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J.E. Campbell[1], M.S. Trommelen[2], M.W. McCurdy[1], C.O. Böhm[2], and M. Ross[3]

[1] Geological Survey of Canada, 601 Booth Street, Ottawa, Ontario, K1A 0E8
[2] Manitoba Geological Survey, 360-1395 Ellice Avenue, Winnipeg, Manitoba R3G 3P2
[3] Department of Earth and Environmental Sciences, University of Waterloo, 200 University Avenue West, Waterloo, Ontario N2L 3G1

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360-1395 Ellice Avenue
Winnipeg, Manitoba R3G 3P2
Ph: (204) 945-4154, 1-800-223-5215
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TILL COMPOSITION AND ICE-FLOW INDICATOR DATA, GREAT ISLAND –CARIBOU LAKE AREA (PARTS OF NTS 54L, 54M, 64I AND 64P), NORTHEAST MANITOBA

Authors: Campbell, J.E.[1], Trommelen, M.S.[2], McCurdy, M.W.[1], Böhm, C.O.[2] and Ross, M.[3]

INTRODUCTION

The Geological Survey of Canada's (GSC) Geo-Mapping for Energy and Minerals (GEM) Multiple Metals–Northeast Manitoba Project was initiated in 2008 in support of the Manitoba Geological Survey's (MGS) Far North Geomapping Initiative to address the geoscience knowledge deficit in Manitoba's far north. This region is a remote, predominantly drift-covered area that last saw systematic geological work more than 25 years ago. Therefore, in an effort to stimulate and support private sector investment in mineral exploration in this region, the project's multidisciplinary team (federal, provincial, university-based collaborators and students) have focussed their mapping activities on high mineral potential areas such as the Great Island–Seal River area in Manitoba's far northeast.

During the 2009 and 2010 field seasons, Quaternary studies involving surficial geological and ice-flow indicator mapping and till geochemical sampling were conducted in conjunction with bedrock geological mapping over about 8100 km^2 extending from south of the Seal River in the Great Island area to Caribou/Kellas lakes to the north (Figure 1). The objectives of the Quaternary component are to 1) compile surficial geology maps at 1:50 000 scale suitable for property-scale exploration (Trommelen and Campbell, 2012a, b, c, d); 2) interpret the glacial and deglacial history at regional and local scales (e.g. Trommelen and Ross, 2011; Trommelen et al., 2011, 2012) and; 3) conduct a regional till-sampling program to establish regional background till compositional data as a means to determine background and threshold element concentrations, sediment-landform relationships, and glacial dispersal distances and directions.

The purpose of this Open File publication is to release the till compositional data sets collected as part of the Quaternary studies to assist with the application of drift prospecting in the Great Island–Caribou Lake study area. This report also includes the regional physiographic and geological setting, regional interpretation of ice-flow chronology, detailed descriptions of the till sampling methods, laboratory procedures and QA/QC for the analytical data. There is no interpretation of the data provided in this report, however, the geochemical results provide up-to-date estimates of background and threshold values characteristic of the region, and fill in part of a gap in regional coverage (Dredge and Pehrsson, 2006; Dredge and McMartin, 2007). Till sampling, especially in combination with lake sediment geochemistry (McCurdy et al., 2010) and the new surficial geological framework, should be an effective exploration tool in this region.

[1] Geological Survey of Canada, 601 Booth St., Ottawa, Ontario, K1A 0E8
[2] Manitoba Geological Survey, 360-1395 Ellice Avenue, Winnipeg, Manitoba R3G 3P2
[3] Department of Earth and Environmental Sciences, University of Waterloo, 200 University Avenue West, Waterloo, Ontario N2L 3G1

REGIONAL SETTING

The study area is located in the northeastern part of Manitoba (Figure 1), between 58°45'W and 59°45'W latitude and 95°N and 97°N longitude. Elevation varies from sea level in the east to 270 m asl in the west. Local relief is up to 30 m high. The Seal River is the major drainage channel in the southern portion of the study area, flowing east from Tadoule Lake into Hudson Bay. Great Island refers to the area where the Seal River splits into north and south channels, before connecting into one river again. Caribou River is the major drainage channel in the northern portion of the study area, flowing east into Hudson Bay. Much of this region is poorly drained. Numerous small streams flow across the drift plains from one lake to another in an immature drainage network, or flow through the muskeg. The area is predominantly mantled by till and/or marine sediments and expansive regions of organic deposits (muskeg). Bedrock outcrops are scarce. The region falls within the zone of discontinuous permafrost. Bogs are commonly frozen and ice-rich below a metre. Periglacial features such as mudboils, peat plateaus, ice-wedge polygons and frost-shattered rock are common.

