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**GEOLOGICAL SURVEY OF CANADA
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2024

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Abstract

This report presents new geochemical data, quality assurance and quality control (QA/QC) results of the reanalysis of lake-sediment samples collected from northern Manitoba (NTS 64-J and 64-K). The original sampling programs were conducted in 1975, whose results are presented in Geological Survey of Canada open files 320 (NTS 64-J) and 321 (NTS 64-K). Lake-sediment samples from 2 005 sites were reanalyzed between 2020 and 2022 and included (i) samples from 1 798 regional-scale sites, covering an approximate area of 26 000 km², with an average density of one sample per 13 km², and (ii) 208 samples from sites from more detailed sampling programs conducted in the same year, in the northwest and northeast corners of NTS 64-J and NTS 64-K, respectively, with an average density of one sample per 7 km² over an area of approximately 3,000 km². In the recent re-analysis, samples were measured for 65 elements via modified aqua-regia – ICP-MS, and for 35 elements by INAA. To ensure high-quality geochemical data, the data were evaluated for accuracy, precision, and fitness-for-purpose. QA/QC results have identified several elements to be monitored carefully for future analyses.

1 Introduction

National Topographic System (NTS) map areas 64-J and 64-K were Manitoba's first (in 1975), along with 64-N and 64-O, to be sampled as part of the National Geochemical Reconnaissance (NGR) program of the Geological Survey of Canada (GSC). The geochemical data were released in GSC Open Files 320 (Hornbrook *et al.*, 1976a) and 321 (Hornbrook *et al.*, 1976b), respectively. Lake-sediment samples were analyzed for Zn, Cu, Pb, Ni, Co, Ag, Mn, Mo and Fe, by flame atomic-absorption spectrometry (FAAS) after reverse aqua regia (3 HNO₃: 1 HCl), also called Lefort Acid digestion; for Hg by cold-vapour atomic-absorption spectrometry (CVAAS), for As by colorimetry, for U by delayed neutron counting (DNC) and loss-on-ignition (LOI) by gravimetry. Lake-water samples were not collected during this work.

With recent improvements in analytical instrumentation, particularly lower detection limits, improvements in analytical precision, and the increase in the number of elements available for analysis, reanalysis of these samples was warranted.

Funding for the modern reanalysis was provided by the GEM-GeoNorth Program, which aims to advance regional geo-mapping of Canada's North to promote sustainable resource exploration and development (Geological Survey of Canada, 2018; Lebel, 2020). Geoscience knowledge produced by the GEM-GeoNorth Program enables governments and communities to make informed decisions regarding the development of resources (Lebel, 2020). The program (2020-2027) is a continuation of the successful Geo-Mapping for Energy and Minerals (GEM) program (2008-2020).

The study area covers about 26 000 km² within NTS map sheets 64-J and 64-K (Figure 1). The region is considered to have significant potential for mineralization of many different deposit types (e.g. Corrigan *et al.*, 2007), including U and critical elements such as Cu, Li and rare-earth elements. Reanalysis using modern analytical techniques provides vital data for mineral exploration and other land uses, and for establishing environmental baselines.

Reanalysis data for lake-sediment samples in two additional adjacent NTS sheets are being published as part of the overall GEM GeoNorth program reanalysis activities for Manitoba. Open File 8948 (Bourdeau, 2024) reports data for NTS map sheet 64-F. A third dataset, covering NTS 64-G, will be published later in 2024.

This open file report presents reanalysis data and QA/QC results for 67 elements: 65 analyzed by induction-coupled plasma mass spectrometry (ICP-MS) and 35 by instrumental neutron activation analysis (INAA) from a total of 2 005 lake sediment sample sites. The results presented here also include analyses originally performed on the samples, for twelve elements plus loss-on-ignition (LOI).

With the publication of this report, we are making available high-quality geochemical data to provide information for the people of Manitoba in general, and northern communities in particular, to make informed decisions about their land, economy and society, and to highlight areas with potential for critical elements used for building renewable energy technologies. Evaluation of geochemical data using adequate quality assurance and quality control (QA/QC) procedures is essential before



Figure 1. Location of the NTS sheets where the re-analyzed lake sediments were originally collected.

interpreting the data (Reimann *et al.*, 2008). The QA/QC process is designed to identify and, where possible, remediate quality issues so that potential users of geochemical data can be fully informed

regarding the data quality. The QA/QC report follows the methodology presented in McCurdy and Garrett (2016) to evaluate the accuracy and precision of geochemical data.

2 Sample Locations

Sample locations are shown in Figures 2 and 3. On checking the original location data, it became apparent that the locations of many samples, as indicated by their previously recorded latitudes and longitudes, did not plot within the lakes to which they had been assigned in the hard-copy sample-location maps in the original open-file releases. There are many possible reasons for this problem, but the two most likely ones are inaccurate geodetic baseline data, and inaccurate digitizing from the field maps. Also, sample collection predated the availability of GPS technology, and the original coordinates of the samples would have been best estimates from paper copies of NTS topographic maps.

After considerable deliberation, it was decided not to undertake the complex task of correcting the sample locations in the new data release. In a survey at this scale any interpretation of the analyses of the sediments and waters would, except in the largest lakes, assume that each analysis was representative of the lake as a whole, rather than at the sample's precise location. Indeed, when regional geochemical data of this kind are examined and interpreted, the symbol assigned to the analysis is frequently larger than the lake itself (e.g., Amor *et al.*, 2019).

3 Bedrock geology of NTS 64-J and 64-K

The sampled area is underlain by the Churchill Structural Province of the Canadian Shield, comprising approximately equal portions of the Chipewyan Domain, to the south, and the Seal River Domain, to the north, with limited occurrence of rocks of the Wollaston Domain in the northwest corner of 64-K (Figure 4).

A summary of the bedrock geology of the sampled area (Figure 5) is presented below with an emphasis of the most recent work carried out by the Manitoba Geological Survey (MGS). Compilations for the individual map sheets can be found in in Schledewitz (1986). The most recent geology compilation at 1:250 000 scale was released in 2022 (Manitoba Geological Survey, 2022).

The Precambrian rocks of the Tadoule Lake and Whiskey Jack Lake regions (NTS 64-J and K, respectively) encompass the northern margin of the Chipewyan domain and the southern margin of the Seal River domain.

The Chipewyan domain is interpreted as an amalgamation of granitoid plutons in an Andean-type continental magmatic arc setting (e.g., Fumerton *et al.* 1984). Emplacement has been considered to have taken place in a short time interval between 1.86 and 1.85 Ga on the margin of the Hearne craton (e.g., Fumerton *et al.*, 1984, Meyer *et al.*, 1992). However, Martins *et al.* (2022) interpret magmatism to have continued in the Chipewyan domain until at least ca. 1.83 Ga.

