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GEOLOGICAL SURVEY OF CANADA
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Geological Survey of Canada till-sampling and analytical protocols: from field to archive, 2020 update

M.B. McClenaghan, W.A. Spirito, A. Plouffe, I. McMartin, J.E. Campbell, R.C. Paulen, R.G. Garrett, G.E.M. Hall, P. Pelchat, and M.S. Gauthier

2020

Canada

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Geological Survey of Canada till-sampling and analytical protocols: from field to archive, 2020 update

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ABSTRACT

For more than 50 years, researchers at the Geological Survey of Canada (GSC) have developed, tested, and refined till geochemical and indicator mineral methods as applied to mineral exploration, provenance studies, and environmental research in glaciated terrain across Canada. This cumulative experience and knowledge were used to produce and publish the GSC's first comprehensive field and laboratory methods protocol manual for till geochemical and mineralogical surveys in 2011. The publication being presented here provides an update and augmentation of this earlier version and presents the major concepts of till as a sample medium, glacial dispersal, and field and laboratory procedures.

These protocols are used by the GSC to guide till sample collection, sample processing, geochemical and indicator mineral analyses, implementation of quality assurance/quality control (QA/QC) procedures, archiving methods, and data reporting by the Geological Survey of Canada. Using consistent sample media and making diligent field notes and observations are also considered fundamental to the protocols, and are presented herein. The protocols will be of value to provincial/territorial government geological agencies, the mineral exploration industry, and academia and we hope they will benefit from the use of this manual.

Adopting a common set of protocols allows the GSC, other researchers, and exploration geologists to directly compare till geochemical and indicator mineral data sets from various parts of Canada and ensures proper minimum levels of QA/QC for all till geochemical and mineralogical data.

1. INTRODUCTION

1.1. Purpose of this protocol manual

From 2008 to 2020, Natural Resources Canada undertook a major initiative of the Canadian government, the Geo-mapping for Energy and Minerals (GEM) Program (https://www.nrcan.gc.ca/earth-sciences/resources/federal-programs/geomapping-energy-minerals/18215). This program was initiated to advance geological knowledge in Canada's North in support of increased exploration for natural resources and to provide information for decisions about land use that balances conservation with responsible resource development. These objectives led to the implementation of numerous projects by the Geological Survey of Canada (GSC), conducted in collaboration with territorial and provincial geological surveys and academia through the supervision of graduate students. Several of these GEM projects involved surficial geological mapping and till sampling.

In the early years of the GEM Program, from 2008 to 2010, a working group of surficial mappers and geochemists from the GSC's Northern Canada Division prepared the first version of this protocol manual (Spirito et al., 2011; Geological Survey of Canada, 2017) to establish consistent methodologies for till sampling studies performed by all GEM projects. The inspiration for establishing these till sampling protocols was the national guidelines already established by the GSC for the collection and analysis of regional lake (Friske, 1991; Friske and Hornbrook, 1991) and stream sediments (Ballantyne, 1991; Friske and Hornbrook, 1991; McCurdy et al., 2014).

This update to the initial 2011 version includes adaptations, modifications, and new methods that have arisen primarily as a result of technological developments. It also ensures that future GSC projects use common methods for till sample collection (sections 3 to 6), quality control (Section 7), geochemistry sample preparation (Section 8), geochemical analysis (Section 9), and metadata (Section 10), as well as common techniques for indicator mineral preparation, analysis, and recording metadata (Section 11). The adoption of these protocols will ensure that consistent sample media and diligent field notes are collected, which will facilitate comparisons of data sets collected from across Canada, regardless of where or when they are collected or under which GSC program or project, such as the numerous projects of the Targeted Geoscience Initiative (TGI). The protocols outlined here may also been used to guide provincial and territorial geological surveys and the exploration industry for till sampling surveys. Two scientific papers that summarize the protocols for till sampling, sample preparation, and geochemical analy-

sis (McClenaghan et al., 2013a) and indicator mineral analysis (Plouffe et al., 2013) have been published in the peer-reviewed journal *Geochemistry: Exploration, Environment, Analysis* to make these protocols available to a wide readership.

The authors envision that this protocol manual will continue to be updated as technology evolves and new ideas and concepts are put forward and tested.

Comments and feedback are welcome and can be communicated to Janet Campbell, Beth McClenaghan, Isabelle McMartin, Roger Paulen, Alain Plouffe, or Wendy Spirito.

1.2. Geological Survey of Canada research

In glaciated terrain, determining the bedrock source of the lithological, mineralogical, or geochemical constituents of the till for the purpose of mineral exploration is known as drift prospecting. This exploration method has a long history, starting with boulder tracing in the 1700s (*see* references in Sauramo, 1924; Kauranne et al., 1992) and 1800s (*see* references in Dreimanis, 1958). Other early pioneers of drift prospecting in glaciated terrain include Milthers (1909), Grip (1953) and Kauranne (1958) in Fennoscandia and Prest (1911), and Lee (1963, 1965) in Canada. Through ongoing research in glaciated landscapes, drift prospecting methods evolved from the 1970s (Shilts 1971, 1972, 1973, 1975; DiLabio, 1979), onward through the 1980s (Shilts, 1984, 1993; Coker and DiLabio, 1989; DiLabio and Coker, 1989) and 1990s (Kujansuu and Saarnisto, 1990; Kauranne et al.,1992; Bobrowsky et al.,1995). Over the past 20 years, GSC research has greatly expanded to meet the growing demand of mineral exploration with significant advancements in analytical methods (McClenaghan et al., 2000, 2001; Paulen and McMartin, 2009; Ferbey et al., 2017; McClenaghan and Paulen, 2018). These studies have provided industry with the knowledge upon which they have been able to adapt drift prospecting techniques and methods that have contributed to many mineral deposit discoveries (*see* Appendix A *in* McClenaghan and Paulen, 2018).

The GSC's study of glacial sediment composition and glacial dispersal mechanisms and patterns since the 1970s has served not only the mineral exploration industry but also environmental geology, for which it has provided baseline data on surficial sediment composition. Till compositional data have been used as a clear indicator of the natural variation of elemental concentrations in the near-surface environment across geological terranes with various environmental applications, such as the study of acid rain (e.g. Kettles and Shilts, 1983; Kettles and Wyatt, 1985) and metals in the environment (e.g. Plouffe, 1995a, 1998; McMartin et al., 1996, 1999, 2002; Henderson et al., 1998).

GSC reconnaissance- to regional-scale till sampling surveys are often undertaken in combination with regional surficial geology mapping projects under the general mandate of stimulating mineral exploration (e.g. Kaszycki, 1989; Plouffe et al., 1995a, 2001a; McMartin et al., 2013, 2015) and, less commonly, for environmental studies (e.g. Plouffe, 1995a; McMartin et al., 2006; McMartin, 2009). GSC local-scale studies near known mineral deposits are completed to provide insights into metal concentrations in till overlying and at various distances down-ice of known mineralization (e.g. DiLabio, 1981, 1982; Coker et al., 1991; McClenaghan et al., 2002, 2011, 2016, 2017a,b, 2018a,b; Parkhill and Doiron, 2003; Plouffe et al., 2016).

Significant methodological developments have occurred over the past 50 years, largely related to new analytical methods (Hall and Bonham-Carter, 1988; Hall, 1991; Noras, 1992; Hall et al., 1996). Focused till studies have identified pathfinder elements for a broad range of mineral deposit types (Table 1) and have demonstrated that elements can be enriched in specific size fractions and that they may be controlled by primary and/or secondary mineralogy (e.g. DiLabio, 1982, 1985, 1988; Shilts, 1984, 1995, 1996; Plouffe, 2001b; McMartin, 2009). At the same time, detailed studies of minerals in specific density fractions of till matrix have yielded advances in mineralogical characterization (Table 1; e.g. McClenaghan et al., 2002, 2016, 2017a,b, 2018a,b; McClenaghan, 2005; McMartin et al., 2011; Plouffe et al., 2016). Although there has been significant methodological advancements, there has been limited consistency in the application of a multitude of chemical digestions, analytical determination methods, size fractions, and mineralogical fractions being utilized.

One of the strengths of regional till sampling surveys is that they cover large areas. This large coverage allows correlations to be made between bedrock geology and glacial transport, as well as observations about the natural variability across major geological entities. However, one of the weaknesses of regional surveys has been the limited comparability among the data sets due to the variability of the methodologies employed by each project. The protocols outlined in this manual, and now formally adopted by the GSC, have eliminated this problem for GSC till survey data sets published after 2010.

1.3. Acknowledgments

This protocol manual is the result of a dynamic collaboration of the Working Group members (authors) and many past and present GSC colleagues including (listed in alphabetical order): S.W. Adcock, R. Fortin, I. Girard, A.G. Grenier, B. Harvey, I.M. Kettles,

Table 1. Common indicator minerals and pathfinder + indicator elements for selected mineral deposit types in glaciated terrain and selected review publications and examples (*modified from* McClenaghan and Paulen, 2018).

Deposit type	Ore elements	Indicator/pathfinder elements	Till geochemistry published reviews and examples	Common indicator minerals	Indicator mineral published reviews and examples
Kimberlite-hosted diamonds	C	Ba, Cr, K, LREE, Mg, Nb, Ni, P, Rb, Sr, Ta, Ti	McClenaghan et al., 2002; Lehtonen et al., 2005; McClenaghan and Kjargsaard, 2007	Cr-pyrope, Cr-diopside, eclogitic garnet, Mg-ilmenite, chromite, diamond	McClenaghan et al., 1996, 1998, 1999a,b, 2002, 2004, 2012; McClenaghan and Kjarsgaard, 2001, 2007; Lehtonen et al., 2005
Volcanogenic massive sulphide	Ag, Au, Cu, Pb, Zn	Ag, As, Au, Ba, Bi, Cd, Cu, Hg, In, Pb, S, Sb, Tl, Zn	McClenaghan et al., 2015b; McClenaghan and Peter, 2016	chalcopyrite, sphalerite, galena, pyrrhotite, gold, pyrite, gahnite, staurolite, cassiterite, spessartine, sillimanite, andalusite, beudantite, jarosite, barite, tourmaline, hogcomite, nigerite	Lalonde et al., 1994; Morris et al., 1997; Averill, 2001; Paulen et al., 2013; McClenaghan et al., 2015b, 2017b
Carbonate-hosted lead-zinc	Ag, Cu, Pb, Zn	Ag, Cu, Pb, S, Zn	Tarplee and van der Meer, 2010; Oviatt et al., 2015; McClenaghan et al., 2018a	chalcopyrite, sphalerite, galena, pyrite, barite, spessartine, smithsonite, anglesite, cerussite	Paulen et al., 2011; Oviatt et al., 2015; McClenaghan et al., 2018a
Gold	Ag, Au	Ag, As, Au, B, Ba, Bi, Cu, Co, Fe, Hg, Mn, Sb, Se, Te, U, W	Peuraniemi, 1991; McClenaghan, 2001; Plouffe, 2001; Sarala et al., 2009; Sarapää and Sarala, 2013	gold, scheelite, tourmaline, rutile, sulphides, tellurides, PGM, barite, cinnabar	Averill and Zimmerman, 1986; Peuraniemi, 1990; Averill, 2001, 2013, 2017; McClenaghan, 2001; Plouffe, 2001; Sarala et al., 2009; McClenaghan and Cabri, 2011; Sarapää and Sarala, 2013; Manéglia et al., 2018
Magmatic Ni-Cu-PGE	Cu, Ni, PGE	As, Au, Cr, Cu, Mg, Ni, PGE, S	Barnett, 2007; McClenaghan and Cabri, 2011; McClenaghan et al., 2011, 2013b, 2019c	pentlandite, chalcopyrite, pyrite, millerite, PGM, chromite, Cr-diopside, enstatite, olivine, Cr-andradite	Averill, 2009, 2011; McClenaghan and Cabri, 2011; McClenaghan et al., 2013b; 2018b, 2019c
Rare metals	Rare metals	Be, Ce, Cl, F, Li, Nb, U, P, REE, Ta,Th, Y, Zr	Batterson, 1989; Batterson and Taylor, 2009	pyrochlore, columbite, Ta-minerals, allanite, zircono-silicates, apatite, monazite, fluorite, rhabdophane, arfvedsonite	Lehtonen et al., 2011, 2015; Sarapää and Sarala, 2013; Mackay and Simandl, 2015; Mao et al., 2015; McClenaghan et al., 2019a
Porphyry Cu-Au-Mo	Ag, Au, Cu, Mo	Au, Ag, Cu, Mo, S	Hashmi et al., 2015; Plouffe et al., 2016	chalcopyrite, chalcocite, pyrite, molybdenite, gold, silver, epidote, tourmaline, apatite, andradite, barite, monazite, rutile, titanite, zircon, jarosite, malachite, pyrolusite	Averill, 2011; Kelley et al., 2011; Hashmi et al., 2015; Chapman et al., 2015; Plouffe et al., 2016; Pisiak et al., 2017; Plouffe and Ferbey, 2017; McClenaghan et al., 2019b
Intrusion-hosted Sn-W	Mo, Sn, W	Ag, As, Be, Bi, Cd, Cu, F, In, Mo, Pb, S, Te, W, Zn	Snow and Coker, 1987; McClenaghan et al., 2014, 2016	cassiterite, scheelite, wolframite, molybdenite, chalcopyrite, Bi sulphides, sulphides, fluorite, topaz, tourmaline	McClenaghan et al., 2016, 2017a
Iron oxide-copper-gold	Au, Cu, Fe	As, Au, Co, Cu, Fe, Mo, Ni, U	McMartin et al., 2009; Normandeau, 2018	magnetite, gold, apatite	McMartin et al., 2009, 2011; Lypaczewski et al. 2013; Sappin et al., 2014; Normandeau et al., 2018
Uranium	U	As, Ba, Cu, F, La, Ni, P, Pb, Th ,Ti, U, Y, Zn, Zr	Boyle, 1982; Geddes, 1982; Simpson and Sopuck, 1983; Campbell, 2009	uraninite (*pitchblende), thorianite, tourmaline, sulphides, monazite, allanite, zircon, baddelyite, niccolite, U-Th anatase, U-Th rutile, brannerite, magnetite	Geddes, 1982

Note: PGE = platinum group elements; PGM = platinum group minerals; REE = rare earth elements
* pitchblende: brown or black pitchy massive form of uraninite

Figure 1. Photographs of till deposited by actively flowing glaciers: **a)** a poorly sorted sediment deposited by glaciers consisting of granule- to boulder-sized clasts in a matrix of clay- to sand-sized particles (knife handle is 8 cm long); **b)** massive, unsorted, stony subglacial till displaying no structure; **c)** weakly jointed, strongly fissile silty-sand subglacial till (pick head is 40 cm wide); **d)** strongly jointed, weakly fissile sandy-silt subglacial till (diameter of the coin is 2.4 cm).

B.A. Kjarsgaard, R. Laframboise, S. Madore, C. Moore, J.M. Rice, I.R. Smith, L.H. Thorleifson, and M. Wygergangs. The manual was produced and updated as a contribution to the GSC's GEM and TGI programs. J. Bond (Yukon Geological Survey) is thanked for providing Figure 5. A. Bajc (Ontario Geological Survey) provided advice about mud rotary coring. Page layout of this open file report was provided by E. Ambrose.

Valuable reviews and comments about this 2020 version of the manual were provided by the following:

- Rod Smith, Geological Survey of Canada;
- Michael Parkhill, New Brunswick Department of Natural Resources and Energy Development;
- Heather Campbell, Geological Survey of Newfoundland and Labrador;
- Denise Brushett, Nova Scotia Department of Lands and Forestry.

2. TILL AS A SAMPLING MEDIUM

2.1. Overview

Till is a non-sorted mixture of sediment, ranging from fine clay to large boulders (Fig. 1a) deposited directly from or by a glacier with little or no sorting by water (Dreimanis, 1989). Till is a product of glacial erosion, entrainment, transportation, and depositional processes.

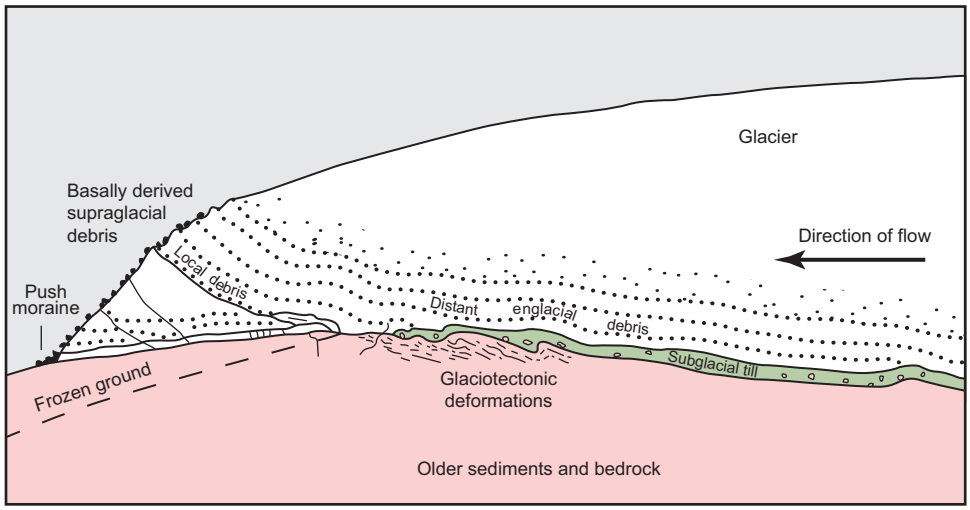

Figure 2. Simplified cross-section through a glacier showing the source areas and the relative positions of supraglacial and subglacial debris relative to the glacier. Pathways of transport of the glacial debris are also shown (*from* Dreimanis, 1990).

In Canada, reconnaissance-scale to local-scale till sampling surveys have been conducted by exploration companies and provincial, territorial, and federal organizations. Till is an important sample medium because it is typically the first derivative of bedrock, that is, it was eroded, transported, and deposited by a single sedimentary process (i.e. glacial ice movement). Till generally has a simpler sedimentary transport history than secondary derivatives or higher sediments (e.g. glaciofluvial sediments, stream sediments, beach sediments, colluvial sediments) that may have been transported by more than one sedimentary process (Shilts, 1976).

Genetically, till is divided into two main groups, subglacial and supraglacial, the latter of which can include englacial and glaciotectonized sediment (Dreimanis, 1990). Lian and Hicken (2017) provide a useful illustrated summary of till types and their distinguishing characteristics. Subglacial till, commonly referred to as basal or traction till, is the optimal sample medium because it was deposited at the base of an actively flowing glacier and is often more locally derived. The characteristics of the two major till types from Lian and Hicken (2017) are summarized below.

2.1.1. Subglacial till

Subglacial refers to the position of emplacement of till at the base of the glacier. There, till can be advected when the force of friction between the deformation front and the sole of the moving glacier becomes stronger than the force of traction, which results in a lodgement till. Till can also be deposited at the base of glacier by melt-out, deformation, comminution, and glaciotectonism (glaciotectonite—bedrock or sediments sheared by overriding ice). Till emplacement and accretion is a time-transgressive process that reflects the glacial dynamics of the ice sheet that forms it (Menzies et al., 2019).

Characteristics: over-consolidated, dense, matrix-supported, massive (Fig.1b,c) or jointed (Fig. 1d), may display moderate to strong subhorizontal fissility, and contain striated and faceted clasts that are typically subangular to subrounded.

Suitability for mapping and drift prospecting studies: the optimal sample medium because it was deposited at the base of an actively flowing glacier (Fig. 2) and in most cases is locally derived. It may occur above bedrock at any depth within an unconsolidated sediment succession (Levson, 2001a; Paulen, 2009).

2.1.2. Supraglacial till

Supraglacial till is deposited within or on top of glacial ice. It may also be referred to as ablation till, as it is generally deposited by melting of stagnant ice. It includes till deposited by supraglacial melt-out and sublimation processes and also can contain subglacial till sequences typically thrusted and buried at glacial margins. It also may include till deposited subglacially by gravity mass flow or at ice margins.

Characteristics: typically massive to crudely stratified, often with structural folding or soft sediment deformation, matrix- or clast-supported, and may contain lenses of sorted silt, sand, and gravel (Fig. 3). Clasts range from angular to subrounded. Striated clasts may be present but are less abundant than in subglacial till.

Suitability for mapping and drift prospecting studies: less optimal for drift prospecting and provenance studies because it represents more of the englacial and supraglacial sediment load of the glacier (Fig. 2) and, in most cases, it is more distally derived.

The above descriptions aid in choosing the optimal material (subglacial till) to sample. This simple classification is useful in till sampling surveys in which detailed sedimentological analyses are not conducted at

Figure 3. Photograph of sandy supra- glacial till exposed in a coastal section. Metre stick in the foreground is 2 m. Photograph courtesy of R. Paulen.

every sampling site. However, it is not meant to exclude or limit detailed measurements (e.g. till fabrics, faults, joints) and observations of sediment and landforms that could serve to classify and interpret sediment genesis. Surficial geology mapping, along with till sampling, can provide additional information about till genesis. For further details about classifications of till, the reader is referred to Dreimanis (1989, 1990), Benn and Evans (1998), Evans et al. (2006), Bennett and Glasser (2009), Lian and Hicken (2017), Evans (2018), and Menzies and van der Meer (2018), amongst others.

2.2. Glacial dispersal

Glacial dispersal trains are three-dimensional bodies comprising till that has been enriched with material eroded from a discrete bedrock source (e.g. mineralized bedrock or specific bedrock lithology) and transported by the glacier in a down-ice direction (DiLabio, 1990a; Parent et al., 1996; Klassen, 2001). They have much larger surface footprints than the original bedrock source and, in most cases, have mineral concentrations that rapidly decrease down-ice. (Fig. 4). Shilts (1976) and others (cf. McClenaghan and Paulen, 2018) have demonstrated that dispersal trains are the products of erosion, glacial transport, and re-entrainment.

Glacial dispersal trains occur at (Fig. 5a,b) or below the till surface (Fig. 5c,d), generally at a distance displaced down-ice of their source. Dispersal trains usually have a defined head—an area of highest dispersed concentration—that is usually immediately down-ice of the mineralized source, and a tail of decreasing concentration of material resulting from down-ice dispersal and dilution by nonmineralized sources. In cross-section, dispersal trains ascend vertically in glacial stratigraphic deposits (Fig. 6; e.g. Drake, 1983; Miller, 1984) as distance down-ice increases and concentrations of the eroded bedrock lithology or mineralization gradually become more dilute (Stanley, 2009). Many dispersal trains have abrupt lateral edges.

In the simplest scenario, glacial dispersal from a single point source, such as a kimberlite pipe (Fig. 7), occurring in a region that was affected by a single sustained ice flow, typically has a ribbon-shaped train of variable length and thickness that is as wide as the bedrock source (Fig. 5a,c) (Batterson, 1989; DiLabio, 1990a; McClenaghan et al., 2002; Stea et al., 2009). Glacial dispersal from a broader mineralized zone, such as a shear-hosted gold deposit, a volcanogenic massive sulphide (VMS) deposit, or a kimberlitic dyke oriented perpendicular or oblique to ice flow, will result in a broader shaped train (Fig. 8) (e.g. Parkhill and Doiron, 2003; Strand et al., 2009).

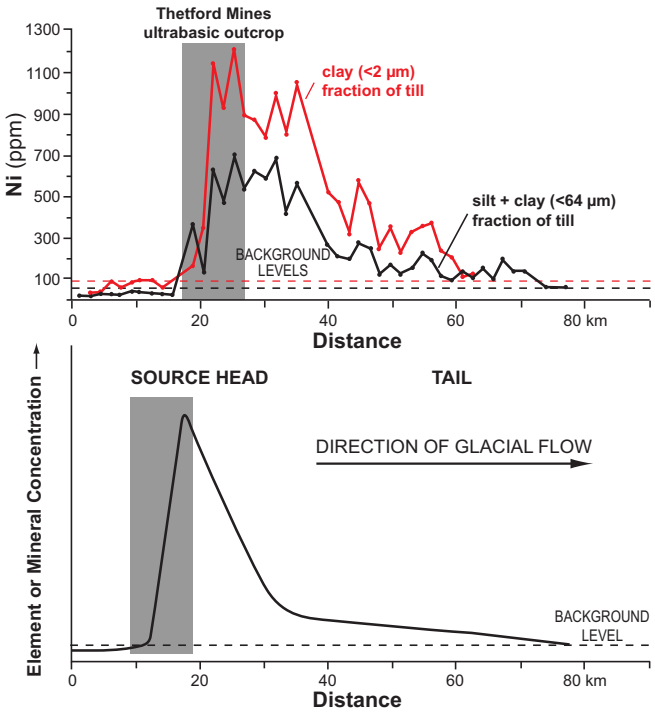

Figure 4. Exponential dispersal curves for nickel (ppm) content in surface till in the Thetford Mines area, central Canada. Actual (top) and idealized (bottom) curves show the relationship between the source, head, and tail of a dispersal train (after Shilts, 1976).

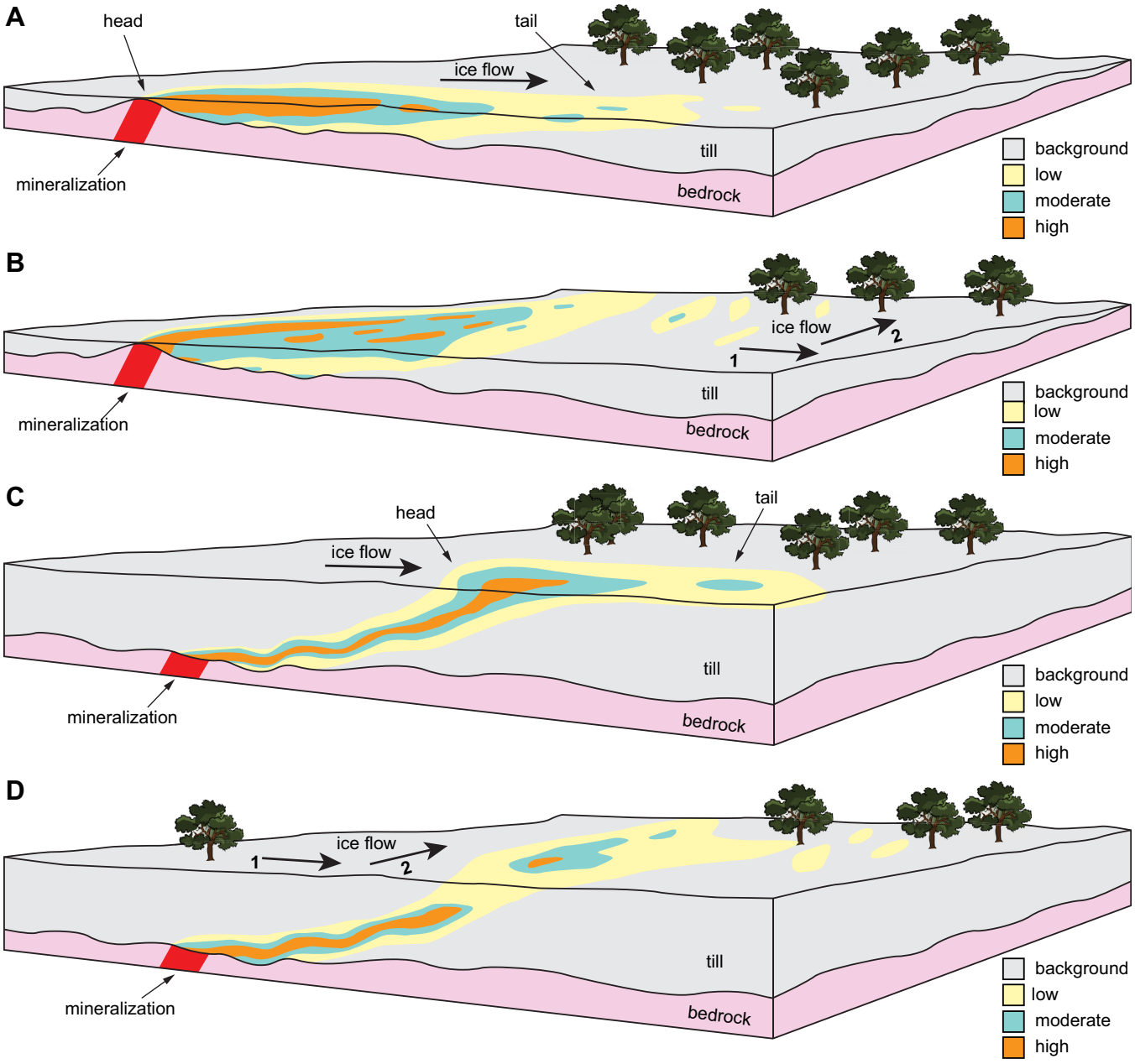

Figure 5. Conceptual models of glacial dispersal trains that exhibit a buried component, a head, and a tail, which can be modified or re-entrained by subsequent ice movements: **a)** thin till cover where mineralized bedrock is dispersed by a single phase of ice flow, creating a dispersal train that is exposed at surface for its entire length and trends down-ice in one direction; **b)** thin till cover where mineralized bedrock is dispersed by two phases of ice flow, creating a palimpsest dispersal train exposed at surface and reflecting two ice-flow directions; **c)** thick till cover where mineralized bedrock is dispersed by a single phase of ice flow, creating a dispersal train that is buried proximal to source and exposed at surface farther down-ice (the train trends down-ice in one direction); and **d)** thick till covers where mineralized bedrock has been dispersed by two phases of ice flow, creating a palimpsest dispersal train, which indicates two ice-flow directions. The train is buried proximal to source and exposed at surface farther down-ice. The original dispersal train created by the earlier ice flow (1) shown in (a) and (c) is diluted and partially redeposited offset by subsequent ice flow (2) as shown in (b) and (d) (*from* McClenaghan and Paulen, 2018).

Multi-phase ice flow across a single bedrock source may (1) produce a fan-shaped train (Fig. 9; e.g. Rogers et al., 1990; Lehtonen et al., 2005; McClenaghan et al., 2012, 2015a,b); (2) significantly modify the original dispersal train and produce a new palimpsest train (Fig. 5b,d; Parent et al., 1996; Stea and Finck, 2001; Stea et al., 2009; McClenaghan and Paulen, 2018) such as shown in the model in Figure 10 or a palimpsest pattern over a broad area (Fig. 11); or (3) minimally modify the original train and produce a new dispersal train in the new ice-flow direction(s), resulting in a bilobate form (Fig. 12) (e.g. Charbonneau and David, 1993; Shilts, 1993; Stea et al., 2009) or multi-lobed (amoeboid-shaped) dispersal pattern (Fig. 13) (e.g. Stea and Finck,

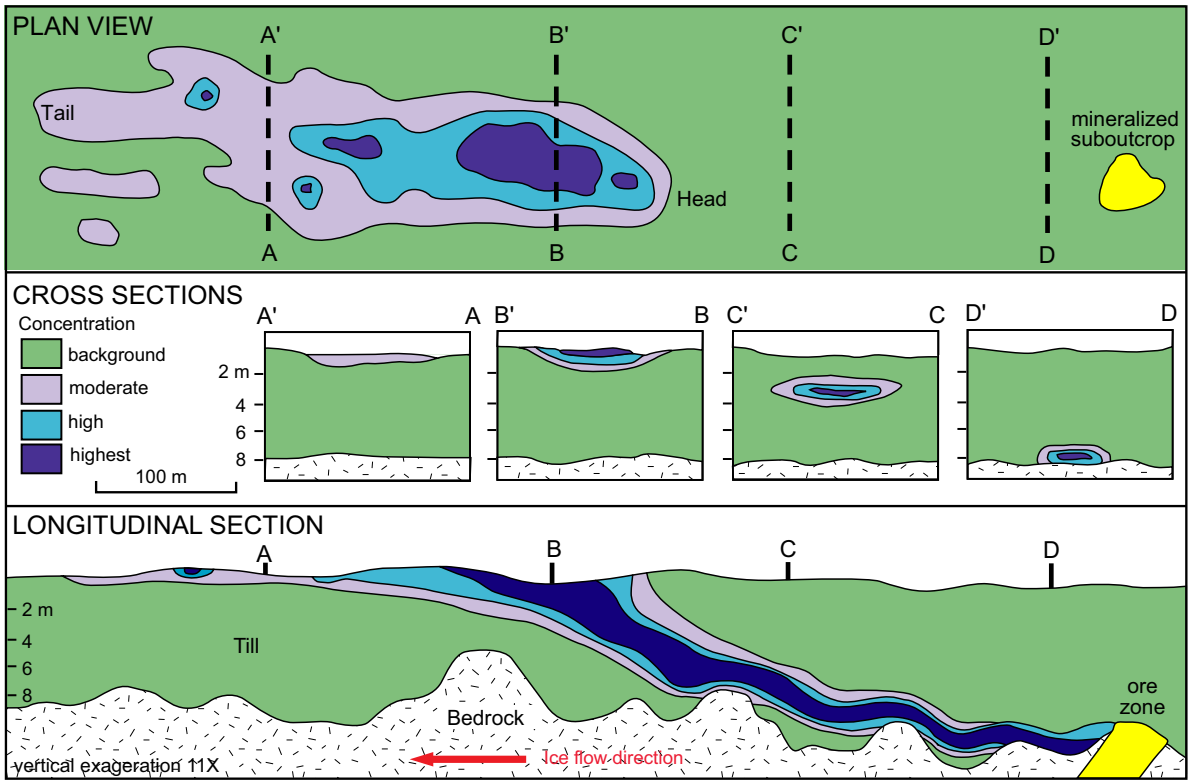

Figure 6. Schematic representation of a geochemical or mineralogical glacial dispersal train formed by a single phase of ice flow across a mineralized bedrock source, colour contoured using hypothetical 90th, 95th, and 98th percentiles: **a)** plan view; **b)** cross-sectional view; and **c)** longitudinal section. *Modified from* Miller (1984).