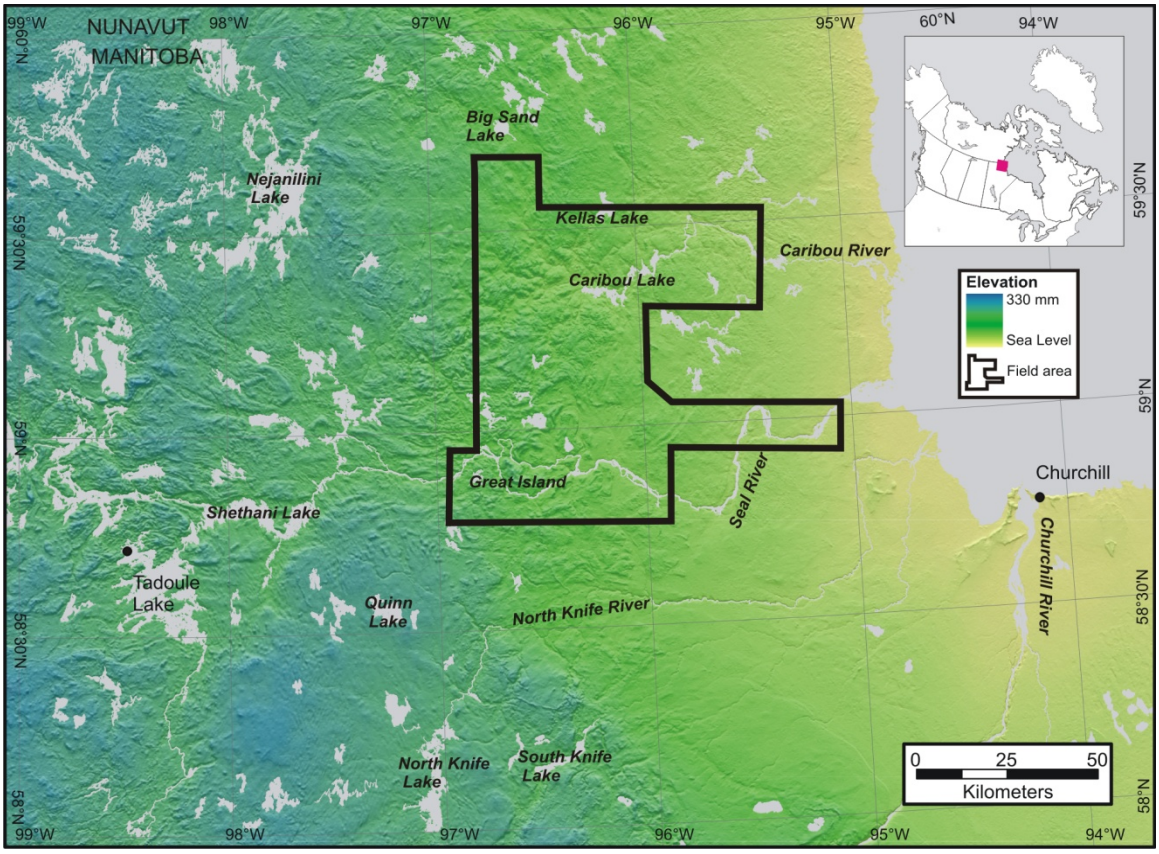

Figure 1. Location of the Great Island–Caribou Lake study area in northeast Manitoba. The background image was generated using the radar-derived digital elevation data from the Shuttle Radar Topographic Mission (SRTM) data set. A hill-shade model has been added with transparency effect to enhance relief.

BEDROCK GEOLOGY

Precambrian bedrock mapping in Manitoba's far northeast, an area that is largely devoid of outcrop, is supported by an aeromagnetic and gamma-ray spectrometric geophysical survey of the Great Island–Seal River area (Fortin et al., 2009) and U-Pb zircon geochronology (Rayner, 2010).

Manitoba's far northeast forms part of the southeast margin of the Hearne craton, which comprises a heterogeneous basement of Archean orthogneiss, granitoid intrusions and rare supracrustal rocks overlain by scattered erosional remnants of latest Archean and Paleoproterozoic siliciclastic cover sequences, all of which have been variably overprinted by tectonothermal and magmatic activity associated with the Paleoproterozoic Trans-Hudson orogeny (Anderson et al., 2010a). The southeast margin of the Hearne craton in northeast Manitoba is subdivided into the Nejanilini and Seal River domains, based on their different proportions of cover and basement rocks, structural trends and metamorphic grade (Anderson et al., 2010b; Fig. 2).

The Nejanilini Domain is dominated by variably deformed Neoarchean metaplutonic rocks, with minor enclaves of early Paleoproterozoic metasedimentary rocks, and Paleoproterozoic sub-circular granitic intrusions. Metamorphic grade increases to the northwest and reached granulite facies conditions in the Archean basement and the cover rocks around 2.5 Ga.

The Seal River Domain, which defines the southeast margin of the Archean Hearne craton in Manitoba, is characterized by a dome-and-basin structural geometry, with the 'domes' defined by Meso- to Neoarchean metaplutonic and metavolcanic rocks, and the 'basins' defined by synforms of latest Archean and Paleoproterozoic continental and marine siliciclastic rocks. The siliciclastic cover rocks have been subdivided into four distinct sequences that provide a discontinuous ca. 2.7 to 1.9 Ga record of intracratonic sedimentation and basin evolution at the south margin of the Hearne craton (Anderson et al., 2010a). Metamorphic grade in the Seal River Domain reached middle amphibolite grade in Archean rocks, whereas sequence 3 and 4 sedimentary rocks are at upper greenschist facies.

To the south, the Seal River Domain is separated from accreted juvenile Paleoproterozoic terranes in the internides of the Trans-Hudson Orogen by the ca. 1.87 to 1.85 Ga Wathaman arc batholith. Southeast of the map area the Precambrian rocks are unconformably overlain by flat-lying Ordovician sandstone, limestone and dolomite at the west margin of the Hudson Bay Basin.

