



**GEOLOGICAL SURVEY OF CANADA
OPEN FILE 6850**

**Till Sampling and Analytical Protocols for GEM Projects:
from field to archive**

**W.A. Spirito, M.B. McClenaghan, A. Plouffe, I. McMartin,
J.E. Campbell, R.C. Paulen, R.G. Garrett, G.E.M. Hall**

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1. Introduction

1.1 Background

From 2008 to 2013, the Canadian government is investing in a major initiative termed Geo-mapping for Energy and Minerals (GEM; <http://gsc.nrcan.gc.ca/gem/>). The objectives of this program are to stimulate economic development in northern Canada and to diversify the economy of northern communities by providing new geoscientific data on energy and mineral potential. These objectives have led to the implementation of numerous projects by the Geological Survey of Canada, conducted in collaboration with territorial and provincial geological surveys as well as students enrolled in graduate programs. Several of these projects involve a surficial geology component and the sampling of till.

In mineral exploration in glaciated terrain, determining the bedrock source of lithological, mineralogical or geochemical constituents of till is known as drift prospecting. This exploration method has a long history, starting with boulder tracing in the 1700s (Sauramo, 1924; Kauranne et al., 1992) and 1800s (Dreimanis, 1958). Other early pioneers include Milthers (1909), Grip (1953), and Kauranne (1958) in Fennoscandia and Prest (1911), Dreimanis (1956, 1958) and Lee (1963, 1965) in Canada.

Drift prospecting methods continue to evolve through ongoing research in glaciated landscapes (c.f. Shilts, 1984; DiLabio and Coker, 1989; Coker and DiLabio, 1989; Kujansuu and Saarnisto, 1990; Kauranne et al., 1992; Bobrowsky et al., 1995; McClenaghan et al., 2000, 2001; Paulen and McMartin, 2009). The study of glacial sediment composition has served not only the mineral exploration industry but has expanded to include the realm of environmental geology, providing baseline data on surficial sediment composition. These data have been used as a clear indication of the natural fluctuation of elemental concentrations in the near surface environment across geological terrains with various environmental applications (e.g. Kettles and Shilts, 1983; Kettles and Wyatt, 1985; Plouffe, 1995b, 1998; McMartin et al., 1996, 1999, 2002; Henderson et al., 1998).

In Canada, reconnaissance-scale glacial sediment sampling surveys covering large areas have been predominantly conducted by provincial, territorial and federal organisations. In most surveys, till is sampled because it is considered the first derivative of bedrock and therefore has been transported by a single process (ice movement) and generally has a simpler transport history than second derivative and higher derivative sediments (e.g. glaciofluvial sediments, stream sediments) which were transported by several processes (ice and water) (Shilts, 1976).

Reconnaissance-scale surveys are often carried out in combination with surficial geology mapping projects with the general mandate of stimulating mineral exploration and, less commonly, for environmental studies (e.g. Kaszycki, 1989; Plouffe, 1995a; Levson, 2001a; Plouffe et al., 2001b; McMartin et al., 2006; McMartin, 2009). In addition, case studies near known mineral deposits of various types have been completed in parallel with these regional surveys (e.g. Dreimanis, 1956; DiLabio, 1981; Batterson, 1989; McClenaghan et al., 2002; Parkhill and Doiron, 2003).

Significant methodological developments have occurred over the years, including the implementation of new analytical methods and digestions (Hall and Bonham-Carter, 1988; Hall, 1991; Noras, 1992; Hall et al., 1996). Furthermore, geochemical analyses conducted on specific grain size fractions of till have demonstrated that elements are preferably enriched in specific size fractions as controlled by primary and secondary mineralogy (e.g. DiLabio, 1982, 1985, 1988; Shilts, 1984, 1995, 1996; Plouffe, 2001; McMartin, 2009). Detailed studies on specific mineralogical fractions have yielded advances in indicator mineral methods (McClenaghan et al., 2002; McMartin et al., in press). These developments have resulted in a multitude of digestions, analytical methods, grain sizes and mineralogical fractions being utilized with limited consistency from one survey to the next.

One of the strengths of regional till sampling surveys is that they cover large areas. This allows correlations to be made between bedrock geology, glacial transport and natural variability across major geological entities. One of the weaknesses of regional surveys is certainly the limited intercomparability of data sets from different surveys due to the variability of the methodologies in each project.