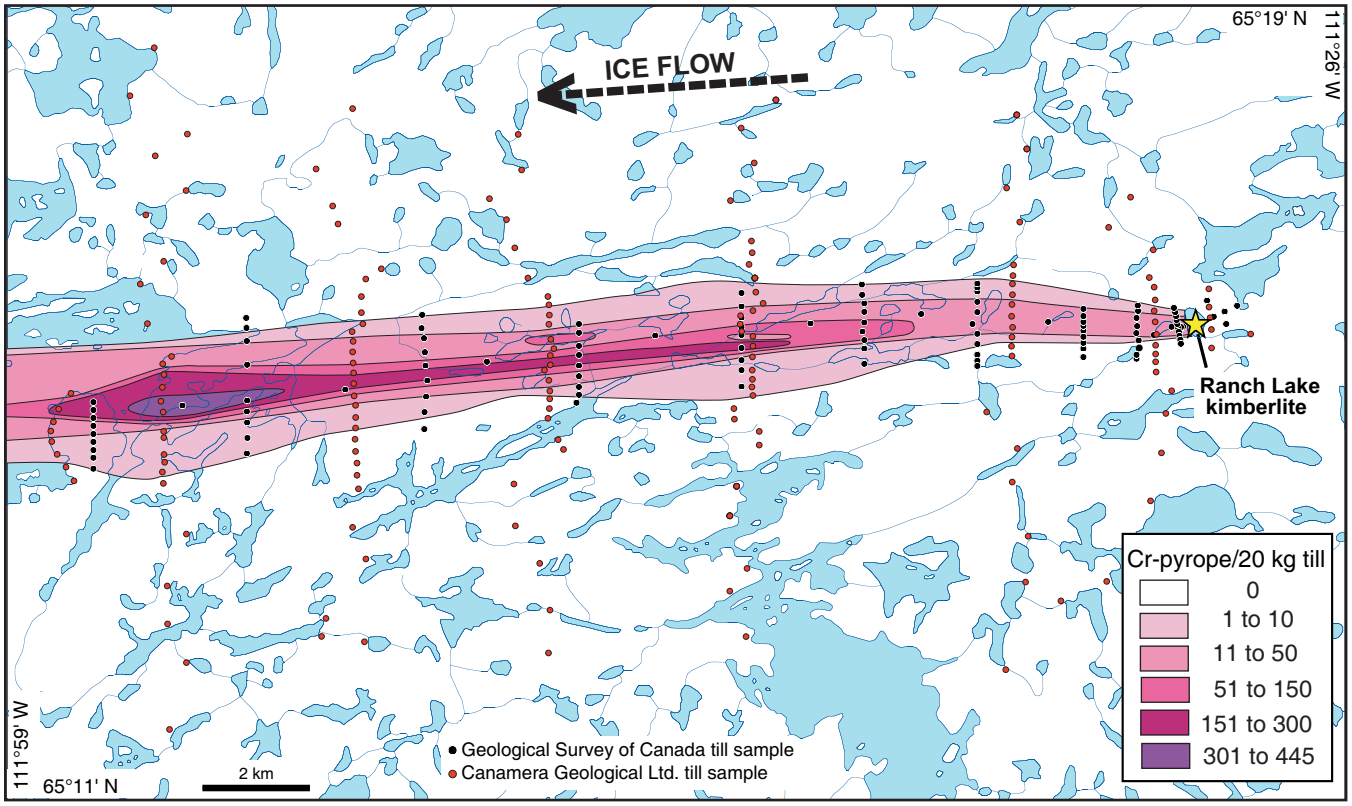

Figure 7. Ribbon-shaped glacial dispersal train trending westward from the Ranch Lake kimberlite pipe, Lac de Gras, NWT, defined by Cr-pyrope concentrations in the 0.25–0.5 mm heavy mineral fraction of 20 kg till samples. The train was formed by a single phase of ice flow towards the west (*modified from* McClenaghan et al., 2002).

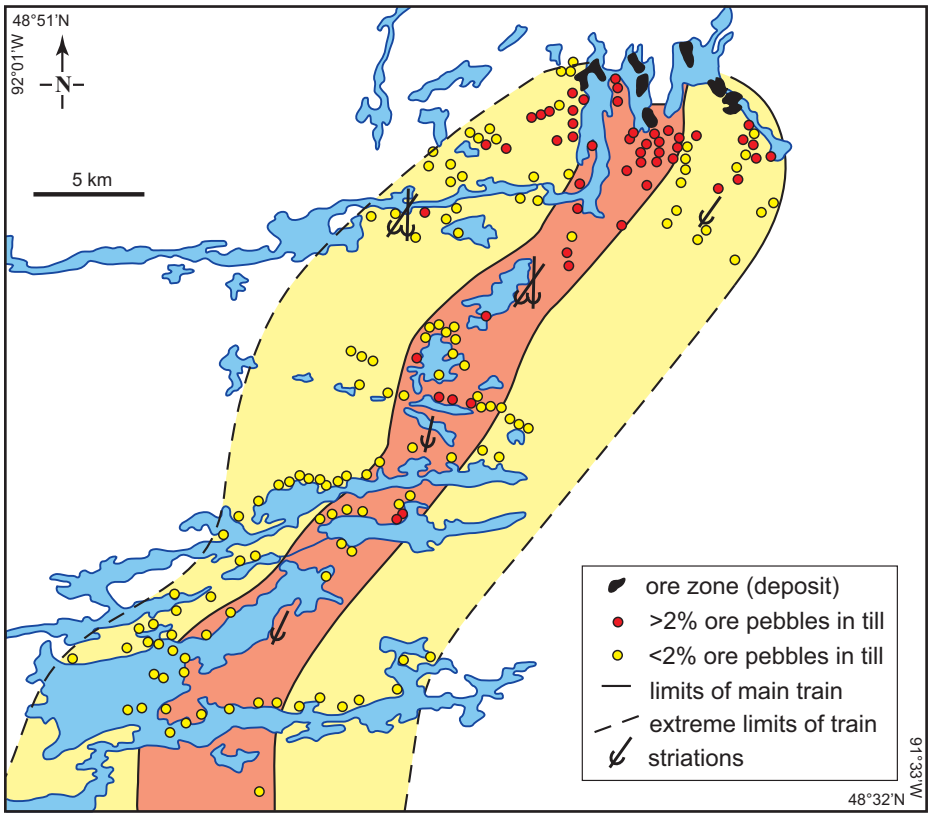

Figure 8. Distribution of iron ore pebbles in till down-ice of the 8 km wide Steep Rock Lake iron deposit in central Canada (*modified from* Nichol and Bjorklund, 1973, *after* Dreimanis, 1958).

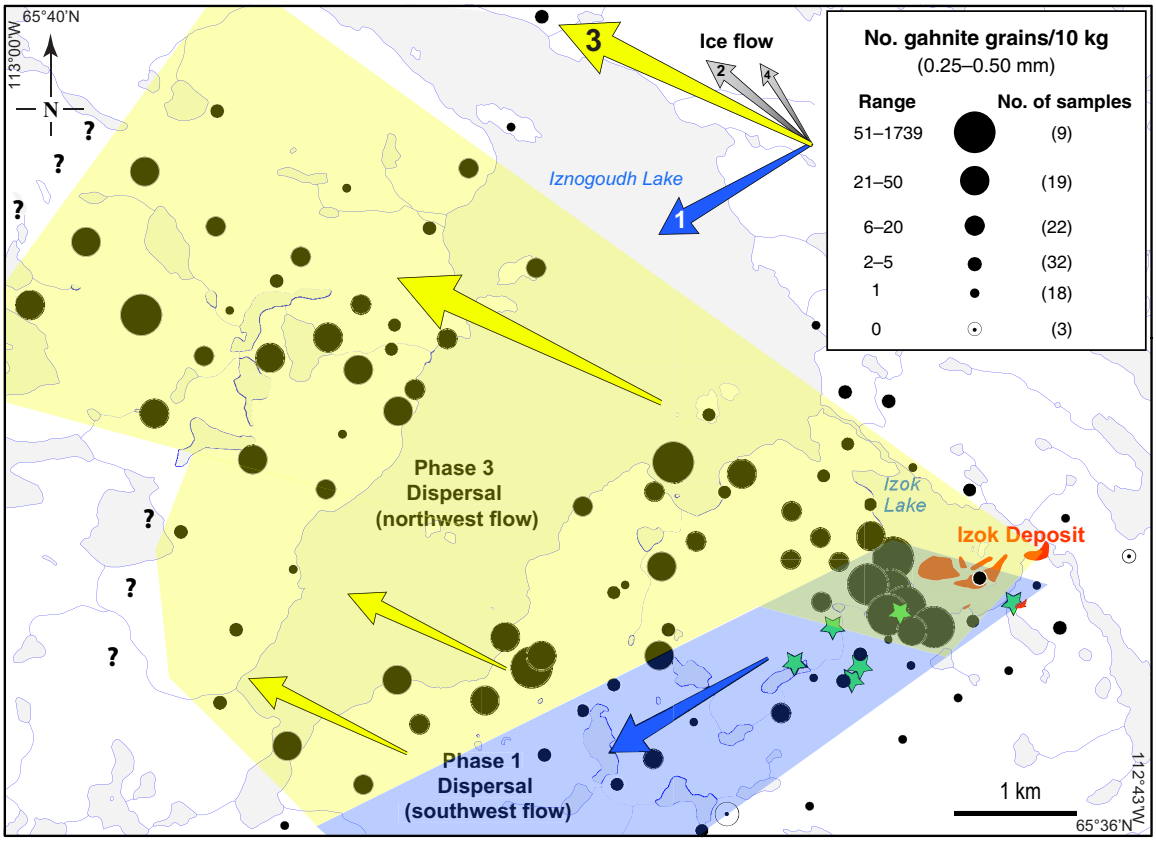

Figure 9. Fan-shaped dispersal train formed by multiple phases of ice flow to the southwest and northwest across the Izok Lake volcanogenic massive sulphide deposit in Nunavut. The train is defined by the abundance of visually identified gahnite (Zn spinel) grains in the 0.25 to 0.5 mm nonferromagnetic heavy mineral fraction of 10 kg till samples (*from* McClenaghan et al., 2015a).

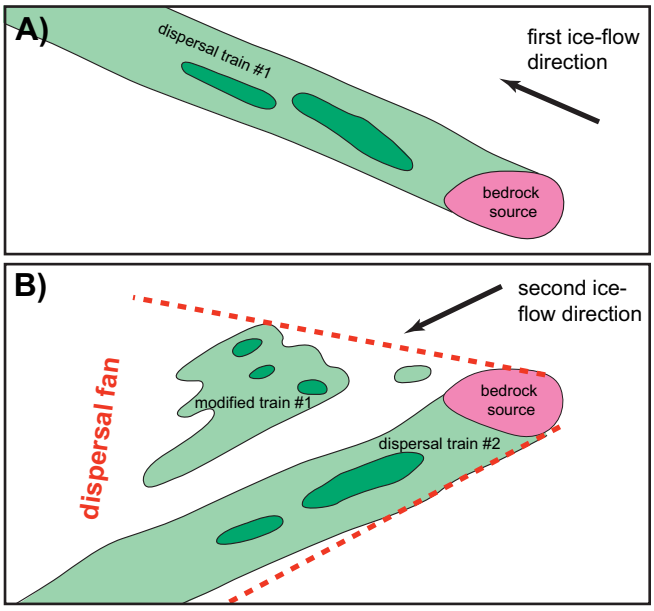

Figure 10. Schematic diagram of the formation of a palimpsest dispersal fan: **a)** initial dispersal from a bedrock source (pink polygon) by northwest ice flow forming a ribbon-shaped dispersal train (#1); and **b)** subsequent dispersal to the southwest forming a new dispersal train (#2) and remobilization of debris from train #1 (*from* Parent et al., 1996).

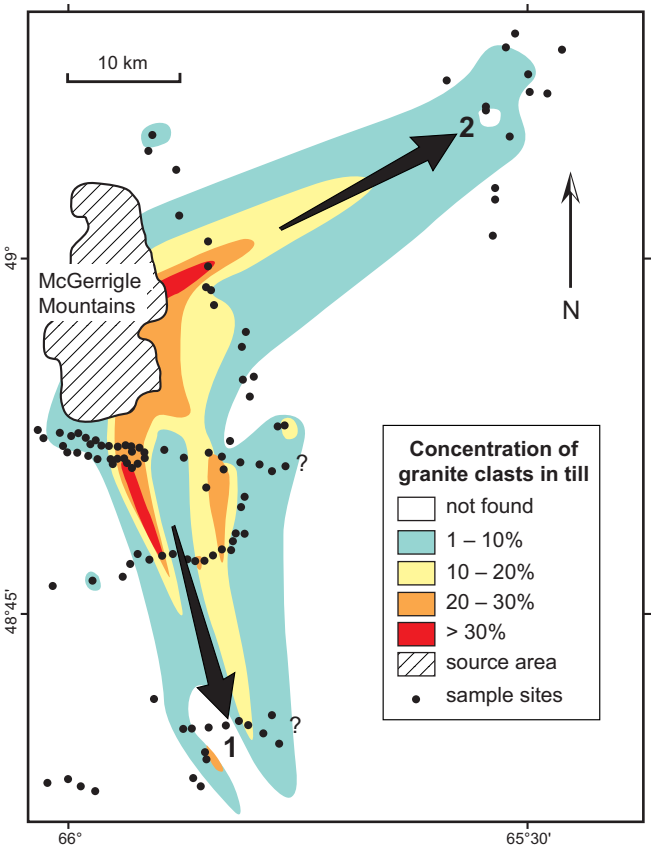

Figure 12. Distribution of granite pebbles (4–64 mm) in till down-ice of the McGerrigle Mountains, Quebec showing a bilobate distribution formed by two phases of ice flow to the (1) south-southeast and (2) northeast (*from* Charbonneau and David, 1993).

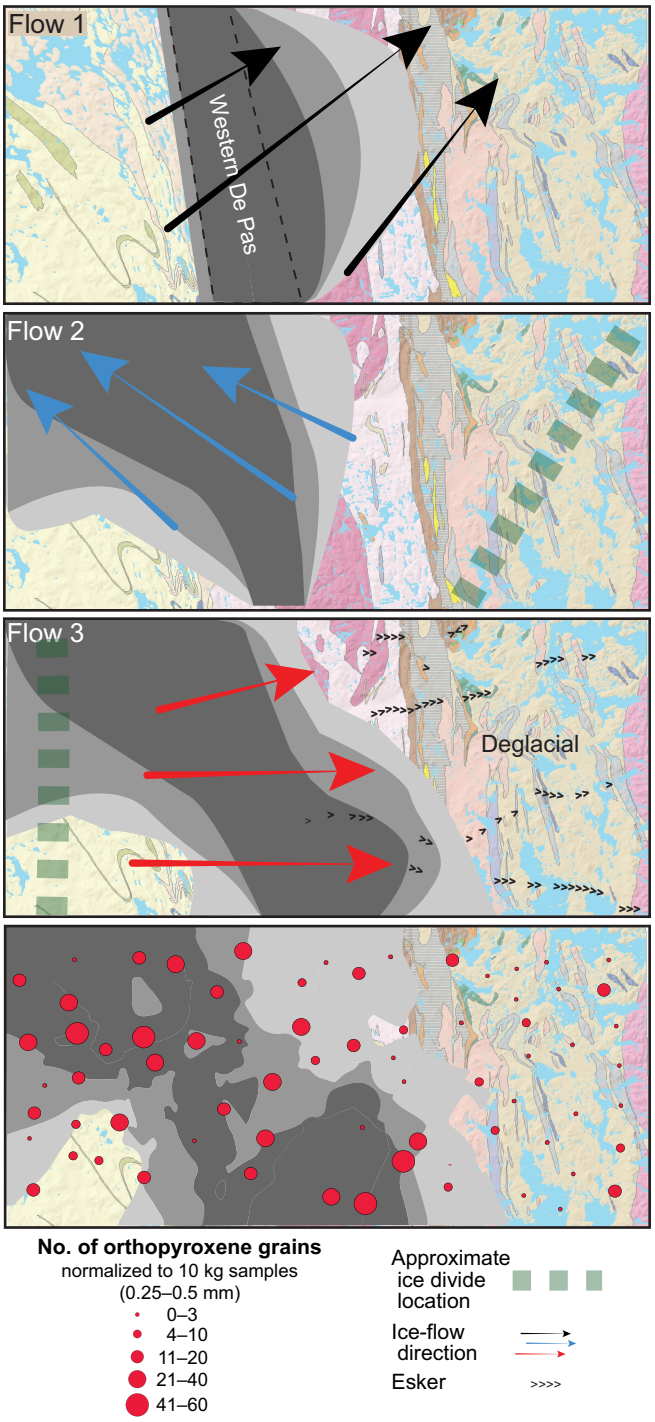

Figure 11. The evolution of dispersal of orthopyroxene grains in the 0.25 to 0.5 mm nonferromagnetic heavy mineral fraction of 10 kg till samples. Grains were derived from the western George River bedrock domain during three major ice-flow phases. The various grey tones for flow 1 (black), flow 2 (blue), and flow 3 (red) depict hypothetical dispersal patterns, with darker tones having the highest concentration and lighter tones the lowest concentrations. The bottom diagram shows the distribution of orthopyroxene grains in till samples normalized to 10 kg (data *from* McClenaghan et al., 2017c), which is the net result of the three phases of glacial dispersal (*from* Rice, in press).

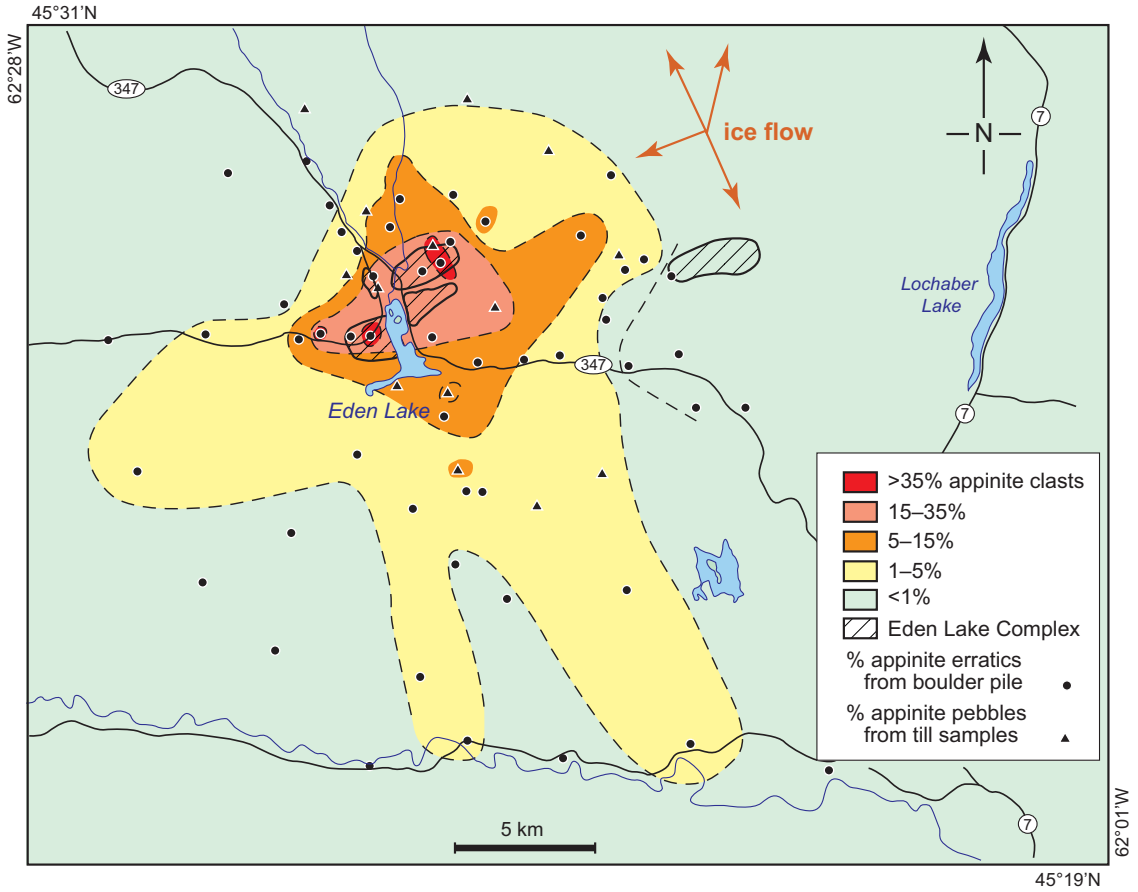

Figure 13. Amoeboid-shaped (multi-lobed) dispersal pattern of mafic pegmatite (appinite) pebbles in till down-ice from the Eden Lake Complex, Nova Scotia (*from* Stea and Finck, 2001).

2001; Parent et al., 2004). Glacial dispersal from a cluster of mineralized zones (e.g. porphyry Cu district) may result in a broad amoeboid-shaped dispersal train (Fig. 14), depending on the complexity of ice-flow movements (e.g. Hashmi et al., 2015; Plouffe et al., 2016).

The purpose of a till sampling survey is to detect patterns of glacial dispersal. The nature and size of a specific bedrock lithology or mineralized source combined with ice-flow history, glacial dynamics, and till stratigraphy will determine the net glacial-dispersal pattern seen at the land surface.

2.2.1. Paleo-ice streams

Ice streams are corridors within an ice sheet that flow more rapidly than the surrounding ice. They form the arteries of ice sheets and are crucial for regulating the flow dynamics of ice masses (e.g. Spagnolo et al., 2016; Stokes et al., 2016). Paleo-ice streams disperse large amounts of ice and debris quickly and have been linked to well defined tracts of far-travelled and exotic debris (e.g. Hicock, 1988; Dredge, 2000; Stokes and Clark, 2001; Bennett, 2003; Dyke, 2008; Ross et al., 2009).

Within an ice stream, the concentration gradient of mineralized debris follows a linear decrease in concen-

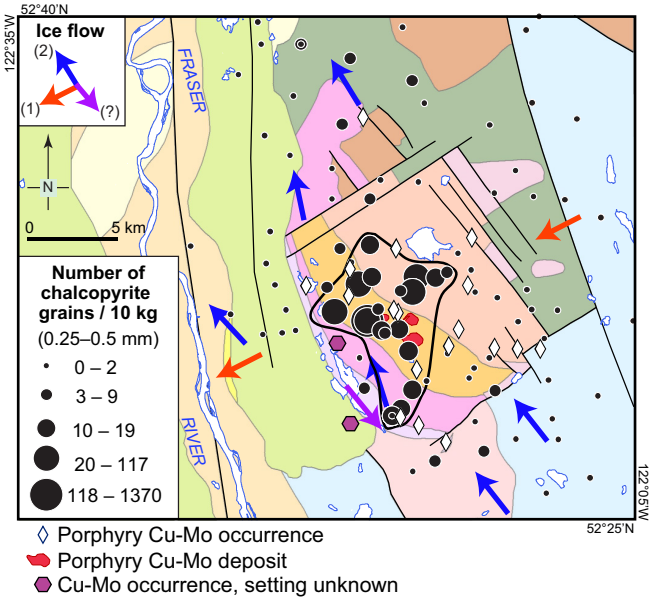

Figure 14. Glacial dispersal patterns of chalcopyrite grains in the 0.25 to 0.5 mm heavy mineral fraction of till (normalized to 10 kg sample mass) in the Gibraltar porphyry Cu district, British Columbia, which shows a broad multi-lobed or amoeboid-shaped dispersal pattern formed by three phases of ice flow across multiple mineralized zones. *See* bedrock geology legend *in*, and *modified from*, Plouffe and Ferbey (2017).

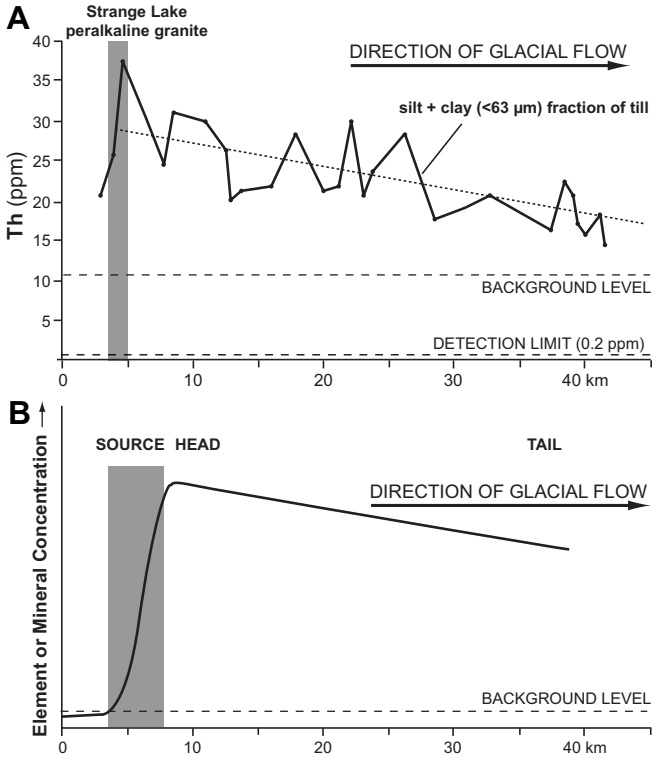

Figure 15. Linear dispersal curve for thorium (ppm) content in surface till down-ice of the Strange Lake deposit, Labrador. **a)** The top curve shows the data (*from* Batterson and Taylor, 2009) plotted with the best-fit curve shown as a dashed line. **b)** The lower curve is an idealized dispersal train (*modified from* Klassen, 1997) that shows the relationships among the source, head, and tail of a dispersal train formed by a paleo ice stream (*from* McClenaghan and Paulen, 2018).

tration down-ice (Fig. 15; McClenaghan and Paulen, 2018). For example, the Strange Lake rare earth element dispersal train in Labrador (Fig. 16a,b) (Batterson, 1989; Batterson and Taylor, 2009) is the result of eastward glacial dispersal during radial flow of the Laurentide Ice Sheet from the Labrador ice centre. The dispersal train was drastically extended in excess of 50 km from the bedrock source by a paleo-ice stream flowing eastward, forming megascale glacial lineations (MSGL; Paulen et al., 2017) (Fig. 16c).

Numerous paleo-ice stream footprints have been identified in areas of the former Laurentide Ice Sheet (e.g. Margold et al., 2015), and the effects of rapidly flowing ice on sediment transport must be recognized when collecting surface till samples in these areas (e.g. McMartin, 2017; McClenaghan et al., 2019a).

2.3. Till weathering and soil development

Postglacial weathering and soil-forming processes can significantly change the chemical and mineralogical composition of sediment in the zone of oxidation above the water table or above the permafrost table (Shilts, 1975; Shilts and Kettles, 1990; McMartin and McClenaghan, 2001; McMartin and Campbell, 2009).

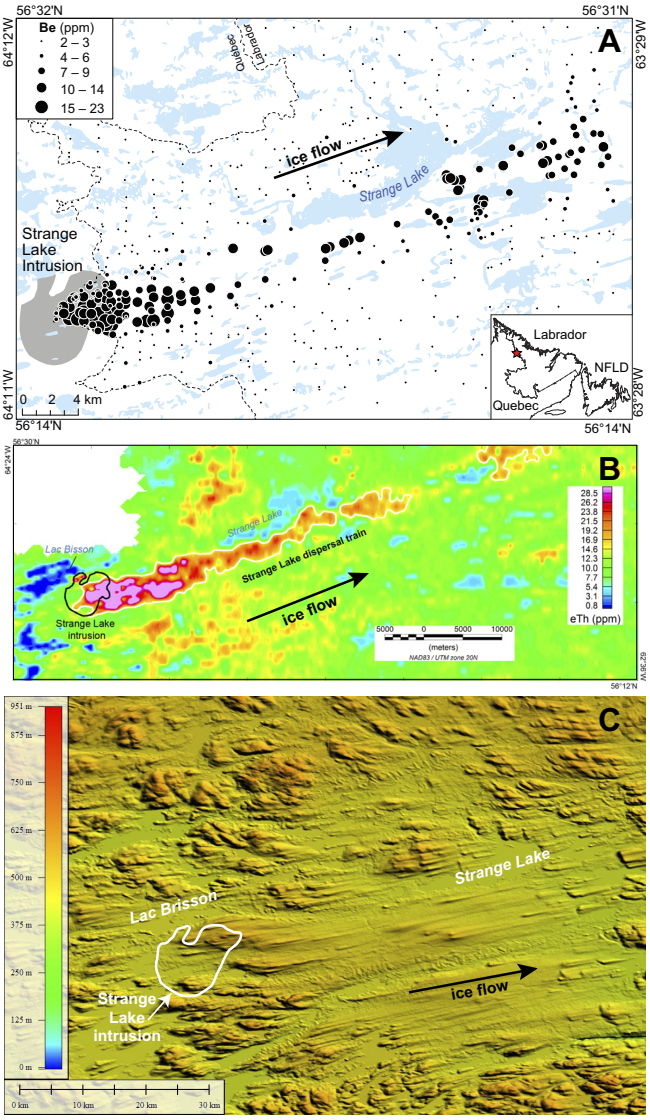

Figure 16. a) A ribbon-shaped glacial dispersal train delineated by the beryllium (Be) content in the <0.063 mm fraction of till trending northeast from the Strange Lake rare earth element deposit in eastern Canada (*modified from* Batterson and Taylor, 2009). **b)** An airborne gamma-ray spectrometry signature defined by high eTh values outlines the dispersal train, which trends northeast from the Strange Lake intrusion (outlined in black). Data *from* Geological Survey of Canada (1980). **c)** Shuttle Radar Topographic Mission (SRTM) hill-shaded DEM image of the Strange Lake intrusion (outlined in white) and mega-scale glacial lineations from the Kogaluk River palaeo-ice stream that eroded the intrusion from left to right (black arrow) and creating the elongated dispersal train shown in (b) (*from* Paulen et al., 2017).

Soils developed on till display variable and decreasing levels of pedogenic alteration that are defined by A, B, and C horizons (Soil Classification Working Group, 1998; University of Saskatchewan, 2019). The A horizon is the organic-rich mineral horizon formed at or near the soil surface. Underlying that is the B horizon, which is a soil horizon formed by either accumulation of material from the A horizon or by alteration or

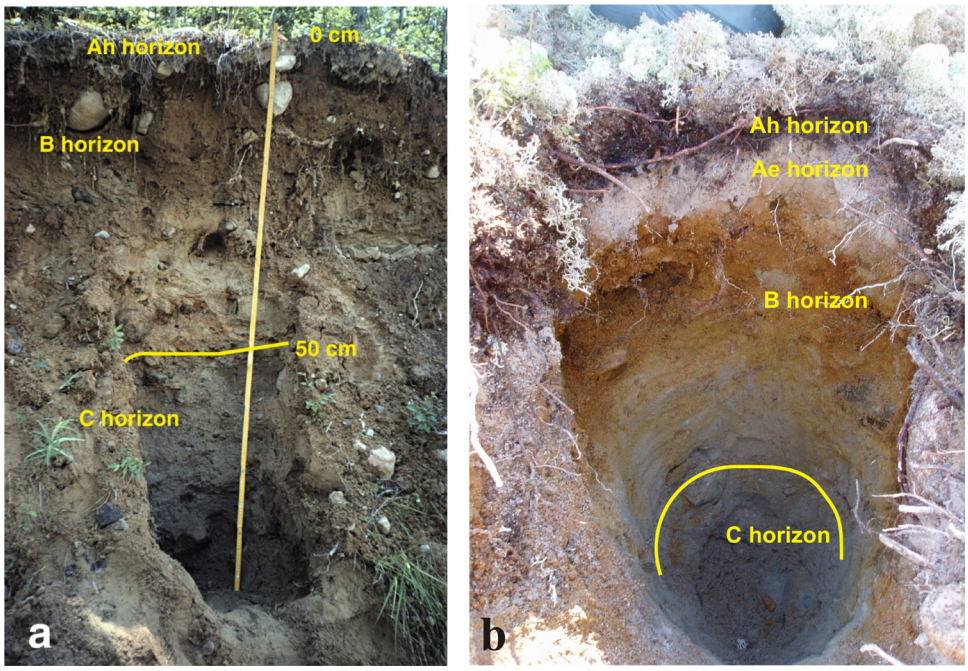

Figure 17. Photographs of **(a)** an Orthic Brunisol developed on a sandy-silt till showing a dark brown-black organic-rich mineral horizon (Ah), an oxidized orange-brown B horizon, and a relatively unoxidized grey C horizon; and **(b)** an Eluviated Dystric Brunisol developed on a well drained sandy till showing a dark brown-black organic-rich mineral horizon (Ah), a light grey leached Ae horizon, an oxidized reddish orange B horizon, and a relatively unoxidized grey C horizon. Till samples should be collected below the B horizon, within the grey C horizon. Photographs *from* McMartin and McClenaghan (2001) and McMartin and Campbell (2009).

eluviation of chemical and sedimentological constituents of the parent material. The C horizon is the underlying parent material (weakly oxidized), and is the target sampling medium to obtain a relatively unaltered signal of clastic glacial dispersal least affected by weathering.

The B horizon is commonly enriched in amorphous Al and/or Fe-Mn oxides/oxyhydroxides (orangey-brown colour), organic matter, and clay (Soil Classification Working Group, 1998). The B horizon is not always easy to identify, and sometimes is not even present in certain soil subgroup types such as Regosol or Gleyed Chernozems (Soil Classification Working Group, 1998). The B horizon often has a different colour than the underlying C-horizon parent material; usually either orange or brown due to oxidation of Fe (Fig.17a,b) or grey due to the reduction of Fe. It can also look identical to the C horizon but be leached of Ca, Na, salts, or clay. When the division between B horizon and C horizon is difficult to identify in the field, a useful rule is to only sample till below a depth of 1 metre where possible.

The oxidation and weathering from soil formation destroys labile minerals such as sulphides and carbonates, and therefore, the geochemical signature in B-horizon soils formed on till reflects a combination of clastic glacial dispersal and geochemical weathering of elements by hydromorphic and other processes. As a result, element contents can be significantly higher or lower in the B horizon compared to the relatively unoxidized C horizon. Thus, clastic glacial dispersal patterns can be distorted and difficult to interpret and follow-up if B-horizon soil developed on till has been sampled instead of C horizon (Hoffman and Woods, 1991; Kaszycki et al., 1996; Paulen, 2001; Lett and Jackaman, 2002; Hall et al., 2003).

2.4. Collect till not soil

The earliest surficial geochemical studies around known mineral deposits/districts in the glaciated terrain of Canada were conducted by sampling 'soil' instead of till (e.g. Bischoff, 1954; Byers, 1956; Ermengen, 1957; Bradshaw, 1975; Brummer et al., 1987; Hicken and Plouffe, 2017). Soil sampling is still used by some exploration companies instead of till sampling (e.g. Kerr and Levson, 1995; Levson and Giles, 1995). In glaciated terrain, soil can develop on a variety of substrates that have different depositional histories, including till, colluvium, glaciofluvial sand, or glaciolacustrine silt and clay. Thus, soil sampling could potentially include any of these different glacial sediment types together in one survey, making interpretation of sediment source and geochemical patterns difficult, masking real anomalies and sometimes creating false ones (Gravel and Sibbick, 1991).