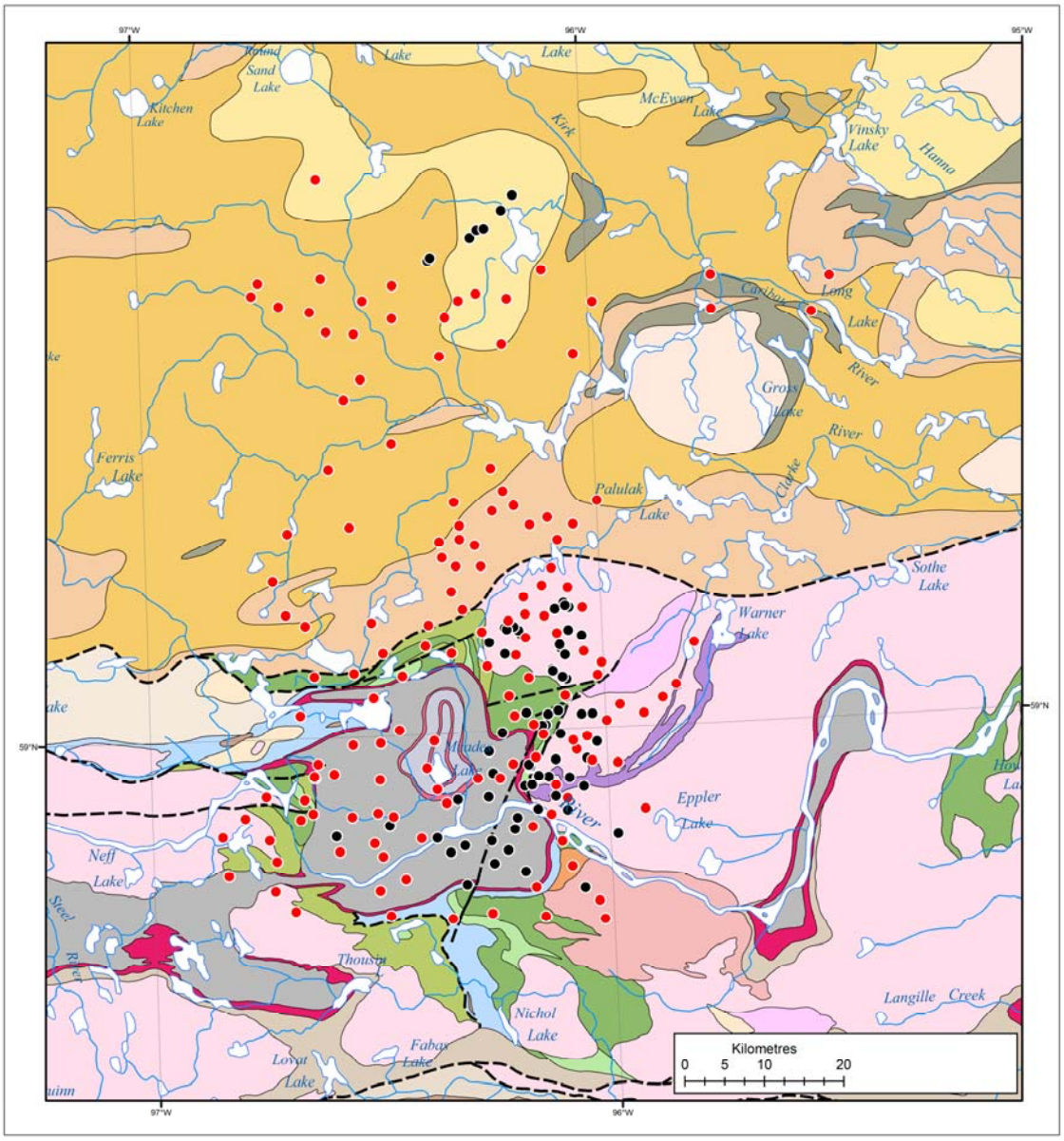

Figure 2a. Map of northeast Manitoba with geology background and legend showing locations of 2009 (black) and 2010 (red) till sample sites. Bedrock geology is modified after Anderson et al., (2010b).

QUATERNARY GEOLOGY

Northern Manitoba was repeatedly covered by the Laurentide Ice Sheet (LIS) and its precursors throughout the Quaternary and likely remained ice covered throughout the Wisconsinan (Nielson et al., 1986; Dredge et al., 1990). The ice predominantly flowed southward across Manitoba from the Keewatin Ice Sector but also was affected multiple times by ice flowing westward from or across Hudson Bay (Hudson Ice; Quebec/Labrador Ice Sector) (Dyke and Dredge, 1989; Dyke et al., 2003). Tills of Keewatin provenance are derived primarily, but not exclusively, from Precambrian

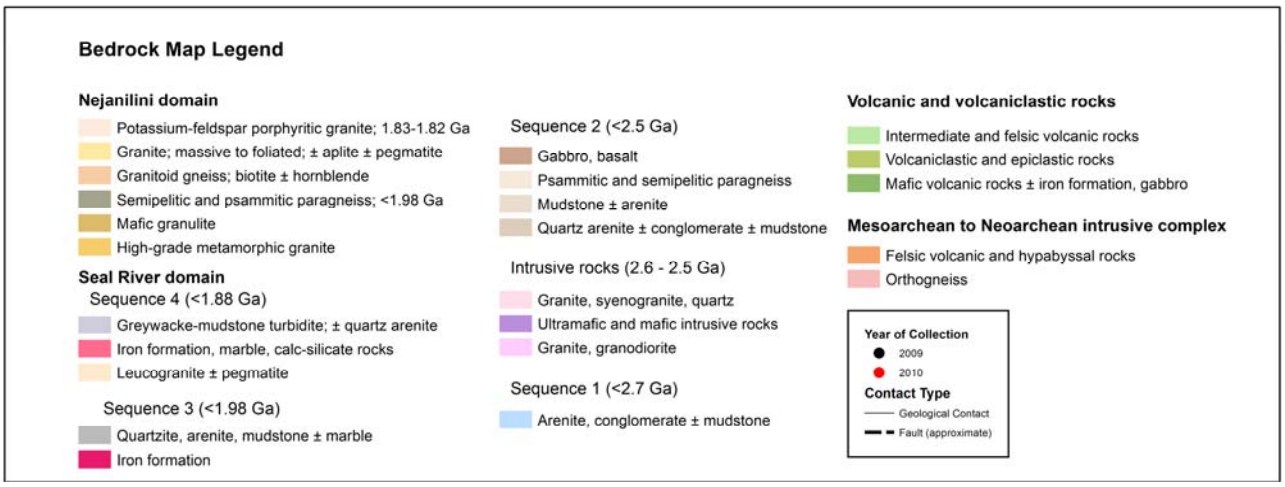

Fig. 2b. Legend to accompany bedrock geology map of northeast Alberta (Fig. 2a, above)

Shield rocks and tend to have a sandy, non-calcareous matrix. Tills of Labradorean/Hudsonian provenance comprise both Precambrian and Paleozoic carbonate bedrock resulting in a predominantly silty and calcareous matrix (Dredge, 1988).

Ice-flow indicator data and stratigraphic records southeast of the map area suggest that the southeastern part of the Great Island–Caribou Lake area may have been within the zone of confluence between the Keewatin and Labradorean ice during the late Wisconsin glaciation. Aside from an outlier on west Great Island, most of the region is draped by Keewatin-source till. Calcareous till (>10% carbonate-bearing clasts) from the Hudsonian ice flow is largely absent, with only scattered carbonate pebbles and calcareous till matrix present (Trommelen et al., 2010). Based on this study's detailed till clast counts, the approximate northwest limit of carbonate clast dispersal from the Hudson Bay Basin (Carbonate Platform) to the east is shown in Figure 3 (dashed blue line).
As the ice retreated from the region, much of the study area was inundated by either postglacial Tyrrell Sea or glacial Lake Agassiz and/or other smaller ice-marginal lakes (Dredge, 1983; Dredge and Nixon, 1992). The approximate limit of marine incursion is shown as a purple line in Figure 3.

Ice-Flow History

Shifting ice-flow patterns in northern Manitoba have been attributed to the migration of an ice-dispersal centre and/or ice divide of the Keewatin Ice Sector during the Wisconsinan glaciation and throughout the Late Wisconsinan deglaciation (Dredge et al., 1986; Dredge and Nixon, 1992; McMartin and Henderson, 2004). This, combined with the influence and interplay of southwest and west flow from Hudson Bay, resulted in a complex ice-flow history and drift dispersal in northeastern Manitoba (Dredge and Nixon, 1992; Dredge and McMartin, 2011; Trommelen et al., 2011; *2012; in review*). The ice-flow history is recorded in part by the fragmented subglacial landscape and by erosional ice-flow indicators. Figure 3 shows the subglacial landforms (eskers, Rogen moraines, streamlined landforms and subaqueous glaciofluvial fans) and erosional ice-

flow indicators (striae, grooves, crescentic fractures and gouges, roches moutonnées) mapped in the study area. Large circles highlight the region-wide relationship between indicators discovered during field mapping and the relative timing of various ice-flow directions at each site. The general ice-flow directions box provides a summary of ice-flow orientations for the entire region.

