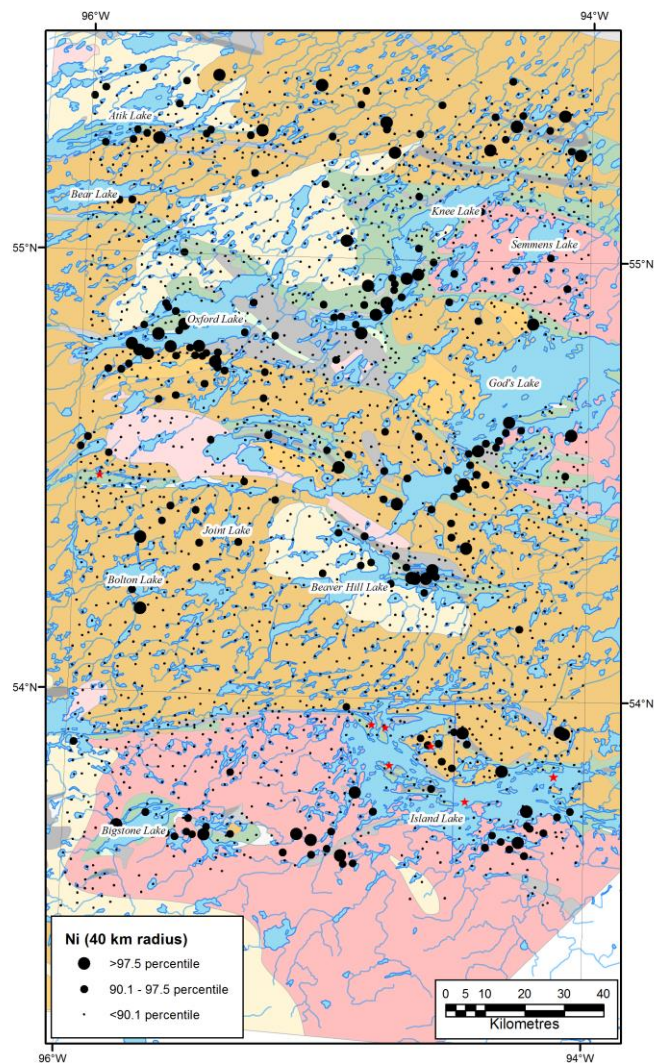
window. The data points on the irregular sampling grid are used to estimate new values on a regular grid which are then used to assign a colour to a raster representing the average or median value. Although these maps can highlight regional trends, local variability is lost as are data at individual sampling points.



**Figure 10** In this figure, a moving ‘window’ with dimensions of 5 km by 5 km is used to estimate a value at the centre of the window from the average of all data points within the window. Within the zone of influence, the value of each point is weighted so that points nearest the centre of the window have relatively high weights while points farthest away exert little influence on the estimated value.

In order to reduce the influence of regional extremes or low variance on individual points or local maxima (Figure 9), a filtering algorithm is used to highlight local maxima in regional datasets, whether local backgrounds are high or low (Amor, 2012; Amor, 2013). The concentration of each element for individual samples is ranked with respect to all of its ‘neighbours’ within a specified radius. The rank is converted to a percentile value that is then assigned back to the sample. The process is repeated for all of the samples in a regional dataset (Amor, 2013). In the example shown below (Figure. 11) for Ni in lake sediments, coordinates at each site identify the centre of a search circle with a radius of 40 km. Within this circle, each sample qualifies as a ‘neighbour’. Values for Ni are sorted and the value of the sample at the centre of the

circle is ranked within the set of values. The rank is converted to a percentile and assigned to this sample. The process is repeated for all the samples. The resulting map narrows the focus in areas of known Ni mineralization around Island Lake (MB mineral occurrence database), highlights elevated Ni values in other explored areas adjacent to Beaver Hill Lake, God's Lake, Oxford Lake and southwest of Knee Lake, and suggests potential in new areas with little or no recorded mineral occurrences east of Bigstone Lake, northeast of Knee Lake, and east of Atik Lake for follow-up work.



**Figure 11** Values for Ni in lake sediments at each sample site represent the rank with respect to all the values within a circle of radius 40 km, preserving location information and highlighting local maxima. Mineral occurrences with Ni as a commodity are marked as red stars.