3. SURVEY DESIGN

The design of a GSC till sampling survey is dependent on the objective of the survey (Table 2), which may be to (1) characterize the regional elemental and mineralogical concentrations within a geological province; (2) delineate geochemical or mineralogical anomalies within a mineralized belt; and/or (3) discover or document glacial dispersal from a mineral deposit, mineralized zone, or specific bedrock lithology (Fig. 18) (Salminen, 1992a; McMartin and McClenaghan, 2001; McMartin and Campbell, 2009). Other regional-scale till sampling projects conducted by the GSC are

Table 2. Summary of factors affecting till sampling survey design.

Target	Survey scale	Sample density	Sample spacing	Sample type and size	Minimum analysis	Comments
Geological province/domain	Reconnaissance	1 sample/ 100 to 500 km^2	10 to 25 km	geochemistry (3 kg) and heavy mineral (10+ kg)	Matrix geochemistry, indicator minerals	Low sample density, often random sample pattern
Mineralized belt, kimberlite field	Regional	1 sample/ 10 to 100 km^2	4 to 10 km	geochemistry (3 kg) and heavy mineral (10+ kg)	Matrix geochemistry, indicator minerals	Low to moderate sample density, often sampled in offset lines perpendicular to regional ice flow
Cluster of deposits	Regional to local	1 sample/ 1 to 4 km^2	1 to 2 km	geochemistry (3 kg) and/or heavy mineral (10+ kg)	Matrix geochemistry, indicator minerals	Moderate sample density in either offset grid or random sample pattern (nearest neighbour)
Individual deposit	Property	100 to 1000 samples/1 km^2	25 to 250 m	geochemistry (3 kg) and/or heavy mineral (10+ kg)	Matrix geochemistry, indicator minerals	High sample density, infilling previous surveys, tight grid or lines perpendicular to dominant direction of transport

designed to characterize the provenance of glacial sediments across major glaciological domains; these sampling surveys typically employ oriented transects to help in paleo-ice sheet reconstructions (e.g. McMartin, 2017; McMartin et al., 2017; Campbell et al., 2019).

The principals of till sampling survey design that are outlined below have been adapted from the principals of geochemical survey design described by Rose et al. (1979), Levinson (1980), Garrett (1983), and Fletcher et al. (1986).

3.1. Factors influencing survey design

- purpose, type, and scale of the survey (Table 2);
- budget and time frame;
- previous work done on the property or in the area;
- size of the survey area;
- ease of access to the area and within the area;
- number of personnel available to conduct the sampling;
- type, distribution, and provenance of surficial materials;
- stratigraphy and overburden thickness (Fig. 18);
- ice-flow history, dominant transport directions, and landform trends;
- geomorphology and topography;
- permafrost distribution;
- land tenure;
- presence of national or provincial parks and First Nations- or Inuit-owned lands;
- sampling density (e.g. reconnaissance versus regional versus property scale);
- nature and size of target (e.g. point source versus elongate mineralized zone);
- method of transport to access sample sites (truck, all terrain vehicle, boat, helicopter, etc.);

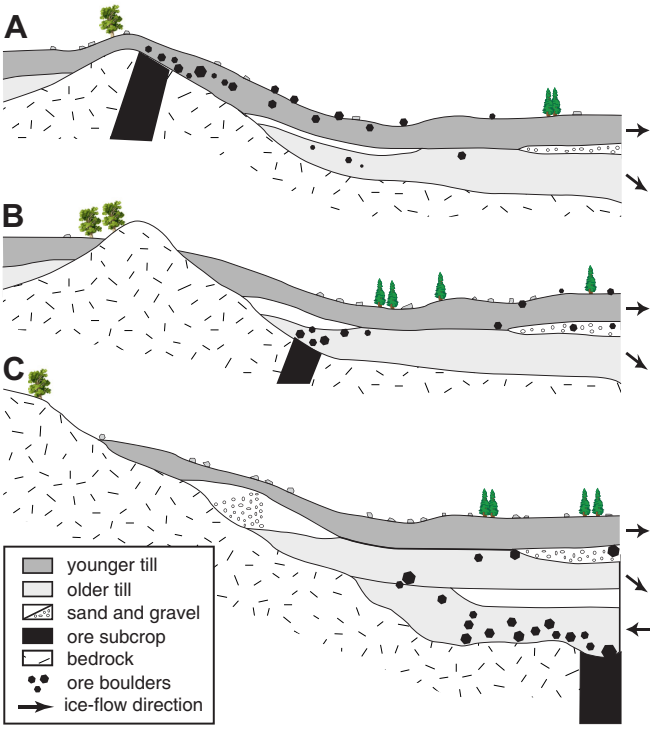

Figure 18. Illustration showing idealized glacial dispersal trains in areas with variable till thickness and till units: **a)** thin (<2 m) till with metal-rich debris dispersed in the surface till; **b)** thicker till with metal-rich debris dispersed in older and younger tills; and **c)** a deeply buried mineralized zone with metal-rich debris dispersed only in the older tills. Note that in this example the ice-flow direction changed over time, leading to a more complex dispersal pattern. Scenarios such as these must be considered when designing till sampling surveys (*modified from* Hirvas and Nenonen, 1990).

- thickness of till: glacial dispersal in areas of thick till may require subsurface sampling as dispersal trains may occur in one or more buried till units without reaching surface (Fig. 18; e.g. Garrett, 1971; Bird and Coker, 1987; Thorleifson and Garrett, 2000);

- predicted shape of the target source (Fig. 7–14): point source(s), broad mineralized zone(s), cluster of sources, or unknown;
- possible federal, provincial, territorial, or industry partners.

3.2. Criteria to establish when designing a survey

The nature and size of a target, combined with ice-flow history and till stratigraphy, will determine the net glacial dispersal train geometry, and ultimately the till sample distribution required to detect the glacial dispersal of debris from a specific bedrock source (Table 2).

Based on the factors listed in section 3.1, three general sample patterns are considered to be most effective:

1. line sampling perpendicular to ice flow (Fig. 19a);
2. grid sampling (Fig. 19b) where ice-flow directions are not known or where multi-phased ice flow is variable in direction;
3. random sample spacing influenced by the availability of till and where site access may be restricted (e.g. only along roads and waterways; Fig. 20);

Criteria to consider when designing a sampling strategy to detect glacial dispersal from a specific bedrock source include the following:

- sampling methodologies (e.g. surface, trenching or subsurface drillholes);
- vertical interval and/or soil horizon to be sampled;
- sample density and spacing (survey scale);
- sample processing and analytical methods to be used: these will dictate the size of the sample to be collected;
- size fraction to be geochemically analyzed, e.g., heavy mineral, <0.063 mm, <0.002 mm;
- consistency of sampling methodology throughout each sample survey.

3.3. Preliminary work to aid survey design

Factors to establish (if possible) during the design of the survey and prior to going to the field:

- distribution and thickness of glacial deposits;
- associated glacial landforms;
- regional ice-flow patterns;
- glacial stratigraphy;
- secondary processes affecting till composition.

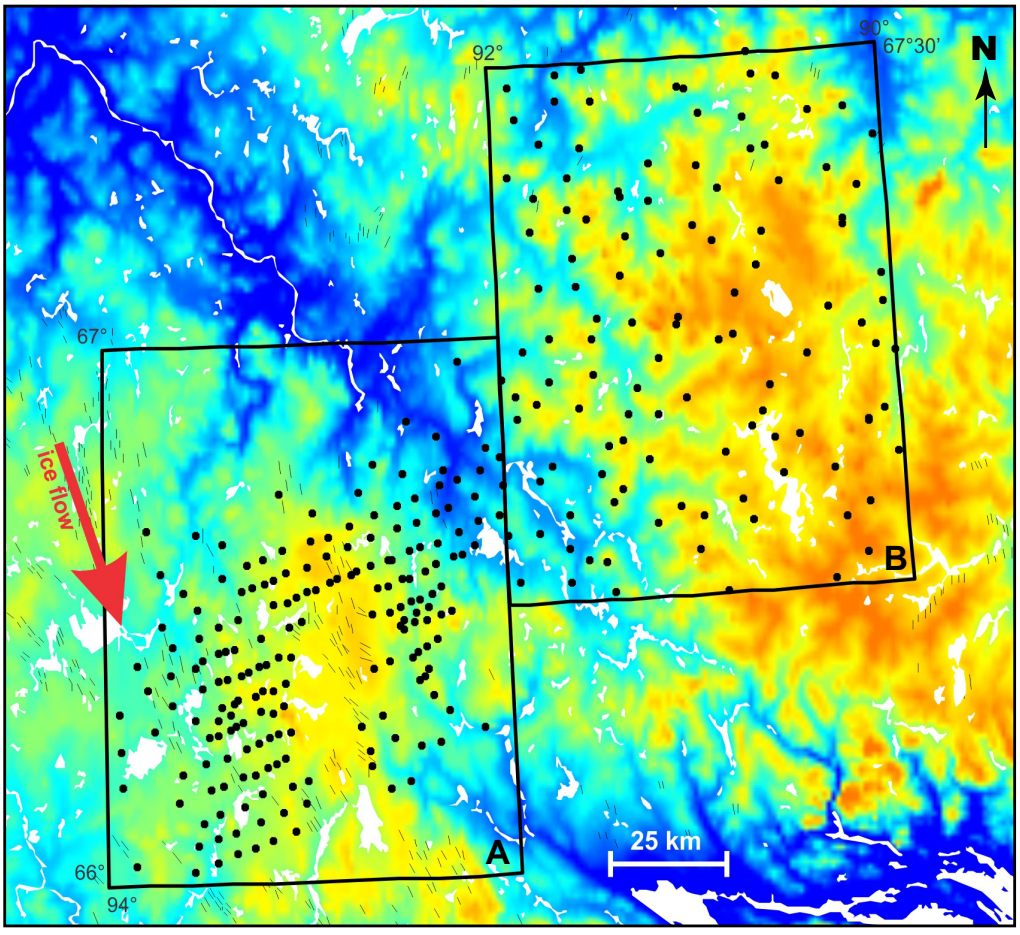

Figure 19. Examples of different sample spacing strategies used by the GSC to collect surface till samples in the Committee Bay supracrustal belt, Nunavut (*adapted from* McMartin et al., 2003); **a)** till samples (black dots) in the western part of the study area were collected at regular spacing intervals along lines perpendicular to the known ice-flow direction (red arrow), and **b)** samples in the northeast part of the study area were collected along a regularly spaced grid because the direction of ice flow was poorly defined in this area prior to the survey. The background colours reflect the topographic elevation: dark blue is the lowest elevation and deep orange-red is the highest.

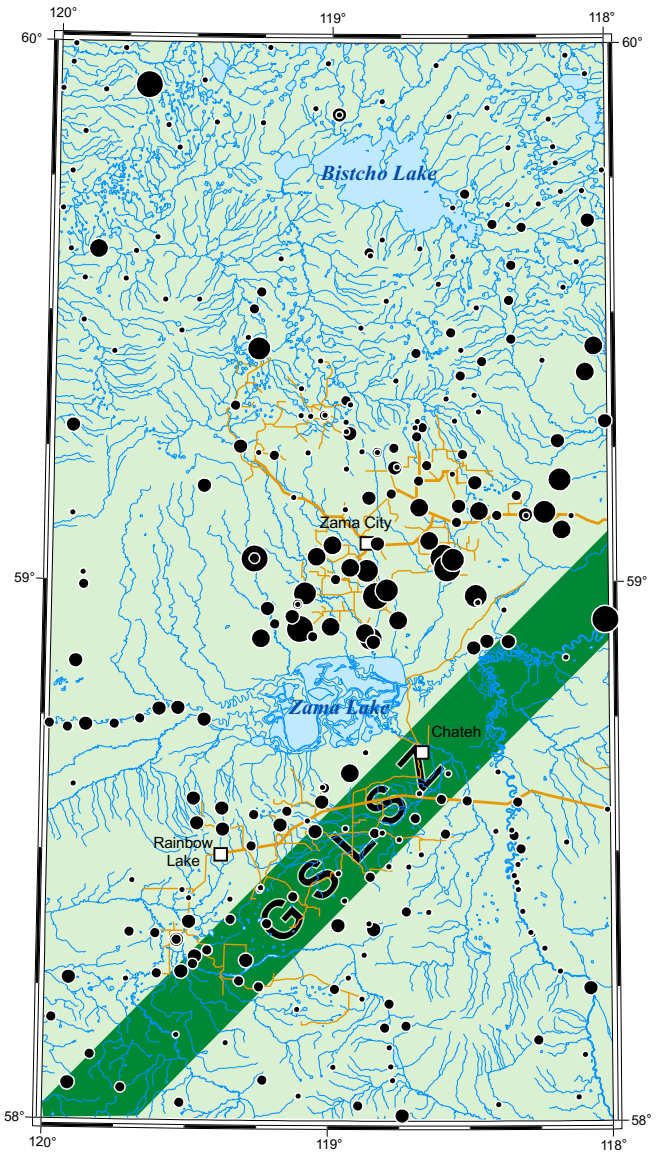

Figure 20. An example of a random sampling strategy that was used to collect surface till samples in northern Alberta along roads and waterways. The orange lines indicate roads. GSLSZ = Great Slave Lake shear zone (*from* Plouffe et al., 2006).

3.4. Information sources for designing fieldwork

When designing fieldwork, information may be gained from consulting the following:

- topographic maps;
- digital elevation models (DEM);
- satellite imagery;
- (light detection and ranging) LiDAR imagery (e.g. Johnson et al., 2015; Sarala et al., 2015a);
- aerial photographs (stereo pairs);
- previous work contained in industry assessment reports, government surficial geology maps, government geological reports, published literature.

4. FIELD EQUIPMENT

The choice of field equipment depends on the objectives and type of survey, together with sample density, depth, and site access. It is important to ensure that all the equipment is in good working condition before travelling to the field and that it is clean and will not contaminate the samples.

4.1. Safety equipment

All GSC fieldwork must comply with Land and Minerals Sector (LMS) Field Safety Guide directives (unpublished NRCan safety manual, available upon request). Increasingly, safety awareness and safety issues permeate all facets of fieldwork, from planning, preparation, execution, to return to headquarters. It is essential that GSC field party leaders ensure that all field personnel have the appropriate Learning Management Systems (LMS) health and safety training. For GSC staff, a *Field Safety Checklist* must be submitted, scrutinized, and approved prior to departure for any fieldwork. Most scientific and exploration field programs have field safety guidelines that are tailored to their field activities. These guidelines are dictated by the nature of the fieldwork and in fulfillment of regulatory and legislative requirements (e.g. Alpay and Paulen, 2014).

Regardless of the type or method of till sampling to be undertaken, it is important to provide and carry the appropriate safety equipment for the job and personnel. In the field, due diligence, proper training, and well maintained equipment are your most important safeguards. Participants should ensure that their personal clothing and footwear are of adequate quality and quantity for a full field season. An emergency response plan must be in place prior to start of the sampling program, and all incidents and/or accidents must be properly documented and reported to Management.

4.1.1. General safety equipment

- first aid kit, checked and stocked;
- SPOT® or inReach® devices, satellite phone, radio, or cell phone communications;
- GPS;
- brightly coloured outer garments (for visibility during rescue and to be seen by hunters);
- safety glasses;
- work gloves;
- flares and flare gun (especially in remote locations);
- if boating: life jackets, boat and motor, anchor, safety equipment (kill switch, bailer, throw rope, flashlight, paddles);
- if being dropped off by helicopter: emergency drop pack;
- bright orange signal flag.

4.1.2 Drilling and backhoe safety equipment

- Rubber gloves;
- hard hats;
- steel-toed boots;
- ear plugs;
- safety glasses;
- safety cage or methods to shore-up walls for trenching when depths are greater than the width of trench. Trench depths beyond which shoring up, wall sloping, or using a cage is recommended vary but the most common is 1.2 m. The depth is dependent on the competence of the sediments being excavated. Snook (2012) lists several tips for keeping safe in trenches.

Use and operation of a backhoe and larger rig-type drills should only be undertaken by licensed and qualified individuals, and usually should be contracted to an established operator.

The Canadian Centre for Occupational Health and Safety (Government of Canada) provides a trenching/excavation fact sheet online: https://www.ccohs.ca/oshanswers/hsprograms/trenching_excavation.html

The province of Ontario provides similar information on a Health and Safety Ontario website: http://www.king.ca/Government/Departments/Building/Documents/27_M026.pdf

4.2. Equipment used for collecting till samples

Field equipment for surface (0–2 m) and subsurface (>2 m) till sampling may include hand-held shovels, hoe picks, Dutch augers, small portable drills, excavators, and truck- or track-mounted drills. The following references provide information overviews of sampling equipment that can be used to collect till samples: Coker and DiLabio (1989), Hirvas and Nenonen (1990), Coker (1991), Kauranne et al. (1992), Salminen (1992b), Plouffe (1995b), Levson (2001a), McMartin and McClenaghan (2001), McMartin and Campbell (2009), and Paulen (2009).

Once again, it is important to note that the choice of sampling equipment depends on the objectives and type of survey as well as the till sample density, depth, and logistics. It is important to ensure that the equipment is in good working condition before travelling to the field and that appropriate safety equipment is used.

4.2.1. Equipment list for till sample site location and documentation

- GPS;
- field-sheet/notebook/handheld computers or tablet;
- digital camera and accessories (e.g. extra batteries, memory cards);
- scale card;
- Silva or Brunton-type compass with inclinometer;
- knitting needles for fabric measurements (non-magnetic);
- 10% HCl in a 15 to 20 ml squirt bottle with a cap, labelled with appropriate Workplace Hazardous Materials Information System (WHMIS) information and stored in 3 mil plastic bag;
- Munsell colour chart (optional);
- hand lens;
- measuring tape (3 m) or stick (2 m);
- water bottle/wet sponge (for cleaning bedrock at the base of the holes to examine for striations and for cleaning the equipment);
- large brush or whisk (for cleaning rock surfaces to examine for striations).

4.2.2. Till sample containers

- Bag closures (thick elastics, cable lock ties, or electrical tape);
- sample number tags to be placed inside each container (e.g. flagging tape, waterproof paper);
- sample labelling: high-quality black permanent markers;
- 19 L (5 gallon) pails for sample storage and shipping;
- shipping labels;

Geochemical samples:

- ~3 kg sample should be stored in 8" x 13" (20 cm x 33 cm) 6 mil clear plastic bags, preferably with no seams (Fig. 21a).

Indicator mineral samples:

i) for clay-rich till: ~20–40 kg (~10–20 L) to be stored in a 20 L (5 gallon) plastic pail (Fig. 21b);
ii) for silty-sandy till: ~10 to 15 kg (~5–8 L) to be stored in 6 mil, 12" x 20" (30 x 51 cm) or larger clear plastic bag, or 10 L (2.5 gallon) plastic pail (Fig. 21a).

Portable XRF (pXRF) sample:

- ~200 g sample stored in thin plastic sandwich bags (Fig. 21a) if pXRF measurements are to be conducted while in the field (*see* Section 9.4 for more details).

4.2.3. Equipment list for near-surface excavation and till sample collection

Surface (<2 m) sampling involves hand excavation and includes dug and augered holes and exposures. Additionally, samples may be collected at or near surface from open trenches, open pits, borrow pits, roadcuts, and river or lakeshore exposures. The following equipment is required for sample collection:

Figure 21. Photographs of **(a)** three samples of till that have been collected from the same site (a large 10–15 kg sample of sandy till for the recovery of heavy minerals; a 3 kg sample for matrix geochemical analyses, determining physical properties, and archiving; and a 300 g sample for pXRF analyses); and **(b)** a 20–30 kg sample of clay-rich till in a 19 L (5 gallon) pail lined with a plastic bag. Note that the sample number is recorded on both the pail and on a tag (flagging tape) inserted in the pail.

- steel shovels with galvanized coatings or paint removed* (D-handle or long handle according to preference);
- Dutch auger for small samples and site reconnaissance, coatings removed;
- grub hoe or pick for digging into exposures, coatings removed*;
- large knife (e.g. hunting knife);
- bricklayer/cement trowel for cleaning sections;
- geological hammer;
- steel trowel with coatings removed* or plastic trowel (optional) for collecting sample from hole or cleared section (note: plastic trowels are inadequate for digging);
- pruners (high quality) to cut plant roots;
- pruning saw to cut thick plant roots;
- sieve (solderless or solder covered with epoxy), ~2.5 cm to separate coarse pebble fraction, if needed;
- fish weighing scale for determining the mass of the sample material;
- J-cloths and paper towels for cleaning hand-held equipment that will be in contact with the sample;
- measuring tape (3 m).

*Notes:

- coatings on new equipment should be removed by sand blasting and/or sanding prior to any sampling to avoid sample contamination (this service may be available from an autobody shop);
- sampling tools wear down with use and produce metal shavings of various unnatural forms that should be expected and recognized in processed samples.

4.2.4. Equipment list for subsurface till sample collection

Subsurface (>2 m) sampling involves the collection of till samples using backhoes, portable drills, or drilling rigs. Equipment required to collect samples using these deep sampling methods includes

- all the equipment listed for near-surface till sampling (*see* Section 4.2.3);
- long measuring tape (10 m);
- selection of tools (utility knife, pipe wrenches, screw drivers, hammers, pliers, etc.);
- safety equipment: hard hat, steel-toed boots, ear plugs, safety glasses.

Some equipment that is drill-rig specific and is not supplied by the drilling company is listed below:

Rotasonic Drills

- core boxes and lids;
- a drill and screws or duct tape to attach lids to core boxes.

Reverse Circulation Drills

- rain suit or waterproof apron;
- rubber gloves, steel-toed rubber boots;
- plastic 20 L (5 gallon) pails for collecting the sample as it comes out of the cyclone;
- #10 mesh sieves;
- wire screening to sit on top of sample bucket and support #10 mesh (2 mm) sieve;

Mud Rotary Drills

- several sieves to catch cuttings;
- 30.5 x 30.5 cm square wooden 0.5 cm and 1.25 cm screens to wash drilling mud off of the cuttings;
- plastic 4 L pails to catch unconsolidated sediments (e.g. sand and gravel);

- muffin trays for small geochemical samples that are to be dried in the field and used for field description, and then bagged for texture description and geochemical analysis;
- aluminum pans for large samples that are to be dried in the field before bagging;
- a board to lay across the water trough;
- rubber gloves;
- steel-toed rubber boots;
- electrical tape.

Diamond drills

Do not collect till samples from diamond drill core: contamination from grease, drilling mud, sample material from further up the drillhole, and/or diamonds from the drill bits is too severe to overcome.

5. SITE SELECTION

It may seem obvious, but the most important thing in till sampling is to ensure that the material sampled is actually till. Based on the experience of the working group members, the best practices to select sample sites have been summarized in five subsections related to different physiographic and surficial geology settings: 1) mountainous terrain, 2) regions once covered by postglacial seas or glacial lakes, 3) specific glacial landforms, 4) organic cover, and 5) areas of anthropogenic disturbance or contamination.

5.1. Selecting till sites in mountainous terrain

Mountainous terrains are regions with rugged topography including steep mountain slopes, peaks, and arêtes separated by highlands, plateaus, and incised valleys.

5.1.1. Steeply sloping terrain

In high-relief terrain, till is often covered by colluvium (Fig. 22). The composition of colluvium depends on the source material and can vary in texture from rubble to diamicton, and can be stratified or massive. Where colluvium is derived from till, differentiating between the two can be challenging and all members of the field sampling crew should be trained to recognize the difference. The following criteria can help to differentiate till from colluvium (Levson, 2001a; Lian and Hickin, 2017):

- Till is generally indurated, less porous, and its matrix contains more fine-grained material than colluvium.
- Colluvium can contain unpolished angular clasts as it is derived from local bedrock without much abrasion and surface modification.
- Till usually contains a wider range of clast lithologies than local-bedrock-derived colluvium, which reflects the longer distance of glacial transport.
- Colluvium can be crudely to well bedded subparallel to the slope, and more oxidized than till.
- Clast fabric and imbrication in colluvium can be weak (widespread in orientation) to strongly developed, reflecting strain during down-slope movement, whereas till fabrics are generally uni- or bi-directional with orientation controlled by the stress imposed by the movement of the glacier.
- The presence of glacial landforms (e.g. lateral moraines) indicates in situ glacial sediments rather than colluvium.
- Colluvium is more likely to contain plant remains associated with burial and incorporation of surface materials.

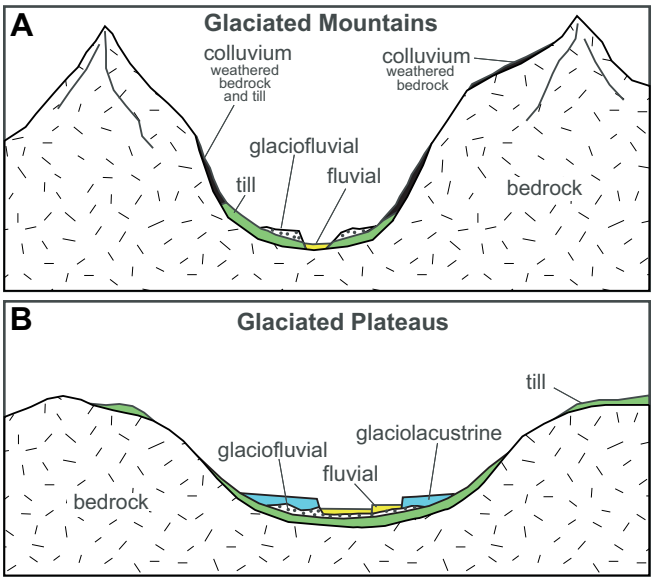

Figure 22. Idealized valley cross-sections in alpine terrain displaying till and other glacial sediment distribution for **(a)** glaciated mountains and **(b)** glaciated plateaus (*from* Yukon Geological Survey, 2007).

Where access roads occur in steeply sloping terrain, unconsolidated sediments are exposed in ditches. Where ditches are deep or where the colluvium cover is thin, till may be exposed beneath colluvium (Fig. 23). In the case of a regional survey for which regional background metal concentrations are being established, till reworked by colluvial processes (colluviated till) can be sampled in the absence of in situ till, but should be described as such in detailed field notes (e.g. Levson and Giles, 1995). In the case of a detailed till survey where a specific mineralized zone is being sought, colluviated till should not be sampled because downslope movement of the sediment is an additional transport vector that needs to be considered when tracing the source of mineralized debris. Colluvium is also deposited in reverse chronology, the youngest glacial

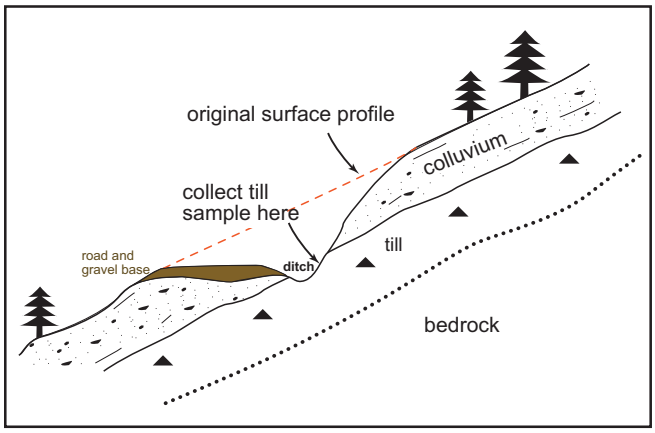

Figure 23. Schematic representation of a sediment exposure in a roadcut along an access road in a mountainous area showing where till may be exposed beneath colluvium and may be accessible for sampling (*from* Spirito et al., 2011).

sediments (often supraglacial till) are the first to be transported downslope, buried by progressively older glacial sediments as they are exposed and impacted by gravity.

5.1.2. Highlands and plateaus

On highlands and plateaus, till is generally the most areally extensive glacial sediment. In these areas, thick till exposures might be present along roads and streams. In such instances, till can be sampled at different depth intervals, depending on the purpose of the project. In general, the top part of a till sequence reflects a more distal and diluted source compared to the bottom. As part of a regional survey, sampling the top part of the till is adequate. For a detailed study of glacial dispersal from a local source, sampling the bottom part of the till, ideally near the underlying bedrock.

More than one till facies (*see* Section 2.1) or bed can be present in natural and artificial exposures (Fig. 24). Samples from each unit should be collected to assess the variability in sediment composition. In such circumstances, detailed structural measurements (e.g. till fabrics, faults) will assist in interpreting sediment genesis. Ferbey et al. (2016) present an example of a till sampling survey where two till facies (subglacial and supraglacial till) were observed within a study area in south-central British Columbia.

On highlands and plateaus in permafrost areas, frost boils are good sites for till sampling.

Boulder concentrations can be present on plateaus where there has been widespread melting at the base of the ice sheet (ablation). Till samples should be collected below the boulder lag (Fig. 25) where the fine-grained component of the till matrix is higher (*see* Ferbey et al., 2016 for an example).

On highlands and plateaus near mountains, where sustained glaciation has deposited till, the top part of

Figure 24. Two till facies observed in an anthropogenic exposure along a forestry road in south-central British Columbia. A maroon-brown till sharply overlies a grey till. The colour differences are attributed to the till provenance. Local intrusive rocks predominate in the lower till and distally derived volcanic clasts are abundant in the upper till. Note the samples that have been collected in each till unit. Photograph courtesy of A. Plouffe.

Figure 25. Discontinuous boulder concentration on the Thompson Plateau of south-central British Columbia. Note the large boulders at surface and at the top of the till section above the dotted line. The shovel for scale is 1.5 m long. Photograph courtesy of A. Plouffe.

the till can be derived from a supraglacial (ice surface) deposition. In other words, avalanche, rock-fall, and other debris derived from steep slopes accumulates on the top surface of the glaciers and is transported down flow until it is eventually deposited on the highland or plateau. In such a setting, a supraglacial till composed predominantly of far-traveled debris could be superimposed on a subglacial till of proximal provenance.

5.1.3. Valleys

The thickest unconsolidated sediments in mountainous terrain are usually found in valleys and may include

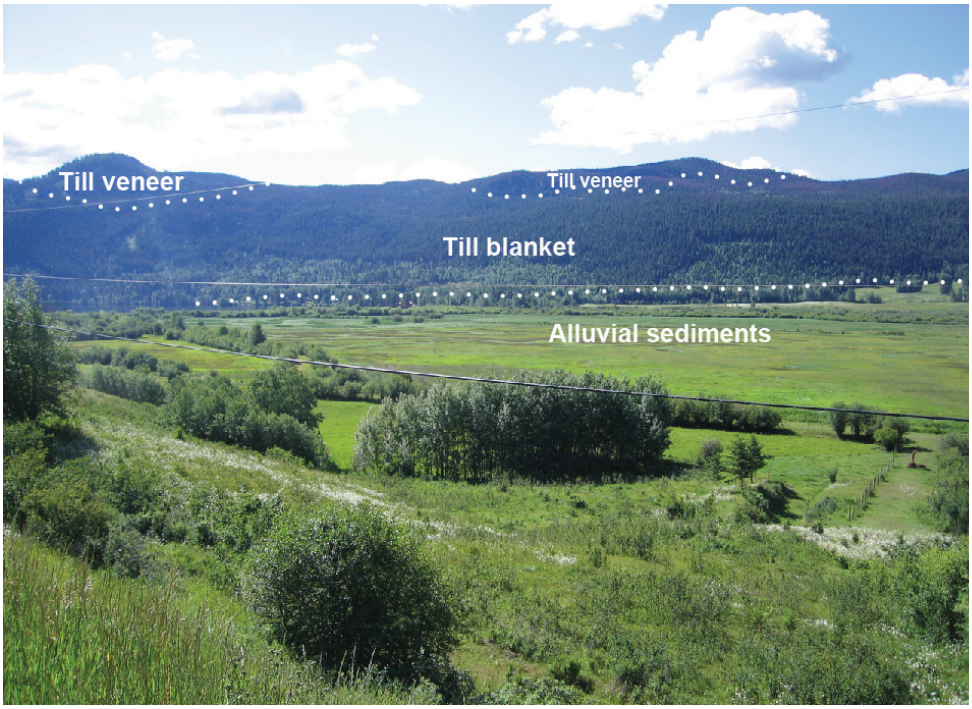

Figure 26. Photograph taken in central British Columbia that illustrates a typical valley-fill sequence of alluvial sediments at the bottom of the valley, a till blanket (>2 m) on the walls of the valley, and a till veneer (<2 m) in the higher and steeper regions where bedrock outcrops are abundant. A dotted line shows the approximate contacts between the sediment types (*from* Spirito et al., 2011).

glacial, glaciofluvial, glaciolacustrine, alluvial, colluvial, and eolian sediments. As part of a till sampling project, differentiating till from these other sediment types is crucial. In valleys, till may be covered by deglacial sediments or may have been reworked or completely removed by glaciofluvial erosion, greatly hampering till sampling. In valleys with an extensive sediment cover (Fig. 26), till can be sampled in natural sections along streams, at the base of roadcuts, or at depth beneath the younger sediment cover using overburden drills. In valleys where till is not present for sampling, collecting glaciofluvial sediments for recovery of indicator minerals can provide limited information for a reconnaissance-scale survey.

5.2. Selecting till sites in low-topography terrain

In areas of flat to gently undulating topography, till may occur at surface or may be deeply buried, and may be part of a complicated thick stratigraphy. It is essential to consult surficial geology maps, 3-D models, and other pre-existing literature before designing a till survey in these areas.

Till samples from forested areas can be collected at surface at depths of between 0.5 and 1 m, preferably above the water table and definitely within the C horizon (below the B horizon) from hand-dug holes (Fig. 27a).

In forested areas, the difficulty in extracting large boulders and penetrating a compact forest root system to reach the till below may result in having to move the sample site and dig another hole, or sample around and/or below a large boulder and into the sides of the hole. Hand-picks, geological hammers, or small axes may be needed to remove boulders, to dig into very hard material, or to cut roots. Small saws or pruning shears are useful for cutting roots. Till samples can also be collected with a shovel from natural sections along rivers or lake shorelines (Fig. 27b), roadcuts (Fig. 27c), borrow pits, or open pits (Fig. 28).