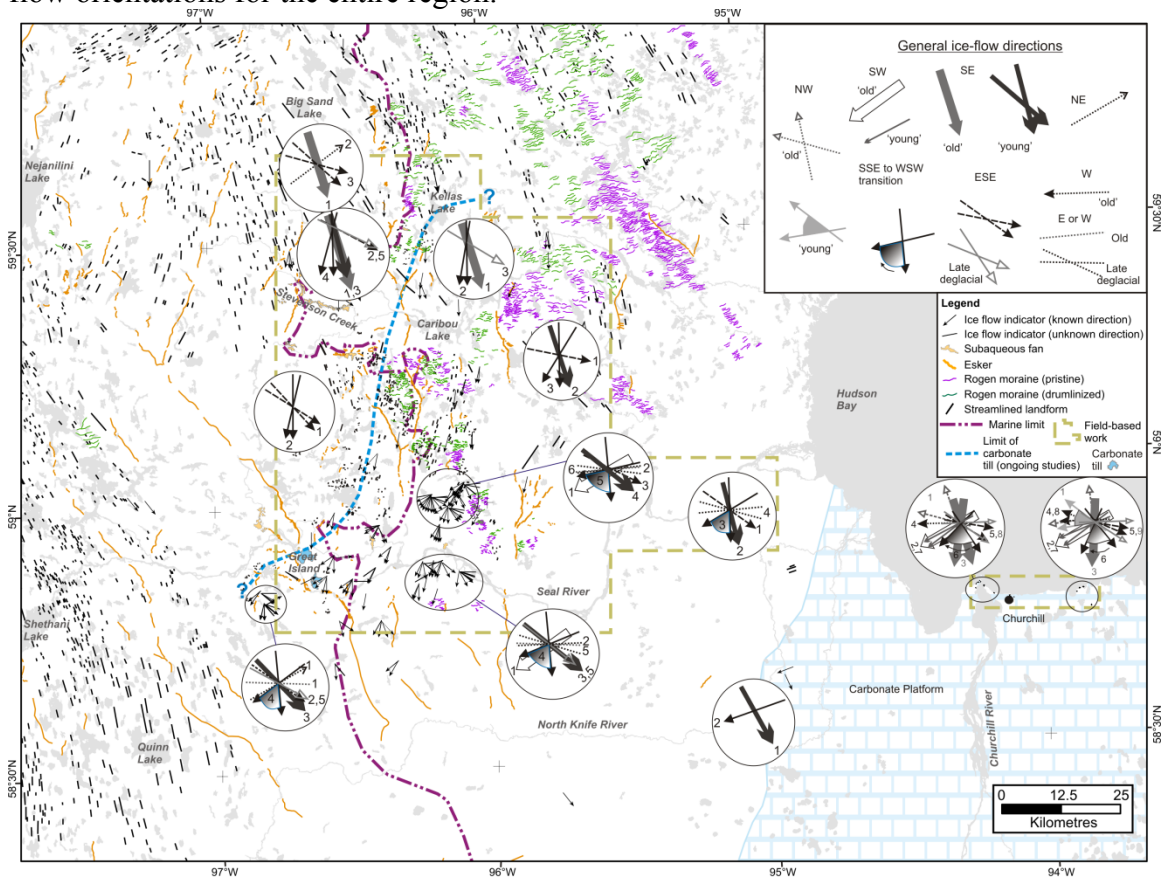

Figure 3. Ice-flow indicators, marine limit (dashed purple), and northwestern limit of carbonate in till (dashed blue) in northeastern Manitoba (Trommelen and Ross, 2011; Trommelen et al., 2012).

Figure 4 illustrates the regional ice-flow history reconstructed from the cross-cutting relationships of the ice-flow indicators shown in Figure 3. This reconstruction indicates large-scale deviations of ice-flow direction through time, but also highlights local isolated fragments of non-matching ice flow orientations. A relative chronological reconstruction from oldest to youngest of the regional ice-flow history for the study area (Churchill flow sets excluded) can be summarized as SW → SE →NE→ E? (Fig. 4a) →ESE →SE →S →SSW (Fig. 4b) followed by late deglacial ice flow to the WSW then E/W and ESE (Fig. 4c). Ice flow in Figure 4b likely represents a swing or shift in ice-flow orientation over time from the southeast to the southwest. The dominant ice-flow is to the south-southwest (blue arrows) ranging between 170˚ and 230˚ (Trommelen et al., 2010). The reader is referred to Trommelen et al. (2010; 2011; 2012) for further interpretation of the ice-flow history.

Glacial Landscapes

The distribution of the various tills and other glacial and postglacial deposits, as well as ice-flow indicators, are shown on the 1:50 000 scale surficial geology maps for this area (Trommelen and Campbell, 2012a, b, c, d). Detailed descriptions of these sediments and the surficial geology of the Great Island–Caribou Lake area are provided by Trommelen and Ross (2009) and Trommelen et al. (2010). The regional Quaternary geology and stratigraphic framework for northeastern Manitoba is available in Dredge and Nixon (1992) and Dredge and McMartin (2011).

The northern part of the map area is characterized by extensive swaths of bouldery drumlinized and pristine (non-drumlinized) Rogen moraine ridges alternating with swaths of streamlined terrain (Trommelen and Ross, 2010) and areas of bedrock outcrops. The remaining area is a mix of till blankets and till veneers over bedrock. Long, large eskers are present throughout the area, at roughly 18 km intervals. The marine limit in the study area is around 180-200 m asl (Figure 3). Below 180 m elevation, till is commonly wave-winnowed, reworked or covered by marine sediments. Where the eskers are located below approximately 200 m asl, they have been partially eroded by glaciolacustrine and/or marine waters. Below 150 m asl, the eskers exist as washed, low-lying sand and gravel blankets rather than ridges. A mix of extensive organic deposits and marine sediments blanket the eastern portion of the study area which is predominantly below 150 m asl.