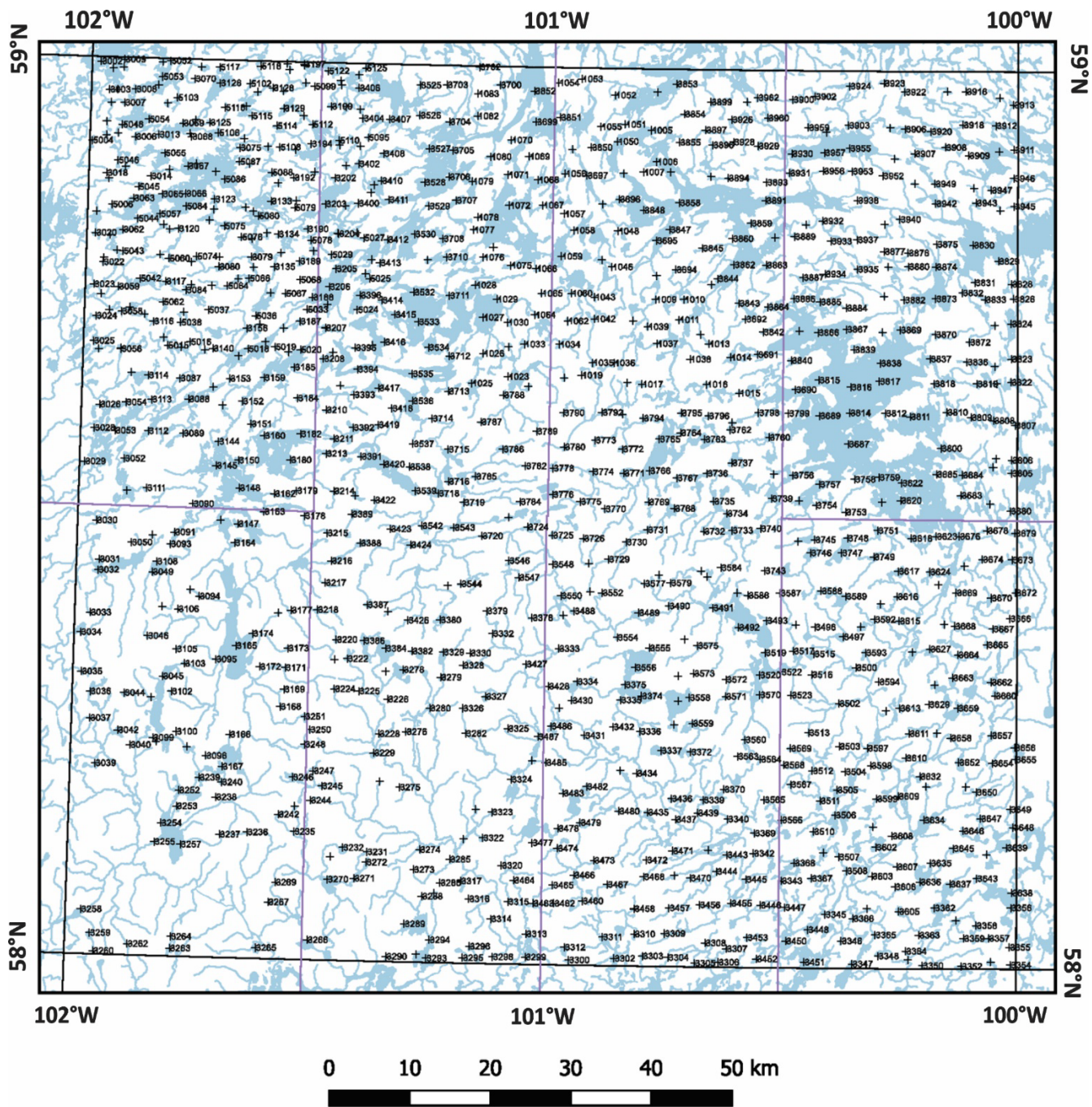


Figure 2. Sample-location map for NTS map area 64-J.

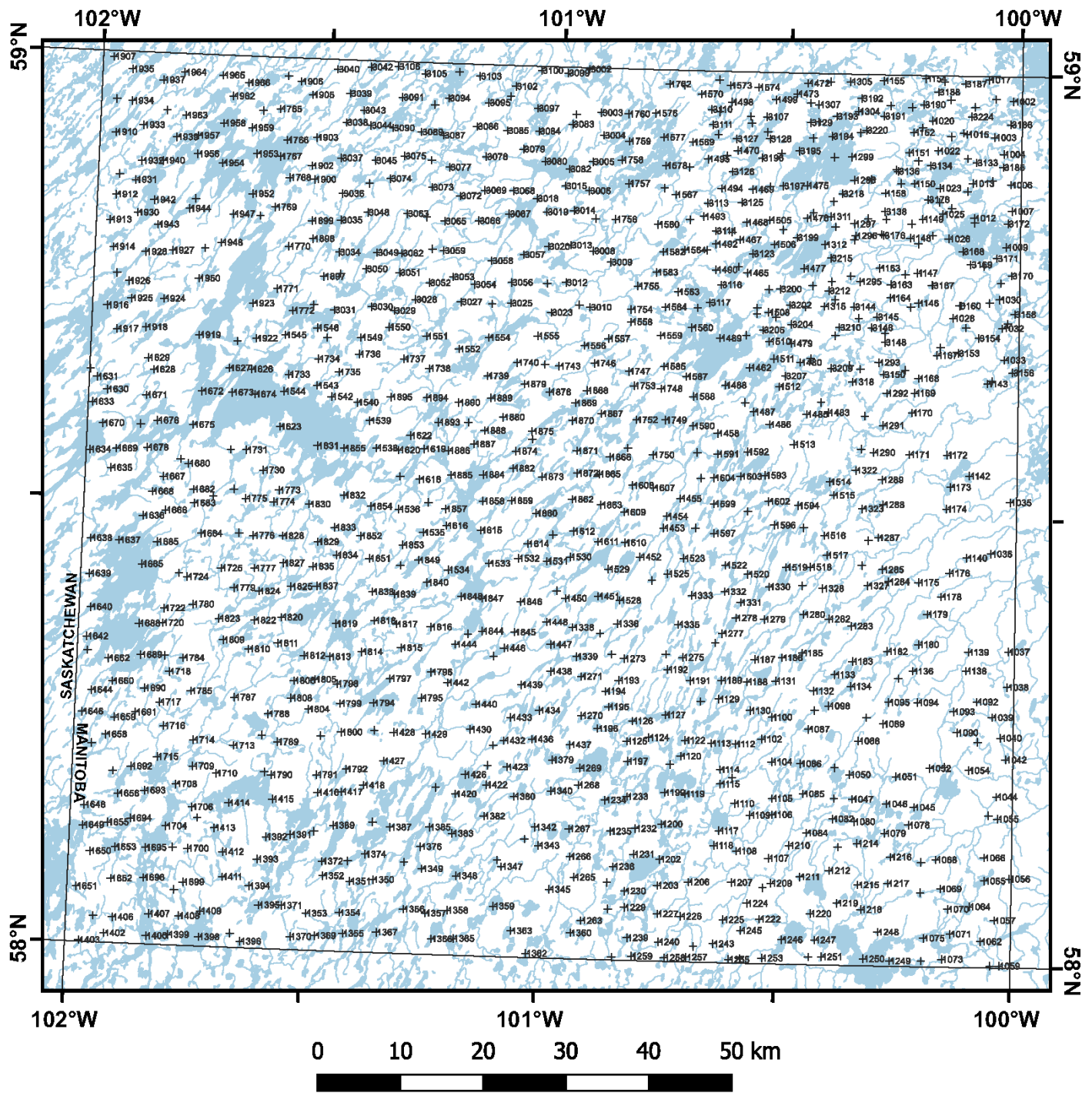


Figure 3. Sample-location map for NTS map area 64-K.