As a first step towards the development of consistent methodologies for the GSC's GEM projects that include till sampling activities, a working group of Quaternary scientists from the Northern Canada Division has prepared this protocol manual. The purpose of the protocols presented in this manual is not to restrict development of or experimentation with new methodologies but rather to ensure that projects use common methods for sample collection, preparation and analysis as a minimum. These protocols will ultimately allow comparison of data sets separated by large distances and gathered through the course of different projects. Hopefully, the protocols presented herein will be adopted in future programs with objectives similar to GEM. However, adaptation and modification of these protocols will be required as technological developments continue and new ideas and concepts are put forward.

Furthermore, provincial and territorial surveys or other agencies conducting till sampling surveys may want to adopt some of the procedures outlined in this protocol manual, although specific details may not be applicable. Our intention is not to enforce these protocols but to suggest a methodology so that final results can be compared. Comments, feedback and corrections are welcome and can be communicated to any of the working group members listed in section 1.2.

Following this chapter, the protocol manual is divided into 9 more chapters presented in the general order that a regional till sampling survey is implemented:

Chapter 2	Survey Design
Chapter 3	Field Equipment
Chapter 4	Site Selection
Chapter 5	Till Sampling
Chapter 6	Field Notes
Chapter 7	Sample Preparation
Chapter 8	Geochemical and Other Analyses of the <0.063 mm Till Fraction
Chapter 9	Quality Assurance/Quality Control

[Chapter 10](#) Sampling and Processing of Glacial and Fluvial Sediments for Recovery of Indicator Minerals

References cited throughout the manual can be found in the last chapter:

[Chapter 11](#) References

1.2 Working Group Members

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2. Survey Design

2.1 Factors Influencing Survey Design

- Budget and time frame;
- Purpose and type of survey (reconnaissance, regional or property-scale; Table 1);
- Size of the survey area;
- Access to the area of interest;
- Type, distribution and provenance of surficial materials;
- Ice-flow history, dominant transport direction and landform trends (Figure 1);
- Stratigraphy and overburden thickness;
- Geomorphology and topography;

2.2 Factors Affecting the Scale and Density of the Survey

- Desired outcome, e.g., defining regional elemental and mineralogical background concentrations, delineating anomalies within a geological province, mineralized belt, or associated with an individual deposit or mineralized zone. The desired outcome will dictate the sampling density (reconnaissance versus regional versus property-scale);
- Sample density (reconnaissance versus regional/local versus property-scale);
- Nature and size of mineralized target and ice-flow history to determine train geometry: point source (e.g., kimberlite) producing ribbon-shaped trains versus a mineralized zone (e.g. stratabound deposit) producing fan-shaped trains versus complex ice-flow history producing palimpsest or ameboid-shaped trains;
- Access, method of transport (e.g., truck, ATV, boat or helicopter) and budget;

2.3 Criteria to Establish at the Onset of a Survey

- Sample processing and analytical methods: these will dictate the size of the sample to be collected;
- Size fraction to be geochemically analysed: e.g., <0.063 mm, <0.002 mm;
- What glacial sediment interval and/or soil horizon and why: element mobility versus logistics and sampling costs;
- Sampling strategy: grid sampling (offset) versus line sampling versus random sampling;
- Survey and sampling methodologies: these must be consistent and used for the entire survey - consistency is essential;

Table 1: Summary of Survey Design Factors

Target	Survey scale	Sample size	Minimum Analyses	Comments
Geologic province/domain	Reconnaissance	Geochemistry and indicator mineral samples	Matrix geochemistry, indicator minerals, heavy mineral geochemistry	Low sample density, often random sample pattern
Mineralized belt	Regional	Geochemistry and indicator mineral samples	Matrix geochemistry, indicator minerals, heavy mineral geochemistry	Low to moderate sample density, often sampled in offset lines perpendicular to regional ice flow
Cluster of deposits	Regional to local	Geochemistry and/or indicator mineral samples	Matrix geochemistry, indicator minerals and/or heavy mineral geochemistry	Moderate sample density in either offset grid or random sample pattern (nearest neighbour)
Individual deposits	Property scale	Geochemistry and/or indicator mineral samples	Single method (matrix geochemistry or indicator minerals and/or heavy mineral geochemistry)	High sample density, infilling previous surveys, tight grid or lines perpendicular to dominant direction of transport