An analysis of the glacial landscape in the study area is presented in Trommelen et al. (2011; 2012). The details and scope of this work are too large to present herein. The data suggest that large areas near the southeast end of the Keewatin Ice Divide (KID) were subject to spatially and temporally variable basal thermal regimes and intensities of erosion/transportation/deposition. This means that the region has glacial landscape zones that are of different ages, and have been emplaced by different combinations of ice-flow direction and intensity. As a result, Trommelen et al. (2011; 2012*)* have partitioned the glacial landscape of northeastern Manitoba into four different *glacial terrain zones (GTZ)*. Each zone has discrete boundaries and a distinct glacial history. The resulting glacial landscape reflects preservation of a long but fragmented glacial record, due to variable degrees of geomorphological inheritance and overprinting. Till composition is a product of its bedrock source(s) as well as the nature, direction and distance of its transport by the ice sheet. The surface till, the material primarily sampled for this report, may be a product of inheritance and reworking by the multiple ice-flow events described above. This can result in amoeboid-shaped relict and/or palimpsest dispersal trains (e.g. Stea, 1994; Stea and Finck, 2001; Stea et al., 2009; Parent et al., 1996; Plouffe et al., 2011; Trommelen et al. *in review*). In general, the relatively thin silty-sandy tills of Keewatin provenance, particularly in areas of veneers and bouldery till with ribbed moraine, appears to be locally-derived and transported over short distances. Consequently, geochemical values in those areas should reflect concentrations in nearby bedrock. Although their genesis is still uncertain, Rogen moraine ridges were likely generated by modification of a pre-existing till sheet rather than by sediment produced

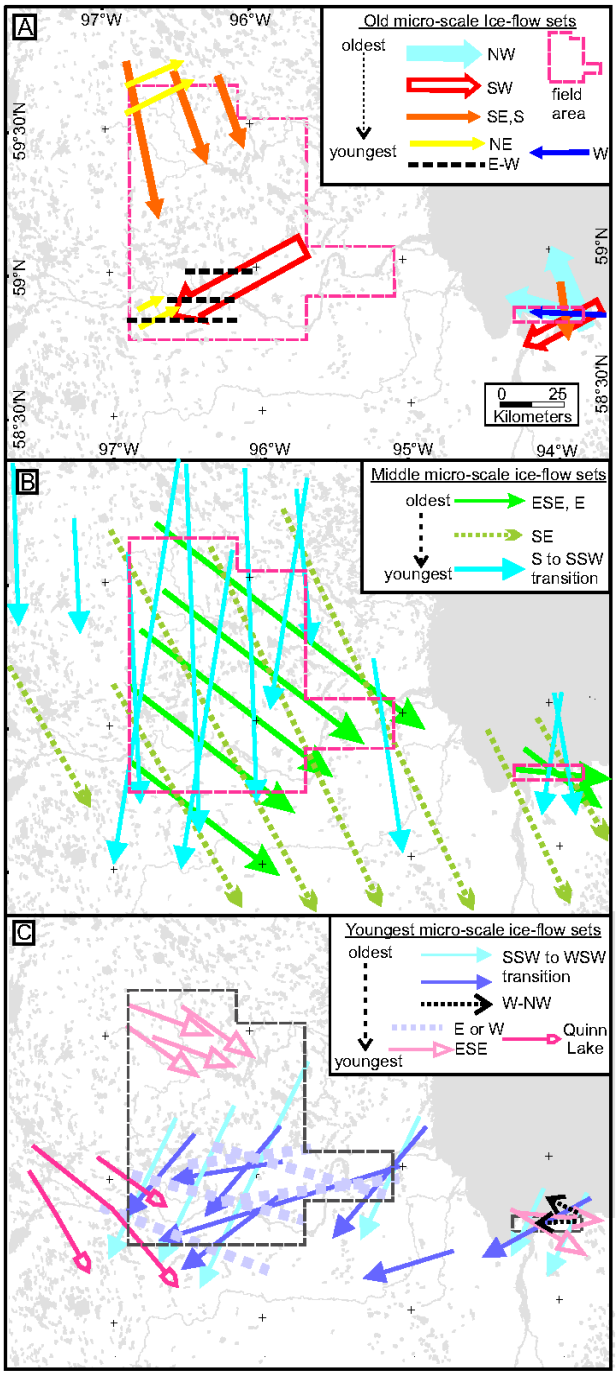

Figure 4. Regional ice-flow chronology based on micro-scale ice-flow indicators (striae, grooves, chattermarks, crescentic gouges, roches moutonnées, and whaleback orientations). Age relationships were determined regionally based on integration of cross-cutting relationships at individual sites as well as knowledge of several old buried tills (Dredge and McMartin, 2011). The arrows outline the minimum spatial extent of each ice-flow set where encountered in the field or displayed on regional maps (Trommelen et al, 2012).

during the younger ice-flow phases, therefore sediment within Rogen moraines may have a different history than sediment from the surrounding regions (e.g. Bouchard, 1989; Hättestrand and Kleman, 1999). In contrast, the less common silty tills of the Hudsonian/ Labradorean Ice contain more components inherited from underlying tills and more far-travelled material. Geochemical values determined from these silty tills may reflect concentrations from local to distant easterly sources. The distribution of carbonate in till (Figure 3) indicates that there has been some transport of material for distances of >175 km westward beyond the Paleozoic carbonate shelf. Also, red shale clasts reported in some till units indicate transport westward from known bedrock sources beneath Hudson Bay (Dredge and Nixon, 1992; Dredge and McMartin, 2011). It is suggested that the till geochemistry data sets should be used for drift exploration within the context of each GTZ and in combination with the ice-flow data for that particular GTZ.

METHODS

Field procedures

Field data collection

Helicopter-supported field work was conducted by M. Trommelen and M. Ross during July 2009 and by M. Trommelen, M. Ross and J. Campbell during July and early August 2010. A total of 325 sites were visited (Figure 2). At each site, geomorphic and terrain characteristics, map unit and geologic interpretation were recorded in addition to location coordinates. The respective site information is included with the sample information in **Appendix 01** and with the ice-flow measurements in **Appendix 02**.

Ice-flow indicator mapping

The orientation and relative ages of erosional ice-flow indicators documented at 74 sites in the study area during the 2009 and 2010 field season include micro-scale non-directional indicators such as striations and grooves, and directional indicators such as rat-tails, chattermarks, crescentic gouges and stoss-lee relationships (Figure 3). Macroform features encountered in the study area include roches moutonnées and whaleback drumlins. Detailed attention was paid at all outcrops to record rare and protected ice-flow indicators, in addition to the dominant indicators. Where cross-cutting patterns were found, the relative ages of flows were determined when possible. Detailed ice-flow indicator measurements for the 74 sites are found in **Appendix 02**.

Till sampling

Till sampling was conducted by M. Trommelen during the 2009 and 2010 field seasons. Helicopter traverses were used to find exposed till during reconnaissance sampling in 2009, based on which more detailed sampling sites were chosen in 2010 to better define

dispersal patterns. In addition, regional sampling continued in 2010 to complete coverage of the study area. A total of 238 till samples of ~2kg each were collected from 218 sites for matrix major/trace-element geochemical, grain-size, carbonate and lithological analyses (Figure 2). Samples were only collected from moderate to undisturbed diamict deposits, and not from extensively wave-washed winnowed till. Site location and description, sample information such as sample material, sample depth and soil horizon, and additional comments related to the sample and/or site are provided in **Appendix 01**. Till samples were collected from both the crest and lee-side of Rogen moraine ridges, the crest of streamlined landforms, till blankets and till veneers. The majority of samples are surface samples (25–135 cm depth) collected primarily with a shovel and trowel from the B, B/C or C soil horizons in hand-dug pits. Where present, samples were collected from mudboils in an attempt to sample the least oxidized and most unweathered material. Detailed profile samples were collected from several sites (i.e.09GSC1010; 10MT108, 10MT114) to test the compositional and provenance variability within Rogen moraines and in comparison to adjacent streamlined terrain.