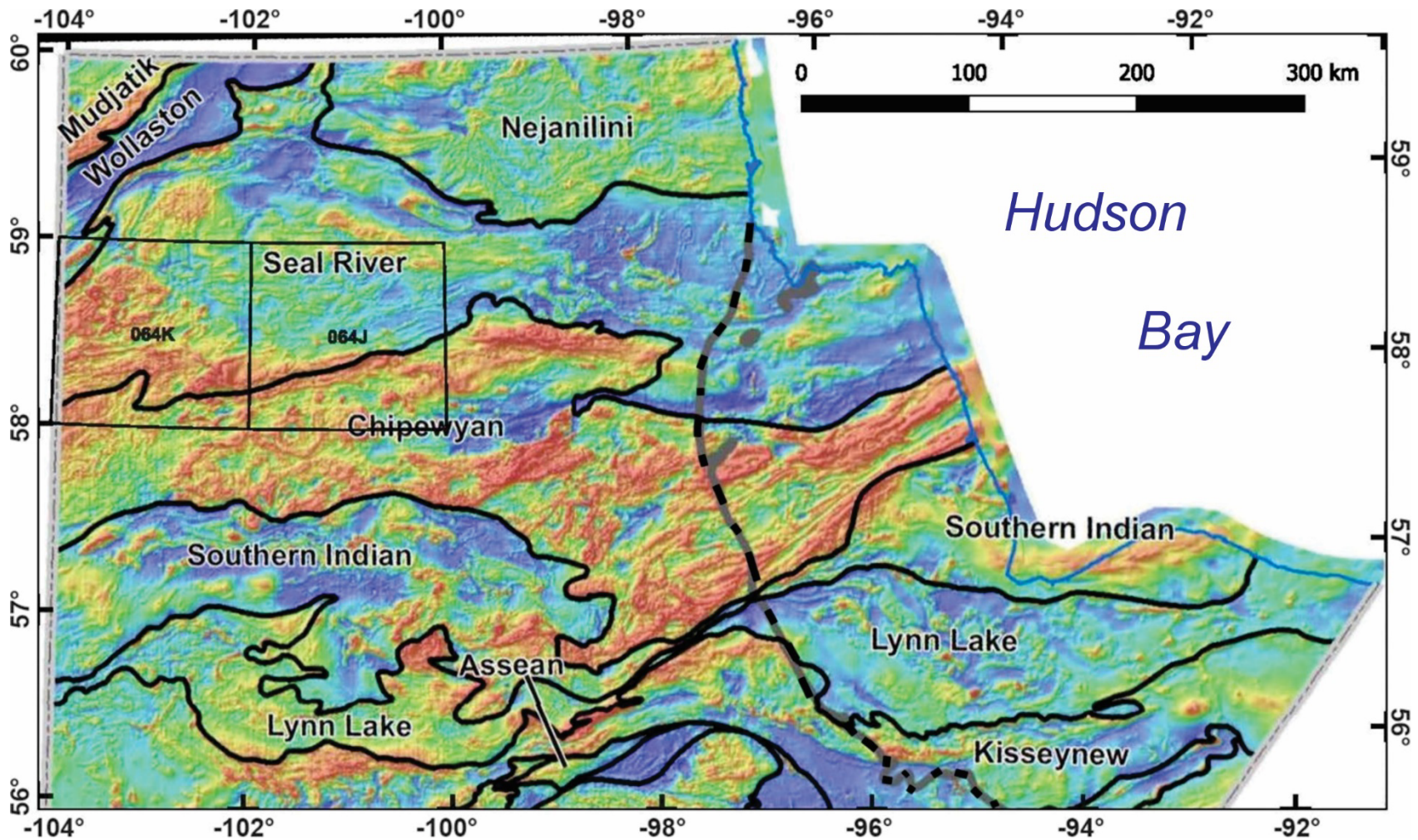


Figure 4. Geologic Domains of northern Manitoba superimposed on total-field magnetics (from Manitoba Geological Survey, 2022). Reds and yellows indicate higher magnetic susceptibility; blues and greens indicate lower magnetic susceptibility. Dashed line indicates edge of Paleozoic cover.

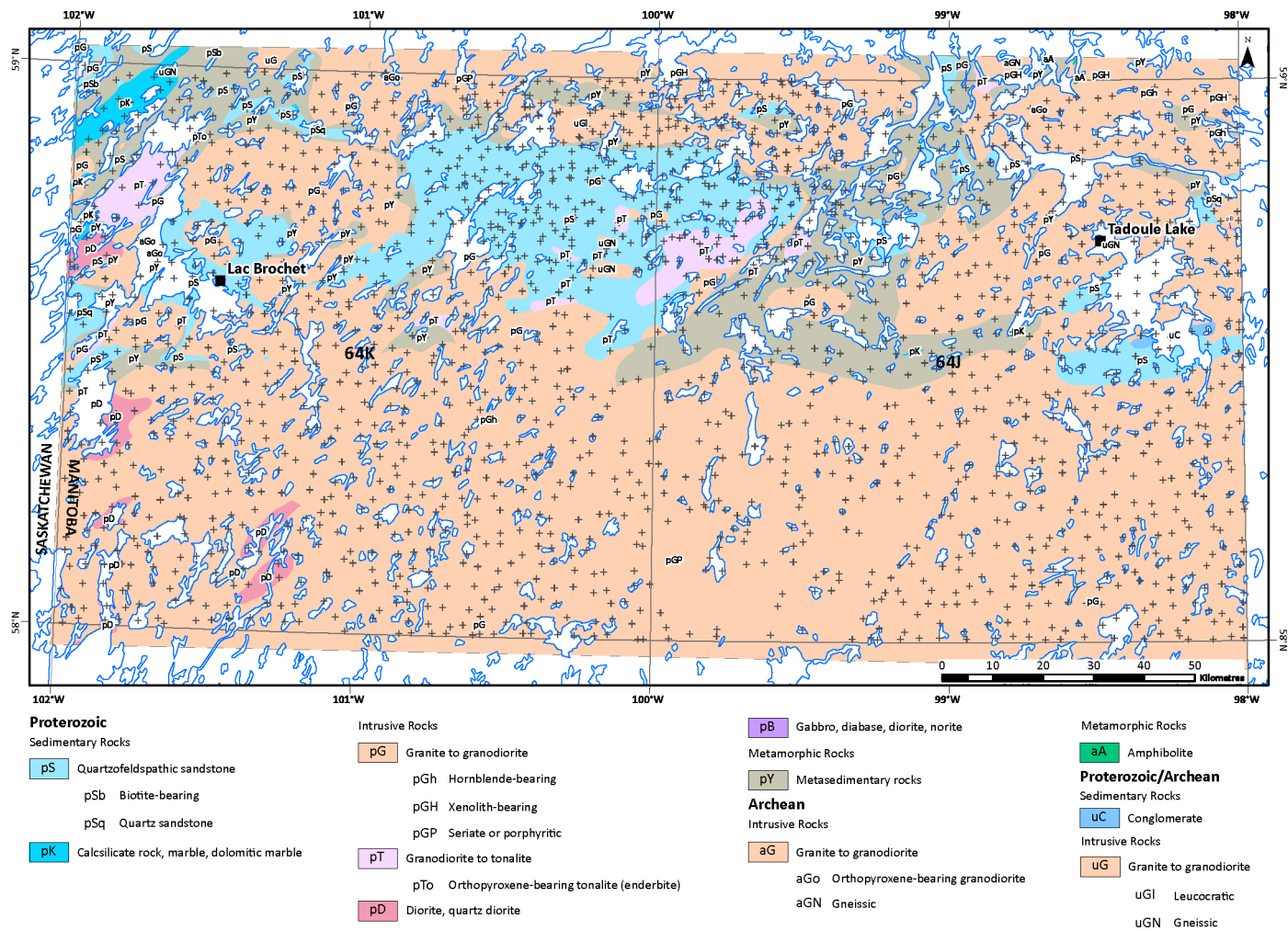


Figure 5. Geological map of the area of coverage (from Manitoba Geological Survey, 2022).

The Seal River domain consists of Neoproterozoic basement rocks of the Hearne craton, overlain and infolded with Paleoproterozoic rift, passive margin, and foreland basin sedimentary sequences of the Wollaston Supergroup. The Neoproterozoic rocks consist dominantly of gneissic granitoids. The Wollaston Supergroup rocks consist of variably metamorphosed wacke to mudstone with minor impure quartzite, rare marble and calc-silicate, conglomerate, and well-bedded arenite to calcarenite (Murphy and Carlson, 2009; Kremer *et al.*, 2010). The metasedimentary rocks are likely part of a <2.5 Ga passive margin–continental rift assemblage and a <2.05 Ga, >1.98 Ga marine deltaic assemblage (sequences 2 and 3, respectively, of Anderson *et al.*, 2010; Böhm and Rayner, 2024). Intrusive rocks, which are interpreted as Hudsonian (ca. 1.83 Ga) based on field relationships with the metasedimentary rocks, are widespread and range in composition from tonalitic to granitic, with rare leucogabbro dikes (Murphy and Carlson, 2009; Kremer *et al.*, 2010). Areas of moderate to intense alkali-calcic metasomatism occur in the Misty Lake area in the northwest corner of 64-K, and result in partial to complete replacement of various host rocks by assemblages containing albite, clinopyroxene and clinoamphibole with or without fluorite, calcite and scapolite (Kremer *et al.*, 2010).