5.2.1. Boreal forest

In the Boreal forest, there is variable association between vegetation type and soil drainage. Generally, pine trees and caribou lichen grow in well drained areas that are formed either due to upland topography or high sediment porosity (i.e. sands, gravels, sand-rich till, thin till over bedrock). Poplar and/or aspen trees typically grow in moderately drained areas, either due to upland topography or moderate sediment porosity. Birch, tamarack (larch), and cottonwood trees generally grow in poorly or very poorly drained areas with flowing water, which are formed either due to lowland topography or patches of low sediment porosity (e.g. clays and clay-rich till) and surface ponding. Black and white spruce trees interspersed with a thick moss cover typically grow in poorly drained terrains, but can drape a range of sediment types.

5.2.2. Areas with organic cover

A cover of organic bog and fen deposits of variable thickness drapes large parts of the Boreal forest. In regions of areally extensive organic cover and where till is near surface, till can often be found in the following locations:

Figure 27. Till may be collected at or near surface from **(a)** hand-dug holes, **(b)** natural sections along rivers or lakes, or **(c)** roadcuts.

Figure 28. A till sample site in a region heavily disturbed by anthropogenic activity at the former Pine Point Pb-Zn Mississippi Valley-type mining district, Northwest Territories (*from* Rice et al., 2013). Till samples were collected in the walls of a former open pit mine, below the natural land surface (*from* Plouffe et al., 2014).

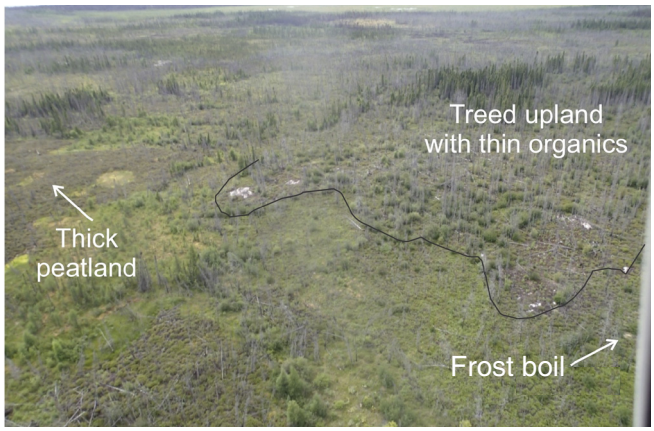

Figure 29. A potential area to collect till in an organic terrain near treed uplands.

- newly-burned areas, where the organic cover has been removed;
- near forested parts of a bog, where either the organic cover is thinner (Fig. 29) at the base of tree wells, or other low-lying depressions within a bog;
- near modern streams or rivers, where the water-course has a base of rock fragments washed from the till;
- at the base of fens, which are often wet but have thin organic cover.

5.3. Selecting till sites in regions once covered by postglacial seas or glacial lakes

These sites include glaciated regions once inundated by marine waters or a proglacial lake during deglaciation, but have since become exposed (i.e. by glacioisostatic uplift or postglacial drainage).

The most useful information for selecting till sample sites is the maximum elevation of the marine or lacustrine submergence. This elevation is crucial in order to determine the extent of inundation, and hence the possibility of the presence of till that has been reworked by waves and currents. This knowledge is also helpful for understanding and identifying marine or lacustrine sediments and their thickness.

Marine/lacustrine limits can either be clearly defined or not obvious. Common features that indicate marine/lacustrine limits include (1) raised deltas, (2) ice-contact deltas, and (3) trimlines, shorelines, and perched boulders. Trimlines are defined by notches cut into till slopes or by sharp contacts between unmodified till and intensively wave-washed bedrock surfaces, or boulder beaches (Fig. 30a). Marine/lacustrine limits are commonly isostatically tilted and may vary in elevation across a sampling region, as a result of differential isostatic rebound, late-glacial ice masses, and/or opening of new outlets (lakes) (e.g. Teller and Thorleifson, 1983).

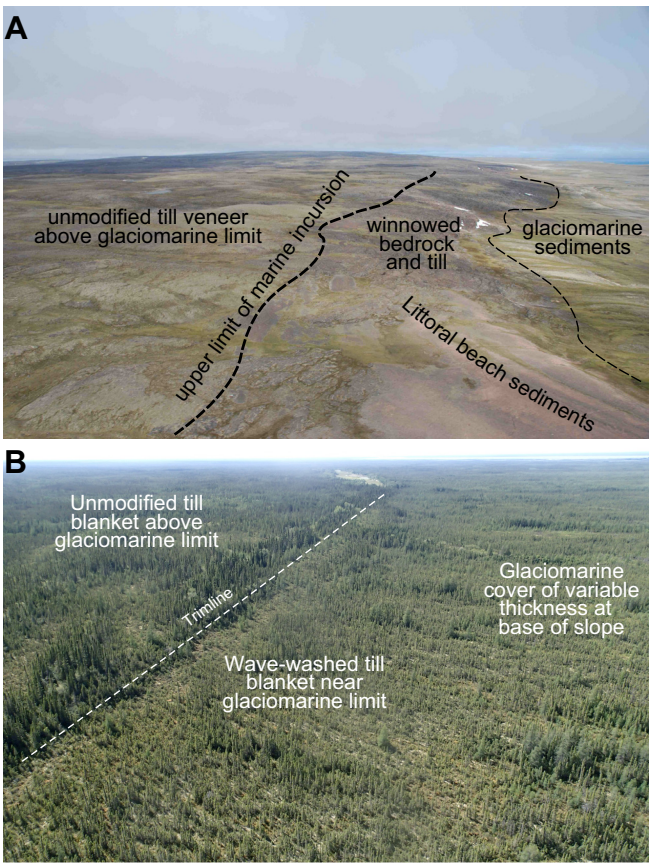

Figure 30. a) Oblique photograph taken from the air showing a marine-limit trimline in till deposits located in permafrost terrain near Baker Lake, Nunavut (the distance between the two black dashed lines is ~350 m). The sharp contrast between the unmodified till and the littoral sediments marks the marine limit at 122 m a.s.l. Wave-washed (winnowed) till underlies the littoral sediments. **b)** A similar setting as is shown in (a) but covered by boreal forest near Gillam, Manitoba. The difference in vegetation demarks the trimline (slope) between unmodified till above (to the left of) the marine limit and till covered by glaciomarine sediments below (to the right of) the limit.

Typically, till surfaces in topographically higher terrain near the limit of submergence have only been weakly reworked by the postglacial sea or glacial lake, and therefore the till is of suitable quality for sampling (e.g. Strand et al., 2009; Randour et al., 2016). In contrast, till surfaces lower than the marine/lacustrine limit may be significantly reworked and winnowed by waves and currents forming a bouldery mantle or beach shingle of poorly sorted debris (washed till) (Fig. 30b). Samples in these areas must be collected beneath the bouldery mantle or lag (Fig. 31).

In areas that were temporarily submerged in a deglacial sea or lake, thin till can be completely reworked into coarse-grained stepped raised beach strandlines. In permafrost areas, suitable till samples can be recovered from rare frost boils between beach crests.

Figure 31. Photograph of boulder lag and reworked stony till above unmodified till (grayish brown colour) in a hand-dug hole. The till surface was reworked by glacial Lake Agassiz following deglaciation in the central Canadian Shield area of Manitoba, west of Thompson (*from* Spirito et al., 2011).

5.3.1. Sampling in areas covered by marine and/or lacustrine blankets

Thick marine/lacustrine blankets (>2 m thick) can completely obscure the surface till or conceal the bedrock, particularly in areas formerly covered by large glacial lake basins.

Choosing a till sampling site within these areas requires an understanding of the marine/lacustrine sediment thickness in order to evaluate the cost-effectiveness of hand sampling at depth below the cover, trenching, or overburden drilling. In areas of extensive marine/lacustrine blankets, acceptable sampling sites may be found on topographic highs where the blanket is thinner (Fig. 32), or at the base of sections along lake shores, streambanks or roadcuts, or at depth using deep overburden sampling methods (*see* Section 6.8).

5.3.2. Sampling in areas covered by marine and/or lacustrine veneers

In areas covered by marine/lacustrine veneers (<2 m thick), potential till sampling sites can be accessed beneath the marine/lacustrine sediments using shallow overburden sampling methods (*see* Section 6.8). Till

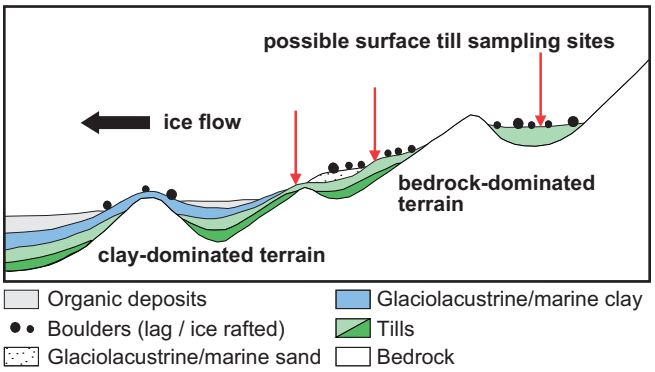

Figure 32. Illustration showing the location of potential till collection sites in Canadian Shield terrain covered by relatively thin (0–2 m) glacial and postglacial sediments that includes upland areas of bedrock-dominated terrain and topographically lower areas dominated by thick glaciolacustrine/marine silt and clay deposits (*modified from* Henderson, 1995).

sampling sites may be found on the uplands (thinner sediment) and down-ice sides of outcrops (Fig. 32) and streamlined landforms. On rocky uplands, till commonly forms a discontinuous veneer with thicker accumulations occurring in depressions from which it can be easily collected. Additional sites may be found in natural or man-made exposures along roads or trails, borrow pits and quarries, or in natural sections along modern river and lake shorelines.

5.4. Sampling in areas of permafrost

In areas underlain by permafrost, physical weathering is the dominant process in soil formation. Cryoturbation, which mixes soil horizons, can be identified by patterned ground features, such as sorted and unsorted nets, circles, polygons, and stripes. In these regions, frost boils, or non-sorted circles, are the preferred till sampling sites (Hornbrook and Allan, 1970; Shilts, 1973, 1977, 1978; Laurus and Fletcher, 1999; McMartin and McClenaghan, 2001; McMartin and Campbell, 2009).

Frost boils are easily recognized on large-scale air photos (≥1:20,000), directly from low-flying aircraft, or on the ground as bare to lichen-covered, round to oval patches, commonly surrounded by ridges of vegetation and rocks, and having either lighter or darker colours than the surrounding sediment/regolith. Due to hydrostatic pressure in the active layer that thaws every summer, relatively fresh till is pushed up to surface in the middle of the frost boil (Fig. 33a). This middle area is free of vegetation (Fig. 33b) and thus is a relatively easy place to dig a hole for sampling. Repeated freezing and thawing of groundwater can force larger stones toward the surface.

Till in frost boils is commonly well homogenized and relatively unweathered, hence representative samples may be collected with a shovel at shallow depth (>0.3 m) from the centre of the patterned ground.

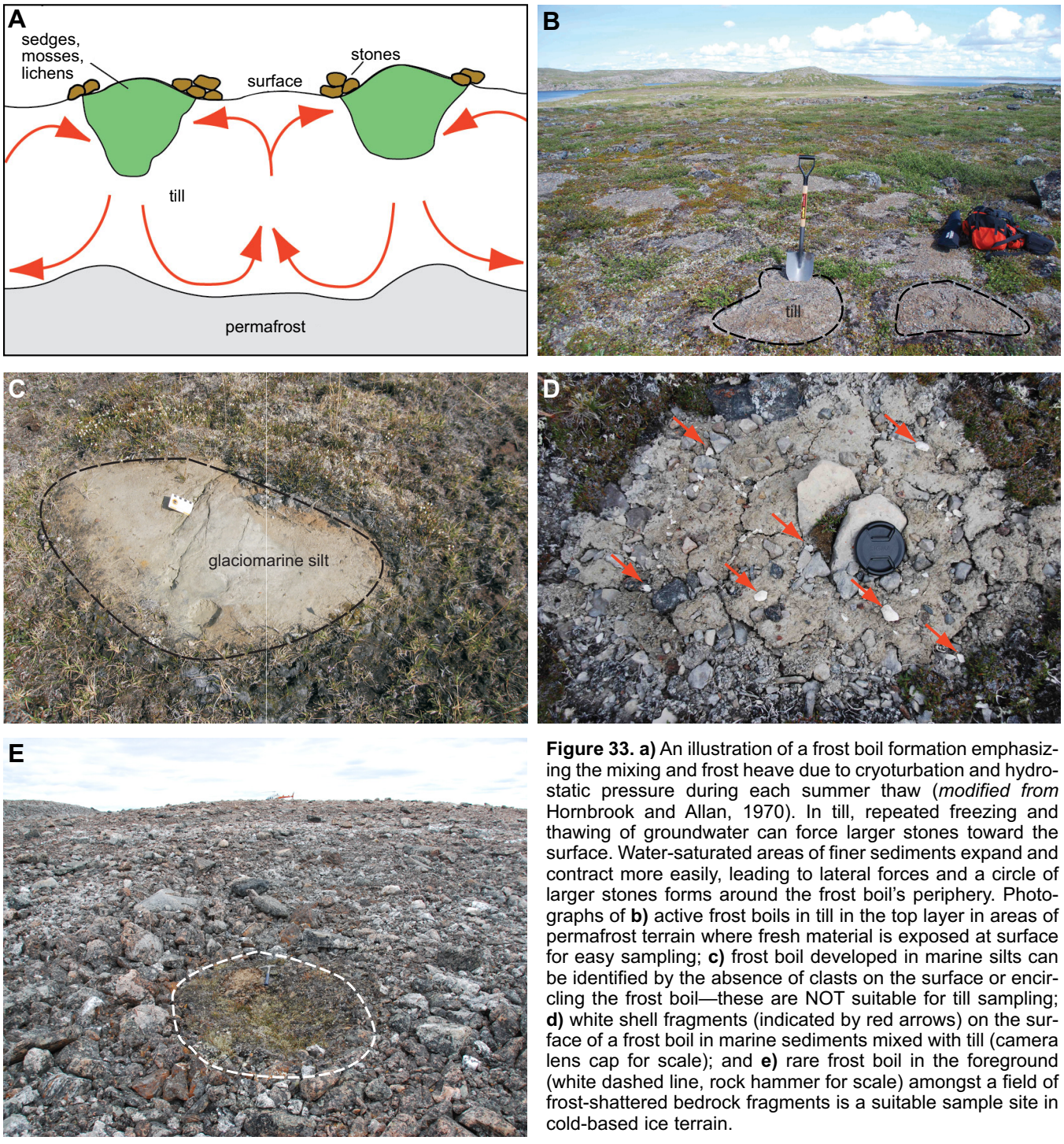

Figure 33. a) An illustration of a frost boil formation emphasizing the mixing and frost heave due to cryoturbation and hydrostatic pressure during each summer thaw (*modified from* Hornbrook and Allan, 1970). In till, repeated freezing and thawing of groundwater can force larger stones toward the surface. Water-saturated areas of finer sediments expand and contract more easily, leading to lateral forces and a circle of larger stones forms around the frost boil's periphery. Photographs of **b)** active frost boils in till in the top layer in areas of permafrost terrain where fresh material is exposed at surface for easy sampling; **c)** frost boil developed in marine silts can be identified by the absence of clasts on the surface or encircling the frost boil—these are NOT suitable for till sampling; **d)** white shell fragments (indicated by red arrows) on the surface of a frost boil in marine sediments mixed with till (camera lens cap for scale); and **e)** rare frost boil in the foreground (white dashed line, rock hammer for scale) amongst a field of frost-shattered bedrock fragments is a suitable sample site in cold-based ice terrain.

Depth of sampling is restricted by the thickness of the active layer, which can extend up to 2 m in depth during the maximum summer thaw period in the southern areas of the continuous permafrost zone or <0.5 m depth in more northerly areas (e.g. Shilts, 1978; Dyke and Zoltai, 1980; Dredge, 2002; French, 2007).

There are special considerations for till sampling in areas covered by marine/lacustrine sediments and underlain by permafrost because frost boils may also form on silt- or clay-rich marine/lacustrine sediments. Therefore, it can be difficult to differentiate tills from fine-grained marine/lacustrine sediments from an aircraft or by airphoto interpretation. Generally, frost boils in marine/lacustrine sediments have few or no rock fragments on the surface (Fig. 33c), except for exotic debris from offshore areas (iceberg dropstones). Frost boils in marine sediments may contain marine shells on the surface or within the sediment (Fig. 33d).

Although not particularly apparent with field observations, till in frost boils directly below the marine

limit may be slightly enriched in sand and depleted in finer particles (silt and clay) as compared to till above the marine limit as a result of winnowing by waves (McMartin et al., 2019a,b). Therefore, marine reworking has the potential to modify till composition as a consequence of changes in texture and resultant mineral partitioning; however, provenance appears to remain the dominant factor controlling composition in reworked till.

In areas covered by marine/lacustrine veneers (<1 m), cryoturbation in the active layer may result in mixing of till with the overlying thin marine/lacustrine cover, severely affecting the sediment composition. In such a scenario, the mixing of debris of local provenance (till) with fine-grained sediments of regional background composition (marine/lacustrine sediments) can mask the glacial dispersal signal. In areas covered by marine sediments, a vigorous reaction to HCl (10%) of an otherwise noncalcareous till may help to indicate the mixing of till with overlying calcareous marine sediments, or leaching and/or mixing with marine shells.

In permafrost regions affected by cold-based glacial conditions, sediment is generally rare and where it is present, it is a mixture of thin older glacial sediments and deeply weathered bedrock consisting of oxidized rock fragments and clay minerals. In these areas, till samples can be collected from rare frost boils (Fig. 33e), cryogenic sediments pushed into the centre of boulder rings (sorted circles) or in between boulders in felsenmeer terrain (e.g. Dredge, 2001; Tremblay and Paulen, 2012).

Vertical mixing of the soil can redistribute surface organic-rich horizons to depths within the active layer. Therefore, it is important not to include layers of organic material, oxidized clasts, or incorporated surface clasts of non-till sediments. The pit should be deep enough to evaluate by eye whether the sample is clear of organics and is representative of the area around it. If cryoturbated organics or oxidized material is found when the hole is dug, another hole should be dug nearby that does not contain these potential surface contaminants. Sometimes several attempts are necessary.

In areas where frost boils are poorly developed or scarce, such as in coarse-grained (sandy) bouldery till or in areas where surface till is covered by a thin organic mat, till samples can be collected from hand-dug holes below any thin soil profile, if present.

5.5. Selecting till sites in areas with specific glacial terrains and landforms

Where possible, till samples for drift exploration should be taken from either till veneers overlying bedrock, or from the base of till blankets. The assumption is that till closest to the bedrock has the highest probability of being derived from the local bedrock. However, in regions overlain by till that is known to be the result of long-distance glacial transport, the till does not reflect the composition of the local bedrock (e.g. Hicock, 1988; Thorleifson and Krisjansson, 1993; Trommelen and Ross, 2014).

The suitability of a landform for till sampling is related to its genesis and the mode and distance of glacial debris transport within the landform. Some landforms are predominantly composed of locally derived material and others contain variable percentages of distally derived material.

It is preferable, when possible, to sample locally derived till that best reflects the underlying bedrock. To assist with the interpretation of the geochemical and indicator mineral results, it is important to make note of the landform sampled and any other indicators, such as clast/boulder lithologies, that would indicate the dispersal distance and provenance of the till. Building on previous summaries by Aario and Peuraniemi (1992) and Proudfoot et al. (1995), Table 3 provides a summary of selected glacial landforms, their characteristics, and preferred till sampling sites.

For a regional-scale study in areas with different glacial landforms, it is good practice to document the glacial landforms from which the till samples are collected (Table 3). This strategy will help to identify any biases that specific landforms could introduce, as well as highlight any multi-phase till-landform generation (e.g. Stea and Brown, 1989).

Some landforms, such as lee-side deposits and De Geer moraines, may be composed of sediments other than till or have a veneer of nontill material. If till is not encountered or confidently identified, sampling is not recommended.

5.6. Selecting till sites in areas of anthropogenic disturbance or contamination

5.6.1. Definition

Anthropogenic deposits are either artificial materials or geological materials modified by human activities so that their original physical properties (e.g. structure, cohesion, compaction) have been significantly altered (Howes and Kenk, 1997). Such sediment should not be sampled for research or mineral exploration purposes for obvious reasons: composition is not related to local surficial geology and has been modified to unknown extents. Near-surface till, near areas modified by humans, even those that appear to be undisturbed, can in fact be highly contaminated (McMartin et al., 1999) and/or bulldozed (e.g. along roadcut shoulders).

Care must also be taken to avoid sampling till that has been contaminated by airborne particulates from

Table 3. Overview of potential till sample sites with respect to till thickness and glacial landforms.

Terrain types	Provenance	Preferred sampling site	Comments	Selected publications
Till veneer (till <2 m thick)	Predominantly local debris	Well drained, top of slope, flat area, *or* Down-ice side of outcrops	Generally best terrain for sampling locally derived debris if till is thick enough (>60–cm) to collect C-horizon soil	Paulen et al., 2013; Kelley et al., 2019
Till blanket or till plain (till >2 m thick)	Variable; generally has significant proportion of local debris	Well drained, top of slope, flat area	Typically, the proportion of local debris increases with depth with the greatest percentage just above the bedrock surface	McMartin, 2000; Averill, 2017; Kelley et al., 2019
Crag-and-tail, Precrag landform		Stoss slope of crag if till is present, otherwise along top of tail	Precrag: tail or rampart formed on the up-ice side of the crag Caution: small-scale crag-and-tails can resemble resemble lee-side deposits; *see* below	Crag-and-tail: McMartin et al., 2015 Precrag: Haavisto-Hyvärinen et al., 1989; Haavisto-Hyvärinen, 1997
Lee-side deposit		Down-ice side of outcrop at top of slope	Cavity-fill deposits beside and down-ice of outcrops; sediments, or portions of, have undergone some degree of meltwater sorting, i.e., fines removed	McMartin et al., 1996
Hummocky moraine – undulating, gentle relief		Well drained, top of slope, flat area	Till composition similar to till plain	Eyles et al., 1999; McMartin et al., 2006; Evans et al., 2014
Ice-pressed landforms: ridges, hills		Top of and between ridges	Basal debris and/or previously deposited sediments squeezed up into cavities (e.g. basal crevasse fills)	Sharp, 1985; Kleman, 1988; Evans et al., 2014, 2016, 2019
Rogen moraine, ribbed moraine	Surface till is local; core (lower till) can have higher proportion of distal de bris	Crest of ridge	Multiple theories on genesis; each type of ridged moraine may be linked to a specific depositional environment, thus will affect how the fill results are interpreted; limited studies suggest the surface deposit is more locally derived	Sarala and Neonen, 2005; Knudsen et al., 2006; Möller, 2006, 2010; Sarala and Peuraniemi, 2007; Trommelen et al., 2014
Drumlin, streamlined ridge	Variable with higher proportion of distal debris	Top of landform, up-ice end	Multiple theories on genesis: surface till often more locally derived; internal composition of drumlins is variable	Stea and Brown, 1989; Campbell, 2007; Plouffe et al., 2011; Stokes et al., 2013b; Evans et al., 2015; Möller and Dowling, 2016; McMartin, 2017; Menzies et al., 2018
Mega-scale glacial lineations (MSGL)	Variable with higher proportion of distal debris	Top of ridge	Generally located in ice stream; typically fast-flowing, long-distance transport	Clark, 1993; Ross et al., 2009; Ó Cofaigh, et al., 2013; Stokes et al., 2013a
Hummocky moraine "kame and kettle"		Between knobs but not in kettles (sorted sediments)	Ice contact deposit; mixture of sand gravel and till; variable sorting; meltout, flow and dump deposits **Caution: sampling not recommended**	
Moraine ridge: recessional, push, minor end, De Geer	Variable, mixed provenances	Top of ridge; where matrix is present	Debris flow, dump, ploughed and meltout debris; in a montane setting, these can provide a regional indication of the mineral potential in the source region of the ice	Day et al., 1987; Evenson and Clinch, 1987
Lateral moraine		Top of ridge; where matrix is present	Restricted to valley glaciers/montane terrain; prospect following moraine up-ice; *see* comment for moraine ridge	Day et al., 1987; Evenson and Clinch, 1987
Medial moraine		Top of ridge; where matrix is present	Rare; restricted to valley glaciers/montane terrain; prospect following moraine up-ice; *see* comment for moraine ridge	Day et al., 1987; Evenson and Clinch, 1987
Thrust moraine (glacial tectonite)		NA	Glaciotectonic ice thrust ridges; plucked, folded and/or stacked sequences **Caution: not recommended for sampling due to deposit complexity**	Evans, 2018
Subglacial meltwater corridors		Crest of mound or ridge if composed of diamicton	Deposits of hummocks and ridges with variable composition and genesis: sand, sand and gravel, and till **Caution: sample till, not ice-contact deposits**	Utting et al., 2009; Haiblen, 2017; Peterson and Johnson, 2018; Peterson et al., 2018

mine sites, smelters (Fig. 34a), refineries (e.g. McMartin et al., 1999; Bajc and Hall, 2000; McClenaghan et al., 2013b), railways, and highways.

Sediments may be affected by overburden drilling waste water and fluids, drill cuttings, drilling mud, grease, and diamonds from drill bits, all of which will contaminate the sample (Fig. 34b) (e.g. McClenaghan et al., 2014).

5.6.2. Common locations of anthropogenic deposits and contamination

Anthropogenic contamination occurs at, and downwind of, the following:

- present and past-producing mines (Fig. 28), mills, smelters, refineries, and tailings ponds;
- present and past-producing granular aggregate and rock quarries, or borrow pits;
- man-made structures and signs;
- transportation corridors (e.g. roads, railways, bridges, dams);
- hydrocarbon infrastructure sites and corridors (e.g. pipelines, drill sites, pumping stations);
- forestry cut blocks and log skidding slopes;
- agricultural areas;
- populated regions.

5.6.3. Criteria to identify anthropogenic deposits

The following criteria may be used to identify anthropogenic deposits:

- landforms—suspicious landforms not related to glacial processes;
- porous sediment texture, poor compaction, and chaotic structures;
- visible contaminants or reworked material in the sediment; scrap metal, stumps, square logs, unusual odour, metal or glass fragments, etc.;
- proximity to locations listed above in Section 5.6.2;
- suspicious paleosol and/or organic matter (buried, truncated, or homogenized modern soil profile/humus layers/wood debris overlain by anthropogenic deposits);
- high-precision digital elevation models such as those constructed using LiDAR (where available) can greatly aid in the identification and avoidance of anthropogenic sites.

6. TILL SAMPLING

6.1. Sample size

6.1.1. Sampling for geochemical analyses

The optimal sample size to allow for matrix geochemical analyses, physical determinations (grain size, colour, carbon content, etc.) and archiving is a mass of

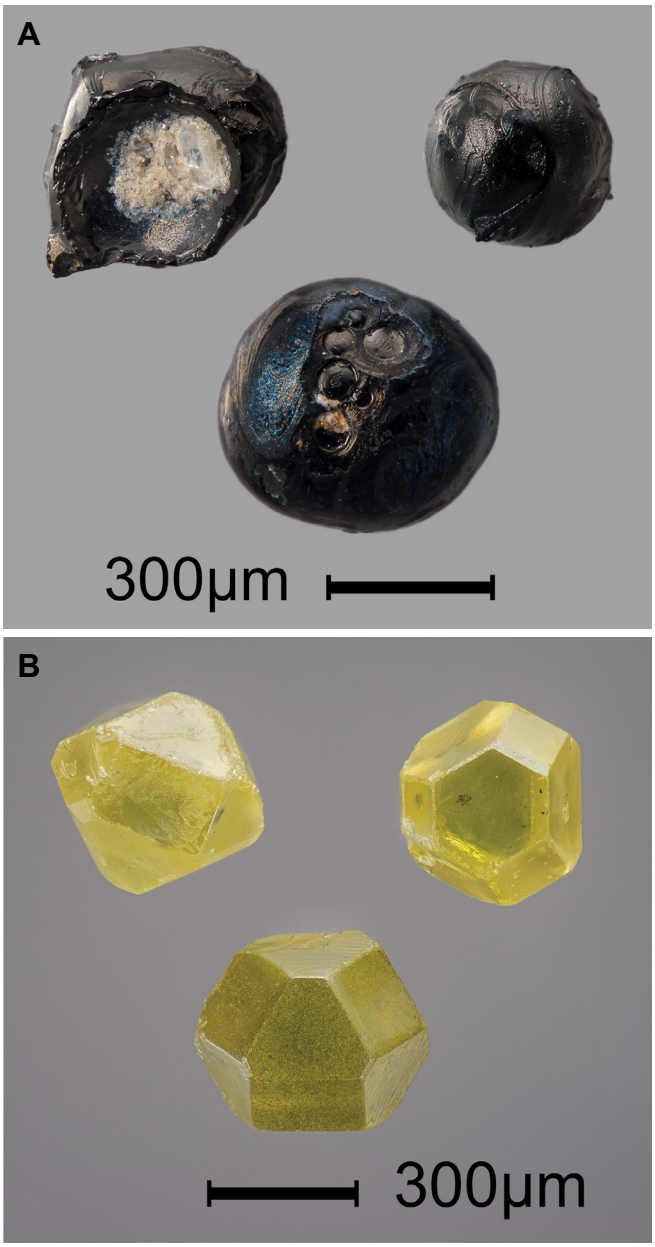

Figure 34. Examples of anthropogenic contamination in the heavy mineral fraction of till: **a)** smelter particles recovered from a till sample collected proximal to the Thompson Ni-Cu mine site in northern Manitoba (*from* McClenaghan et al., 2013b); and **b)** industrial diamonds recovered from the heavy mineral fraction of a till sample that was collected from diamond drill core at the Sisson W-Mo deposit, New Brunswick (*from* McClenaghan et al., 2014). Photographs provided by Michael J. Bainbridge Photography.

~3 kg (Fig. 21a). This small sample should be collected from the same hole as the large indicator mineral sample used for heavy mineral separation. The sample should be collected in a bag that is separate from the heavy mineral sample to allow for simultaneous shipping to different laboratories if required. The small bag should be filled 2/3 to 3/4 full, and weighed with a portable scale (e.g. hanging fish weighing scale) to

ensure 3 kg has been collected. Larger, obvious clasts (>10 mm) should be excluded when filling the geochemical sample bag. For tills containing abundant clasts, sample size may need to be increased to recover sufficient volume of the finer till matrix material (<0.063 mm).

6.1.2. Sampling for indicator mineral recovery

A large till sample weighing between 10 and 40 kg, should be collected for recovery of indicator minerals.

i) For sandy till: 10 to 15 kg (~5–8 L) of material should be collected for indicator mineral surveys (Fig. 21a);
ii) For silt- or clay-rich till: 20 to 40 kg (~10–19 L) of material should be collected for indicator mineral surveys (Fig. 21b).

6.1.3. Sampling for portable X-ray fluorescence analyses

A small (~200 g) sample (Fig. 21a) should be collected if pXRF measurements are to be conducted while in the field (*see* Section 9.4 for more details).

6.1.4. Sampling for examining the pebble fraction of till

If the pebble fraction is to be used for till provenance studies, pebbles can be collected from the pit or the surface of a sorted circle or a non-sorted circle (frost boil) and put in a separate sample bag. Alternatively, >2 mm clasts (including pebble-sized) may be recovered during sample processing at the laboratory from either the small 3.0 kg sample (granules only) and/or from the large >10 kg indicator mineral sample (granules and pebbles).

6.2. Till sample depth

In reconnaissance- and regional-scale surveys for which hand tools are utilized (e.g. shovel and hoe), till samples are commonly collected from the C horizon developed on till in the upper part of the surface till unit (0.5–1.0 m below the natural land surface).

In local-scale surveys designed to locate buried mineralization with the greatest possible precision (e.g. prior to drilling), where possible, samples can be collected at various depths below the visibly intensely oxidized zone (B horizon; *see* Section 2.3). Samples collected close to bedrock (<1 m above bedrock surface) are preferred, as the lower part of the till generally reflects a more local provenance.

Where till is thick (>2 m) and where possible, additional samples should be collected at different depths (0.2–0.5 m intervals) at a small number of sites in sections or trenches, to characterize compositional variations related to till stratigraphy and/or weathering.

In some prospective areas covered by thick till, such as the Abitibi Greenstone Belt in the Canadian Shield (e.g. Bird and Coker, 1987; Sauerbrei et al.,1987; McClenaghan, 1994, 2001), or the central Interior Plateau of the Canadian Cordillera (e.g. Ferbey and Levson, 2009; Averill, 2017), multiple till samples should be collected throughout the till sequences because dispersal trains may be intersected at any depth and in more than one till unit (e.g. Garrett, 1971; Paulen, 2009).

In areas of continuous permafrost, the depth of till sampling is restricted by the thickness of the active layer above the permafrost. Depending on the area being investigated, active layer depths may vary from 0.5 to 2 m during the maximum summer thaw. Till in frost boils is commonly well homogenized and relatively unweathered, hence representative samples may be collected with a shovel at shallow depth (>0.3 m) from the centre of the feature (McMartin and McClenaghan, 2001; McMartin and Campbell, 2009).