Field duplicates were collected from 8 sites to test sediment heterogeneity and field variability. Each field duplicate was taken from the same sample pit approximately 10–20 cm lower than the original sample. The duplicate samples were bagged separately and given unique sample numbers.

Laboratory preparations and analytical procedures

The 2009 samples were processed for compositional analyses by M. Trommelen at the University of Waterloo. A portion of each sample (~400 grams) was dried in a low temperature oven then gently disaggregated with a mortar and pestle. The sample was then dry-sieved to obtain the <63 μm (silt + clay) size fraction. Approximately 50–60 g of the <63 μm fraction was reserved and sent for geochemical analyses.

The 2010 samples were prepared at the GSC Sedimentology Laboratory following procedures described by Girard et al. (2004) and by McMartin and Campbell (2009). Approximately 1kg of till samples were air-dried and dry-sieved, using a stainless steel 230 mesh screen to obtain the <63 μm fraction for geochemical analyses and carbonate content determinations. The remainder (<800 g) of the original samples was archived at the University of Waterloo (2009 samples) and the GSC (2010 samples). All remaining geochemical <63 μm fraction pulps are archived at the GSC.

Till matrix textural analysis

Till matrix textures of the 2009 samples were analyzed by M. Trommelen at the University of Waterloo. Textural analyses of the 2010 samples were completed by the GSC Sedimentology Laboratory. Fifteen of the 2009 samples were sent to the GSC for textural analysis to provide calibration between labs and methods, and between the 2009 and 2010 samples. The matrix textural results for all samples were calculated as weight percent sand, silt and clay of the < 2mm fraction and are presented in **Appendix 03**.

At the University of Waterloo, once the samples were oven-dried, each sample was gently disaggregated then dry-sieved in stainless steel sieves consisting of the following sizes: 8 mm, 4 mm, 2 mm, 1 mm, 500 or 600 µm, 250 µm, 125 µm, and 63 µm. Each sieve fraction was weighed, and grain sizes are reported in weight percent. Sieves were cleaned and dried between every sample. Additionally, after every 20th sample, quartz sand was run through the sieve set to test for comtamination. The <63 µm size fraction was later run through the laser diffractometer to obtain the distribution by volume of silt and clay.

The GSC Sedimentology Laboratory procedure for grain size analysis of the 2010 and selected 2009 till samples includes sand-silt-clay and complete grain-size analysis of the <2 mm size fraction as outlined by Girard et al. (2004). Approximately 200–300 g of samples were used for the analysis. The classes of sizes greater than 63µm were determined using wet sieving (to obtain the >45µm to <2mm size fraction) followed by dynamic digital image processing using a CAMSIZER Particle Size Analysis System. The classes of sizes smaller than 63µm were determined using a Lecotrac LT-100 Particle Size Analyser.

Combining results from two different methods of analysis (digital imaging and laser diffraction), as well as using different subsamples, introduced a discrepancy in the results for the < 63µm fraction, of the 2009 samples. Weight percent sand-silt-clay results in this report are assumed to be equivalent to volume percent results from Particle Size Analyzer System. It is not recommended to combine the datasets from University of Waterloo and the GSC labs.

Till matrix colour determination

The 2010 till samples were submitted to the GSC Sedimentology Laboratory for dry soil-colour determination. Soil colour of geological samples is described using a standardized system of colour standards and terminology known as the Munsell Soil Color Chart, a colour-order system that has gained international acceptance and is widely used for colour determination of geological samples. Soil colour is one of the initial physical properties analyzed in the GSC Sedimentology Laboratory and is performed on the dry bulk sample.

In the GSC Sedimentology Laboratory, Munsell Color is determined using an SP64 Series X-Rite Spectrophotometer linked to Color iControl software. When Munsell values are obtained using the software, results are generated for the following colourimetric systems: CIE XYZ, CIE Yxy, CIE L*a*b*, Hunter Lab, CIE L*c*h, CMC, CIE94, Whiteness and Yellowness per ASTM E313-98, Metamerism Index and DIN 6172 + AATCC Gray Scale. Results cannot be restricted solely to the Munsell Soil Color sub-group. The colour determinations of the samples are provided in **Appendix 04**.

Till matrix carbonate analyses

The analyses of the carbonate content and calcite-dolomite ratio of the <63μm size fraction of the till matrix were carried out in the GSC Sedimentology Laboratory in a two-step process using the CM 5014 Coulometer. Total CO_2 associated to the carbonate content is first measured, followed by the determination of the CO_2 associated to calcite. The difference between the two measurements is used to characterize the amount of CO_2 associated to the presence of dolomite. Depending on the expected CO_2 content the sample-size ranges from 0.02 to 0.50 grams.

Total carbonate is determined by decomposition of the carbonated minerals using hot dilute hydrochloric acid, evolving the carbonate carbon in the form of carbon dioxide. The gaseous carbon dioxide is bubbled to react with a solution of ethanolamine. Through the use of a coloured indicator that fades as the ethanolamine is consumed, a potential is applied to electrodes to replenish the ethanolamine being consumed by redox reaction. The Faraday law is used for the calculation where each Faraday corresponds to one microgram of carbon dioxide.

The calcite fraction of the carbonate minerals is also determined by decomposition as described above. The quantification of dolomite requires the determination of total carbonate carbon. Dolomite is quantified by the difference.

For the 2010 batch, the samples were first screened for inorganic carbon content to prevent the saturation of the analytical instrument and determine the size of sample required for the calcite-dolomite analysis. The determination of inorganic carbonate content involves a LECO (Lincoln Electric Holdings, Inc.) CR-412 Carbon Analyser that relies on combustion of the sample. The released CO_2 is determined by infrared detection. A sample split is ashed at 500°C for 1 hr to remove the organic carbon. The inorganic carbon is determined on the residue. The results include the loss-on-ignition (LOI) at 500°C which are expressed as % weight loss of the dry weight. LOI helps to give a measure of the degree to which the sample geochemistry has been modified by post-depositional weathering. Inorganic carbon is a useful provenance indicator for tills as well as the presence of a buffering agent which could affect the sample geochemistry.