4 Surficial Geology of NTS 64-J and 64-K

Surficial geology within the study area was mapped in the 1980s and published as a series of 1:250 000 scale black and white preliminary maps (Figure 6; Dredge *et al.*, 1982; Richardson *et al.*, 1982). Initial mapping was finalized as reconnaissance-scale 1:500 000 scale colour maps (Dredge *et al.*, 1985, 2007). Given the lack of detail, any local-scale work to follow-up on lake-sediment geochemistry results should also include new detailed local-scale mapping of the surficial geology.

Using the reconnaissance-scale surficial geology mapping, the western half of the study area is dominantly covered by till (69.6%) and organics (15.4%), with only about 4.9% bedrock outcropping at surface (Table 1). Contrastingly, the eastern half of the study area is covered by a mix of lacustrine sediments (26.1% sandy and 10% clayey), till (29.4%) and organics (27.4%, Table 1). Bedrock outcrops at map-scale are even rarer in the eastern half of the study area (2.1%). The organic-covered area (about 27.4% of NTS 64-J) is likely also covered by lacustrine sediments. These sediments were deposited within Lake Agassiz, a large proglacial lake that once covered the entire study area (cf. Teller and Leverington, 2004). At its greatest extent, Lake Agassiz may have covered an area of 440 000 km² (Fisher *et al.*, 2002) meaning that those lacustrine sediments could have been sourced from far away. Till, a diamict deposited directly by a glacier, underlies the lacustrine sediments across most of the study area. In some areas, extensive wave-washing by Lake Agassiz has completely removed the underlying till or turned it into a sand and gravel lag.

Drift exploration using lake-sediment geochemistry is most likely to be successful in areas with low relief and disorganized drainage systems (Friske, 1991; Davenport *et al.*, 1997), where sediment cover is thin and derived from the underlying bedrock. Hence, if the reconnaissance-scale mapping is correct, lake-sediment geochemistry will mostly reflect the chemistry of the underlying Lake Agassiz sediments or the till, and not that of local bedrock. As such, any local-scale work to follow-up on these reanalyses should include new detailed local-scale mapping of the surficial geology.

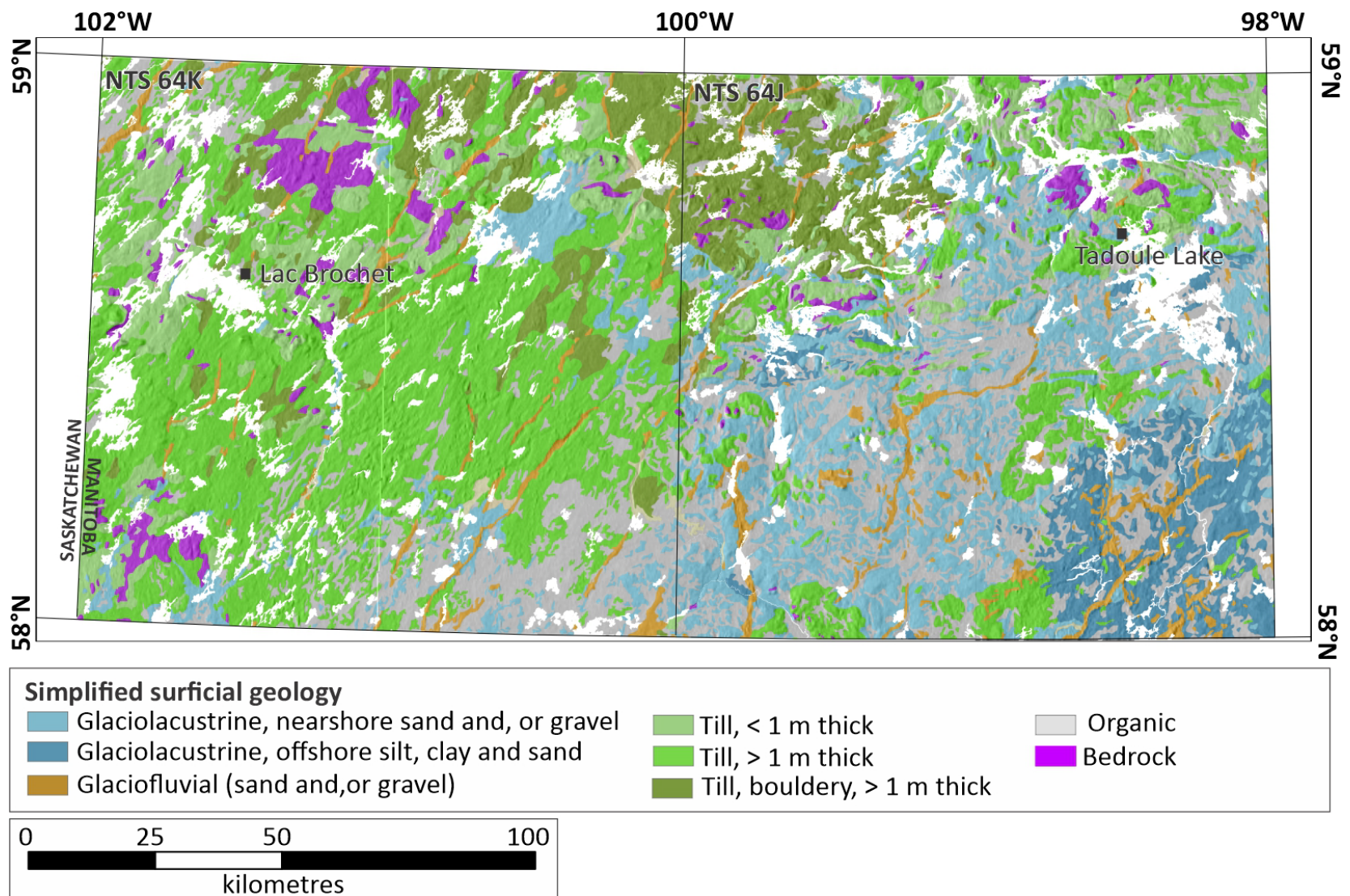


Figure 6. Simplified surficial geology of the area of coverage (Dredge et al., 1985, 2007).

Table 1. Relative cover of surficial sediments in NTS 64-J and 64-K.

Surface sediment type	NTS 64J		NTS 64K	
	% of map area	Area covered km ²	% of map area	Area covered km ²
Bedrock	2.1	269	4.9	639
Till	29.4	3821	69.6	9035
Organic	27.4	3561	15.4	1998
Lacustrine - sandier	26.1	3388	6.1	796
Lacustrine - clayier	10.0	1294	0.0	0.0
Glaciofluvial	4.8	627	3.4	444
Fluvial	0.1	18	0.5	71
Total %	100.0	12979	100.0	12983

Attention must be paid to the variety and thickness of Quaternary sediments in the area, and the data analyzed with these factors in mind.

At least six ice-flow phases are recorded in parts of the study area. Ice-flow indicators used to make these conclusions include erosional outcrop-based ice-flow indicators like striations (Figure 7) and streamlined-landform flowsets (Figure 8), which are groups of linear landforms that form parallel to ice-flow (Gauthier *et al.*, 2019).