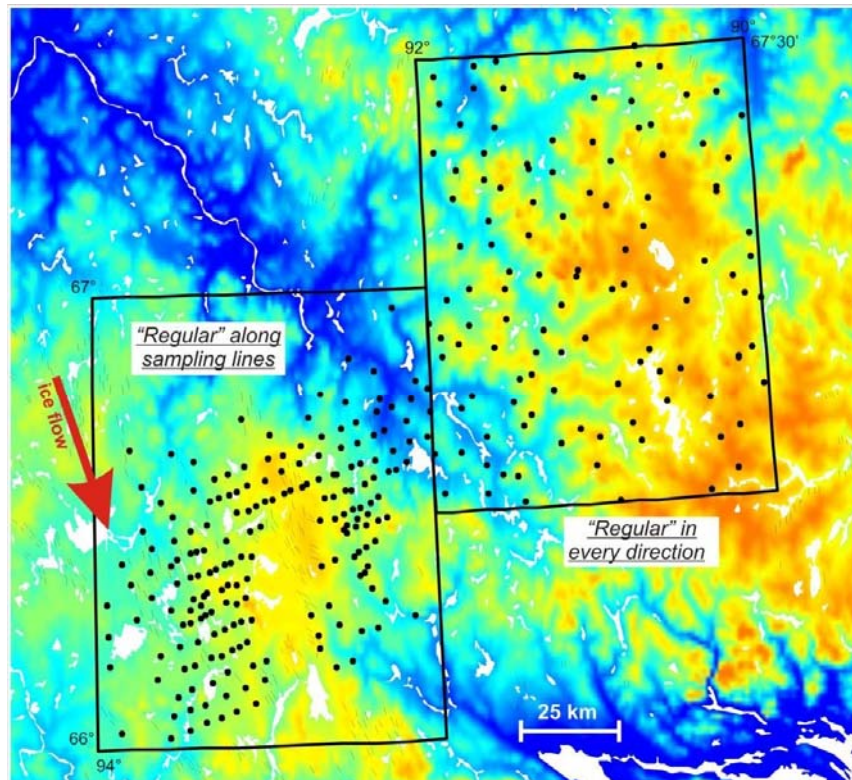


Figure 1: Example of different till sample grids used in the Committee Bay Supracrustal Belt, Nunavut. Samples in the western part of the study area were collected at a regular spacing along lines perpendicular to the known ice-flow direction. Samples in the northeast were collected along a more regular sampling grid because the direction of ice-flow was poorly known prior to the survey. From McMartin and Campbell, 2009 (adapted from McMartin et al., 2003).

3. Field Equipment

3.1 Choosing the Right Equipment

The choice of field equipment depends on the objectives and type of survey as well as sample density, depth and site access.

It is important to make sure that your equipment is in good, working condition before heading into the field.

3.2 Safety Equipment

All field work must be carried out in accordance with Earth Sciences Sector Field Guide 2010 which can be found at: http://ess.nrcan.gc.ca/tfss-sstt/pdf/fieldguide_e.pdf . It is essential that Geological Survey of Canada field party leaders ensure that all field personnel have the appropriate ESS Health and Safety training (http://intranet.ess.nrcan.gc.ca/fasd-dsfa/admin/health_safety/courses/upcoming_e.php).

For Geological Survey of Canada staff, the ESS Field Checklist, Field Season Preparations and other field safety related information can be found at: http://intranet.ess.nrcan.gc.ca/fasd-dsfa/admin/health_safety/field_safe_e.php

No matter what type or method of sampling is proposed, it is important to provide and carry safety equipment which matches the job and personnel. Common sense, proper training and equipment are your main safety factors. Participants should ensure that their personal clothing and footwear is adequate in quality and quantity for a full field season. An Emergency Preparedness plan must be in place prior to beginning the sampling program.

3.2.1 General safety equipment

- First aid kit checked and stocked
- Satellite, radio, cell phone communications
- Brightly coloured outer garments (for visibility during rescue)
- Safety glasses
- Work gloves
- Flares and flare gun (especially if in remote locations)
- If boating - lifejackets, boat and motor safety equipment (kill switch, bailer, throw rope, paddles or oars)

3.2.2 Drilling and backhoe

- Rubber gloves
- Hard hats, steel-toed shoes
- Ear plugs

- Safety cage or methods to shore-up walls for trenching when depths are greater than width of trench. Recommended depths for shoring up, sloping or using a cage vary but the most common is 1.2 m. The depth is dependent on the competence of the sediments being excavated.

An excellent reference can be found on the Construction Safety Association of Ontario web site at: http://www.csa.org/UploadFiles/Safety_Manual/Hazards/Trenching.pdf

Manitoba provides a booklet on trenching at:
<http://safemanitoba.com/uploads/guidelines/excavationwork.pdf>

A recommended statement of policy for trenching is provided by University of Guelph at:
<http://www.uoguelph.ca/ehs/sites/uoguelph.ca.ehs/files/06-12.pdf>

3.3 Equipment Lists

3.3.1 Near surface sampling

Near surface sampling involves hand excavation and includes dug and augered holes and exposures (i.e. open trenches, open pits, borrow pits, road cuts, river or lake shore exposures).