6.3. Till sampling in areas of anthropogenic disturbances and other contamination

Contaminated areas can be sampled if done so with extreme caution and diligence. Some guidelines include the following:

- avoid suspected disturbed/contaminated areas;
- fresh exposures can be found along road construction sites, including trenches and pits; discussion with machinery operators can provide valuable information;
- fresh exposures can be found in the walls of open pit mines (Fig. 28);
- samples may be collected at the base of granular aggregate pits, if the pit has been excavated down to/into till;
- avoid areas stained by oil or hydraulic fluid;
- if an area is suspected of being physically disturbed (e.g. bulldozed), look for an intact soil profile to indicate the material is in place and suitable for sampling;
- contamination around anthropogenic sites (mine site, smelter, etc.) can be airborne and penetrate the B-horizon soil depending on the distance from the point source; contamination may also occur at depth in C-horizon soils, particularly in coarse-grained till or where the surface organic layer is thin (McMartin et al., 1999, 2002), or it may fall onto the sample as it is being collected. Exercise caution with exposed horizontal and vertical surfaces;
- dig deep vertically (>1 m) into a hand-dug pit or natural sediment before collecting a sample and pay attention to any surface material that could

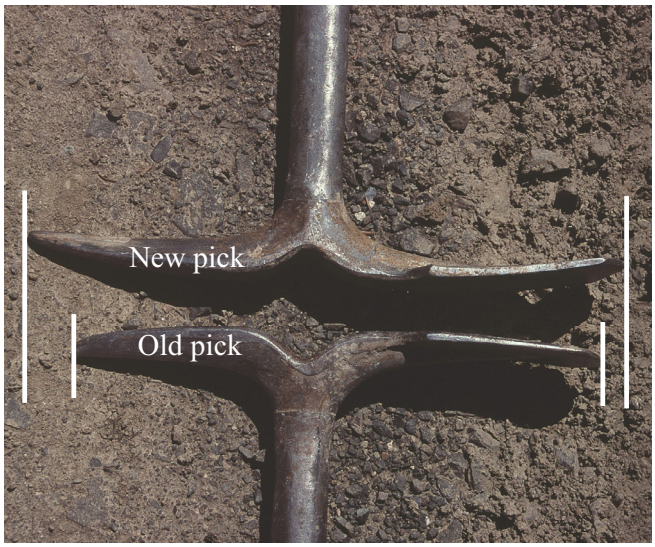

Figure 35. Two identical hoe-picks used to excavate and collect till samples; the top pick is nearly new and the bottom pick has been used for three field seasons. Note that the older one has visibly lost metal (distance between the white lines) that has, in part, ended up in the collected till samples (*from* Plouffe et al., 2014).

inadvertently fall into the sampled material obtained at depth;

- dig deep horizontally (>1 m) into open pit walls or roadcuts before collecting a sample to avoid anthropogenic material that may reside in the outer layers of the vertical face;
- sampling equipment requires extra cleaning;
- keep sample containers closed until the last possible moment before filling them with sediment;
- rinse sample containers with distilled water just before sampling to remove mining/smelter dust;
- coveralls and other types of clothing that have been used around heavy machinery, drilling, or rock-cutting operations must not be worn;
- sampling tools should be monitored for wear as small metal flakes may break off tools and end up in the sample as contamination (Fig. 35).

6.4. Composite till samples

A composite till sample is one in which several samples are combined into one sample that is then analyzed. In contrast to some reconnaissance surveys carried out as part of the national and international geochemical mapping programs (e.g. Edén and Björklund, 1995; Salminen et al., 1998, 2005), the GSC does not use composite till samples for routine regional till sampling programs.

However, the GSC does use composite till samples when sampling till in drill core in local areas for focussed studies. For example, a till sample collected from drill core may be a composite taken along a specific vertical interval (usually 1.5 m). In these cases, composites should be of similar characteristics and not from across lithological contacts.

The GSC also uses composite till samples when reanalyzing archived material in small focussed studies, where the archived till samples are of insufficient mass for reanalysis. For example, several smaller 1 to 2 kg samples from adjacent locations may be combined and mixed thoroughly to make one larger heavy mineral sample (e.g. McClenaghan et al., 2019a).

6.5. Field data and notes

Collection of field data for each till sample is essential to the success of any sampling program. The need for high quality measurements and observational records cannot be stressed enough. Field data are now commonly captured in digital format using mobile data collectors such a field computers, tablets, or personal digital assistants (e.g. Salminen, 1992b; Buller, 2004; Shimamura et al., 2008; Schlatter et al., 2010; Allard et al., 2018). In addition to using a mobile data collector, the location of each sample site may be recorded on a hard copy of an airphoto and/or topographic map or as coordinates in a field notebook.

Photographs should be taken of each sample site and sample hole and should also include a sample number label and scale in the photograph. Additional photographs should be taken to show context of the sample site within the area. For example, if the sample site is associated with a glacial landform or nearby anthropogenic influences, and what the surrounding vegetation cover looks like. Small digital cameras can be useful for taking photographs inside of dug holes allowing for proper exposure and resolution of photographs of the sampled and overlying sediments.

Ensure an accurate site location is determined using a GPS and ensure everyone uses the same datum for locational data collection.

The minimum field data that must be recorded for each till sample collected for heavy mineral and geochemical analyses are listed below.

- site number;
- sample number;
- site and sample photographs;
- location: UTM easting and northing, or latitude and longitude;
- UTM zone;
- datum: NAD83 or 27;
- NTS map sheet (1:250,000 or 1:50,000 scale);
- province/territory;
- name of geologist who collected the sample;
- material type: diamicton, otherwise describe;
- sediment genetic interpretation: till, subglacial till, reworked till, unknown genesis, etc.;

- landform/map unit: till blanket, till veneer, streamlined till, moraine, etc.;
- sample site type: river section, lake shore, road-cut, open pit, gravel pit, dug hole, trench, frost boil, etc.;
- purpose of sample: for analysis of heavy minerals, matrix geochemistry, grain size, etc.;
- sample depth (top) from natural land surface (m);
- sample depth (bottom) from natural land surface (m);
- degree of sample oxidation: unoxidized, light, moderate, or heavy;
- sample moisture content: dry, moist, or wet;
- sample texture: silty sand, sandy, clay-rich, etc.;
- sample colour: grey-brown, reddish, orange-brown, etc.; Munsell colour;
- clast comments: lithologies observed, as well as size (maximum, minimum, mode), angularity, abundance, presence of striae, etc.;
- general notes, if required;
- site sketch if required;
- site location marked on a paper topographic map.

6.6. Numbering till samples

The till sample numbering scheme employed by the GSC must include a three-letter officer's code; each GSC scientist has a unique three-letter code.

Typically, the three-letter code is preceded by two digits to indicate the year of sampling: e.g. 20-MPB-001, for samples collected in 2020 by a geologist with an officer code of MPB. The sample numbering scheme should allow for insertion of quality assurance/quality control (QA/QC) samples during the sample preparation stage. Preplanning this scheme before going into the field is encouraged.

If the GanFeld digital field sample data capture system is used, it is not as critical to reserve sample numbers while in the field because the QA/QC samples can be added as an extra sample number at a station (e.g. 07-PMA-230-1 and 07-PMA-230-2 could be a routine and a QC/QA sample, respectively).

Regardless of the numbering system chosen, it is critical to communicate with the preparation laboratory and to indicate how sample numbers for QA/QC samples are handled.

Section 7.2.5 provides more details about inserting QA/QC samples into the sample numbering scheme.

6.7. Till sample quality in the field

A significant source of variability in the till geochemical or mineralogy data can be due to poorly defined field protocols (e.g. Hoffman and Woods, 1991). Some basic sampling and data recording procedures can be followed in the field to reduce the possibility of geochemical or mineralogy data variability and to ensure that the sample is well documented and is not contaminated:

- ensure that the samplers and assistants are well trained and experienced;
- to avoid contamination, do not use coloured, coated or painted sampling equipment;
- do not wear jewellery on hands that may come in contact with the sample material or containers (cf. Kontas, 1991);
- thoroughly clean sampling tools between each sample site, preferably with a water rinse, but if this is not possible, then wipe the tools with a clean rag or moist moss;
- sample numbers should be written at more than one location on the outside of sample bags/pails with a high-quality permanent marker and labelled waterproof tags should also be inserted inside each sample bag/pail. A mislabeled or unlabeled sample can result in the loss of two samples;
- ensure sample bags or buckets are new and clean inside;
- use a standardized field collection data form so that the same minimum level of information will be collected by everyone, e.g., GanFeld (*see also* Section 6.5);
- review the field notes and sample locations at the end of each sampling day to be sure there are no errors or omissions;
- crosscheck sample numbers on the bags against the sample list on a daily basis, and check the master sample list against the samples in the pails before shipping. Note: checking sample numbers on a daily basis increases the chances of being able to successfully correct sample numbering mistakes or notice that samples are missing;
- anthropogenic contamination of glacial sediments can affect the geochemical and indicator mineral signature (*see* Section 6.3 for guidelines for sampling in these areas);
- sample numbers may be assigned to a till sample site beforehand for preselected sites or selected in the field using a digital field note-taking system;
- sample numbers should be reserved for later insertion of QA/QC samples (*see* Section 7.2.5 for details).

Field duplicate samples are used as part of an overall QA/QC scheme to monitor and ensure data quality. Refer to Section 7.1 for instructions for the collection of field duplicate samples.

6.8. Methods for sampling till

6.8.1. Hand excavation

The most cost-effective procedure to collect surface till samples at shallow depths is from pits dug with a hand shovel (Fig. 27a). Hand-picks, geological hammers, or small axes can also be used to remove boulders, to dig into very hard material, or to cut roots. Small saws or pruning shears are useful to cut roots. Till samples can also be collected with a shovel from natural sections along rivers or lake shorelines (Fig. 27b), roadcuts (Fig. 27c), borrow pits, or open pits (Fig. 28).

Depending on accessibility and spacing between sample sites, about 5 to 8 holes can be hand-dug in an 8-hour day by a two-person crew in forested areas. Pits should be filled with excavated material after the till sampling is completed to avoid leaving hazards for humans or wildlife.

In permafrost terrain, as many as 20 samples per day can be collected by a two-person crew with helicopter support. Frost boils are the most common sampling site in permafrost terrain (Fig. 33), but sometimes river sections can also provide excellent opportunities for sampling by hand. On river sections, only undisturbed till, i.e., till that has not been slumped or soliflucted should be collected.

6.8.2. Dutch auger

The Dutch auger (Fig. 36) is a light, portable hand tool that cuts through heavily rooted A- and B-horizon soils to test the nature of soil/sediments at depth prior to digging a hole with a shovel. It can be used identify the proper sampling material (till), required depth, and optimal sampling site.

The use of a Dutch auger saves time in forested areas as it can be used to determine if the overlying sediments (glaciomarine/glaciolacustrine clays or glaciofluvial sand) are thin enough (<0.5 m) to dig a hole through in order to reach the unoxidized till layer below. Dutch augers are rarely used in permafrost areas as they generally cannot penetrate the frozen sediment.

Dutch augers can also be used to collect small till samples (1–2 kg) suitable for geochemical surveys. They are ideally suited for collecting fine-grained surface tills (e.g. on the Prairies) at sites where the water table is very close to surface and hand-dug holes become immediately flooded, for sampling at depths unreachable with a hand shovel, or where small quick till samples are needed for pXRF analysis.

6.8.3. Trenching/pitting

The use of a wheeled or tracked excavator for digging holes or trenches (Fig. 37) can be economical in areas of thicker (>3 m) drift and where terrain conditions permit. A typical backhoe machine can dig trenches

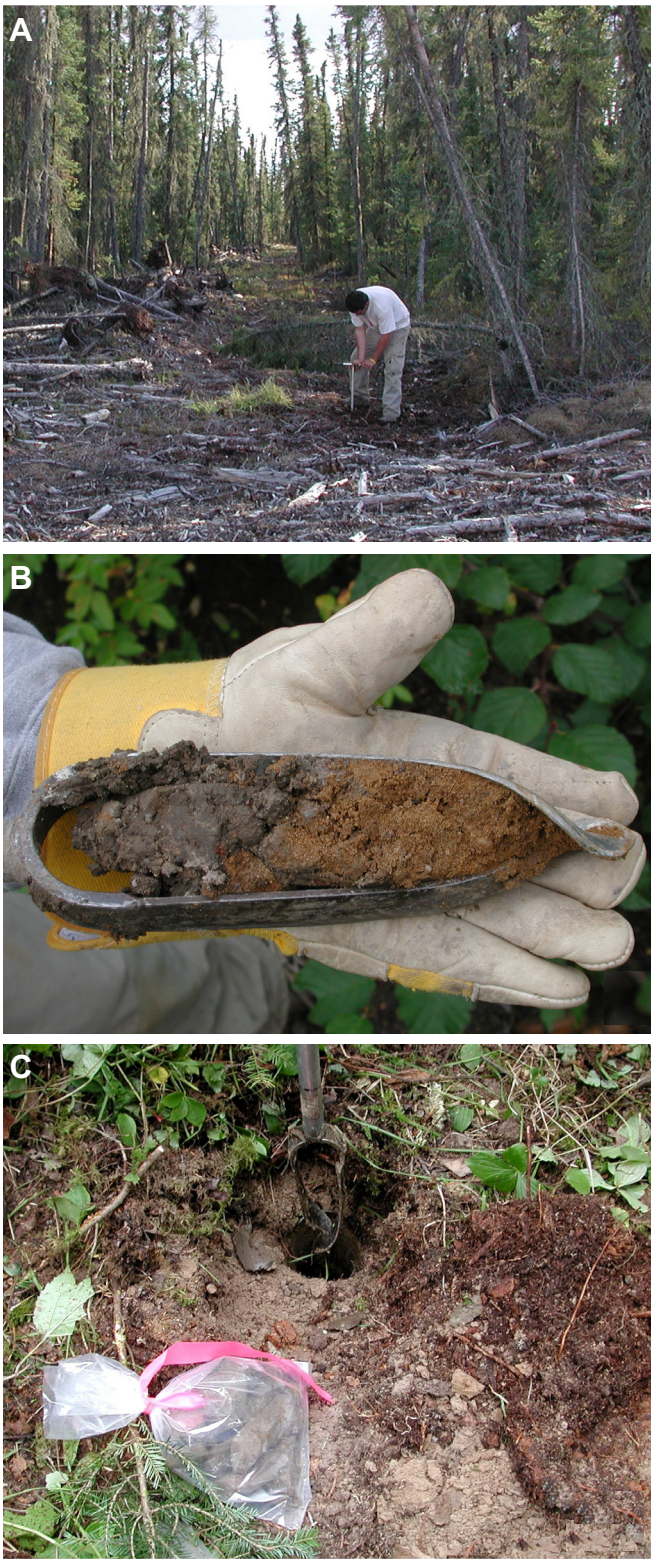

Figure 36. Photographs of till samples being collected using a Dutch auger: **a)** augering to obtain a sample below a thick cover layer of peat; **b)** material recovered in a Dutch auger displaying the contact between the till and the underlying glaciofluvial sediment; and **c)** till material obtained using a Dutch auger after the LFH layer was removed by digging a shallow hole with a shovel. At this type of site, the auger with extensions was used to recover till several metres deeper than the bottom of the dug hole (*from* Paulen, 2009).

Figure 37. A small-tracked excavator used to dig trenches up to 3 m deep to collect till samples within and beneath highly oxidized surface till. Photograph courtesy of R. Paulen.

from 3 to 5 m deep. The use of an excavator is particularly helpful for collecting closely spaced samples in a small area or study site (e.g. McClenaghan et al., 2014, 2018a). Direct observations of the bedrock and till features are possible in the trench walls or floor, and large and representative till samples can be obtained across vertical profiles or along a longitudinal profile at regular intervals. Environmental impacts can be significant when using excavators, particularly if trails have to be cut in areas of dense tree cover.

Hazards of working in a trench are significant and walls should be stepped or reinforced (*see* Section 4.1.2 for trenching safety advice). The pits must be back filled with excavated material after the till sampling is completed to avoid leaving hazards for humans or wildlife.

6.8.4. Portable drills (Wacker Neuson, Pionjär, Cobra®, Sipre, CCREL)

Portable drilling equipment (Fig. 38) is an alternative for till sampling where drift is moderately thick (<10 m), or where accessibility, costs or environmental impact restrictions prohibit the use of a backhoe or truck or track-mounted drills (Gleeson and Cormier, 1971; Gleeson and Sheehan, 1987; Lestinen et al., 1991; Hartikainen and Nurmi, 1993; Gustavsson et al., 1994; Sarala, 2015a,b).

Percussion drills use short (1 m), small-diameter rods and a short (0.3 m) flow through ejector bit (2–3 cm diameter) to collect a single till sample at the bottom of a drillhole, to a maximum of 8 to 10 m depth in sandy till with few boulders (Hirvas and Nenonen, 1990). Till samples are small (150–300 g) and small 'buttons' of bedrock (2–3 cm) can be collected where bedrock is crushed, loosened, or weathered (Plouffe, 1995b). Till samples are collected from a flow-through bit.

Figure 38. Photographs of portable drilling equipment: **a)** a portable percussion drill used to collect till samples in Finland; **b)** small (150–300 g) samples collected using the flow-through sampler on a portable percussion drill (photographs (a) and (b) *from* McMartin and McClenaghan, 2001); and **c)** till sampling below thick peat using a semi-portable Sipre auger corer in northern Manitoba (*from* McMartin and Campbell, 2009).

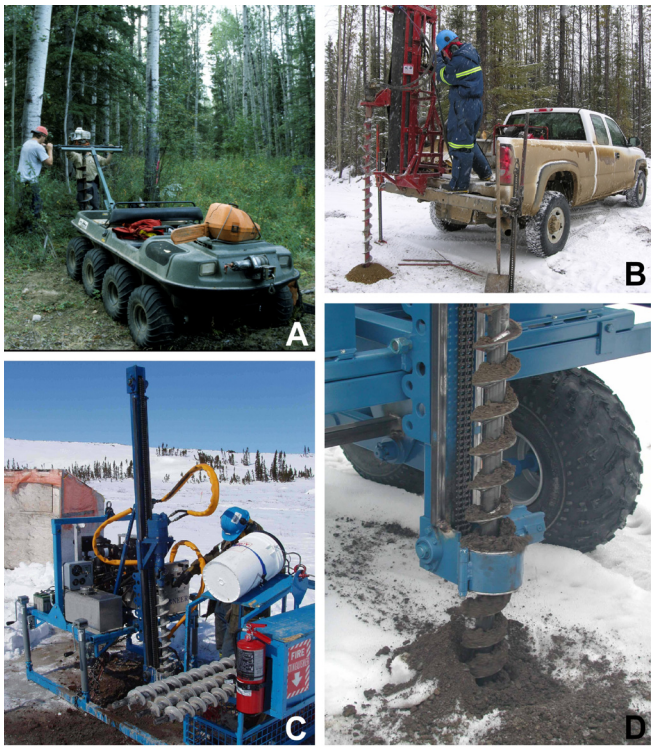

Figure 39. Photographs of solid-stem auger drills being used to recover till: **a)** a two-person auger with 15.2 cm (6 inch) diameter flights mounted on the back of an Argo® terrain vehicle to quickly obtain samples at 1 m depth; **b)** a Big Beaver® auger drill mounted on the back of a pick-up truck; **c)** a Pioneer® auger drill brought to a remote site in the Yukon by helicopter (photo courtesy of Multi-Power Products Ltd.); **d)** till recovered on 10.1 cm (4 inch) auger flights with a Pioneer® auger drill mounted on the back of an all-terrain vehicle (photo courtesy of Multi-Power Products Ltd.) (*from* Paulen, 2009).

The stratigraphy of sediments can be established with portable drills, but this requires the recovery of material for every interval equivalent to the sampler length. Samples collected are typically small (150–300 g) and can be used for fine-fraction till geochemistry, but are not sufficient for heavy mineral studies.

The lightest of the percussion drills weighs 10 to 25 kg, and can be carried into the field along with its extension rods. The advantages of track-mounted percussion drills are that they have minimal impact on the environment, they are portable, and are inexpensive to operate. However, they cannot drill deep holes or penetrate compact material, such as dry sand over consolidated interglacial clay or very stony and bouldery till (Averill, 1990).

6.8.5. Power auger drills

A solid-stem auger drill (Fig. 39) can be used in 1 to 100 m of unconsolidated sediments if the near surface stratigraphy is not overly complex and the cobble/boulder content is low (e.g. Canadian Prairies: Paulen, 2009). An auger stem is drilled into the ground at specific intervals and then pulled; till is collected from the auger stem flights (Fig. 39d). Commonly used auger flight diameters are 10.1 cm, 15.2 cm, and 20.3 cm (4 inch, 6 inch, and 8 inch, respectively), depending on the drill power and the depth of drilling required. Some sidewall contamination of the till being sampled is unavoidable, however the material closest to the drill stem is usually reliable enough for discerning stratigraphy and undertaking sampling. Once the groundwater table is penetrated, mixing of sediment on the auger stems is unavoidable, recovery is poor, and control of sample depth is unreliable.

A hollow-stem auger (Fig. 40) can be used through thick till sequences to collect continuous core and samples for indicator minerals (Pawlowicz et al., 1996,

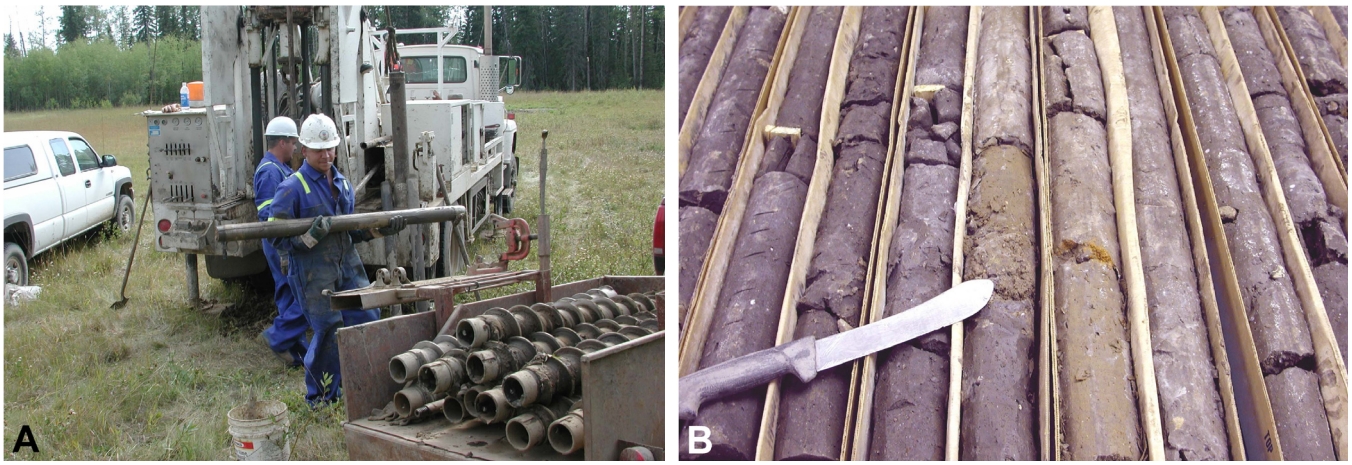

Figure 40. Photographs of a hollow-stem auger drill being used to recover till core for heavy mineral sampling in Alberta: **a)** removing the 1.5 m split core barrel, note the large 15.2 cm (6 inch) diameter hollow auger stems in the foreground; and **b)** an example of till core recovered by hollow-stem auger drilling (*from* Paulen, 2009).

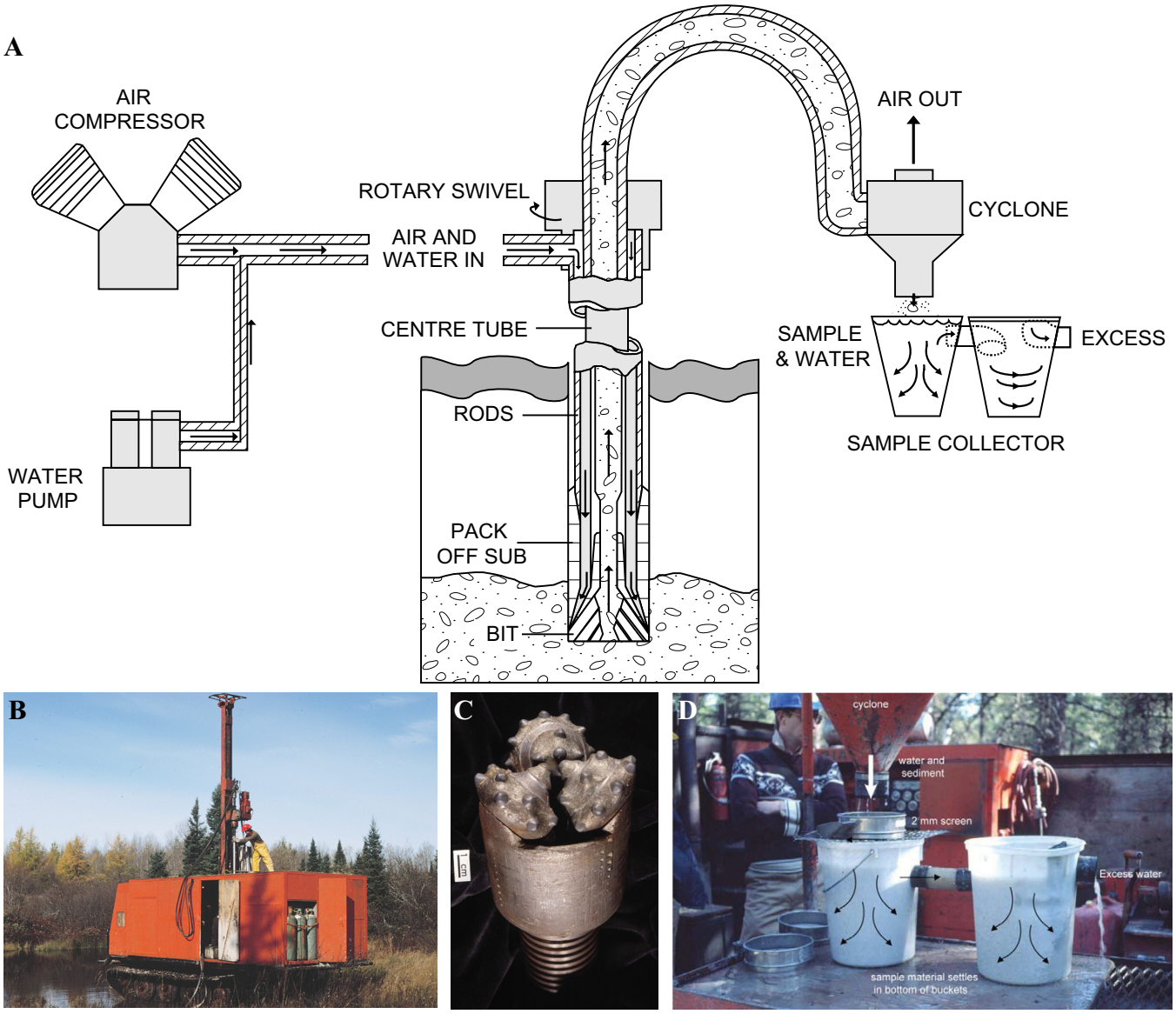

Figure 41. a) Schematic cross-section of reverse circulation (RC) drilling equipment, which is used to collect a mud+chip slurry from thick overburden sequences. Photographs of RC equipment: **b)** a track-mounted drill rig for summer drilling; **c)** a tricone bit used to drill the hole; and **d)** a cyclone used to decrease velocity of the slurry returned from the drillhole and the two-bucket system used to collect the sample and allow excess water to flow off. A sediment sample is collected from the bottom of both buckets at specific depth intervals. All photographs and images *from* McMartin and McClenaghan (2001).

1998; Paulen, 2009). Typically, truck-mounted drilling rigs are used. These are equipped with a 15 cm (6 inch) diameter hollow-stem auger with a fitted split-core barrel designed to retrieve core in segments up to 1.5 m long with a diameter of 7.6 cm. The core barrel precedes the hollow-stem drill bit by about 2 cm, coring as the auger flights drill into the ground. The hollow stem of the auger acts as a casing while the core barrel is retrieved every 1.5 m. Within the Interior Plains physiographic region, this is often the most economical means of obtaining shallow sedimentary core large enough for sampling. As with solid-stem auger drills, indurated beds, large cobbles, and boulders are impediments.

In areas of continuous and discontinuous permafrost, semiportable auger drills (e.g. CCREL) have been used for mineral soil sampling.

6.8.6. Reverse circulation rotary drills

Reverse circulation (RC) rotary drilling (Fig. 41a,b) can be used to collect till samples from units that are deeply buried and/or units that are thick (>5 m) and where till is stony and bouldery (McMartin and McClenaghan, 2001). Some Canadian examples of deep till sampling using RC drilling surveys include those conducted in prospective areas such as the Abitibi Greenstone Belt (e.g. Garrett, 1971; Skinner, 1972; Bird and Coker, 1987; Harron et al., 1987;

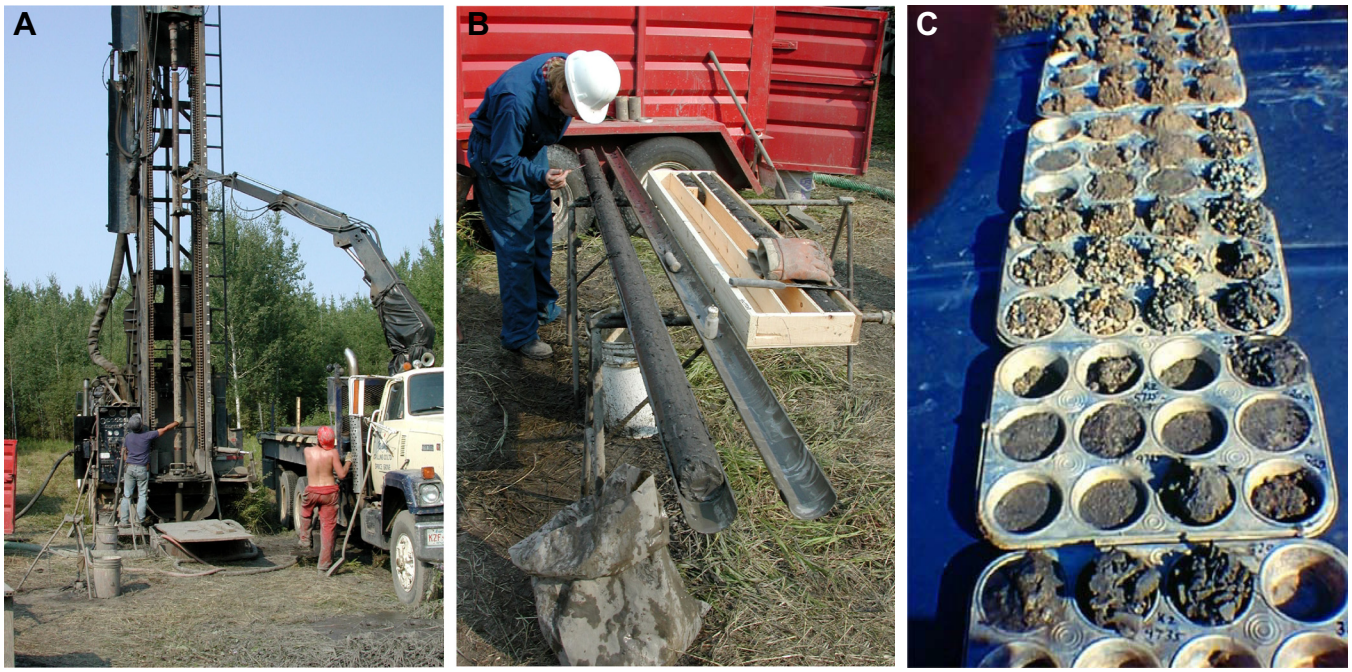

Figure 42. Photographs of **(a)** a mud rotary drill, which is a cost effective method for subsurface sampling of fine-grained, matrix-rich tills; **(b)** drill core from a mud-rotary drill laid out for sampling (*from* Paulen, 2009); and **(c)** cleaned samples collected from a mud rotary drilling rig, which range from clays and silts (cuttings), fine sands, sands and gravels, to fine-grained till (cuttings; extreme back) (*from* Spirito et al., 2011).

Sauerbrei et al., 1987; McClenaghan and Wyatt, 1997), the central Interior Plateau of British Columbia (e.g. Ferbey and Levson, 2009; Averill, 2017), the Athabasca Basin (Geddes, 1982; Wilson, 1985), and the central Slave kimberlite field (Kelley et al., 2019).

In RC drilling, dual tube rods are employed to drill a continuous 7 cm diameter hole through glacial sediments and into bedrock. Air and water are injected at high pressure down the outer tubes of the drill rods (Fig. 41a) to a tricone bit (Fig. 41c) at the bottom of the hole, which directs the compressed air and water mixture onto the bit as it cuts. Drill cuttings are carried up to the surface through the inner tube as a continuous slurry of <1 cm diameter chips and mud. The material delivered to the surface passes through a cyclone, to slow down the velocity of the slurry, through a 4.0 mm screen, and then into a two-bucket system (Fig. 41d) to allow the sediment to settle and excess water to flow off.

A 10 kg till sample is collected from material in the bottom of both buckets for approximately every 1 to 2 m drilled. Most clay-sized material and approximately 30% of the silt-sized material in the till is lost using this drilling method. Also, till samples can become cross-contaminated by the recirculating water.

Recovery is generally good for all sediment types and the drill can penetrate boulders and bedrock fairly quickly. The quality of stratigraphic interpretation is limited because the sample is a disturbed slurry of mud and rock chips and the geologist has only one chance to describe and sample the material as the drill rapidly penetrates the ground.

6.8.7. Mud rotary drills

Mud rotary drills (Fig. 42a,b) are typically used for water wells and groundwater investigations in areas of extremely thick glacial sediments (10–300 m), particularly where the tills are fine-grained and matrix-rich (Andriashek, 2003; Paulen, 2009). A bentonite slurry is used as circulation fluid and is pumped down the centre of the rod, out through the drill bit, and back up the borehole in the annulus between the drill stem and the borehole wall. A carbide-tipped insert or wing bit is commonly used because it provides drill cuttings. Depending on the hardness, a tricone bit is used to drill through boulders or into bedrock, however, the speed of penetration is reduced substantially. Drill cuttings are carried up to the surface as a continuous slurry of cuttings and drilling mud. The tricone returns <1 cm diameter chips and mud.

Cuttings of the sediments are collected over 1.5 m (5 ft) intervals (Fig. 42c). Compacted samples (i.e. clay, till) are collected using screens and loose sediments, such as sand and gravel, are collected in buckets. Continuous sampling from the surface collar allows for collection of large representative samples (>10 kg) for indicator mineral studies.

Recovery is generally very good for all sediment types with loose, unconsolidated sediments having the poorest recovery. The quality of the sample and strati-

graphic interpretation is variable depending on the drilling conditions, which can affect the return rate as well as the size and abundance of the cuttings.

Mud rotary drilling may also be used to core sediments for sampling. The process uses a diamond-surfaced or tungsten-carbide core bit with a locked split-core barrel that is attached with a wireline core retrieving system. The drill stem acts as a casing and the core is retrieved in 3 m (10 feet) segments (Fig. 42b). Recovery is excellent in clay, silt, and fine-grained tills but due to the circulating water and mud during the drilling process, recovery is poor in sand and gravel. Core size is commonly 76 mm (3 inch) diameter, but can be up to 152 mm (6 inch) diameter.