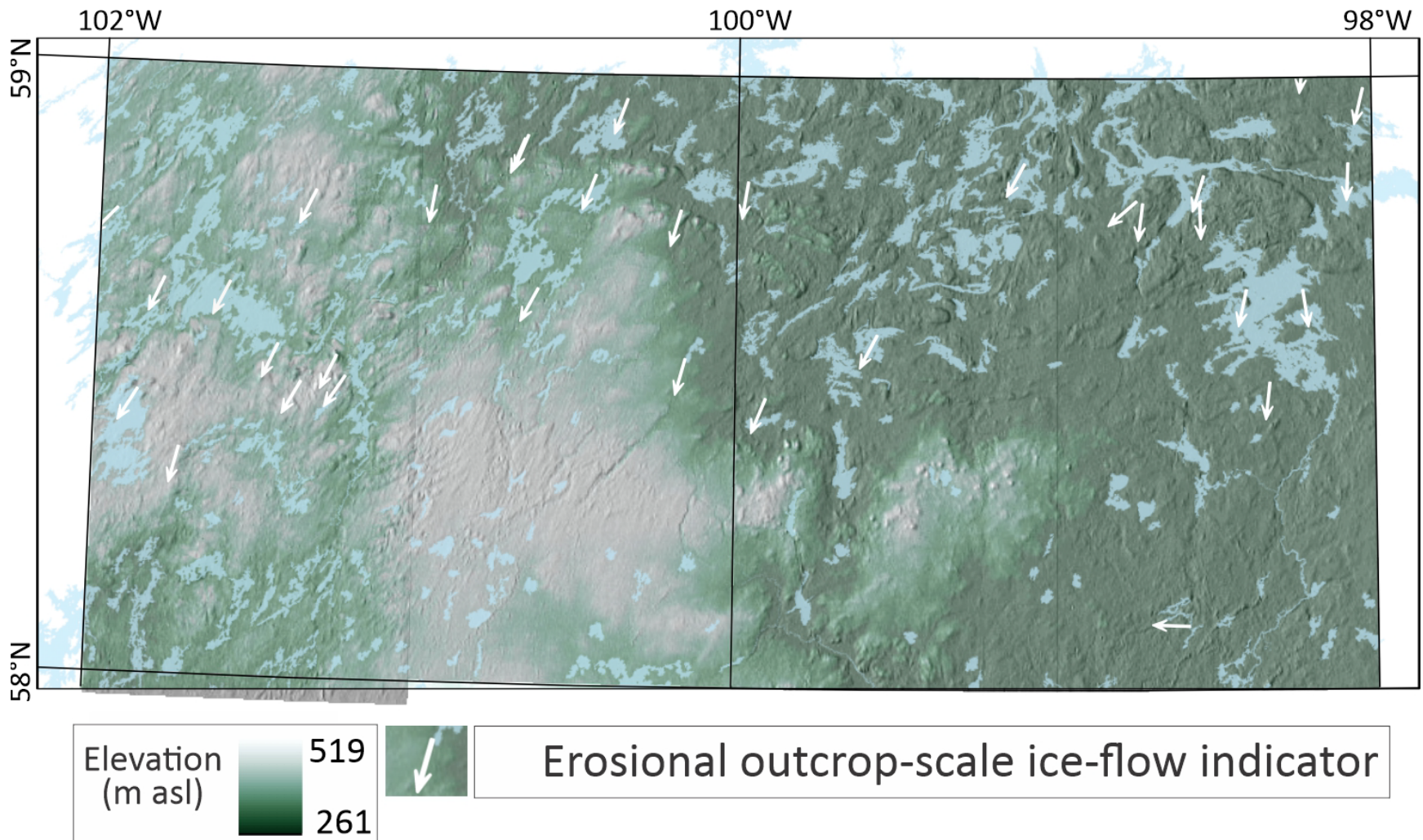


Figure 7. Digital terrain map of the area of coverage with erosional ice-flow indicators.

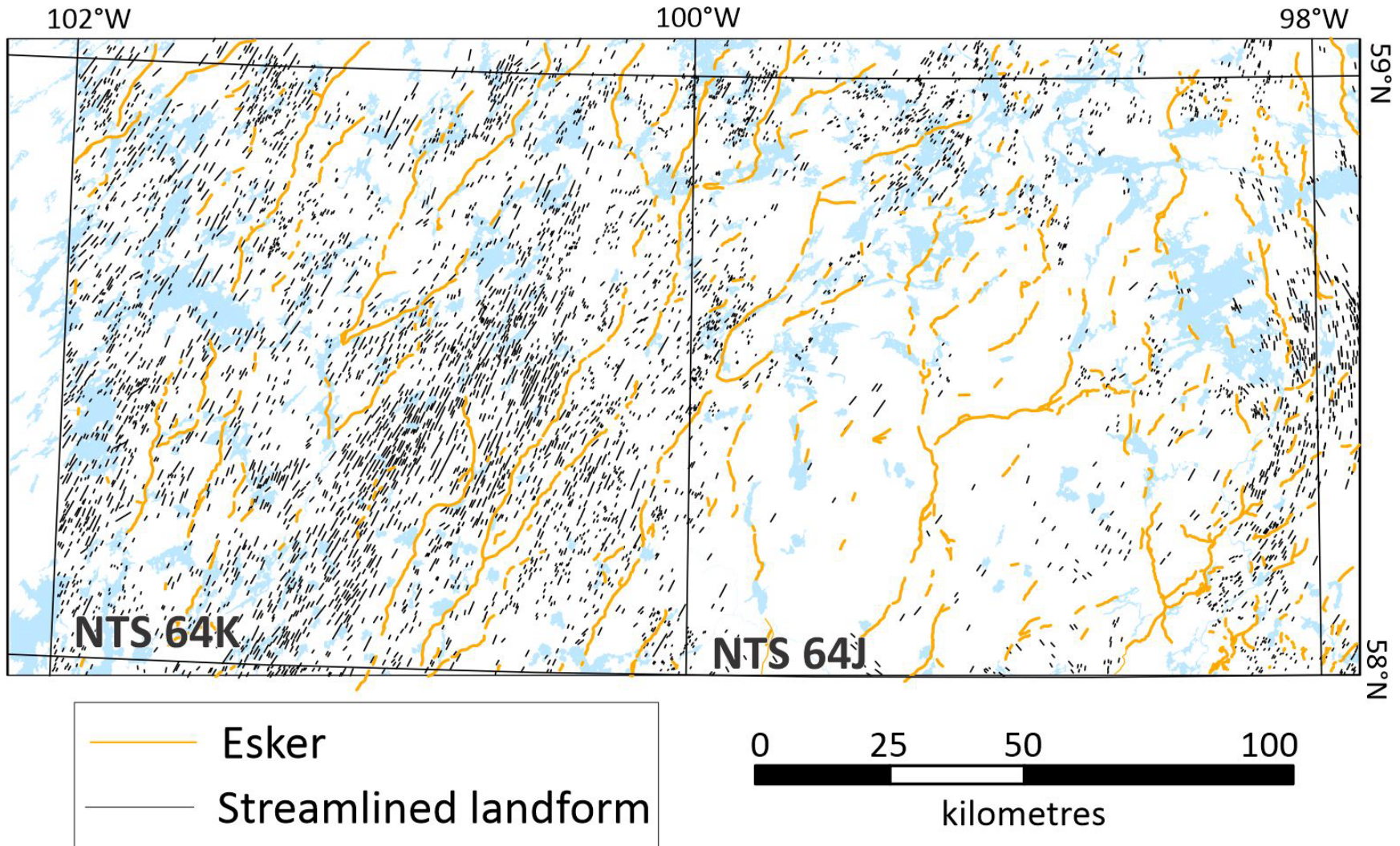


Figure 8. Eskers and streamlined landforms.

Figures 9a-9f show the locations of the ice-flow phases' effects in NTS 64-J and 64-K (this report), 64-G (Amor *et al.*, in prep.) and 64-F (Bourdeau, 2024).