3.3.1.1 Site location and documentation

- Handheld GPS
- Field sheet /notebook/ handheld (GanFeld)
- Digital camera and accessories (e.g., extra batteries, memory cards)
- Scale card
- Silva or Brunton-type compass with inclinometer (a Brunton is best for fabric work)
- Knitting needles for fabric study (make sure they are not magnetic; aluminum is preferable as they do not warp)
- 10% HCl in a 15 to 20 ml squirt bottle with a cap plus extra acid depending on the length of the project
- Munsell colour chart (optional)
- Hand lens
- Measuring tape (3 m) or stick (2 m)
- Water bottle/wet sponge (for cleaning off bedrock at base of hole for striations and cleaning the trowel)
- Large brush or whisk (for cleaning rock surface for striations)

3.3.1.2 Excavation and collection

- Steel shovels with coatings removed (D-handle or long handle according to preference)
- Dutch auger for small samples and site reconnaissance - coatings removed

- Grub hoe or pick for exposures
- Knife (e.g. hunting knife)
- Bricklayer/cement trowel for cleaning off sections
- Geological hammer
- Steel trowel with coatings removed and plastic trowel(optional) for actual sampling (note: plastic trowels are inadequate for digging)
- Pruners or pruning saw – high quality - to cut plant roots
- Sieve, ~ 2.5 cm to separate pebble fraction, if needed
- Fish scale (optional)

*Note - coatings on new equipment should be removed by sand blasting/sanding prior to any sampling (this service may be available from an auto body shop)

3.3.1.3 Sample storage

- Till geochemical sample:
 - ~3 kg (~1.5 L) samples: store in 8" x 13" (20 x 33 cm) 6 mil clear plastic bags, preferably with no seams
- Indicator mineral sample:
 - a) clay-rich tills – 20-40 kg (~10-20 L): store in 20 L plastic pails
 - b) silty, sandy tills – 10 to 15 kg (~5-8 L): store in 6 mil, 12" x 20" (30 x 51 cm) or larger clear plastic bags or 10 L plastic pails
- Bag closures - # 64 elastics, cable lock ties (indicator mineral samples)
- Sample number tags (e.g., flagging tape, waterproof paper) inside bag
- Permanent markers - black chisel point – high quality
- 20 L pails for sample storage and shipping
- Shipping labels (not necessary but useful)

3.3.2 Sub-surface sampling

Sub-surface sampling includes samples taken using backhoes, portable drills or drilling rigs.

3.3.2.1 Backhoe and portable drills

The equipment list is the same as for near-surface sampling but also includes a longer tape measure

- Measuring tape (10 m)

3.3.2.2 *Drilling rigs*

The equipment needed depends on the type of drilling. In addition to the Site Location and Documentation list in section [3.3.1.1](#), it is also useful to have:

- Tape - duct, electrical, masking, fibre, packing
- Various knives (at least 1 large kitchen knife)
- Cement trowel
- Plastic bags appropriate for the sample size being collected
- 20 L pails
- J-cloths and paper towels
- Sample number tags (e.g., flagging tape, water proof paper) inside bag
- Permanent markers - fine point and chisel point – high quality
- Selection of tools (utility knife, pipe wrenches, screw drivers, hammers, pliers, etc.)
- Safety equipment - hard, 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

- Roll of plastic sleeve for core collection;
- pre-ordered core boxes + lids
- drill and screws to close boxes

Reverse Circulation Drills

- Rain suit or waterproof apron
- Rubber gloves
- Rubber boots
- White plastic 20 L pails for sample collection
- 10 mesh sieves
- Wire screening

Mud Rotary Drills

- Conductivity meter
- Several sieves – to catch cuttings
- 30.5 x 30.5 cm square wooden 0.5 cm and 1.25 cm screens - to wash cuttings

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- Plastic 4 L pails - to catch unconsolidated sediments
- Muffin trays
- Several aluminum dishes
- Oven to dry samples (optional)
- Kraft bags for dried samples
- Board for across water trough
- Rubber gloves
- Rubber boots
- Electrical tape – many rolls

Diamond drills

Do not use - contamination too severe to overcome

4. Site Selection

Based on the experience of the working group members, best practices to apply in different physiographic and surficial geology settings are summarized into four sections relating to: 1) mountainous terrain; 2) regions once covered by postglacial sea and glacial lakes; 3) selected glacial landforms; and 4) areas of anthropogenic disturbance. Additional sections describing other environments will be added to this first set based on the cumulative experience of till sampling survey participants.