A variation of mud rotary drilling employed by the Ontario Geological Survey (e.g. Marich et al., 2017) collects core in 5 foot increments (instead of 10 foot) and obtains PQ-size (85 mm diameter) core using a core barrel that does not split. Instead, core is extruded by gravity and slides vertically out of the core barrel onto split PVC tubing. They use a modified Christiansen core barrel retrievable by wireline. This drilling method is well suited to highly consolidated tills, fine-grained sediments (silt and clay), and sand. Stony sediments such as cobble-boulder gravel and loose, stony diamicton often result in poor recovery. Compared to rotasonic drilling (described below), this method preserves sedimentary structures well. Drilling rates are, however, much slower than rotasonic, with a good drilling day returning approximately 30 to 40 m of core (A. Bajc, pers. comm., 2019).

Down-hole geophysical logs, which measure single-point resistance and spontaneous potential (SP) of the materials, are helpful for the identification and correlation of lithological units.

6.8.8. Rotasonic drills

Rotasonic drilling (Fig. 43a) is an optimal drilling method for areas of extremely thick glacial sediments (10 to >125 m) where natural exposures are rare or absent, and where till is stony and bouldery. It is used instead of reverse circulation drilling when more detailed stratigraphic information is needed or concerns about loss of the fine matrix fraction of the till are significant. Some Canadian examples of deep till sampling using rotasonic drilling include those conducted in the Abitibi Greenstone Belt (e.g. Averill et al., 1986; Smith, 1990; McClenaghan 1994; McClenaghan et al.,1996, 1998, 1999a,b, 2001), the Rainy River greenstone belt (Bajc, 1991; Averill, 2013), and New Brunswick (Lamothe, 1990, 1992).

Rotasonic drilling (Fig. 43b) uses a combination of high frequency resonant vibration and rotation to advance a hollow drill bit with tungsten carbide buttons (Fig. 43c) through glacial sediments, boulders, and

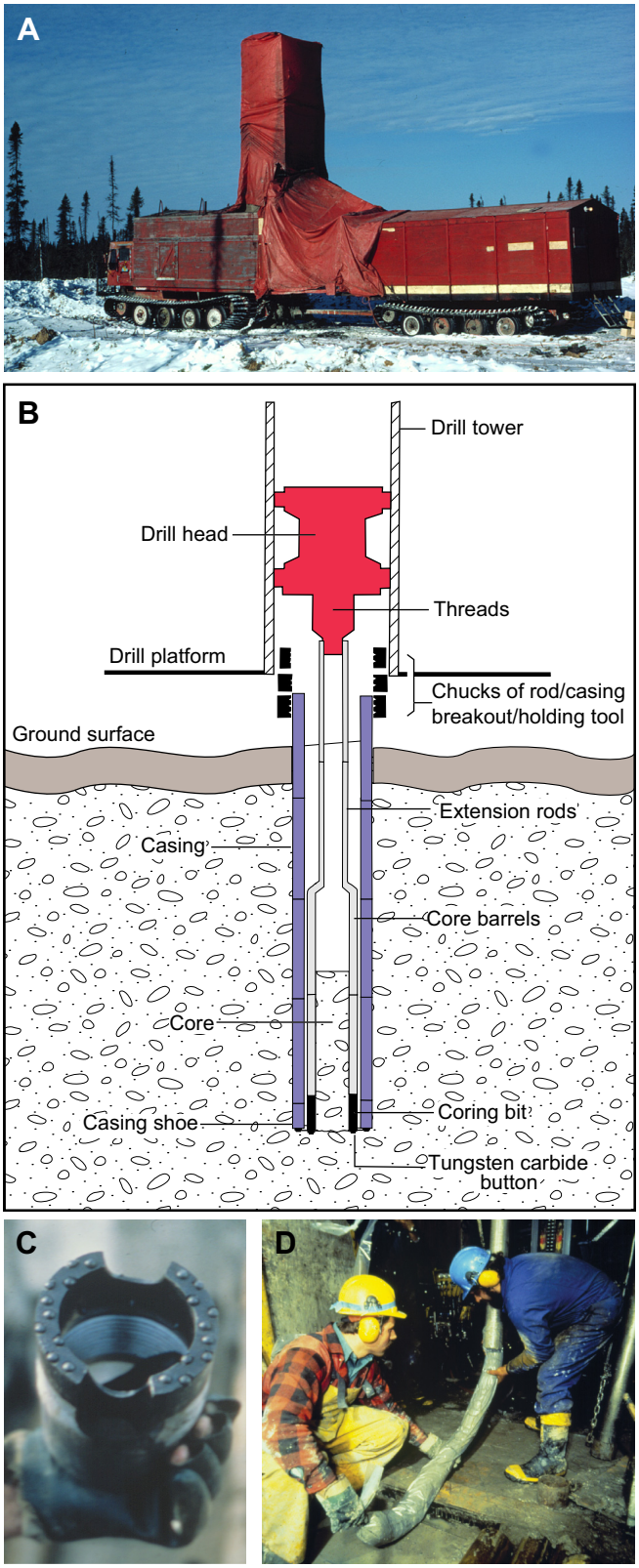

Figure 43. Photographs of a large rotasonic drill used to collected continuous core from thick overburden sequences: **a)** track-mounted drill mounted for winter drilling; **b)** schematic cross-section of a rotasonic drill; **c)** drill bit with tungsten carbide buttons; and **d)** continuous core being extruded from a core barrel into a plastic sleeve (*modified from* McMartin and McClenaghan, 2001).

bedrock with minimal compaction or disturbance to recover a continuous core 9 cm in diameter (Averill et al., 1986; McMartin and McClenaghan, 2001). Casing is used to prevent collapse of the borehole when the rods and core barrels are pulled out of the hole to retrieve the core. The core is vibrated from the core barrels into plastic sleeves (Fig. 43d), usually in 1.5 m increments, and then the rods and core barrels are put back down the hole for addition drilling.

The major advantage of a rotasonic drill over a reverse circulation drill for collecting till samples is that it recovers high-quality drill core. However, in gravelly units, recovery is variable. One disadvantage of this method over reverse circulation drilling is the higher cost. On an hourly basis, rotasonic and reverse circulation drilling costs are approximately equal, however, the need to use casings, and the requirement to pull core barrels and rods to recover core and re-enter the hole significantly reduces the productivity of rotasonic drilling.

6.8.9. Shipping till samples from the field

When shipping samples to the GSC Sedimentology Laboratory or a commercial laboratory for preparation for geochemical analysis, ensure that every sample pail or other shipping container that holds samples is clearly labelled with the name and address of the receiving laboratory and the name and address of the sending geologist. Include a hard copy of the sample list with the shipment and simultaneously send a digital copy of the sample list to the laboratory. If using metal pails, it is recommended to line the pail with a 4 mil plastic bag to avoid wet samples corroding the inside of the pail during transit or storage. Prior to shipment, photographs of the sample pails with the sample numbers visible may be taken to ensure traceability.

7. QUALITY ASSURANCE/ QUALITY CONTROL

Geochemical surveys include many stages and it is important to control the quality at each stage (Garrett, 1969, 1983; Fletcher, 1981; Thomson, 1983; Gustavsson, 1992; McCurdy and Garrett, 2016). Having a well thought out quality assurance/quality control (QA/QC) program for all steps, from sample collection and analysis to data management and archiving, ensures that no systematic bias is introduced into the project. The data can then be relied upon to be consistent for comparison with other data sets. A robust QA/QC program also saves time and money as it eliminates the need to repeat sampling and/or analyses.

7.1. Quality control in the field

A significant source of variability in till geochemical or mineralogical data can be due to poorly defined field protocols. Some basic sampling and recording procedures can be followed in the field to reduce the possibility of geochemical data variability and to ensure that the sample is well documented and not contaminated. Refer to Section 6.7 for sampling guidelines to follow in the field.

A means of monitoring quality control in the field is the collection of a field duplicates, which are collected from a second hole or from sediments exposed up to 5 m away from the original site. The amount collected for a geochemical sample should be two times larger (6 kg) than the normal sample size (3 kg) because it will be split to create a laboratory duplicate after the entire field duplicate sample has been prepared in the laboratory. The field duplicate sample must not be homogenized with the original sample. The original and field duplicate samples are collected into separate bags and labelled with different sample numbers. A number consecutive to the original sample can be given to the field duplicate sample.

7.2. Quality control in the laboratory

7.2.1. Precision and accuracy

The GSC has always been responsible for publishing large volumes of geochemical data and for ensuring their quality. Precision and accuracy are used to describe the data quality. It is required that the data be both precise and accurate, however, one does not automatically imply the other. For example, it is possible for data to be precise, but inaccurate.

Accuracy reflects how close the analytical result is to the true value. Accuracy is evaluated by analyzing certified reference materials or internal reference standards that have been inserted among the routine samples sent to the analytical laboratory and comparing the results with accepted average reference values for these standard materials.

The choice of reference materials to be used depends on the anticipated concentration levels. For example, is your survey area highly mineralized? Are you aware of what metalliferous commodities might be present in the local bedrock? Whenever possible, the commodities of interest in the survey area should be above background concentrations in the reference materials chosen. Whenever possible, the matrix of reference samples should match the survey samples, i.e., use till standards when analyzing till samples. For a detailed explanation of methods for evaluating the accuracy of GSC data, refer to McCurdy and Garrett (2016).

Precision is a measure of the ability of an analytical method to reproduce the same value each time a sample is analyzed. Precision can be evaluated by analyzing duplicate samples prepared from the same field

sample (Garrett, 1991). Precision can also be estimated from replicate analyses of the same sample. For a detailed explanation of methods for evaluating precision of GSC data, refer to McCurdy and Garrett (2016).

7.2.2. Types of samples used for quality assurance/quality control

The GSC uses a sample collection scheme based on blocks of 20 samples (e.g. Friske and Hornbrook, 1991) for monitoring till geochemical analyses. In each block of 20 samples, there should be one field duplicate, one certified reference material or internal reference standard, and one laboratory duplicate. Occasionally, a block of 20 samples will also include a blank sample. Definitions of the various QA/QC sample types are explained below and are modified from McCurdy and Garrett (2016):

Routine sample: A routine sample is a sample of material (lake sediment, stream silt, soil, till, etc.) collected at a specific location for the purpose of a scientific investigation, from which physical characteristics (weight, colour, grain size, etc.) and geochemical data (concentration of elements, oxides, organic material or some other chemical constituent) are determined by observation and analysis.

Field duplicate: A field duplicate sample is a second sample taken at or within a few metres of a routine site, depending on the scale of the survey, and is collected to test sample site variability. It is prepared and analyzed using the same methods as the routine sample.

For the 3 kg geochemical sample, the mass of the field duplicate (second sample of the field duplicate pair) collected should be 2x larger than the original first sample of the duplicate pair because it will be subsequently split in half to make a laboratory duplicate during sample preparation.

Laboratory duplicate: A laboratory duplicate sample is produced by splitting the field duplicate after the field duplicate sample has been prepared for geochemical analysis but before the sample is submitted for geochemical analysis. It is analyzed using the same methods as the routine samples. This duplicate allows for a more powerful QA/QC analysis using unbalanced analysis of variance (UANOVA; *see* Garrett, 1983, 2013a). The laboratory duplicate should only be created from a routine sample if there is insufficient material collected for the field duplicate. A laboratory duplicate was formerly termed a blind duplicate in Spirito et al. (2011).

Certified reference material: A certified reference material (CRM) is used to monitor analytical accuracy. One or more of its properties has been certified by a technically valid procedure accompanied by, or traceable to, a certificate or other documentation issued by a certifying body (Horowitz, 1991). CRMs are homogeneous materials and have been analyzed by a large number of laboratories using various techniques. Section 7.2.4 discusses the CRMs used by the GSC for till geochemistry.

Table 4. Composition of silicic acid that is used as a 'blank' in till sample batches submitted for geochemical analyses, as reported by the supplier.

Assay (as SiO_2)	100%
Non-volatile with HF	0.07%
Chloride (Cl)	<0.01%
Sulphate (SO_4)	<0.005%
Heavy Minerals (as Pb)	<0.002%
Iron (Fe)	<0.003%
Loss on Ignition (as H_2O)	12%

Internal reference standard: A standard is used to monitor analytical accuracy and the geochemical values have been established as being suitable by analyzing multiple times along with CRMs and the CRM results agree with published CRM values. The standard should be prepared and analyzed in the same manner as the sample material from the survey (Garrett, 1991).

Blank: A blank is material that is inserted into the batch to monitor cross-contamination between sample batches and between samples within a batch and usually has very low concentrations of the key elements of interest. Not only does this help measure contamination that may have occurred during sample preparation it will also help purge preparation equipment between metal-rich samples. However, these blanks should not be considered as a substitute for a reliable cleaning procedure. Section 7.2.3 discusses the blank material used by the GSC for till geochemistry.

7.2.3. Silicic acid blank

The GSC uses silicic acid as blank material to monitor cross-contamination between sample batches and between samples within a batch that may have occurred during sample preparation. The blank material consists of silicic acid n-hydrate powder (not sand) and is available from J.T. Baker (Fisher Scientific) in 10 L (2.5 gallon) pails. Its composition is listed in Table 4. Each pail of silicic acid has a unique identification number that is assigned by the supplier and the GSC Sedimentology Laboratory records and tracks this number.

Once a sample preparation order has been established, based on the suspected metal content of the samples, the number of silicic acid blanks to be inserted can be determined. It is recommended that a minimum of three blank samples should be inserted within a sample batch: one at the beginning, one in the middle, and one near the end. The more metal-rich samples there are in a batch, the more silicic acid

blanks should be inserted throughout the batch. Silicic acid blanks are provided and inserted into a sample batch by the GSC Sedimentology Laboratory prior to sieving the sample batch.

Approximately 400 g of silicic acid is sieved to create one 64 g laboratory preparation blank. If a contamination problem is suspected, this amount will allow for replicate analyses by methods requiring up to a 30 g aliquot. The amount needed for an analytical aliquot (1–30 g) is then placed in a sample container and labelled using the sample numbering scheme being used for the routine samples. Analytical results for each silicic acid blank are reported to the project scientist by the GSC Sedimentology Laboratory as part of their routine QA/QC report. Silicic acid blanks can be obtained from the GSC Sedimentology Laboratory if using a commercial laboratory for till sample preparation.

7.2.4. Certified reference materials

A suitable certified reference material (CRM; e.g., Horowitz, 1991) is one whose grain-size characteristics and possible mineralogical composition of the matrix is similar to the tills being analyzed. Certified reference materials are included with real ('routine') samples submitted for geochemical analyses to estimate the accuracy of laboratory results. Although insufficient data are available for certification under International Organization for Standardization (ISO) guidelines, provisional elemental values with standard deviations for CANMET till CRMs have been published by CANMET (*see* websites listed below) with additional data published by Burnham and Schweyer (2004). Analytical data from CRMs in a sample batch are compared with accepted or provisional results to estimate the accuracy of the batch samples, and to monitor drift and systematic changes in determinations with time.

The following certified reference materials are used by the GSC to monitor analytical accuracy for till geochemical analyses. One of these materials should be inserted in every block of 20 till samples.

TILL-1 is a CANMET certified reference standard (Lynch, 1996). It consists of till that was collected 25 km northwest of Lanark, Ontario and contains low levels of metallic elements. For a listing of recommended values, *see* http://www.nrcan.gc.ca/mms-smm/tect-tech/ccrmp/cer-cer/till-1-4-eng.pdf.

TILL-4 is a CANMET certified reference standard (Lynch, 1996). It consists of till that was collected near the Sisson W-Mo deposit (McClenaghan et al., 2016) in New Brunswick. For a listing of recommended values, *see* http://www.nrcan.gc.ca/mms-smm/tect-tech/ccrmp/cer-cer/till-1-4-eng.pdf.

OREAS-46 is a certified reference standard. It consists of till that was collected near Chibougamau, Quebec and its composition reflects the composition of the local Archean metavolcanic and intrusive rocks. For a listing of recommended values, *see* http://www.ore.com.au/crm/oreas-46.

OREAS-47 is a certified reference standard. It consists of till that was collected near Chibougamau, Quebec and its base composition reflects the composition of the local Archean metavolcanic and intrusive rocks. It was augmented with minor amounts of various ore (PGE, REE, Li) and concentrates (base metals) to give it higher metal levels. For a listing of recommended values, *see* http://www.ore.com.au/crm/oreas-47.

7.2.5. Sample insertion and numbering for quality assurance/quality control

Considerations for inserting QA/QC samples into a GSC till geochemistry sample batch and the sample numbering scheme are listed below:

1. As noted in Section 6.6, using the GanFeld system allows for extra sample numbers to be added at a field station, so it is not as critical to reserve sample numbers when in the field.
2. If the numbering scheme does not allow for easy insertion of samples in the numerical suite after returning from the field, two sample numbers should be left free per block of 20 samples while geologists are in the field. This allows for a laboratory duplicate and a CRM or standard to be inserted (Friske and Hornbrook, 1991).
3. The first sample number in the block of twenty is used for the laboratory duplicate. The result is sample numbers 1, 21, 41, etc. are left unused when geologists are in the field.
4. A second number in the block of 20 is reserved for the CRM or internal reference standard. Randomize the position of the CRM or internal reference standard between blocks so that it falls in a different location in each block of 20. It is important that the standards are not grouped together, for example, at the end of the batch being submitted to the analytical laboratory.
5. A few sample numbers throughout the batch should be reserved for the insertion of blanks by the preparation laboratory. This may result in <17 routine samples in some blocks of 20 samples. At a minimum, blanks should be inserted at the beginning, middle, and end of each batch. The silicic acid blanks are inserted more frequently if many of the samples are suspected to be metal-rich, i.e., every ~25 samples.

8. PREPARATION OF TILL SAMPLES FOR GEOCHEMICAL ANALYSES

8.1. Procedures for submitting till samples

8.1.1. Using the GSC Sedimentology Laboratory for sample preparation

The services of the in-house GSC Sedimentology Laboratory, based in Ottawa, are available to all GSC scientists. This laboratory provides sample receiving, archiving, and preparation services, in-house analyses, arranging of and shipping for commercial laboratory analyses, and QA/QC reporting. The following procedure is used to access the services of the Sedimentology Laboratory:

1. Enter the sample data into the GSC's digital Sample Management System (SMS).
2. Fill out a GSC Sedimentology Laboratory requisition form; check "Archive" on the requisition to ensure that an archive split is taken before the samples are prepared.
3. Flag the field duplicates on the request form so laboratory personnel can use the samples to prepare laboratory duplicates; communicate to the laboratory how the numbering of the QA/QC samples should be handled.
4. The GSC Sedimentology Laboratory will insert silicic acid blanks into the batch prior to sample preparation.
5. Always indicate a preparation order to the GSC Sedimentology Laboratory and the laboratory will keep track of this order—the default preparation is numerical order. If the samples are suspected to be metal-rich, order the samples from least to most metal-rich to minimize the possibility of cross-contamination during preparation.
6. Samples must be physically prepared, as indicated in the Section 8.2.
7. Before submitting samples to the analytical laboratory, the GSC Sedimentology Laboratory will insert certified reference materials or internal reference standards and laboratory duplicates.
8. When the remainder pulps are returned to the GSC Sedimentology Laboratory from the analytical laboratory, they will be archived at the GSC's collections facility and reported as such in the SMS database.

8.1.2. Using an external laboratory for sample preparation

Using an external sample preparation laboratory is recommended only if the GSC Sedimentology Laboratory is unable to process your samples in a timely manner. The GSC Sedimentology Laboratory will communicate the specific preparation instructions to the commercial laboratory based on the information that is provided to them when the samples are submitted:

- All steps and instructions listed above in Section 8.1.1 will be followed when using a commercial laboratory for sample preparation.
- After the samples are prepared, they are to be shipped back to the GSC Sedimentology Laboratory for insertion of QA/QC samples prior to geochemical analysis.
- Consult with the staff of the GSC Sedimentology Laboratory for an estimated timeframe for sample preparation, and only use a commercial laboratory for sample preparation if a backlog at the GSC laboratory will severely hinder delivery of results.
- For samples collected as part of an orientation survey (e.g. near a known mineral deposit), preparation should be carried out only at the GSC Sedimentology Laboratory. This allows for more control over the preparation stage, minimizing the possibility of cross-contamination.
- If some samples are suspected to be metal-rich, specify the processing order to the laboratory preparing the samples, with the most metal-rich samples processed last. This order will minimize the possibility of cross-contamination.
- If no processing order is specified, the default processing order is numerical order.

8.2. Procedures for commercial geochemical analyses with sample preparation completed at the GSC

Sample preparation protocols for the archiving and geochemical analysis of a 3 kg till sample are detailed below and summarized in Figure 44.

8.2.1. Dry the sample

- Entire sample is to be air dried in a metal pan at <40°C in a room or cupboard dedicated to only sample drying.
- Freeze dry samples with high clay content if clay sticks to the sand-sized grains after drying.

8.2.2. Archive the split

- After the sample has been dried, split off 800 g or the equivalent for filling a fixed-size (1 pint / 470 ml) archive container of unprocessed and dried sample material for archiving.
- Label the container with a unique sample number and bar code.
- Store the archive sample at the GSC Ottawa collections facility, which provides an environment that is cool and dark (i.e. out of direct sunlight).

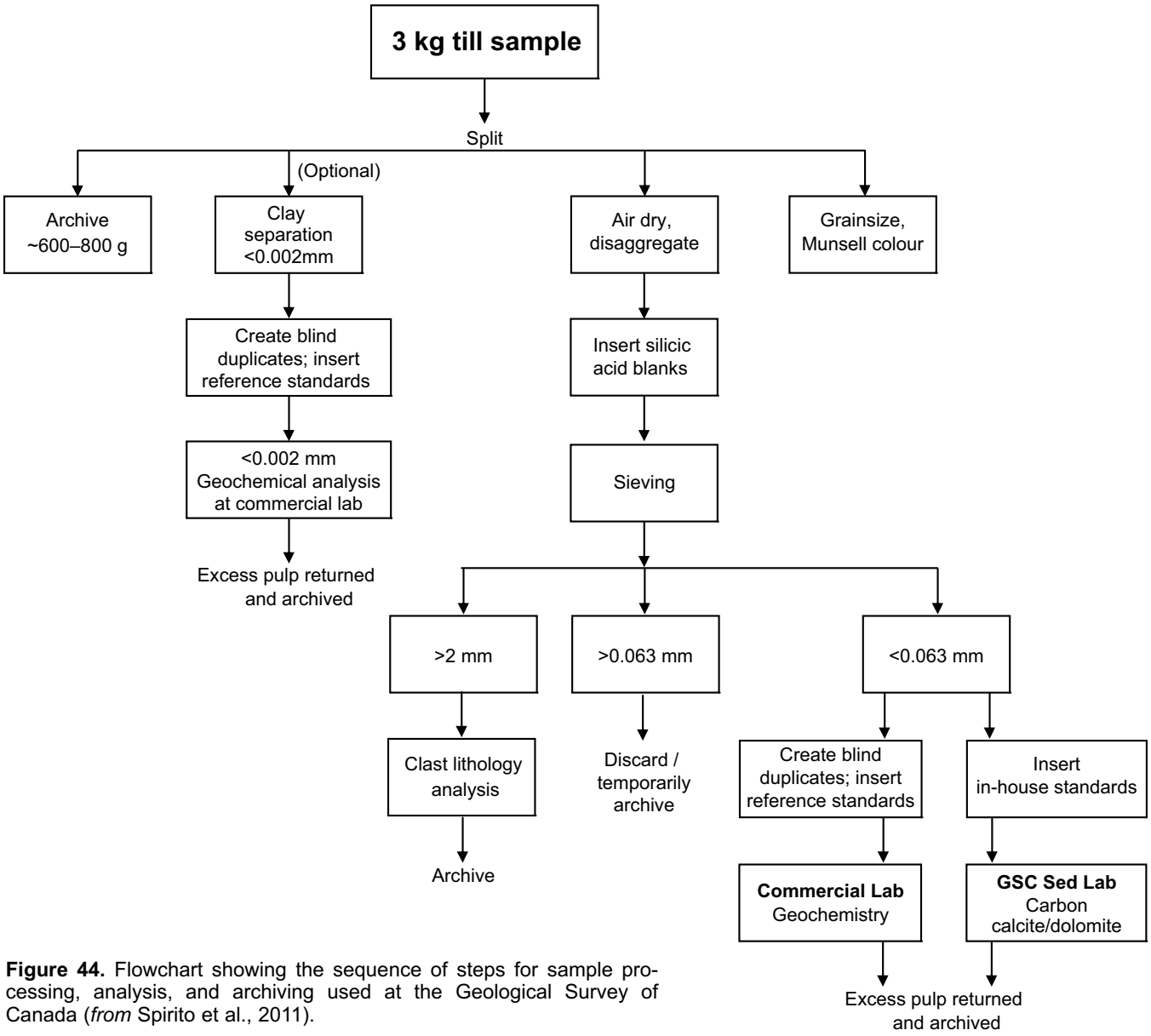

Figure 44. Flowchart showing the sequence of steps for sample processing, analysis, and archiving used at the Geological Survey of Canada (*from* Spirito et al., 2011).

8.2.3. Process the sample

- If required, disaggregate the dried sample in agate (not porcelain) mortar and pestle; alternatively, the sample (within the original sample bag) can be placed in a clean plastic bag and hit with a rubber mallet until the samples is disaggregated; a new clean outside bag is required for each sample.
- Prepare a laboratory duplicate in the following manner: sieve the entire field duplicate and then split into two subsamples of equal size, which will become the field and laboratory duplicates. Number samples as per instructions in Section 7.2.5.
- To monitor possible cross-contamination during sample preparation, silicic acid blank samples should be added to a sample batch prior to sieving. Section 7.2.3 describes what this material is and its purpose. Silicic acid blank samples are inserted by the GSC Sedimentology Laboratory at regular intervals within the batch of routine samples and sieved using the same equipment as the routine samples.
- If you choose to randomize and renumber the samples after sample preparation and before geochemical analysis, ensure that the blanks are interspersed within the batch after randomizing.

8.2.4. Sieve to -250 mesh (<0.063 mm)

The -250 mesh (<0.063 mm) fraction (silt + clay) is the most commonly used size fraction for till geochemical analysis for provenance studies and mineral explo-

ration (e.g. McEachern and Stea, 1985; Saarnisto and Taipale, 1985; Pronk and Burton, 1988; Batterson, 1989; Smith, 1990; Koljonen et al., 1992; Plouffe and Ballantyne, 1993; McClenaghan, 1994; Edén and Björklund, 1995; Tarvainen, 1995; Friske et al., 2001; Woodruff et al., 2004; McClenaghan et al., 2011; Paulen et al., 2011) for a number of reasons:

- sulphide and some precious metal minerals are easily comminuted to the <0.063 mm fraction over short distances and thus are enriched in this size fraction (Shilts, 1975, 1995, 1996; Coker and DiLabio, 1989; Nevalainen, 1989);
- the <0.063 mm fraction contains abundant phyllosilicates that will scavenge cations released during weathering (Shilts, 1993,1995);
- quartz and feldspar can comprise up to 95% of all grains in the fine sand-sized fraction (0.063–0.125 mm) of till (Shilts, 1977), and thus removing this fine sand-sized material avoids the dilution of the geochemical signal;
- many studies have shown that gold is most abundant in the silt-sized (<0.063 mm) fraction (e.g. DiLabio, 1982; Shelp and Nichol, 1987; Tarvainen, 1995; Laurus and Fletcher, 1999), therefore, including coarser material that is >0.063 mm will dilute the gold signal;
- the <0.063 mm fraction can be prepared rapidly and inexpensively for analysis (Lett, 1995; Levson, 2001a,b) because this fraction can be easily prepared by dry sieving;
- a significantly smaller mass (200 g) needs to be processed to recover the <0.063 mm fraction compared to the larger mass (1000 g) that is required to generate sufficient material for analysis of the <0.002 mm fraction.

For all sieving, use stainless steel sieves that are solderless or that have an epoxy or silicon seal covering solder on both the upper and underside edges of the screen. Do not use any sieve with exposed solder because solder, which is typically a soft alloy, can be a source of metal contamination. Sieve the sample into a stainless steel pan, not onto paper or a plastic sheet. Transfer the sieved material into a sample envelope or polypropylene vial.

Between each sample, the sieve and pan are to be cleaned with a brush and air hose, wiped clean with a low-lint precision wipe moistened with distilled water, and then inspected to ensure no grains are stuck in the sieve. The sieve is to be cleaned after every 10th sample within a batch using an ultrasonic cleaner and before the first sample of a new batch. Testing conducted by the Sedimentology Laboratory on sieving of metal-rich till has demonstrated these procedures are sufficient to clean the sieves (Grenier et al., 2015).

Between sample batches, all sieve(s) being used to sieve the till samples in a batch are to be cleaned in an ultrasonic cleaner before the first sample of a new batch.

In order to avoid the artificial concentration of gold, the entire sample should be sieved to completion because gold grains (due to their high density) will preferentially pass through the sieve mesh at the beginning of the sieving process.

Each field duplicate sample should be sieved to completion. This will ensure that there is sufficient material to create a laboratory duplicate from the field duplicate.

To monitor contamination from previously processed samples, the first sample in a batch should be a silicic acid blank prepared in the following manner: 400 g of silicic acid is sieved to obtain a minimum of 62 g of <0.063 mm material. This provides sufficient material should a replicate analysis be required if contamination from a previous sample batch is suspected.

For routine samples, sieve sufficient sample material (~2 kg if the till is derived from the Canadian Shield) to generate at least 62 g of <0.063 mm fraction material (2 x 30 g aliquots plus 2 x 1 g aliquots). This 62 g portion allows replicate analyses if required and archiving of <0.063 mm material for future reanalysis.

8.2.5. Sieve to -80 mesh (<0.180 mm)

The -80 mesh (<0.0180 mm) fraction (clay+silt+fine sand) has been used for exploration-focussed geochemical analysis of soils and stream sediments since the 1930s and 1940s (Garrett, 2019). The -80 mesh fraction was used in the early years (1960s–1970s) of till geochemistry as a direct adaptation of soil geochemical methods recommended by Bloom and Crowe (1953) and others. Some of the earliest reports of the use of -80 mesh fraction for till geochemistry were made by Bolviken (1967), Garrett (1969), and Mehrtens et al. (1972).

Since these very early days of till geochemistry, numerous till geochemical studies have demonstrated that the <0.063 mm (-250 mesh) is the optimal size fraction for till geochemical analysis because it generally contains higher gold and base metal contents than the coarser size fractions, avoids the dilution effect from sand-sized quartz and feldspar, and is still cost effective to recover.

The price difference for sample preparation at commercial laboratories between sieving of the -80 mesh (<0.180 mm) and -250 mesh (<0.063 mm) fractions is generally between $1.00 and $1.50 per sample. The added cost of preparing the <0.063 mm of till samples is minimal when compared to the cost of the fieldwork to acquire the samples and the overall analytical costs of

the project. For all of these reasons, the -80 mesh fraction is not recommended for GSC regional till surveys.

8.2.6. Separate the clay (<0.002 mm) fraction (optional)

The <0.002 mm fraction (clay-sized material) of till may also be analyzed, although its is more costly and time consuming to recover. Analysis of this finer size fraction is used for specific survey areas or when exploring for specific commodities such as uranium, because of its greater capacity to retain elements released during weathering, and to avoid textural bias in matrix geochemistry (Shilts, 1975, 1995). Disadvantages of using the <0.002 mm fraction include (1) a large mass of material (up to 1 kg) is needed to produce a sufficient mass (1–2 g) of clay-sized material for analysis (Lindsay and Shilts, 1995; Klassen, 2003), and (2) this fraction is most influenced by hydromorphic processes such that it may distort or completely mask the signature of clastic glacial dispersal (Pronk, 1987). Examples of GSC surveys and studies that have analyzed the clay-sized fraction of till include Kettles (1992), Kaszycki et al. (1996), McMartin et al. (1996), McClenaghan et al. (2002), and Plouffe et al. (2011, 2016).

Clay separations should be completed following the procedures outlined in GSC Open File 4823 (Girard et al., 2004). The GSC procedures and considerations are outlined below.

Before separation, samples should be mixed with distilled water or a deflocculant such as sodium hexametaphosphate $(NaPO_3)_6$. If P and Na are specific elements of interest, or if selective extractions are to be carried out (e.g. Plouffe et al., 2001b), then sodium hexametaphosphate should not be used because it will artificially enrich samples with P and Na, and may act as a weak leach for certain metals present in labile phases, resulting in their loss during the clay separation process.

Centrifuge bottles and decanting equipment must be rinsed and cleaned with distilled water between samples.

Check with the GSC Sedimentology Laboratory for the number of times a bottle can be used before a new bottle is required — this may require adjustment. Two sets of bottles are used in the Sedimentology Laboratory, one set only for distilled water and the other set for sodium hexametaphosphate. Bottles are discarded when they no longer come clean. If a bottle becomes stained, it is discarded immediately after the sample has been processed.

The desired aliquot for geochemical analysis is 2 g. To recover this mass, between 500 and 800 g of silty-clay till or 1 to 2 kg of sand-rich till will have to be processed to obtain 2 g. The mass of material to be processed will have to be adjusted based on the clay content of the till.

The entire oversized field duplicate sample should be processed to ensure that there is sufficient material to create a separate 2 g laboratory duplicate from the field duplicate.

Keep track of the order that samples were centrifuged, in order to monitor for possible cross-contamination.

8.3. Procedures for commercial geochemical analyses with sample preparation also carried out at the commercial laboratory

8.3.1. Archive and dry the samples

- Instruct commercial laboratories that the samples are to be inidivually air dried in metal pans at <40°C in a room or cupboard dedicated to drying.
- Instruct commercial laboratories to set aside an 800 g bulk split of each sample prior to preparation and to ship the splits directly to the GSC Sedimentology Laboratory. These splits will be placed in labelled sample containers and archived by the GSC.

8.3.2. Process the samples

If required, disaggregate any dried samples using an agate (not porcelain) mortar and pestle; alternatively, dry samples may be left within their original sample bags and be placed inside a clean plastic bag and hit with a rubber mallet; a new clean outside bag is required for each sample.

For case studies around mineralized sites, arrange field samples in a list from expected metal-poor to most metal-rich, and have the samples sieved in this order to minimize the possibility of cross-contamination.

8.3.3. Use of silicic acid blank

To monitor possible cross-contamination during sample preparation at a commercial laboratory, silicic acid blanks should be used (*see* Section 7.2.3). Unsieved blank material will be provided to the GSC scientist by the GSC Sedimentology Laboratory.