- **Southeast:** the earliest observed regional ice-flow indicators (Phase 1, Figure 9a) trend southeast (120–160°, Trommelen, 2015; Hodder, 2018), and are based on observations on NTS map sheet 64-G; however, this event likely covered all four of the map areas.
- **Northwest and west:** Phase 2 ice (Figure 9b) then flowed northwest and west (Kaszycki and Way Nee, 1990a; Kaszycki *et al.*, 2008; Trommelen, 2015; Hodder, 2018). The extent of this ice-flow phase into the sampled areas is unknown but needs to be considered as a possibility.
- **Southwest and south:** ice then flowed to the southwest (Phase 3, Figure 9c) and south (Phase 4, Figure 9d), across the study area.
- **South-southwest and southwest:** Phase 5 ice flowed south-southwest and then southwest (Figure 9e), likely forming the abundant streamlined landforms in the western half of the study area (Figure 8).
- **Deglaciation:** the Quinn Lake ice stream readvanced across the eastern part of the study area, into glacial Lake Agassiz (Gauthier *et al.*, 2022). Elsewhere, ice generally continued to flow southwest during deglaciation, approximated by the dashed ice-margin lines on Figure 9f (Gauthier *et al.*, 2022).

Additional till-matrix geochemistry datasets, the relationship between ice-flow and sediment transport, and implications for the interpretation of lake-sediment geochemistry are described in Appendix A.

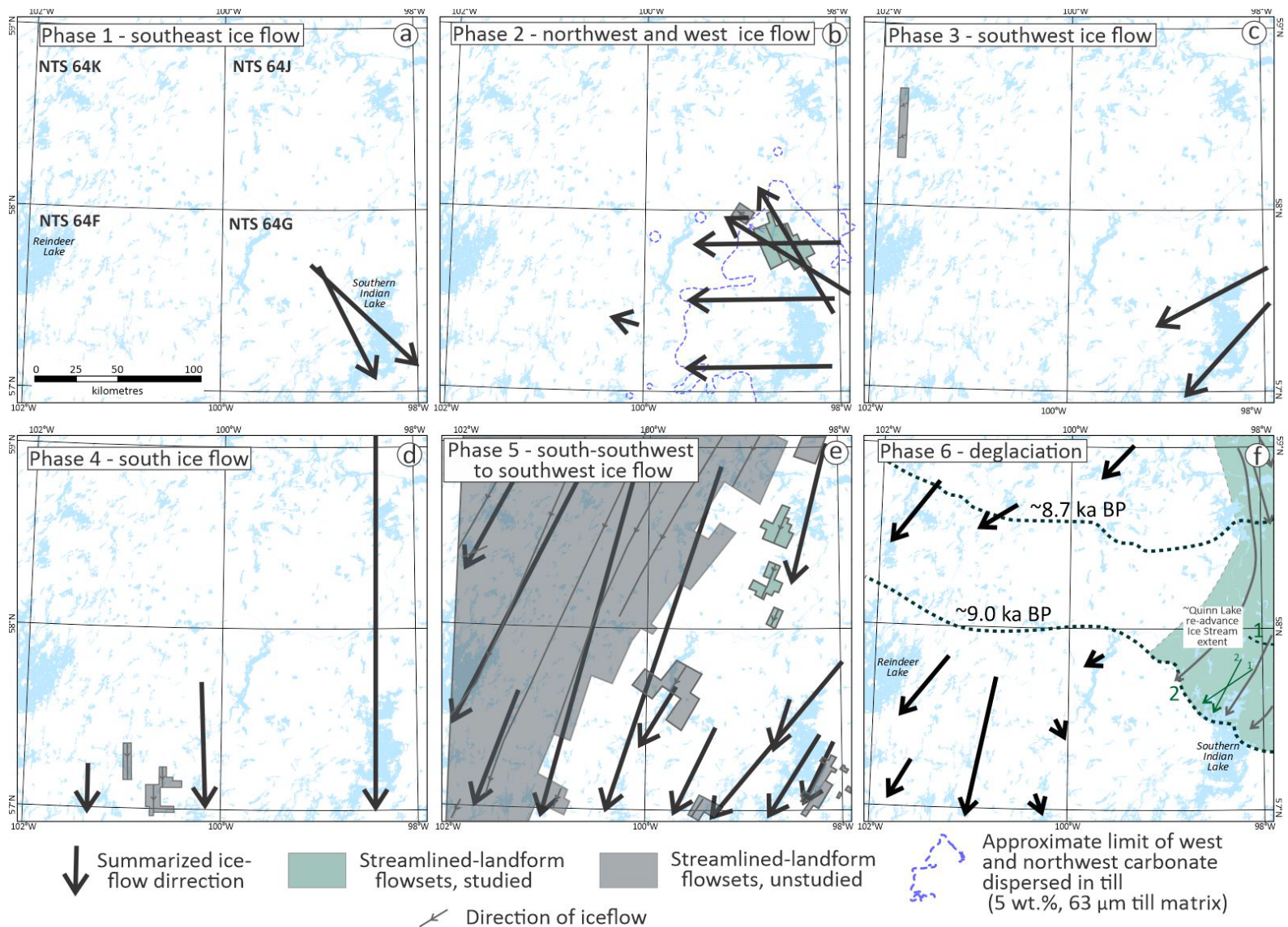


Figure 9. Indicators of glacial events for NTS map areas 64-F, 64-G, 64-J and 64-K (from Gauthier et al., 2019).

5 Sampling and analytical techniques

5.1 Description of surveys and sample management

A total of 2 368 samples, including samples used to evaluate data quality (control reference samples, analytical, field duplicate samples, and five silica blanks), were re-analyzed. There was sufficient material for aqua regia-ICP-MS analysis of all the samples; thus, analytical results were returned for 2 005 site samples (Figs. 2 and 3) and 363 QA/QC samples.

Reanalysis by instrumental neutron activation analysis (INAA) was not completed for 100 samples from NTS 64-J, comprising 85 routine samples and 15 QA/QC samples, due to their being lost in transit. For the samples that were submitted for INAA, three were not reanalyzed because of insufficient material; these comprised two routine samples and one for QA/QC, all from NTS 64-J.

Lake-sediment samples were collected using a hollow-pipe, bottom-valved sampler, a tool developed by the GSC (Friske and Hornbrook, 1991). During retrieval of the sample, the top few centimetres of sediment were washed out while the remainder, the organic-rich gyttja ('mud'), was retained. Approximately 1 kg of wet lake sediment was collected and placed into high wet-strength paper bags. Samples were then labelled, and field observations for each site were recorded on field cards used by the GSC (Garrett, 1974). Next, samples were shipped to the GSC where they were air-dried and sieved through an 80 mesh (177 µm) screen before being milled using a ceramic mill with ceramic balls. Typically, 1 kg of organic-rich gyttja yielded about 50 g of suitable material for analysis. After the initial preparation and subsequent geochemical analysis, the unused prepared sample material was placed in plastic containers and archived at GSC Ottawa.

During the original survey, samples were numbered consecutively in blocks of 20. Within each block of 20 samples, one site duplicate pair (two samples from the same site) was collected. Each block also contained an empty slot to place an analytical duplicate split comprising a sample split from a routine site. Additionally, a control reference standard sample was added at a random pre-selected position into each block.

Samples selected for reanalysis were retrieved from the GSC archival facility in Ottawa and shipped to a commercial laboratory for reanalysis. The samples were already sieved and milled from sample processing conducted during the original survey. The samples' group (or block) structure and quality control measures described in the above paragraph were preserved during reanalysis. For the reanalysis, new control reference standards (LKSD-1 and LKSD-3) replace the original standards used during the previous analysis (Lynch, 1990, 1999; Hechler, 2013).

Before publication of this report, a thorough inspection of the field and analytical data was made to check for any missing or mislabeled samples, as well as for any analytical errors. These checks were carried out both at the commercial laboratory and upon receipt of the data at the GSC.

5.2 Analytical Procedures 1975

The analytical parameters for the 1975 lake-sediment samples are summarized from Hornbrook *et al.* (1976a), that applies to both the original files and 2017 re-release of the original files, and from Friske

and Hornbrook (1991). For the determination of Zn, Cu, Pb, Ni, Co, Ag, Mn and Fe, a 1 g sample was reacted with 6 mL of a mixture of 4 M HNO₃ and 1 M HCl (“Reverse Aqua Regia” or “Lefort Acid”) 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 2 hours with periodic shaking. The sample solution was then diluted to 20 mL with metal-free water and mixed. Determinations were done by flame atomic absorption spectrometry (FAAS) using an air-acetylene flame. Background corrections were made for Pb, Ni, Co, and Ag. For further information regarding the original analyses can be found in the Canadian Database of Geochemistry (Adcock, et al., 2013; https://geochem.nrcan.gc.ca/cdogs/content/main/home_en.htm).