The results for carbonate content and LOI at 500°C are provided in **Appendix 05**. LOI at 1000°C, total carbon and total sulphur are part of the ACME Laboratory's total digestion analytical package (see "total digestion" under section "Geochemical analyses"). The results for the LOI at 1000°C are included in **Appendix 08** and for total carbon and sulphur see **Appendix 09**.

Till clast lithology

Clast lithology data from the till samples were analyzed to assess transportation distance, as well as to help identify major directions of dispersal and till provenance. Clasts (2–80 mm) were sieved from a portion of each till sample collected, and further separated into

clast-size fractions of 2–4 mm, 4–8 mm and 8+ mm (up to 80 mm). Granules or pebbles within each clast-size fraction were then separated according to lithology by M. Trommelen, under an optical microscope. These data are supplemented with field-based counts (20–80 mm), taken at 35 sites from mudboils at the till surface, or from within the sample hole when no mud boils were present (washed and counted in camp). In total, an average of 750 to 800 clasts were counted for each till sample (2–4 mm av = 600; 4–8 mm av =150; 8–80 mm av = 29). For the field count sites, averages of 145 pebbles were counted. The lithological class results are expressed as the number of clasts and are given in **Appendix 06**.

Based on the diverse regional geology (Figure 2), clasts were separated into 23 rock-types (**Appendix 06**). The local sedimentary and volcanic rock types are at low to medium metamorphic grade. Local sedimentary clasts are typically friable, angular to sub-angular in shape, sometimes striated, and coloured brown, grey or black; with very rare burnt-umber red mudstone. Local volcanic clasts are typically resistant, angular to sub-rounded in shape, and coloured black/dark green (mafic to intermediate) or off-white/grey (felsic). Volcaniclastic rocks consist of matrix-supported, poorly sorted felsic to mafic and heterolithic end members, with clasts that generally reflect the matrix composition but occasionally contain rounded clasts of white or orange chert, and quartz. The exotic metasedimentary, metavolcanic and metaconglomerate clasts are typically resistant, rounded to sub-rounded in shape, and coloured red or purple. These rock types include porphyries with white phenocrysts (identified as the Pitz Formation of the Dubawnt Supergroup (Peterson, 2006)) or with phlogopite phenocrysts (identified as the Christopher Island Formation of the Dubawnt Supergroup). Paleozoic carbonate clasts are tan, rounded to subrounded, soft and often partially decomposed, and react with 10% hydrochloric acid when crushed. Iron formation (oxide facies, Anderson et al., 2010a) clasts are typically sub-angular to sub-rounded in shape, rusty brown, and magnetic.

Geochemical analyses

'Total' digestion

For the determination of 48 elements or oxides listed in Table 1, a 0.2 g sample was dissolved by mixing with 1.5 g of lithium metaborate ($LiBO_2$) and lithium tetraborate ($Li_2B_4O_7$) 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 inductively coupled plasma-emission spectrometry (ICP-ES). The same sample solution was analysed for rare-earth elements and refractory elements by inductively coupled plasma-mass spectrometry (ICP-MS).

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.

Element/Oxide	Lower Detection Limit	Element/Oxide	Lower Detection Limit
Al_2O_3	0.01%	Ho	0.02 ppm
CaO	0.01%	La	0.1 ppm
Cr_2O_3	0.002%	Lu	0.01 ppm
Fe_2O_3	0.04%	Mo	1 ppm
K_2O	0.01%	Nb	0.1 ppm
LOI (1000°C)	0.1%	Nd	0.3 ppm
MgO	0.01%	Ni	20 ppm
MnO	0.01%	Pb	1 ppm
Na_2O	0.01%	Pr	0.02 ppm
P_2O_5	0.01%	Rb	0.1 ppm
SiO2	0.01%	Sc	1 ppm
TiO_2	0.01%	Sm	0.05 ppm
Total C (LECO)	0.02%	Sn	1 ppm
Total S (LECO)	0.02%	Sr	0.5 ppm
Ba	1 ppm	Ta	0.1 ppm
Be	1 ppm	Tb	0.01 ppm
Ce	0.1 ppm	Th	0.2 ppm
Co	0.2 ppm	Tm	0.01 ppm
Cs	0.1 ppm	U	0.1 ppm
Cu	5 ppm	V	8 ppm
Dy	0.05 ppm	W	0.5 ppm
Er	0.03 ppm	Y	0.1 ppm
Eu	0.02 ppm	Yb	0.05 ppm
Ga	0.5 ppm	Zn	5 ppm
Gd	0.05 ppm	Zr	0.1 ppm
Hf	0.1 ppm		

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

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 07.** In appendix 7, analytical results which are below detection limit are shown with "<".

Aqua regia ('partial') digestion

For the determination of 65 elements listed in Table 2, a 30 g sample was leached with a mixture of concentrated HCl, HNO_3 and demineralised water (2:2:2 v/v) at 95° C in a beaker for one hour. After cooling the solution (6 mL solute per gram sample) was made up to a final volume with 5% HCl. The ratio of sample weight to solution volume was 0.5 g per 10 mL. The sample solution was analysed by ICP-ES and ICP-MS. The analytical results are presented in **Appendix 08**. Analytical results which are below detection limit are shown with "<".

Geochemical maps for selected elements and LOI are included in **Appendix 10**.

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

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

QUALITY CONTROL FOR GEOCHEMICAL RESULTS

Reliability (accuracy or 'trueness', and precision) of analytical data returned from commercial laboratories was determined by including analytical ('blind') duplicates (BD), 'blanks' and control reference (CR) samples within the sample suite submitted to the labs. Due to the small number of quality control samples analyzed over a two-year period (see Table 3), accuracy, in the sense of the presence or absence of bias (Reimann et al., 2008) could not reliably be determined. Table 3 provides information on the number of each quality control sample within each sample suite, based on the year of collection. Note that (due to insufficient sample material) no control references were analyzed with the 2009 suite of samples sent for trace-metal analysis using an aqua regia digestion, and thus no estimate of trueness is made for these samples. Blanks were inserted to monitor contamination occurring during analysis. Analytical data for control reference standards, analytical and field duplicates, and blanks are included with this report in **Appendix 11**.

	2009				2010			
n	71				159			
Analytical Method	FD	BD	CR	Blank	FD	BD	CR	Blank
$LiBO_2$ Fusion	2	3	4	5	6	8	4	8
Aqua regia/ICP	2	3	0	4	6	8	4	8
Leco	2	3	4	5	6	8	4	8

Table 3. Quality control samples included with 2009 and 2010 till samples. Note that 2009 samples analyzed by aqua regia/ICP did not return quality control data because of insufficient material for analysis (FD – field duplicate; BD - blind duplicate; CR – Control reference).