The blank samples should be prepared/sieved by the commercial laboratory using the same equipment as was used for the routine samples.

If the samples are being shipped from the field directly to a commercial laboratory for preparation, there are two options for inserting the silicic acid blanks into your batch:

1. Insert 400 g aliquots of the unsieved blank in the sample batch and label each using the sample numbering scheme already being used; this

requires that the silicic acid has been brought to the field as pre-measured, bagged 400 g aliquots and that sample numbers have been reserved for the blanks. Possible contamination may occur if the silicic acid aliquots are not premeasured in the GSC Sedimentology Laboratory.

or

2. Ship the required number of 400 g aliquots of unsieved silicic acid blanks to the commercial laboratory with instructions about how the blanks are to be inserted into the sample batch and sieved with the routine samples. Before shipping these aliquots to the commercial laboratory, label each aliquot using the sample numbering scheme already being used for the routine samples.

At a minimum, the silicic acid blanks should be inserted at the beginning, middle, and near the end of the sample batch. If the samples are metal-rich, it is advisable to insert blanks more frequently throughout the batch. The decision of how many silicic acid blanks to insert might not be determined until after a scientist returns from the field, therefore it is more difficult to decide how many sample numbers to reserve for blanks while still in the field. In this case, assign the silicic acid blanks sample numbers with dashes so that more than one sample is assigned to a station. It is not critical to hide these samples in the batch because the preparation laboratory will likely recognize them as blanks given their white colour compared to the grey to brown colour of the till. However, it is critical to ensure blanks occur throughout the batch and not all at the end.

8.3.4. Sieving to -250 mesh (<0.063 mm)

For all sample sieving, the commercial laboratory must use stainless steel sieves that are solderless or that have an epoxy or silicon seal covering solder on both the upper and underside edges of the screen. They must not use any sieve with exposed solder; solder, which is typically a soft alloy, can be a source of metal contamination. Samples should be sieved into a stainless steel pan, not onto paper or a plastic sheet. The sieved material should then be transferred into a sample envelope or polypropylene vial.

All sieves to be used for a GSC sample batch need to be cleaned in an ultrasonic cleaner before the first sample is sieved.

Between each sample, the sieve and pan should be cleaned with a brush and air hose. The sieve will need to be cleaned after every 10th sample within a batch using an ultrasonic cleaner.

To monitor contamination from previously processed samples, the first sample in a batch should be a silicic acid blank prepared in the following manner at the commercial laboratory: 400 g of silicic acid is sieved to obtain a minimum of 62 g of <0.063 mm. This provides sufficient material should a replicate analysis be required if contamination from a previous sample batch is suspected.

Request that the commercial laboratory sieve each sample to completion, including the oversized field duplicate samples. This will ensure that there is sufficient material to create a laboratory duplicate from the field duplicate; this large mass will allow for replicate analyses by methods using a 30 g aliquot, if a problem is suspected.

For routine samples, submit sufficient sample material (~2 kg if Shield-derived till) to generate at least 62 g of <0.063 mm fraction material (2 x 30 g aliquots plus 2 x 1 g aliquots). This 62 g portion allows replicate analyses if required and archiving of <0.063 mm material for future reanalysis.

After sieving is completed at the commercial laboratory, the sieved material should be shipped to the GSC Sedimentology Laboratory, where laboratory duplicates will be created from the oversize field duplicate samples and QA/QC samples will be inserted.

The GSC Sedimentology Laboratory will submit the routine samples together with the QA/QC samples for geochemical analysis.

If requested, the GSC Sedimentology Laboratory will renumber and randomize the samples after sample preparation and before geochemical analysis. In doing so, the Sedimentology Laboratory will ensure that the QA/QC samples are not all analyzed in one group, but remain interspersed within the batch.

8.3.5. Separate the clay (<0.002 mm) fraction

It is important to document and understand the methods that the commercial laboratory will use to separate the clay (<0.002 mm) fraction of the till samples, as their methods may be different from those of the GSC methods. Request that the commercial laboratory prepare a minimum of a 2 g aliquot for geochemical analysis.

If the till samples are clay-rich, request that the commercial laboratory process approximately 500 g of sample material. If the till samples are sand-rich, request that the commercial laboratory process 1 to 2 kg of sample material.

Request that the commercial laboratory prepares the entire oversized field duplicate sample, which will ensure that there is sufficient material to create a laboratory duplicate from the field duplicate.

Request that the commercial laboratory rinse and clean centrifuge bottles, tubes and decanting equipment in a sonic bath with distilled water between each

sample. Document and understand the commercial laboratory protocols for replacing the centrifuge bottles and tubes. Request that bottles and tubes that do not come clean after washing in the sonic bath not be used for GSC samples.

Request that the commercial laboratory ship samples back to GSC Sedimentology Laboratory for insertion of reference standards and for the preparation of the laboratory duplicates from the oversized field duplicates.

Request that the commercial laboratory record the order in which the samples were centrifuged, in order to be able to monitor for possible cross-contamination during sample preparation.

9. GEOCHEMICAL AND OTHER ANALYSES OF THE TILL MATRIX

9.1. Recommended minimum requirements for GEM projects

The standardization of analytical protocols and reference standards for all GEM program activities (and other programs) has allowed direct comparison of analytical data sets among projects over the long term. Therefore, the same suite of standards should be used for each batch over multiple years. The analytical methods and protocols suggested and recommended in this section have been used by the GSC as well as other jurisdictions and industry for many years and are well documented in the literature (e.g. Chao, 1984; Chao and Sanzolone, 1992; Kauranne et al., 1992).

Two digestions are recommended as a minimum for analysis of the <0.063 mm till fraction: (1) aqua regia, and (2) borate fusion total digestion.

9.1.1. Aqua regia digestion

Aqua regia (3 HCl + HNO_3) is a partial digestion that is particularly effective for digesting base metals hosted in sulphide minerals and some clay minerals (e.g. Koljonen and Malisa, 1991; Räisänen et al.,1992; Klassen, 2001). As a fairly weak digestion, it highlights the signal from the sulphide, sulphate, and oxide content of the sample without dissolving the more refractory rock forming elements (Gaudino et al., 2007). As such, this digestion is not suitable for digesting minerals such as barite, chromite, gahnite, cassiterite, ilmenite, rutile, sphene, monazite, zircon, and garnet.

The use of aqua regia is recommended because of the long history the GSC has of using this digestion to analyze till in mineral exploration-focused surveys. This digestion is particularly useful in identifying base metal content for mineral exploration or environmental purposes, and the ongoing use of this method allows for direct comparisons of current and previously published data sets from 1) the GSC (e.g. Shilts, 1975; Kettles and Shilts, 1983; Smith, 1990; McClenaghan and DiLabio, 1993; Plouffe and Ballantyne, 1993; Thorleifson and Kristjansson, 1993; Friske et al., 2001; Parkhill and Doiron, 2003; Dredge et al., 2005; McClenaghan et al., 2011; Lett and Rukhlov, 2017); 2) other geological surveys (e.g. Eriksson, 1976; Koljonen et al., 1992; McClenaghan, 1994; Sarala et al., 1998; Bajc and Hall, 2000; Lett, 2001; Barnett, 2007; Ferbey, 2010; Ward et al., 2011), and 3) the mineral exploration industry (e.g. Sinclair 1979; Snow and Coker, 1987; Brereton et al., 1988; Coker et al., 1988).

Method: Digest a 30 g aliquot of <0.063 mm till matrix material in aqua regia, and analyze by ICP-ES and ICP-MS for 65 elements, including the rare earth element (REE) suite. The addition of REEs to this package was developed by B.A Kjarsgaard (writ. comm., 2009) and Kjarsgaard et al. (2013a,b). Note, this analytical package may also be applied to the <0.002 mm fraction.

Note: An aqua regia digestion involves a mixture of 3 parts hydrochloric (HCl) and 1 part nitric (HNO_3) acids (Hall, 1991). Some commercial laboratories reporting an aqua regia digestion use the same acids but in a 1:1 ratio (modified aqua regia).

9.1.2. Borate fusion total digestion

Borate fusion is a total digestion that determines major and minor elements and trace elements, and is useful for identifying signatures related to specific bedrock lithologies at both the local and regional scale (Koljonen et al., 1992). It allows the determination of the elemental abundances present in mineral phases not dissolved in aqua regia. It is effective for characterizing total element abundances that are related to bedrock lithology and the proportion of the major rock-forming minerals present in the sample. This method is ideal for determining the total concentrations of REEs and other high field strength elements.

Determining total element concentrations in till and examining till clasts have been used for more than 30 years to identify provenance from specific bedrock lithologies (e.g. Broster, 1986; Taipale et al., 1986; Graves et al., 1988; Lahtinen et al., 1993; Tarvainen, 1995; Tarvainen et al., 1996; Stea and Pe Piper,1999; Plouffe et al., 2006; Lett, 2008; Lett and Rukhlov, 2017) and in some cases to map the composition of the underlying bedrock in areas where till cover is thick and bedrock cannot be accessed. Abundances of important pathfinder elements that are hosted in mineral phases that are not dissolved by an aqua regia digestion are best determined by 'total' methods; these include barite (Ba), chromite (Cr: McClenaghan et al., 2011), scheelite (W: McClenaghan et al., 2014), and cassiterite (Sn: McClenaghan et al., 2016). Total digestion methods are useful for determining REE contents in till, which can then be compared to patterns character-

istic of kimberlite (e.g. McClenaghan et al., 2002, 2004; McClenaghan and Kjarsgaard, 2007), carbonatite, or peralkaline granite (B. Kjarsgaard, pers. comm., 2010). Its use by GSC scientists and others has been increasing since the late 1990s (e.g. Bobrowsky et al., 1997; Stea and Pe Piper, 1999; McClenaghan et al., 2002, 2004, 2011, 2017a; McMartin et al., 2003; Plouffe et al., 2006; Kjarsgaard et al., 2013a, 2014a,b).

Method: Fuse a 0.2 g aliquot of <0.063 mm till matrix material with lithium metaborate/tetraborate, digest in nitric acid, and determine major oxides by ICP-ES and trace elements by ICP-MS. This method has higher detection limits for some elements compared to 4-acid digestion. The addition of Cu, Mo, Ni, Pb, Sc, and Zn by ICP-MS to this analytical package was developed by B.A. Kjarsgaard (writ. comm., 2009) and Kjarsgaard et al. (2013a,b). Note, this analytical package may also be applied to the <0.002 mm fraction.

9.2. Additional geochemical methods

9.2.1. 4-acid digestion

Another near total digestion is 4-acid digestion and has been used by the GSC and other geological surveys since the late 1990s (e.g. Parkhill and Doiron, 2003) to provide insights into the lithological provenance of till. For example, the Geological Survey of Finland used total (4-acid) digestion of till to map the composition of bedrock at both local and regional scales as part of the Geochemical Atlas of Finland (Nikkarinen et al., 1984; Koljonen et al., 1992). The Geological Survey of Newfoundland and Labrador, in eastern Canada, uses 4-acid digestion for their regional till geochemical surveys (S. Amor, pers. comm. 2011). This digestion is used for Cu, Ni, Co, Pb, Zn, Mo, and Ag assays. Note, this analytical package may also be applied to the <0.002 mm fraction.

This method is not suitable for determining total abundance of rare earth elements. The digestion is considered to only partially digest some Cr and Ba minerals as well as oxides of Al, Fe, Hf, Mn, Sn, Ta, and Zr. Volatilization during fuming may result in some loss of As, Sb, and Au.

Method: Digest a 0.25 g aliquot of <0.063 mm till matrix material using four acids (HF-$HClO_4$-HNO_3-HCl) and determine concentrations using ICP-ES and ICP-MS for 55 elements, including REEs. The addition of REE to this analytical package was developed by B.A. Kjarsgaard (writ. comm., 2009) and Kjarsgaard et al. (2013a,b).

9.2.2. Fire assay determination of Au, Pt, and Pd

Lead fire assay is an additional total analysis that is used to determine the total concentration of Au, Pt, and Pd in till samples from areas with potential to host gold or magmatic Ni-Cu-PGE deposits. The method is described in more detail by Hall et al. (1989) and Hall and Oates (2003). Note, this analytical package may also be applied to the <0.002 mm fraction.

Method: Use a 30 g aliquot of <0.063 mm till matrix material to determine the total content of Au, Pt, and Pd. Fire the sample + PbO flux mixture, separate the lead button from the slag, and cupel the button to isolate the Ag doré bead, which will contain the Au, Pt, and Pd. Dissolve the doré bead in HNO_3 and determine Au, Pt, and Pd by ICP-MS.

9.2.3. Instrumental neutron activation analysis for non-destructive analysis of the <0.25 mm heavy mineral concentrate

Instrumental Neutron Activation Analysis (INAA) is a simultaneous, multi-element, total, automated technique that does not require digestion and provides good precision and accuracy as determined by measurements of standard materials. The non-destructive nature of INAA makes it advantageous for samples requiring further analyses or mineralogical examination. For example, heavy mineral concentrates analyzed by INAA can be later examined for specific minerals. A cooling period is required for the reduction of radioactivity emitted from the samples prior to their release from the analytical laboratory. Note, this analytical package may also be applied to the <0.002 mm fraction.

Method: Use a 30 g aliquot of <0.063 mm till matrix material to determine total concentrations of major, minor, and trace elements. A sample is weighed and encapsulated for irradiation at the analytical laboratory. Samples are irradiated together with neutron flux monitors in a pool-type reactor. After a seven-day decay period, samples are measured with a high-resolution germanium detector. Typical counting time is 500 seconds (McCurdy et al., 2019). Note detection limits are elevated compared to the methods described for aqua regia digestion (*see* Section 9.1.1) and for borate fusion total digestion (*see* Section 9.1.2).

9.3. Archiving the analytical remainders

The return of pulps (i.e. extra material after geochemical analyses) from a commercial analytical laboratory must be requested on a Sedimentology Laboratory requisition form. Archiving of the pulps at the GSC's collections facility is done automatically if samples are prepared by the GSC Sedimentology Laboratory. Archiving of pulps must be specifically requested if sample preparation is done by an outside laboratory. Archiving of pulps greatly facilitates future reanalysis of samples if new analytical methods become available and if new commodities are sought.

9.4. Portable X-ray fluorescence analysis

Portable X-ray fluorescence (pXRF) spectrometry can be used to determine metal contents of till in the field and/or in the laboratory using hand-held (Fig. 45a) or bench-top (Fig. 45b) equipment. The method can be used to detect anomalies and actively guide till sampling and follow-up while still in the field (Arne et al., 2014). In the laboratory, pXRF analysis can be used to order samples prior to submitting them for conventional laboratory-based geochemical or mineralogical analysis. Several recent studies report the advantages and disadvantages of applying pXRF analysis to dry versus moist till, unsieved versus sieved till, and making determinations through plastic sandwich bags if nothing else is available (Peter et al., 2010; Hall and McClenaghan, 2013; Plourde et al., 2013; Kjarsgaard et al., 2014a,b; Sarala et al., 2015b; Hall et al., 2016; Sarala, 2016). Knight et al. (2013) and Rukholov (2013) have tested the operating conditions of, and reference materials for, pXRF analysis of sediment samples.

The following protocols are recommended:

- samples should be air-dried prior to analysis;
- if sample drying in the field is not an option, moist (but not completely wet) samples may be analyzed;

Ideally, 2 to 5 g of pebble-free unsieved sample material is placed into a disposable pXRF cup or plastic vial and then covered with Prolene® film (Fig. 45c). The sample is then ready for analysis. If pXRF cups are not available in the field, then approximately 200 g of sample material can be placed into a new, thin, inexpensive plastic sandwich bag (Fig. 45b). Samples are then analyzed by placing the film-covered cup or sandwich bag in front of or on the flat surface of the pXRF analytical window (Hall et al., 2016).

Reference standards and blanks (described below) should be analyzed along with the routine samples for QA/QC. These QA/QC samples should be prepared in pXRF cups covered by Prolene® film prior to going to the field. The GSC's Inorganic Geochemistry Research Laboratory (IGRL) can provide GSC scientists with a set of prepared pXRF standards and blanks to take to the field.

The median value of at least two readings should be used as the final value for each sample. The sample should be tapped and repositioned in front of the analytical window before taking the second and/or additional readings.

GSC scientists conducting their own pXRF readings in the field, require GSC in-house training on the pXRF instrument before leaving for the field to ensure the instrument is used safely, appropriately, and is optimized for the users' specific matrix/results intent (qualitative/quantitative). The serial number and type of pXRF unit used and date of readings should be recorded with all field readings.

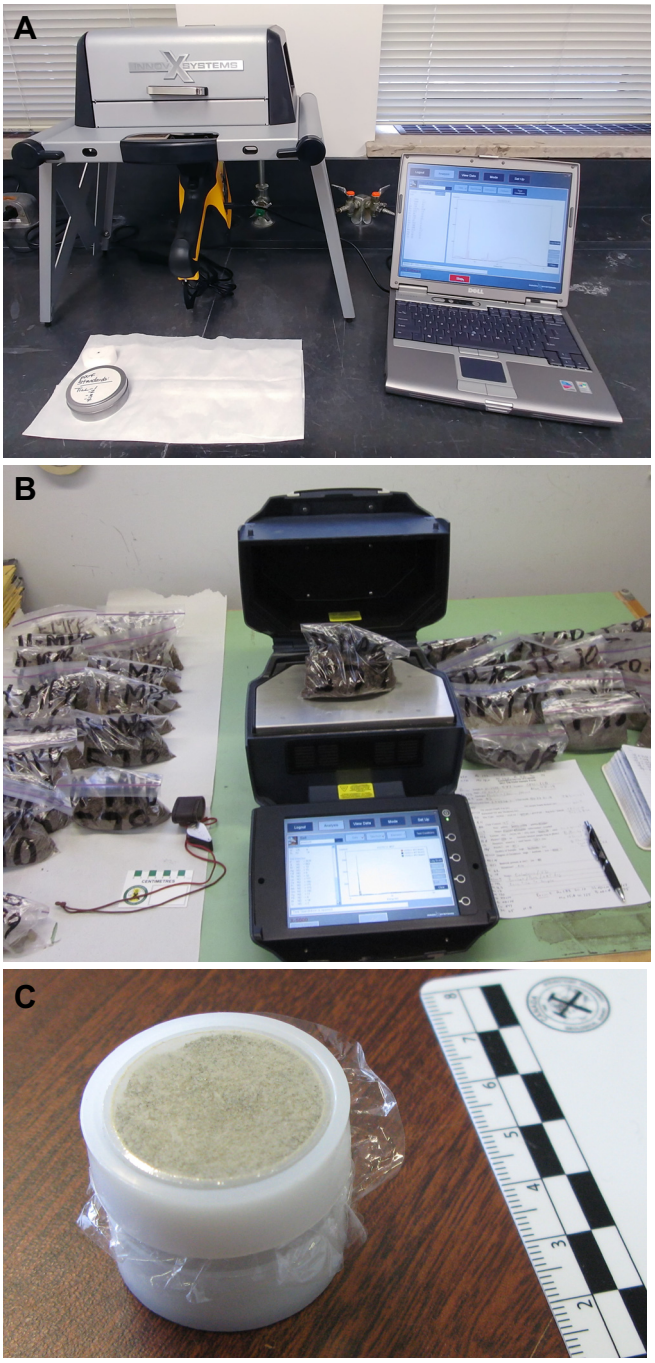

Figure 45. The unsieved fraction of till can be analyzed by portable X-ray fluorescence (XRF) in the field and/or in the laboratory using **(a)** a hand-held model (left side of photograph), or **(b)** a bench-top model. Measurements are made on dry material that is in a pXRF cup **(c)** covered with 4 µm prolene film, or if that is not available, inside of a thin plastic sandwich bag as shown in (b). Photograph (b) courtesy of M. Parkhill.

Safety training and certification required for GSC personnel to safely operate the pXRF unit include the following:

- Users of analytical X-ray equipment should follow Health Canada Safety code 32 and its addendum. This document provides regulatory and user requirements, guidance, and information specific to portable, hand-held, X-ray tube-based open beam XRF devices related to non-destructive testing (NDT) applications. (https://www.canada.ca/en/health-canada/services/environmental-workplace-health/reports-publications/radiation/addendum-safety-code-32-portable-hand-held-tube-based-open-beam-devices.html).
- A person operating a hand-held XRF device in open beam mode must also have a valid NRCan NDT XRF level-1 certification to operate the analyzer (https://www.nrcan.gc.ca/mining-materials/non-destructive-testing/19572).

pXRF instruments are factory calibrated. The Innov-X DP-4000 uses an optimized multibeam "Soil Mode" (lower concentrations) or "Mining Mode" (higher concentrations) factory calibration. Prior to going into the field, the calibrations should be tested for accuracy in the laboratory with certified reference materials (CRMs) or if available, samples of the matrix of interest for the study area. Limitations can be evaluated and custom standardization made to adjust the calibration to the matrix of interest. A series of CRMs and a blank can also be tested in the laboratory and provide part of a good QA/QC protocol.

Complete pXRF testing of till samples is also available from the GSC's Inorganic Geochemistry Research Laboratory (IGRL) via a Laboratory Study Agreement. The GSC's Sedimentology Laboratory will prepare a split for pXRF testing in the IGRL Laboratory after sample drying as described in Section 8.2.1.

Whether samples are analyzed in the field or in a laboratory setting, a separate split of each sample should be submitted for geochemical analysis at a commercial laboratory to monitor the accuracy and precision of the pXRF analyses.

9.5. GSC Sedimentology Laboratory testing

9.5.1 Munsell colour

The Munsell colour of till is determined as part of the assessment of the degree of till sample oxidation and provenance. The GSC Sedimentology Laboratory has developed a new application to determine the Munsell colour of sediment samples, including till. A Ci64 Spectrophotometer UV Spectro linked to Color iControl software is used for the measurement. Although Munsell notations can be calculated via the Color iControl software, the values computed represent the entire Munsell colour chart and are not restricted solely to the Munsell soil colour subgroup. The laboratory has built its own Munsell soil colour database by scanning each Munsell soil colour chip contained in the handbook. Samples are analyzed and the results computed by the software are compared to the database to generate the nearest Munsell colour subgroup match. Determinations of Munsell colour using the spectrophotometer are carried out on air-dried samples. Note: this method is not described in the GSC Sedimentology Laboratory Manual, Open File 4823 (Girard et al., 2004).

9.5.2. Grain size analysis

Textural analysis of the <2.0 mm fraction (% sand, silt, and clay) of till is conducted to assess potential textural influences on till geochemistry and to assist with provenance determinations. The minimum requirement is to determine the percentages of sand, silt, and clay.

Particle size distribution is determined using a Beckman Coulter™ LS 13 320 Laser Particle Size Analyzer for the <0.063 mm fraction and a CAMSIZER® Particle Size Analysis System for the 0.063 to 2 mm fraction.

Quality assurance/quality control duplicates are run for ±5% of each batch.

9.5.3. Total carbon, organic carbon, and loss on ignition

Total carbon, organic carbon, and loss on ignition (LOI) are determined to assess carbonate content and provenance, and organic carbon/material content. Carbon content and LOI are determined using a LECO® and CM 5014 Coulometer.

The QA/QC for the CM 5014 Coulometer involves the analysis of 10 to 20% standards of known $CaCO_3$ content, including 5% as duplicates.

The QA/QC for the LECO® involves 5 to 10% duplicates, and 5% replicates. A LECO® 12% standard (502-902) is used for calibration of the machine (note that the usage of the previous LECO® standard 501-034 was discontinued in April 2019). CANMET standard SO-3 is used to monitor accuracy.

For information about additional tests that are available, refer to the GSC Sedimentology Laboratory Manual, GSC Open File 4823 (Girard et al., 2004).

9.5.4. Clay mineralogy

Clay minerals present in the <0.002 mm fraction of till can be determined by X-ray diffraction (XRD) in the GSC-Ottawa Mineralogy Laboratory. Initial mineral identification is accomplished using EVA (Bruker AXS Inc.) software with comparison to reference mineral patterns. Quantitative analysis is carried out using TOPAS (Bruker AXS Inc.), a computer-based program that performs Rietveld refinement of XRD spectra. A

minimum of 40 mg of dry <0.002 mm material is prepared for analysis in the GSC Sedimentology Laboratory using the clay separation methods described in Section 8.3.5. Data can be used to assess the degree of weathering and determine sediment provenance.

9.5.5. Gamma-ray spectrometry

Gamma-ray spectrometry can be used for mapping the concentrations of K, U, and Th in surface till over large areas to aid uranium and REE exploration (Fig. 16b) and regional bedrock mapping (e.g. Campbell et al., 2007; Fortin et al., 2015; Hagedorn et al., 2018, McClenaghan et al., 2019a). The gamma-ray signature of till samples can be measured to ground-truth airborne gamma-ray spectrometry data sets. At the GSC, this process consists of two stages.

In the first stage, a till sample is air dried in the Sedimentology Laboratory and unsieved (pebble free) or sieved (<2 mm) till is used to completely fill a 236 ml (8 oz) circular metal tin (10 cm diameter) (Fig. 46), which is approximately 300 to 500 g of material. The tin is covered with a metal lid and sealed with black electrical tape and left to sit for three weeks to allow radioactive equilibrium to be reached. In the second stage, the till sample is processed at the GSC Gamma-Ray Laboratory, where the gamma radiation being emitted is measured and the data are converted into concentrations of the radioelements potassium (K), uranium (eU), and thorium (eTh) (Fortin et al., 2015; Hagedorn et al., 2018) using respective standards IAEA-RGK-1, IAEA-RGU-1, and IAEA-RGTh-1. Additional details are provided in Grasty et al. (1991) and Friske et al. (2013).

10. DATA EVALUATION

10.1. Evaluation of quality control data

Digital geochemical results that are received from commercial analytical laboratories by the GSC Sedimentology Laboratory are distributed to the scientist that requested the analysis and recorded in 1) the Sedimentology Laboratory database, and 2) the GSC's Canadian Database of Geochemical Surveys (CDoGS) geochemical database.

QA/QC reports are generated from the results of the control samples that were inserted into the batch of routine samples. A QA/QC module developed for the Sedimentology Laboratory is used to extract the results for the control samples and automates the creation of this QA/QC report. The module integrates simple statistical tools to calculate the mean, the standard deviation, and the relative standard deviation. Sedimentology Laboratory staff and the scientist use the QA/QC report to identify any potential analytical problems. A quick review of the reports validates the reliability of the data. The module generates two types of reports:

Figure 46. Photograph of metal tins filled with dried sieved till and sealed in preparation for gamma-ray spectrometry measurements to determine concentrations of the radioelements potassium (K), uranium (eU), and thorium (eTh). Photograph courtesy of B. McClenaghan.

1. accuracy reports, which are generated from the results of reference materials; and
2. precision reports, which are generated from the results of duplicate samples.

10.1.1. What if you suspect errors?

If a scientist suspects inconsistency, drift, or spurious results in the analytical data, she/he should meet with the manager of the GSC Sedimentology Laboratory. The manager will review the report with the scientist and contact the analytical laboratory to have the samples in question reanalyzed. Another sample from the original material can be prepared and analyzed if it is suspected that the problem arose from the sample preparation.

10.1.2. Other quality assurance/quality control data

Analysis of the QA/QC data can be carried out using statistical packages (e.g. SPSS, SYSTAT, ioGAS), but it is often more time consuming to set up and complete than using the existing Sedimentology Laboratory QA/QC module. An explanation of how to use analysis of variance (ANOVA) to determine both sampling and analytical variability is outlined in Reimann et al. (2008) and software to undertake these calculations is provided in Garrett and Chen (2007). McCurdy and Garrett (2016) present a procedure to calculate the analytical precision and to conduct an analysis of variance between field and laboratory duplicates using open source R Project computing software (R Core Team, 2015) and 'rgr' functions (Garrett, 2013b) written specifically for QA/QC tasks using geochemical data. The ANOVA determines if the between-site variability is greater than the within-site variability, which is nec-

essary for meaningful spatial pattern determination in the geochemical data.

10.2. Metadata

When the analytical data are published, it is important that the publication also includes the essential metadata for the survey and for the till samples because metadata give context to the data and increase the confidence others have in it (Spirito et al., 2013). Appropriate metadata should be reported in all GSC publications releasing geochemical data for GSC till samples. To facilitate the scientist in providing the essential metadata for their survey, a metadata template (Appendix A1) has been created and is to be completed and published with all GSC reports. An example of a completed metadata form is included in Appendix A2. The template represents the minimum metadata that should be reported; a scientist may choose to include more information than the minimum. The template is available for download from the GSC website containing the latest publication formatting instructions of the Scientific and Technical Publishing Services section. The template will be updated occasionally as needed.

Categories of essential metadata that are found on the template (Appendix A) are outlined in the following sections.

10.2.1. Sample and project metadata

- geologist's name;
- province/territory;
- project name;
- funding source;
- datum for sample location coordinates;
- context of the current work as it relates to earlier or ongoing work;
- citations of supporting publications;
- sampling access method;
- sampling design/pattern;
- sample medium/media;
- sampling method;
- number of samples for each medium;
- sample density;
- sample collection date range.

10.2.2. Sample preparation metadata

- laboratory name;
- work order number or certificate name;
- screening: mesh size;
- screening: Wentworth-scale grain size;
- methodology: describe in as much detail as possible;
- number of samples prepared (i.e. was every collected sample prepared?);
- include a published reference for the techniques, if possible (e.g. Percival and Lindsay, 1997; Girard et al., 2004);
- commercial laboratory preparation package code.

10.2.3. Geochemical analysis metadata (bulk geochemistry)

- laboratory name;
- work order number or certificate name;
- date the samples were submitted to the laboratory;
- date the sample data were reported to GSC;
- size fraction analyzed;
- analytical digestion (if applicable) – be specific about ratio and type of acid(s);
- analytical method/aliquot mass (e.g. ICP-MS, 0.5 g);
- laboratory analytical package name – use the abbreviation and package name the laboratory advertises in its brochure;
- upper and lower detection limits for each element (provide an Excel® table);
- digital file (e.g. PDF) of the laboratory's brochure – previous years' brochures are often difficult to find, as are brochures if the laboratory has been sold or has been amalgamated with another company;
- deviations from methods described in the brochure, e.g., different sample mass, extra analyses of additional elements;
- list the types of QA/QC samples (e.g. field and laboratory duplicates, standards, and blanks) inserted in the sample batch;
- if the samples have also been collected for indicator mineral processing, also review the list of metadata required for indicator mineral processing in Section 11.6.3.

10.2.4. A note about publishing data

Publishing the analytical data provides a permanent archive. Databases (corporate and personal), spreadsheets, and backups (CD, DVD, network server, external hard drive, Cloud) are all possibilities of how to archive your data, but they are not necessarily permanent. Publishing the data ensures that there is a permanent record of the work that will be publicly available and accessible in perpetuity through the internet even after a scientist leaves the GSC or any other geoscientific institution.

Publication of GSC Open Files should include the following:

- all raw analytical data files, unaltered, and as originally reported by the laboratory (unless received with errors);
- any modified data files derived from the raw laboratory data.

If some or all of the samples were reanalyzed because of spurious results (*see* Section 10.1.2), both the original and reanalyzed data should be published in the report, together with a clear explanation and identification of the errors.

11. SAMPLING AND PROCESSING OF GLACIAL AND FLUVIAL SEDIMENTS FOR RECOVERY OF INDICATOR MINERALS

The term 'indicator minerals' is used in this report to refer to mineral species present in transported detrital sediments (till, stream sediments, and glaciofluvial sediments) that are indicative of a specific type of mineralization, alteration, or bedrock lithology (McClenaghan, 2005). Most indicator minerals can be physically separated and concentrated from their host sediments using a combination of gravity, sizing, and magnetic separation methods. They typically have medium to high density (>2.8 specific gravity) and usually are relatively stable in the surface environment (Averill, 2001; McClenaghan, 2005).

The protocols described here for the collection of samples and recovery of indicator minerals are applicable to till, glaciofluvial sediment, and fluvial (stream) sediment samples. Refinements of this methodology may be necessary for detailed studies of indicator minerals near mineralized zones. The procedures outlined and recommended in this report are written for GSC scientists. If these procedures are used by provincial and territorial geologists or exploration companies, some specifics will not be applicable (e.g. the filing of information in the GSC heavy mineral database). In the text of Section 11, the term "laboratory" refers to the heavy mineral processing laboratory, unless otherwise noted.

11.1. Field sample collection

11.1.1. Quality control measures in the field

Basic quality control measures for till sample collection are outlined in the Section 6.7.

11.1.2. Tool maintenance

Ensure that sampling tools to collect large heavy mineral samples are thoroughly cleaned between sample sites to avoid cross-contamination. This cleaning can be accomplished by rinsing the tools in water, otherwise, a steel brush or a hard bristle brush and clean cloth can be used to ensure that no material adheres to the shovel between sample sites. Tools should be monitored for wear as small metal flakes can break off tools and end up in the sample as contamination (Fig. 35).

11.1.3. Sample size

Recommended sample size is dependent largely on the texture of the glacial sediment, but also may be dictated by the range and type of analyses to be performed. In the field, a consistent sample size should be collected based on volume (e.g. full pail, full sample bag, etc.), not weight as the weight of the sediment will vary depending on moisture content, sediment compaction, and composition. As a general guide, a glacial sediment sample must contain 5 to 10 kg of sand-sized material to obtain an adequate number of indicator mineral grains to be useful for mineral exploration (Clifton et al., 1969; Averill, 2001). If gold or platinum group minerals (PGMs) are targeted, the silt component is also important because these minerals tend to be silt-sized (Averill, 2001, 2009).

In regions where the till is sandy (e.g. Canadian Shield, Appalachians, Cordillera), a 10 L (2.5 gallon) pail or large sample bag of 10 to 20 kg is collected for heavy mineral recovery (Fig. 21a). In regions where the till is clay-rich (e.g. Western Canada Sedimentary Basin), commonly a full 22 L (5 gallon) rock pail, equivalent to 20 to 40 kg, is collected for heavy mineral recovery (Fig. 21b).

Approximately 12 to 25 kg should be collected for glaciofluvial and stream sediment samples. For the same volume, glaciofluvial and stream sediment samples are usually heavier than till samples. They should be collected using a coarse sieve (i.e. 2.5 cm) to remove the coarse fraction. Sieves should be thoroughly cleaned between samples. Pebbles sieved or picked from the till or glaciofluvial sediments can be retained for subsequent examination.