Arsenic was determined colorimetrically using silver diethyldithiocarbamate. Decomposition was accomplished by heating a 1 g sample in 20 mL of 6 M HCl at 90° C for 1½ hours. Colorimetric measurements were made at 520 nm.

Mercury was determined by the Hatch and Ott Procedure with some modifications. The method is described by Jonasson *et al.* (1973). A 0.5 g sample was reacted with 20 mL concentrated HNO₃ and 1 mL concentrated HCl in a test-tube for 10 minutes at room temperature prior to 2 hours of digestion with mixing at 90° C in a hot water bath. After digestion, the sample solution was cooled and diluted to 100 mL with metal-free water. The Hg present was reduced to the elemental state by the addition of 10mL 10% w/v SnSO₄ in 1 M H₂SO₄. The Hg vapour was then flushed by a stream of air into an absorption cell mounted in the light path of an atomic absorption spectrophotometer. Absorption measurements were made at 253.7 nm.

Molybdenum was determined by FAAS using a nitrous oxide acetylene flame. A 0.5 g sample was reacted with 1.5 mL concentrated HNO₃ for 30 minutes. 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 was added to suppress interferences, and the sample solution diluted to 10 mL before aspiration.

Uranium was determined using a neutron-activation method with delayed neutron counting (DNC). In brief, a 1-gram sample was weighed in a 7-dram polyethylene vial, capped and sealed. The irradiation was provided by the Slowpoke reactor with an operating flux of 10¹² neutrons cm⁻² second⁻². The samples were pneumatically transferred from an automatic loader to the reactor, where each sample was irradiated for 20 seconds. After irradiation, the samples were again transferred pneumatically to the counting facility where, after a 10 second delay, the sample was counted for 20 seconds with six BF₃ detector tubes embedded in paraffin. Following counting, the samples were automatically ejected into a shielded storage container. Calibration was carried out twice a day as a minimum, using natural materials of known U concentration.

Loss-on-ignition (L.O.I.) 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 2-3 hours. The sample remained at this temperature for 4 hours and was then allowed to cool to room temperature for weighing.

5.3 Analytical Procedures 2023

5.3.1 Induction-Coupled Plasma Mass Spectrometry (ICP-MS)

Samples selected and prepared for reanalysis were analyzed at Bureau Veritas in Vancouver (British Columbia) using a modified aqua regia digestion followed by inductively-coupled plasma-mass spectroscopy (ICP-MS) analysis. A total of 65 elements were determined via ICP-MS (Table 2). A small portion (0.5 g) of the sample was taken and digested using a modified aqua regia solution (1:1:1 HNO₃: HCl: H₂O) for one hour on a heating block. Afterwards, the sample was brought up to volume with dilute HCl. The solution was then aspirated into a mass spectrometer for the determination of elements. The commercial laboratory QA/QC protocol included the addition of external CRM OREAS262 and internal CRM DS11, and total procedural blanks. The latter do not contain any sample material (i.e., empty vials), but are brought through the entire digestion and analysis process in the same manner as the remainder of the samples. Analytical results were verified by a British Columbia Certified Assayer prior to being delivered to the GSC.

Table 2. Lower detection limits published by Bureau Veritas for aqua regia digestion followed by ICP-MS analysis.

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

Table 3. Lower detection limits published by Bureau Veritas for instrumental neutron activation analysis (INAA).

Element Lower Detection Limits			
Ag	2 ppm	Ni	10 ppm
As	0.5 ppm	Rb	5 ppm
Au	2 ppb	Sb	0.1 ppm
Ba	50 ppm	Sc	0.2 ppm
Br	0.5 ppm	Se	5 ppm
Cd	5 ppm	Sm	0.1 ppm
Ce	5 ppm	Sn	100 ppm
Co	5 ppm	Ta	0.5 ppm
Cr	20 ppm	Tb	0.5 ppm
Cs	0.5 ppm	Te	10 ppm
Eu	1 ppm	Th	0.2 ppm
Fe	0.2 %	Ti	500 ppm
Hf	1 ppm	U	0.2 ppm
Ir	50 ppb	W	1 ppm
La	2 ppm	Yb	2 ppm
Lu	0.2 ppm	Zn	100 ppm
Mo	1 ppm	Zr	200 ppm
Na	0.02%		

5.3.2 Instrumental Neutron Activation Analysis (INAA)

Samples of the <177 µm fraction were analysed by INAA at Maxxam Analytics (formerly Becquerel Labs, now affiliated with Bureau Veritas), Mississauga, Ontario. A 30 g aliquot of each sample was encapsulated and packaged for irradiation along with certified reference materials, field and analytical duplicates. Samples and quality control insertions were irradiated together with neutron flux monitors in a two-megawatt pool type reactor. After a seven-day decay period, samples were measured with a high-resolution germanium detector. Typical counting time per sample was 500 seconds. Elements determined by INAA are listed in Table 3; the sample weight is also reported. The commercial laboratory did not provide internal QA/QC data for the neutron-activation analyses.

6 QA/QC of geochemical data

Lake sediment control reference standards are used to quantify accuracy, while analytical duplicates are used to quantify precision. The fitness-for-purpose for mapping is determined using variance analyses of in-site versus between-site variability using the analysis of variance (ANOVA) of field duplicate samples.

Project samples and laboratory metadata are presented in Appendix B. Data resulting from the re-analysis of lake sediment samples by the commercial laboratory are presented in Appendix C. Elements in Appendix C are listed in the order that they were reported in the laboratory certificates and can be used to independently verify the results presented in Appendix D. Results for accuracy, precision, and

fitness-for-purpose are presented in Tables D-1, D-2, D-3 and D-4 in Appendix D. Table 4 (below) lists the contents of the Appendix D workbook.

Table 4. Contents of Appendix D workbook. The workbook contains the QA/QC results for this study.

Worksheet	Contents
D-1 Accuracy LKSD-1 ICP-MS	Compares accepted values for CRM LKSD-1 with results from re-analysis via ICP-MS.
D-2 Accuracy LKSD-3 ICP-MS	Compares accepted values for CRM LKSD-3 with results from re-analysis via ICP-MS.
D-3a Accuracy LKSD-1 INA	Compares accepted values for CRM LKSD-1 with results from re-analysis via a 'total' analytical method (Instrumental Neutron Activation analysis).
D-3b Accuracy LKSD-3 INA	Compares accepted values for CRM LKSD-3 with results from re-analysis via a 'total' analytical method (Instrumental Neutron Activation analysis).
D-4a Precision ICP-MS	Provides an estimate of precision using analytical duplicate pairs for re-analysis via ICP-MS.
D-4b Precision INA	Provides an estimate of precision using analytical duplicate pairs for re-analysis via Instrumental Neutron Activation analysis (INAA).
D-5a ANOVA ICP-MS	Provides an estimate of fitness-for-purpose using field duplicate pairs for re-analysis via ICP-MS.
D-5b ANOVA INA	Provides an estimate of fitness-for-purpose using field duplicate pairs for re-analysis by Instrumental Neutron Activation analysis (INAA).

6.1 Accuracy

The accuracy of analytical data was evaluated by inserting Canadian Certified Reference Lake Sediments LKSD-1 and LKSD-3 at random locations within each block of 20 samples throughout the analytical suite (McCurdy and Garrett, 2016). LKSD-1 combines lake sediments from two lakes located in central Ontario (Brady Lake, NTS 31-M and Joe Lake, NTS 3-1F). 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 31-M, 41-P, 42-A), eastern Quebec (NTS 31-N, 32-C, 32-D) and northeastern Saskatchewan (NTS 64-L, 64-M).