Unexpected values for several elements (Pt, Bi, Hg, Pb, Ag, Au, Cd, Zr) detected by aqua regia ICP-ES/MS in the silica sand used for blank samples in the 2010 suite led to the reanalysis of 35 samples by the same method. Reanalysis indicated that rather than contamination, an analytical shift during calibration of the ICP-MS system for those analyses was the likely cause of the values above detection limit. Reanalysis data were returned with values at or below the detection limit. New values from reanalysis replaced the original values in the data file.

The variability and high values of the LOI at 1000° C may be the result of sample media variability (i.e., oxidation state, degree of post-glacial weathering, the inclusion of organic material, clay-rich samples). The abundant presence of any of these materials in a sample could affect the concentrations of some elements.

Data quality was evaluated using control reference materials to evaluate trueness, 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 total estimated sampling and analytical variability ('At Sites') and the variability between sample sites ('Between Sites') for mapping purposes. In addition, samples submitted to laboratories were randomised to spread any possible time-dependent errors (bias) at the laboratory between all samples when they were returned to order. This was done in order to randomize any systematic analytical drift across the survey area so that it could not be interpreted as a systematic spatial trend (Reimann et al., 2008.

Tables 1 through 4 in **Appendix 11** can be used to estimate the quality of analysis for almost every analyte found in Tables 1 and 2. Elements (or analytes) are grouped based on their position in the Periodic Table.

Accuracy ('Trueness)

Accuracy in the sense of 'trueness', or closeness of agreement of standard reference materials with accepted reference values of analytical data, was evaluated by inserting Canadian Certified Reference Materials Till-3 (soil) and Till-4 (till) at random locations throughout the analytical suite. These two standards were incorporated into both 2009 and 2010 samples. Till-3 is a combination of B- and C-horizon soil collected 8 km east of Cobalt, Ontario. Till-4, consisting entirely of C-horizon till, was collected near Sisson

Brook, New Brunswick and augmented with a molybdenite-bearing soil collected near Gatineau, Quebec (Lynch, 1996).

In Tables 1 and 2 in **Appendix 11**, means and standard deviations (MEAN ± SD) for control reference standards Till-3 and Till-4, for which provisional values have been published by Lynch (1996), are shown with detection limits (DL), standard deviation (SD) and Relative Standard Deviation (RSD) for elements by total (n=6) and partial (n=4) methods from repeated analyses of reference standards Till-3 and Till-4. Relative Standard Deviation (RSD), expressed as a percentage, facilitates comparison of the repeatability of elements measured in different units and varying means (Reimann et al., 2008. RSD is independent of both the magnitude of the data and the units. Accepted values in square brackets are derived from unpublished data ($n \geq 10$) collected from recent projects at the GSC.

For Till-3 and Till-4, for analytes for which an accepted mean exists, almost all analytes are within one Standard Deviation of an accepted mean. Elements with possible analytical problems, as indicated by a relatively high (>33%) Relative Standard Deviation (RSD), are shown in bold type. However, a relatively high RSD, suggesting poor repeatability, may also be an indication that analytical results are close to the detection limit for the element or analyte.

Precision

Precision is considered in terms of the closeness of agreement between analytical duplicate samples analyzed by the same method, i.e., independent test results obtained using the same equipment within short intervals of time on duplicate project samples. In order to provide an estimate of precision for each element or analyte, the squared difference between two analytical duplicates was calculated for N = 11 duplicate pairs. The sum of these values was divided by the number of samples ((2*N) = 22) to estimate a measure of variability (variance). A Standard Deviation was then obtained by calculating the square root of this variance. The resulting numerical estimates of precision are shown in Table 3 in **Appendix 11** represented by the Relative Standard Deviation, where 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 3.

Elements exceeding a precision of 20% in Table 3 in **Appendix 11** 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 Au, Be, Hg, Na, Cd, Sb, In, B, Ge, Te and Se by partial methods, and Be, Ni, Mo, Pb and Sn by total methods.

Analysis of Variance (ANOVA)

Field duplicates are used to estimate the combined variation due to sampling and analysis between samples collected within a few metres of each other. Field duplicate samples were collected at eight field sites over a two-year period to provide means of estimating variability introduced by field sampling procedures and by sediment heterogeneity. The combined analytical and sampling variability can be estimated from these sample pairs using Analysis of Variance (ANOVA). Using the 'anova2' function found in the 'rgr' package running under the R system, a random effects model Analysis of Variance (ANOVA) determines the combined sampling and analytical variability between sets of duplicate field samples (Garrett, 2011). This combined variability is more important than analytical variability alone because if the combined sampling and analytical variability is not significantly smaller than the field survey variability, it cannot be stated that there are statistically significant spatial patterns in the data, and thus the data are likely not suitable for mapping (Garrett, 2011), nor are sophisticated methods of data manipulation recommended (Reimann et al., 2008; Garrett, 1969).

Table 4 in **Appendix 11** shows results for all elements in Tables 1 and 2 (above). Elements (or analytes) are grouped based on their position in the Periodic Table.

The Analysis of Variance (ANOVA) of field duplicates partitions variability into two components, 'Between Sites' and 'At Sites' in **Table 4, Appendix 11**. The fourth column, 'F', lists the results of formal F-tests to determine the significance of the variability of the previous two columns, while the last column, 'p-value', confirms the significance of the F-test.

For this set of till data, a value for 'F' of 3.1 or greater suggests that sampling variation significantly exceeds the analytical variation, with a 95% or better degree of confidence that data can be used to prepare maps. Generally speaking, confidence increases with increasing F and decreasing p-values. It should be noted that in cases where an element or oxide is evenly distributed throughout all samples, as is the case with SiO_2, 'F' and 'p-values' may fall below the level of confidence.

The ANOVA indicates that the sampling and analytical variability is significantly lower than the field survey variability, at the $p < 0.05$ level (>95% confidence level) for analytes not in bold print in Table 4, **Appendix 11**. From this it is inferred that maps of the distribution of these elements will display the true spatial variability of those analytes.

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Graduated symbols plotted over bedrock geology are used to illustrate relative values of elements in tills. Data are classified into ranges using the Jenks natural breaks classification method available in Arcmap®.

The distribution of data for selected elements is graphically illustrated using histograms, cumulative normal percentage probability (CPP) plots, and Tukey boxplots, together with a table of selected percentiles of the data and summary statistics. Histograms, CPP plots and summary statistics were generated using the 'inset' function found in the 'rgr' package running under the R system (Garrett, 2011). Tukey boxplots were generated using the 'bxplot' function from the same source. For further information and a discussion of these graphic methods of displaying data distribution, see Reimann *et al*. (2008).

11. Till matrix geochemistry – quality control

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