## HIGHLIGHTS

### Gold

Five gold deposits (indicated by a yellow star) are recorded within the survey area shown on Figure A3-3 (Au) in **Appendix 3 ELEMENT DISTRIBUTION MAPS**, two at Island Lake, and one each at God's Lake, Knee Lake and Oxford Lake. Multiple showings are recorded in the MGS Mineral Occurrence Database (<http://web33.gov.mb.ca/mapgallery/mgg-gmm.html>) around Island Lake, God's Lake and Atik Lake (Figure A3-3 (Au)). Clusters of elevated gold values in lake sediments not associated with known deposits or occurrences are present around Joint Lake, Bigstone Lake and northeast of Knee Lake. Given the positive geochemical response of gold around known deposits in the survey area, these elevated values warrant further investigation.

Most of the silver showings listed in the MGS database are located around Island Lake and there is a positive geochemical response in the lake sediments in this area (Figure A3-1). Silver can be a useful pathfinder element for other metals such as gold, lead-zinc and nickel and there are clusters of elevated silver values in the proximity of Beaver Hill Lake, west Oxford Lake and southeast of Knee Lake that bear further investigation.

### **Base Metals (Ni, Cu)**

Geochemical results for nickel shown in Figure A3-18 (Ni) narrow the focus in areas of known nickel mineralization around Island Lake, highlights elevated nickel values in other explored areas adjacent to Beaver Hill Lake, God's Lake, Oxford Lake and southwest of Knee Lake, and suggests potential in areas with few or no recorded mineral occurrences east of Bigstone Lake, northeast of Knee Lake, and east of Atik Lake.

In addition to multiple recorded occurrences at Island Lake, God's Lake and northwest of Joint Lake there are a number of other areas where elevated copper values are clustered, around Beaver Hill Lake, Oxford Lake and Knee Lake (Figure A3-12 (Cu)). Further investigation is suggested in these areas.

### **Rare-Earth Elements and Rare Metals**

The existence of lithium pegmatites has recently been reported in the survey area by the MGS at God's Lake and southwest of Knee Lake (Figure 2). Typically, these pegmatites are enriched in lithium, cesium, tin, rubidium and tantalum and form tabular dikes, sills, lenticular bodies or irregular masses. Individual pegmatites are small, usually less than 1 km<sup>2</sup>, but they can occur in groups covering up to a few tens of square kilometres (Bradley et al., 2013).

Figure A3-14 (Li) includes the locations of two reported pegmatites in the survey area along with geochemical concentrations of lithium in lake sediments. The God's Lake pegmatite is associated with elevated lithium in only one adjacent lake, not surprising, given the typically small surface areas of pegmatites. The pegmatite southwest of Knee Lake is not associated with elevated lithium; however the adjacent lake sediment sample site is elevated in both cesium (Figure A3-11 (Cs)) and beryllium (Figure A3-5 (Be)), both geochemical indicators of pegmatites.

Carbonatites are intrusive carbonate mineral-rich igneous rocks that may contain economic or anomalous concentrations of rare earth minerals. The geochemical signal is typically given by elevated thorium, barium, niobium, copper, iron and associated rare earth metals (Verplanck et al., 2014).

Two carbonatites identified by the MGS are shown in Figure 2, at Knee Lake and Oxford Lake. The geochemical signal at each site is variable, with niobium (Figure A3-17 (Nb)), cesium (A3-11 (Cs)) and thorium (Figure A3-26 (Th)) elevated in lake sediments sampled near the Oxford Lake carbonatite and niobium and beryllium elevated southwest of Knee Lake. Ytterbium, a heavy rare-earth element (Figure A3-29 (Yb)), is elevated at both sites and the cluster of elevated sites extends well beyond the immediate area around the location of the carbonatites.

## **SUMMARY**

Lake sediments originally collected in 1986 from 2,485 sites in east-central Manitoba, within the Superior geological province of the Canadian Shield, were re-analyzed for 65 elements in 2016.

In summary:

- Used in combination with bedrock and surficial geology maps, lake sediment geochemistry is a useful tool for mineral exploration for Au, Cu-Ni-Ag and rare-earth elements in the northern Superior geological province of Manitoba
- Geochemical maps for gold and base metal concentrations in lake sediments outline significant areas for follow-up
- Several elements (Li, Be, Cs, Nb, Th, Yb) highlight areas with potential for rare metals associated with carbonatites and Li-pegmatites (Be, Li, Ta, Nb, Sn, Cs, Zr, REEs)



- Rare-earth metal targets (Li-pegmatites and carbonatites) are small; only one or two sites might be elevated relative to surrounding lakes

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