The accepted values for LKSD-1 and LKSD-3 were published by Lynch (1990, 1999) and Hechler (2013). Accepted values published by Lynch (1990, 1999) were derived from analyses at several national and international participating laboratories, where samples were digested using a strong acid (concentrated HNO₃-concentrated HCl) for partial digestion but with varying fuming times, acid ratios, and sample weights. It is assumed that the resulting standard deviations of the accepted values reflect variabilities in the analytical methodology across the laboratories. Comparatively, accepted values published by Hechler (2013) were measured at the Geoscience Laboratories of the Ontario Geological Survey (Sudbury). Samples were digested using a modified (nitric acid-rich) aqua regia solution for partial digestion and analyzed via ICP-MS. Taking into account lower detection levels resulting from instrument improvements over the last decades, accepted values from Lynch (1990, 1999) and Hechler (2013) using

partial digestions are in agreement with all published certificates and are considered to be comparable. Whenever possible, the most recent accepted value was compared to this study's measured value.

Equation (1) Mean

$$\bar{x}_i = \frac{\sum_{i=1}^n x_i}{n}$$

Equation (2) Standard Deviation

$$S_i = \sqrt{\frac{\sum_{i=1}^n (x_i - \bar{x}_i)^2}{n - 1}}$$

To determine accuracy, we compare the means (Equation 1) and standard deviations (Equation 2) of a CRM to that of the samples for each determined element. We also calculate the relative standard deviation (RSD; Equation 3), which indicates precision at the stated mean. As an additional measure, we also calculate the relative error (RE), which provides a semi-quantitative indication of how close the measured mean (\bar{x}_i) is to the accepted one (\bar{x}_a). Here, we report the relative error in percent (%) in Equation 4. As some of the accepted values are decades old, the relative error can only be used in a semi-quantitative manner. A value of $\pm 20\%$ indicates that, on average, 95 out of 100 analyses should fall within two standard deviations on either side of the mean (Fletcher, 1981). An RSD exceeding 20% combined with a relatively narrow range of concentrations could result in overlooked anomalies (Howarth and Martin, 1979).

Equation (3) Relative Standard Deviation

$$RSD = \frac{S_i}{\bar{x}_i} \times 100\%$$

Equation (4) Relative Error

$$RE = \frac{|\bar{x}_i - \bar{x}_a|}{\bar{x}_a} \times 100\%$$

The accuracy of analytical results for LKSD-1 and LKSD-3 from ICP-MS are presented for each element in Tables D-1 and D-2, Appendix D. Of 65 elements determined, 6 (B, Ge, Pd, Pt, Ta, and Te) in LKSD-1 and 5 (B, Ge, Pd, Pt, and Ta) in LKSD-3 are at or below laboratory detection. Furthermore, several elements could have accuracy issues (i.e., RSD >20%). For CRM LKSD-1, these elements include Au, Be, Bi, Hf, Te, and W. For the CRM LKSD-3, the elements of concern include Au, Re, and W.

Elements at or below laboratory detection or that have a high RSD are in low abundance within the CRM (both for accepted and measured values). Low detectable concentrations and subsequent relatively high RSD values (>20%) 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). The apparent low concentrations in elements such as Au, B, Be, Bi, Ge, Hf, Pd, Re, Pt, Re, Se, Ta, Te, and W in samples are probably caused by the inability of the digestion method to dissolve these elements in the above minerals. Additionally, the RSD % for Au in both LKSD-1 and LKSD-3 is relatively high (>20 %) due to the difficulty of creating homogeneous standard materials (Harris, 1982; Clark, 2010; Dominy, 2014). Lastly, for element means falling outside the accepted standard deviations (RE %), it should be noted that the accepted values for these elements were measured in 1990 and may not be as accurate as current determinations of sample means. Thus, the element means falling outside the accepted standard deviations reflect improved measurements of that element by modern instrumentation.

The accuracy of analytical results for LKSD-1 and LKSD-3 from Instrumental Neutron Activation analysis (INAA) are presented for each element in Tables D-3a and D-3b, Appendix D. Of 35 elements determined, 8 (Zr, Ag, Cd, Ta, Ir, Sn, Se, and Te) in LKSD-1 and 7 (Zr, Ag, Cd, Ir, Sn, Se, and Te) in LKSD-3 are at or below laboratory detection. Furthermore, several elements could have accuracy issues (i.e., RSD >20 %). For CRM LKSD-1, these elements include Ni, W, Au, Eu, Yb and Lu. For the CRM LKSD-3, the elements of concern include Zn, Mo, W, and Au. These elements are present in low concentrations in samples (at or just above laboratory detection limits). In addition, Au has homogeneity issues that can result in higher (>20) RSD values.

6.2 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 time intervals on duplicate project samples). The estimation of the analytical precision follows the procedure of Youden (1951) for up to 122 duplicate pairs analyzed by ICP-MS and 115 duplicate pairs analyzed by INAA, with results in the case where both members of a pair are above the respective detection limits. The resulting numerical estimate of precision for variables is listed in Tables D-4a and D-4b in **D1 Quality Control Data.xlsx** as a % Relative Standard Deviation (Standard Deviation 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 %)' and is presented in Tables D-4a and D-4b.

Elements with precisions poorer than 20 % in Tables D-4a and D-4b tend towards generally low concentrations in samples, as indicated by the number of duplicate pairs removed, the range, the mean, and the percentage of data below the detection limit. Such is the case for elements As, Au, Bi, Ge, In, Pt, Re, Sb, and Te that underwent an aqua regia ('partial') digestion and Ni, Zn, Zr, W, Au and Sb in Table D-4b (Precision INAA). 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 B, Ge, Pd and Ta, analyzed by ICP-

MS, less than two pairs of analytical duplicates have both samples above detection, and no ANOVA results are calculated. Similarly, INAA results for Ag, Cd, Ir, Sn, and Te were not calculated.

6.3 Analysis of Variance (ANOVA)

Field duplicate data were used to test the hypothesis that the combined sampling and analytical variability (s^2_{sa}) was equal to the 'regional' variability (s^2_r), across the areal extent of the field duplicates (i.e., $H_0: s^2_{sa} = s^2_r$) using a one-way Analysis of Variance (ANOVA; Garrett, 1983). It is desirable that this test fails, and the sampling and analytical variability is not 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. If this 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 of the 'rgr' package in 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). Tables D-5a and D-5b 'ANOVA' in **D1 Quality Control Data.xlsx** shows results from an ANOVA undertaken on up to 118 field duplicate pairs analyzed by ICP-MS and 113 field duplicate pairs analyzed by INA 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 Tables D-5a and D-5b. The variance ratio, F, is calculated in 'anova2' within the 'rgr' package 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 Tables D-5a (ICP-MS analysis) indicate that the sampling and analytical variability is lower than the field survey variability, at the $p < 0.05$ level (>95 % confidence level) for all elements except Au and Te. For the elements B, Ge, Pd, Pt and Ta, less than two field duplicate sample pairs with both members above detection are available for calculations, and no ANOVA results are reported. The ANOVA statistics in Tables D-5b (INA analysis) indicate that the sampling and analytical variability is lower than the field survey variability, at the $p < 0.05$ level (>95 % confidence level) for all elements

except Au and Sb. For the elements Zr, Ag, Cd, Ir, Sn, and Te, less than two field duplicate sample pairs with both members above detection are available for calculations, and no ANOVA results are reported.

From these results, 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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9 Appendices

Appendix A Supplementary Surficial Data

A1 Supplementary Surficial Data.docx

Appendix B Survey Metadata

B1 Metadata.docx

B2 Bureau Veritas Fee Schedule 2023.pdf

Appendix C Raw Data

C1 ICP-MS & INAA Raw Data.xlsx

C2 Bureau Veritas Certificates of Analysis.pdf

Appendix D Quality Control Data

D1 Quality Control Data.xlsx