11.1.4. Heavy mineral field duplicate samples

Protocols for the collection of field duplicates are described in Section 7.1.

11.1.5. Sample contamination in the field

Considerations for anthropogenic and other types of sample contamination are discussed in sections 5.6 and 6.3.

11.1.6. Sample labelling

All samples should be labelled on the outside of the sample bag or rock pail in more than one place, as well as inside the sample bag or pail with numbers written on flagging tape or waterproof sample tags. It is good practice to label sample bags in two locations. including near the top of the bag where there is less chance of the number to being rubbed off during shipping.

11.2. Preparing samples for heavy mineral separation and identification

Several commercial indicator mineral processing laboratories in Canada have developed their own internal QA/QC measures and their data should be considered when monitoring QA/QC (Averill and Huneault, 2003; Baumgartner, 2006; de Souza, 2006; Hozjan and Averill,

Figure 47. Photographs of GSC "Bathurst blank": **a)** an unconsolidated weathered Silurian-Devonian granite (grus) collected near Bathurst, New Brunswick and used as the Geological Survey of Canada's blank sand heavy mineral sample; **b)** the sample material in a 2.5 gallon bucket; and **c)** a close-up view of the blank sample material (*from* Plouffe et al., 2014).

2009; Michaud and Averill, 2009). In addition to insisting on internal laboratory QA/QC, GSC users of commercial heavy mineral laboratories should independently and consistently follow protocols to measure the quality of the data obtained from those laboratories.

11.2.1. Quality control using blank samples

Blank sand samples are inserted into GSC sample batches to monitor potential carry-over contamination from a previously processed batches and cross-contamination between samples within a batch. The GSC uses blank sample material referred to as the "Bathurst blank".

The Bathurst blank consists of weathered Silurian-Devonian granite (grus) of the South Nepisiguit River plutonic complex (Wilson, 2007) collected in the Miramichi Highlands, 66 km west of Bathurst, New Brunswick. The material is unconsolidated and has the appearance of well sorted beach sand (Fig. 47; McClenaghan et al., 2012; Plouffe et al., 2013). It does not contain any precious or base metal indicator minerals. Due to easy access, its consistent nature, and the large area of outcrop, this material was bulk sampled (200 buckets weighing 20 kg each) and used as a GSC in-house blank sample. It was collected in 9.5 L (2.5 gallon) pails and each pail has a unique sample identification number for tracking results. It has been analyzed by the GSC more than 30 times and contains a rare/occasional pyrite or gold grain.

A blank sample should be inserted and analyzed as the first sample of each submitted sample batch. This blank sample should be labelled with a sample code similar to the rest of the samples. This first blank sample is used to monitor cross-contamination from previously processed batches in the laboratory. The use of the blank does not prevent contamination; it is used to monitor it. Contamination may extend beyond the first sample if laboratory processing equipment is highly contaminated. In addition to an initial blank sample, one additional blank sample should be inserted for every 15 to 30 samples to assess the potential for cross-contamination between samples. A Bathurst blank should not be inserted immediately after a 'spiked' sample.

11.2.2. Quality control using in-house standards

The GSC uses a silty-sand till sample bulk samples (70 buckets weighing 20 kg each) from a borrow pit (Fig.

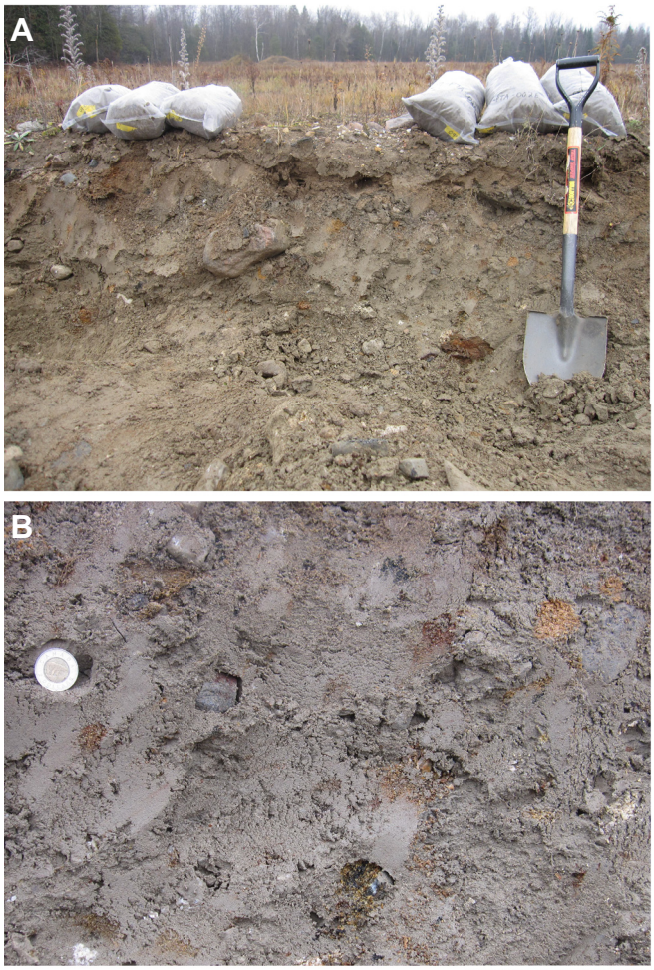

Figure 48. Photographs of **(a)** till section samples at a site near Almonte, Ontario, the samples are to be used as the Geological Survey of Canada's blank till heavy mineral sample ("Almonte till"); and **(b)** a close-up view of the till in place prior to sampling (diameter of the coin is 2.8 cm; *from* Plouffe et al., 2014).

48) in Almonte county, Ontario ("Almonte till") as a GSC in-house standard and as a base material for spiking (Plouffe et al., 2013). Bulk samples of the in-house till standard have been analyzed at least 20 times. This sample has the typical texture and colour of Shield-derived till. Table 5 lists the average composition of the Almonte till as reported from six analyses following processing and picking methods routinely used for GSC samples at a commercial heavy mineral laboratory. Processing methods, including tabling and heavy liquid separation, are described in more detail below.

11.2.3. Spiked sample material

The GSC uses the Bathurst blank and/or the Almonte till as base material for spiking with known indicator minerals. A spiked sample should be inserted every 30 to 50 samples. The mineral species that are added to the blank should be the ones expected to be encountered in the survey area. Spiked samples are inserted into batches to assess a commercial laboratory's ability to recover and identify specific indicator minerals.

Table 5. Average content of heavy minerals in the 0.25–0.5 mm non-ferromagnetic heavy mineral fraction of the GSC in-house till standard known as the Almonte till (n= 6; *from* Plouffe et al., 2013).

Minor constituents[1]		**Major constituents**[2]	
Mineral	Content	Mineral	Content
Gold grains[3]	≤1 grain	**Silicates**	
Sulphides		Hornblende	49%
Chalcopyrite	trace[4]	Garnet (almandine and grossular)	17%
Pyrite	≤70 grains	Epidote	2%
Silicates		Clinopyroxene	25%
Garnet (purple to red)	trace[4]	Orthopyroxene	1%
Olivine	≤3 grains	Sillimanite	trace[5]
Low Cr-diopside	≤14 grains	Tourmaline	trace[5]
Chondrodite	≤22 grains	Staurolite	trace[5]
Oxides		Titanite (sphene)	4%
Chromite	trace[4]	**Oxides**	
Spinel	≤6 grains	Limonite/Goethite	trace[5]
Corundum	≤2 grains	Hematite	1%
Red rutile	≤60 grains	**Phosphate**	
Ilmenite	trace[4]	Apatite	trace[5]

[1]Complete examination of the heavy mineral concentrates; grain counts normalized to 10 kg
[2]Based on counting of a 100 grain split
[3]Pristine and modified grains observed
[4]Under minor constituent trace means 1 grain or less on average
[5]Under major constituent, trace means <1%

It is recommended that mineral grains recovered from glacial sediments be used as spiking grains. Alternatively, mineral grains obtained from crushed bedrock can be used but these grains are generally easy to recognize due to their fresh angular surfaces, which can bias the assessment of the mineral identification. Ideally, mineral grains used for spiking should be etched with a laser and/or well documented using SEM backscattered images so that each grain for spiking can be identified when it is recovered from the processed spiked sample. Density beads (e.g. zirconia beads) and cubes can also be used as spiking grains.

The spiking mineral grains should be inserted into the middle of the till sample and not at the top of the pail or the bag. Part of the sample may need to be temporarily removed from the bag or pail to insert the spiking grains. Insertion in the middle of the sample will ensure that the spiking grains are not removed if a commercial processing laboratory removes a split for archiving. The vial holding the spiking grains should be rinsed with deionized water to ensure that all grains have fallen out of the vial and into the sample.

Only clearly identified mineral grains should be used as spiking grains. The mineralogy of uncertain grains should be verified (e.g. with scanning electron microscope) before using them for spiking.

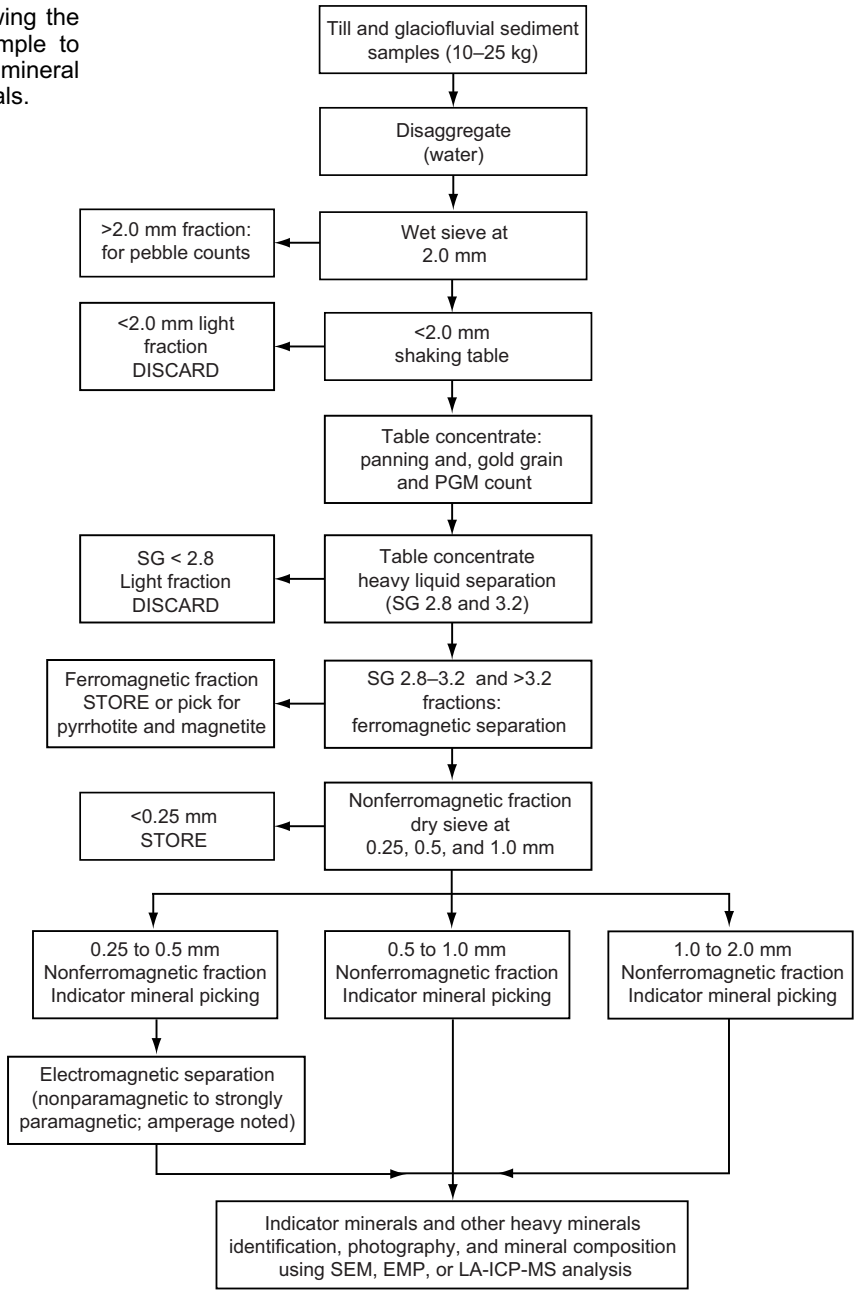

Figure 49. Schematic flowchart showing the main stages of processing a till sample to recover the heavy and mid-density mineral fractions and to count indicator minerals.

The number of spiking grains added to each sample and the number of grains recovered from each spiked sample should be reported in the same publication with the raw heavy mineral data from the commercial laboratory.

11.2.4. Numbering system and order of analysis

The order in which the samples are to be processed should be provided to the laboratory in a spreadsheet. If known, the least mineralized samples should be processed first to limit the possibility of cross-contamination. The order of sample analysis can be randomized. The numbering system for the blank and spiked samples should be the same as for the routine samples.

11.3. Recommended laboratory procedures

Sample processing and indicator mineral picking of GSC samples are usually conducted at a commercial laboratory. In recent years, standing offers with commercial laboratories have been established. Consult with the GSC Sedimentology Laboratory about active standing offers for heavy mineral separation and analyses.

Heavy mineral concentrates from large indicator mineral till (or stream sediment) samples are to be prepared using a shaking table and heavy liquids (McClenaghan, 2011) following the procedures outlined in Figure 49. These procedures have been used to

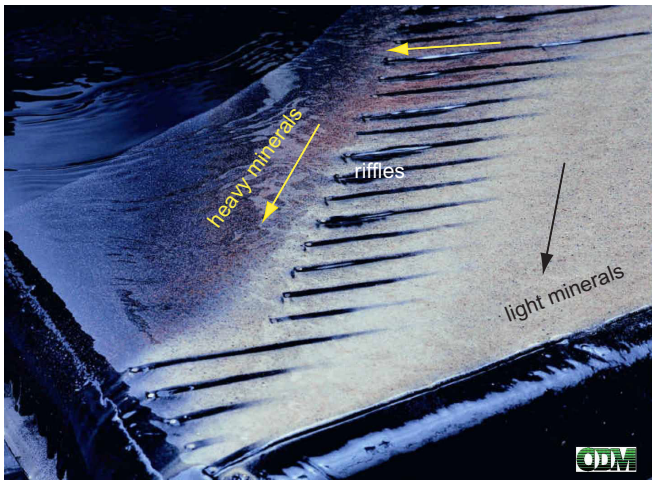

Figure 50. Photograph of a till sample being processed across a shaking table in the first stage of processing to recover the heavy mineral fraction. Photograph courtesy of Overburden Drilling Management Ltd.

process GSC till samples for more than 30 years (e.g. Thorleifson and Kristjansson, 1993; McClenaghan et al., 1995, 2004, 2015a; Plouffe, 1995c). Heavy liquid separation at a specific gravity (SG) of 3.2 is used to process GSC till samples and isolate the >3.2 SG heavy mineral fraction for indicator mineral identification and counting. However, separations at 3.3, 2.8, or other SGs have also been used depending on project objectives. For example, porphyry and REE indicator minerals are usually identified and counted in the 2.8 to 3.2 SG fraction in addition to the >3.2 SG fraction of till (e.g. Plouffe et al., 2016; McClenaghan et al., 2019b). The commercial laboratory can be instructed to process the entire bulk sample or a portion thereof. Arrangements can be made for archiving or disposing of unused sample material.

The heavy mineral processing laboratory can collect an ~800 g representative sample of unprocessed material prior to the heavy mineral separation that can then be used for till geochemical analysis. This procedure is not necessary if a separate sample for geochemical analysis was collected by the geologist while in the field.

The sample processing scheme used for GSC till (or stream sediment, glaciofluvial sediment) samples is shown in Figure 49. In the first phase of the sample processing, a sample is wet sieved to remove the >2 mm fraction. This >2 mm material can be further sieved into specific size fractions (2–4 mm granules, >4 mm or 4–16 mm pebbles, etc.) for future clast lithological examination. An oxalic acid wash of the clasts can be performed to remove oxidation or other coatings from clasts to facilitate their lithological identification.

The <2.0 mm material is passed over a shaking table (Fig. 50) and the table preconcentrate is recovered and micro-panned to recover gold, platinum group minerals, and other small heavy minerals. These panned minerals are examined and counted.

Panned gold grains are characterized according to their shape and surface features using the scheme of DiLabio (1990b) (Fig. 51; *pristine/modified/reshaped*), which describes conditions and surface textures of gold grains as they relate to glacial transport distance and builds on Averill's (1988) descriptions. The scheme is widely used in Canada. *Pristine* gold grains (Fig. 51a) retain primary shapes and surface textures, usually occur as angular wires, rods and delicate leaves, as crystals with grain molds, and as inclusions in sulphides. Glacial transport distance is generally short; tens to hundreds of metres. *Modified* gold grains (Fig. 51b) retain some primary surface textures but all edges and protrusions have been crumpled, folded and curled during glacial transport; they are commonly striated. Glacial transport distance is moderate, i.e., hundreds of metres. *Reshaped* gold grains (Fig. 51c) have under-

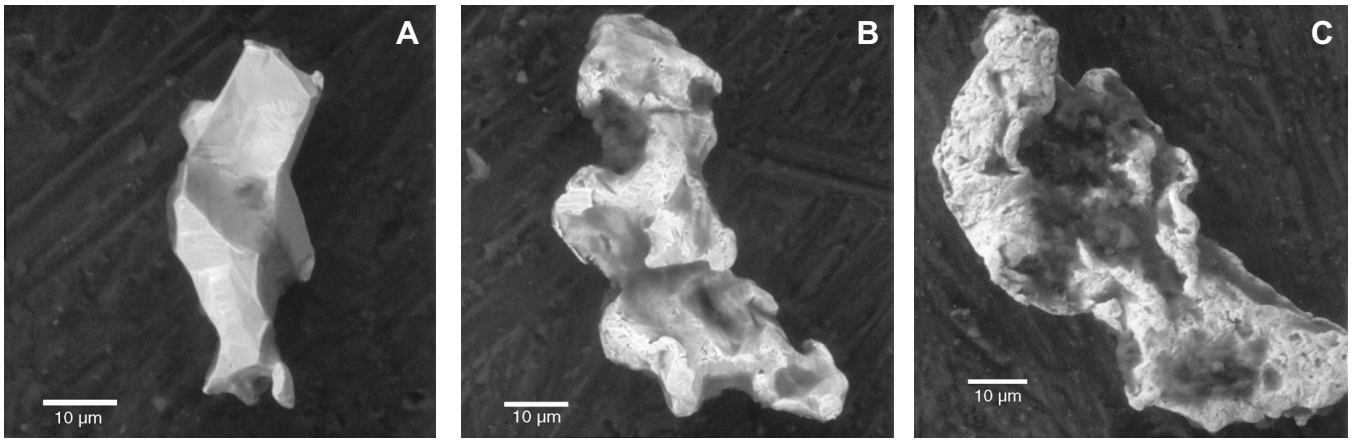

Figure 51. Scanning electron microscope secondary images of gold grains recovered from till showing the three "conditions" routinely documented: **a)** pristine gold grain with equant molds suggestive of former gangue minerals; **b)** modified gold grain with vestiges of equant gangue molds and edges that are slightly curled; and c) reshaped gold grain showing pitted surfaces and well curled edges (*from* McClenaghan, 2001).

gone sufficient glacial transport that all primary surface textures have been destroyed, grains are flattened to round due to repeated folding of leaves, wires and rods, and surfaces may be pitted from impact marks from other grains. Glacial transport distance is usually kilometres. Most background gold grains have a reshaped morphology (Averill, 1988).

All panned grains are returned to the table preconcentrate, which is then sieved at 0.25 mm and the 0.25–2.0 mm fraction is further refined using heavy liquid separation in methylene iodide that has been diluted to a SG of 3.2. The <3.2 SG fraction is stored and the >3.2 SG fraction is processed further to remove its ferromagnetic fraction, which is then set aside. The nonferromagnetic heavy mineral (>3.2 SG) fraction is dry sieved into three size fractions (0.25–0.5 mm, 0.5–1.0 mm, and 1.0–2.0 mm), which are then examined visually for indicator minerals. Heavy mineral identification of smaller size fractions (e.g. 0.25–0.18 mm) or ferromagnetic fractions can be completed if required for the research project and can be requested in consultation with the laboratory.

If the recovery of mid-density indicator minerals (e.g. tourmaline, apatite, jarosite) are important to the research project, then the 2.8–3.2 SG nonferromagnetic fraction can be separated from the <3.2 SG fraction using methylene iodide heavy liquid separation that has been diluted to 2.8 SG. The resulting 2.8–3.2 SG fraction will be examined and the <2.8 SG fraction stored.

If heavy mineral concentrates are unusually large, only a split of the concentrate picking fraction may be examined. In this case, the picking laboratory must report the weight of the split that is picked. It is the GSC project geologist's responsibility to determine if the reported grain abundances are for the split or have been normalized to the weight of the entire concentrate. If the counts have not been normalized to the weight of the entire concentrate, this fact must be reported in GSC publications that accompany the raw laboratory data files.

Gold, platinum group minerals (PGM), sulphide, and uranium mineral grain counts also may be conducted on the panned concentrate fraction immediately after tabling (e.g. McClenaghan et al., 2019c). This procedure can be completed on the entire shaking table concentrate prior to heavy liquid separation, or on the <0.25 mm heavy mineral fraction after the complete heavy mineral separation and geochemical analyses (non-destructive instrumental neutron activation) are completed.

Indicator minerals for a broad range of mineral deposit types can now be identified in till, stream sediment, and glaciofluvial samples (McClenaghan, 2005, 2019c; Sarala and Peuraneimi, 2007; Plouffe and Ferbey, 2017; McClenaghan and Paulen, 2018). Selected examples of indicator mineral suites for different deposit types are listed in Table 1. For regional-scale till surveys, samples should be examined for all the indicator mineral suites listed in Table 1. For detailed case studies around a specific deposit type, the samples can be scanned for specific indicator mineral suites of interest.

11.4. Mineral chemistry

11.4.1. Scanning electron microscope

A scanning electron microscope (SEM) can be used to examine indicator minerals recovered from till samples to (1) confirm the identity of the minerals that have been selected for study; (2) document mineral associations within the grains; (3) document morphology and surface textures of the grains; and (4) identify mineral grains for additional mineral chemical characterization (Layton-Matthews et al., 2017).

Grains mounted on a SEM stub or in a circular epoxy grain mount are then examined using either backscatter secondary electrons (BSE) to document shape and surface textures, or energy dispersive spectrometry (ed.) to identify relative element concentrations.

11.4.2. Electron microprobe microanalyzer

Indicator minerals are commonly analyzed to determine their major and minor element contents using an electron probe microanalyzer (EPMA), which uses wavelength dispersive spectroscopy (WDS) to collect spectra. The lower detection limit is ~0.01% (100 ppm) for most elements (Layton-Matthews et al., 2017).

11.4.3. Rapid scanning electron microscope techniques

The rapid SEM techniques, Quantitative Evaluation of Minerals by SCANning electron microscopy (QEMSCAN) and Mineral Liberation Analysis (MLA) are used to rapidly digitally identify and characterize (e.g. mineral chemistry, size, shape, inclusions, liberation) very small (<0.25 mm) indicator mineral grains in heavy mineral concentrates of till or stream sediments (e.g. Wilton and Winter, 2012; Lehtonen et al., 2015; Mackay et al., 2016; Layton-Matthews et al., 2017; Simandl et al., 2017; Lougheed et al., 2019; Mao et al., 2019).

Both methods collect EDS spectra and high-resolution BSE images. In post-collection data processing, the full X-ray spectrum is compared with a user-defined EDS mineral library and the BSE image to create a digital data set that includes a false-colour mineral map (Fig. 52) and tables of modal mineralogy, grain size, mineral associations (occurrence and interlock-

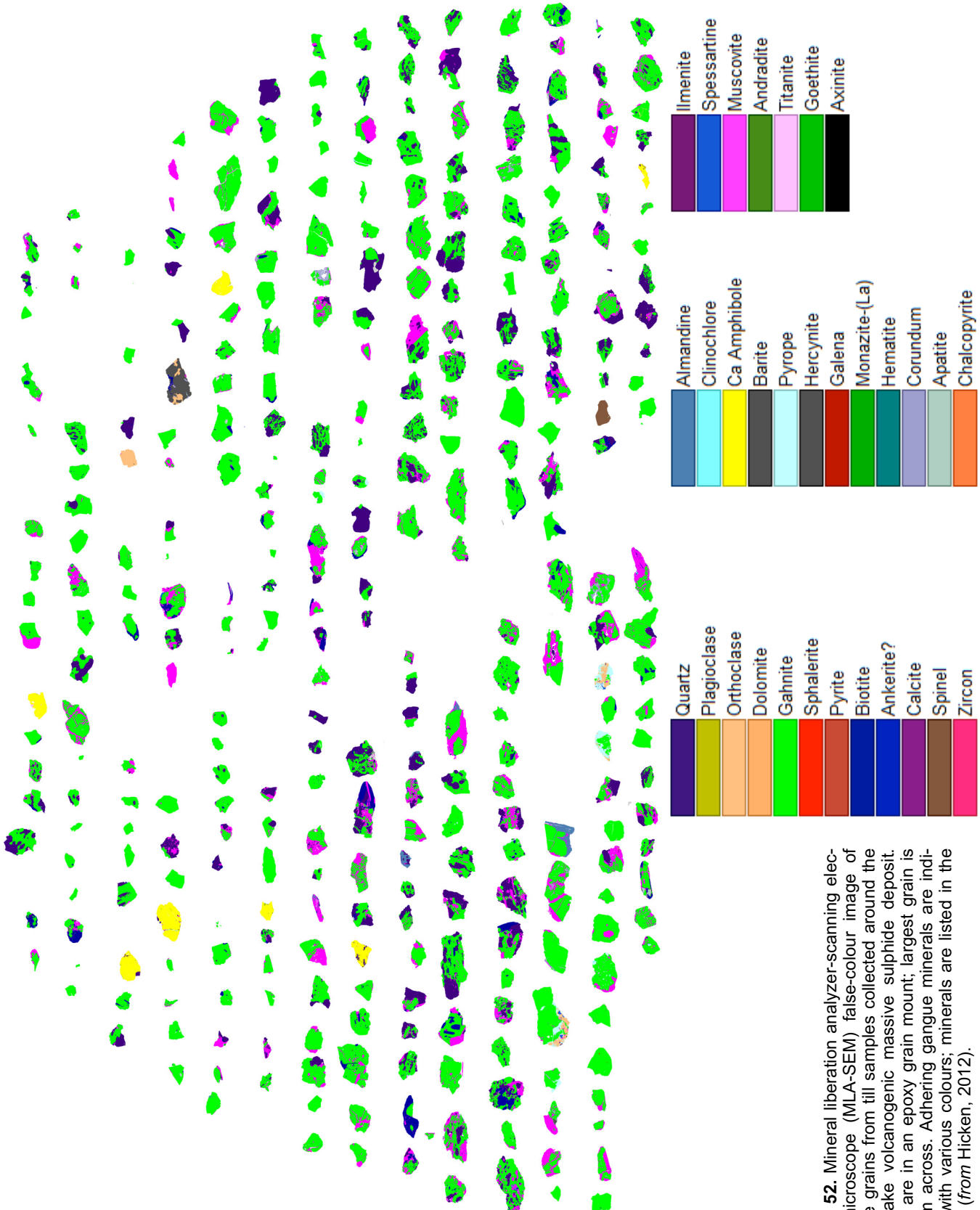

Figure 52. Mineral liberation analyzer-scanning electron microscope (MLA-SEM) false-colour image of gahnite grains from till samples collected around the Izok Lake volcanogenic massive sulphide deposit. Grains are in an epoxy grain mount; largest grain is 0.5 mm across. Adhering gangue minerals are indicated with various colours; minerals are listed in the legend (*from* Hicken, 2012).

ing), particle properties (roundness, area, shape), and mineral liberation.

Mineral grains are mounted in circular epoxy pucks in order to analyze thousands of grains from a single sample. The main advantage of using rapid SEM scanning is that all minerals in the sample can be identified, not just those thought to be indicator minerals.

Lougheed et al.'s (2019) recent study of automated mineralogical characterizations of the <0.25 mm fraction of till heavy mineral concentrates has documented many aspects that must be considered if rapid SEM scanning techniques are to be used. These include the extremely small size of the grains, sample processing methods, representative splits, grain mounting, loss of mineral grains, and various ways to report the mineral abundance data.

11.4.4. Laser ablation inductively coupled mass spectrometry

Laser ablation inductively coupled mass spectrometry (LA-ICP-MS) measures very low concentrations (ppb to ppm) of elements and isotopes using a short-pulsed laser to ablate a small volume of a mineral over tens of seconds during which the material is converted into vapour and aerosol components (Jackson et al. 1992; Layton-Matthews et al., 2017). These components are continually transferred in a carrier gas to be ionized in an inductively coupled plasma and analyzed in either a quadrupole or magnetic-sector mass spectrometer (Layton-Matthews et al., 2017). The method is used to analyze specific point location on grains (e.g. Normandeau et al., 2018; Mao et al., 2019) and to make chemical maps of entire mineral grains to identify chemical zonation (e.g. Poulin et al., 2016, 2018; Duran et al., 2019).

11.5. Reporting of heavy mineral laboratory data

The processing laboratory must report to the GSC the following information:

- laboratory report number;
- digital data file listing the masses of each fraction produced and counts for various indicator minerals in each fraction;
- the types of magnetic separations performed and the equipment used, with its settings;
- the sample processing order;
- the laboratory's internal QA/QC procedures and their results;
- the percentage of the total concentrate that was picked for indicator minerals;
- a PDF file with the sample processing flowchart for every sample batch, including any modifications to the standard heavy mineral separation method.

11.6. After receiving data from the laboratory

11.6.1. Replicate mineral counts

To verify the reproducibility of indicator mineral grain counts, heavy mineral concentrates should be resubmitted for picking. If this procedure is followed, the sample numbers should be changed so that the mineralogy laboratory does not know which samples are being resubmitted.

The optimal approach is to relabel original vials with new numbers and resubmit the samples to the laboratory. Transferring samples to different relabelled vials could result in grain loss. At least 5% of the original samples should be resubmitted for repicking, ensuring that the samples selected for repeat analysis reflect the original range of concentrations of indicator minerals.

11.6.2. Sample archiving

Once picking and analytical work is completed, project or activity leaders are responsible for submitting to the Sedimentology Laboratory all the fractions that were produced and provided to GSC by the commercial laboratory. The Sedimentology Laboratory will archive the various concentrate fractions for future research.

11.6.3. Metadata for indicator mineral samples

In addition to the metadata described in Section 10.2.1, GSC publications reporting indicator mineral data should include the following metadata in an appendix, as shown in the completed metadata template in Appendix A2 under Indicator Mineral Metadata (parts 1 to 4):

- sample medium, i.e., till, stream sediments, beach sand, glaciofluvial sediments, etc.;
- number of samples of each type of medium;
- name of processing laboratory;
- name of picking laboratory;
- work order number;
- date the samples were submitted to the laboratory for processing;
- date the sample data was reported to the GSC;
- flowchart of the processing steps, as a PDF file;
- initial sample mass before processing, stated as a range, e.g., 10–15 kg;
- grain size range used for sample processing, e.g., <2.0 mm;
- preconcentration method(s);
- rock disaggregation method;
- rock disaggregation laboratory name;
- name and density of heavy liquid(s);
- ferromagnetic separation method, e.g., hand magnet, Frantz, roll magnet;

- size fraction(s) that were examined and picked for indicator minerals;
- percentage of heavy mineral concentrate examined for each sample (usually 100%);
- mineral identification method(s) (e.g. binocular microscope, electron microprobe (EMP), SEM);
- mineral grain picking criteria (e.g. kimberlite indicator mineral (KIM), metamorphic/magmatic massive sulphide indicator minerals (MMSIM), other custom suites);
- mineral chemistry determination method(s) (e.g. EMP, SEM, MLA, other);
- mineral chemistry laboratory name;
- mineral count data as the raw data reported by the picking laboratory (use a separate appendix).

11.6.4. Data publication

GSC open files or other publications releasing GSC indicator mineral data should include the following:

- metadata form (*see* the template in Appendix A1 and the completed example in Appendix A2);
- sample location data;
- raw heavy mineral data files as reported by the laboratory;
- edited/modified heavy mineral data, normalized to a consistent sample mass;
- flowchart showing the sample processing steps.

12. CONCLUSIONS

Scientists at the GSC have developed, tested, and refined till geochemical and indicator mineral methods for mineral exploration, provenance studies, and environmental research in glaciated terrain. A collaborative team of scientists from the GSC with vital assistance from provincial geological agencies and the exploration industry have compiled their cumulative experience and knowledge to produce this protocol manual. The major concepts of till as a sample medium, some key concepts of glacial dispersal, and field and laboratory procedures have also been explained to provide a foundation for the methods outlined here.

By adopting this common set of protocols, researchers at the GSC and other facilities as well as exploration geologists will be able to directly compare till geochemical and indicator mineral data sets from anywhere in Canada with the assurance of proper minimum levels of QA/QC for all till geochemical and mineralogical data. This protocol manual is intended for use by provincial/territorial government geological surveys, the mineral exploration industry, and academia.

13. AUTHORSHIP

All authors contributed to the writing and editing of this open file. McClenaghan, Spirito, Plouffe, McMartin, Campbell and Paulen contributed photographs and figures.

B. McClenaghan was the senior author and coordinated the activities of the large team of authors, solicited new figures and tables, oversaw editing, and was the lead author of the sections describing GSC research, till as a sample medium, sample preparation for geochemical analysis, analytical methods, and mineral chemistry methods. W. Spirito was the lead author of the sections describing quality control, data evaluation, and metadata reporting requirements. A. Plouffe was the lead author of the sections explaining heavy mineral sampling and analysis. I. McMartin lead the writing of sections that describe survey design and till sampling methods. J. Campbell was the lead author of the sections that explain field and till sampling equipment, site selection, and rotary drilling. R. Paulen led the writing of the sections describing glacial dispersal principles, detailed sample media (till) descriptions in their glacial context, and requirements for field safety. R. Garrett assisted with the writing of the quality control section and overall writing. G. Hall cowrote the geochemical analytical methods section. P. Pelchat cowrote the pXRF methods section and wrote the pXRF safety requirements. M. Gauthier wrote the section on till sampling in low topography terrain, and contributed to the writing of other sections and one new figure.

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