4.1 Selecting Till Sites in Mountainous Terrains

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

4.1.1 Steeply sloping terrain

- In steeply sloping terrain, till is often covered by colluvium. The composition of colluvium depends on the source material. It can vary in texture from rubble to diamicton, and can be stratified to massive;
- Where colluvium is derived from till, differentiating both sediments can be challenging. The following criteria can be used to differentiate till from colluvium (Levson, 2001b):
 - till is generally more compact, less porous and its matrix contains more fines than colluvium;
 - colluvium can be crudely to well-bedded subparallel to the slope, and more oxidized than till;
 - clast fabric and imbrication in colluvium can be strong and indicate a down slope movement;
 - the presence of glacial landforms (e.g., lateral moraines) indicates *in situ* glacial sediments as opposed to colluvium;
- Where access roads are built 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 (Figure 2);
- In the case of a regional survey for which regional background metal concentrations are being established, colluviated till can be sampled in the absence of *in situ* till, but should be described as such in the field notes (e.g. Levson and Giles, 1995);
- However, in the case of a detailed 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 which needs to be considered when tracing the source of mineralized debris;

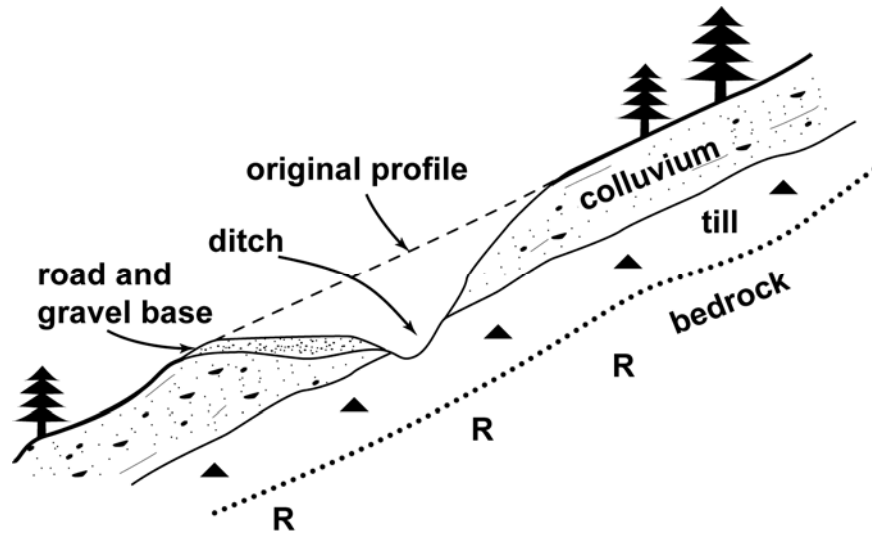


Figure 2. Schematic representation of a sediment exposure in a ditch along an access road in a mountainous area. Till can be exposed underneath colluvium.

4.1.2 Highlands and plateaus

- On highlands and plateaus, till is generally the most areally abundant glacial sediment (Figure 3);
- Thick till exposures might be encountered along roads and streams;
- In such instances, till can be sampled at different depth intervals depending on the purpose of the project. As a general rule, the top part of a till sequence reflects a more distal source compared to the bottom. As part of a regional survey, sampling the top part of the till is adequate but for a detailed study on glacial dispersal from a local source, sampling the bottom part of till, ideally near the underlying bedrock, might be more appropriate;
- On highlands and plateaus in permafrost areas, mudboils are good sites for till sampling;
- Boulder lags can be present on plateaus where there has been widespread melting at the base of the ice sheet. Till samples should be collected below the boulder lag;
- On highlands and plateaus near mountains which have sustained glaciation, the top part of the till can be derived from a supraglacial position. In other words, debris might have fallen on the ice from the steep slopes of the mountainous terrain and remained in a supraglacial position until its deposition on the highland or plateau;



Figure 3: Typical plateau region in British Columbia with an extensive forestry road network accessing cut blocks (treeless regions in picture). Till is generally well exposed along these roads which facilitates sampling.

4.1.3 Valleys

- The thickest unconsolidated sediments are usually found in valleys and may include 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 might be covered by late glacial sediments or might have been reworked or completely removed by glaciofluvial erosion greatly hampering till sampling;
- In valleys with an extensive sediment cover, till can be sampled in natural sections along streams;
- In valleys with an extensive sediment cover and road access, till can be exposed at the base of road sections;
- In valleys with an extensive sediment cover, till may be accessed at depth using overburden drills (Figure 4; see also drill descriptions in [section 5.5](#));
- In valleys where till is not present for sampling, collecting glaciofluvial sediments for recovery of indicator minerals can provide valuable information for a reconnaissance scale survey;

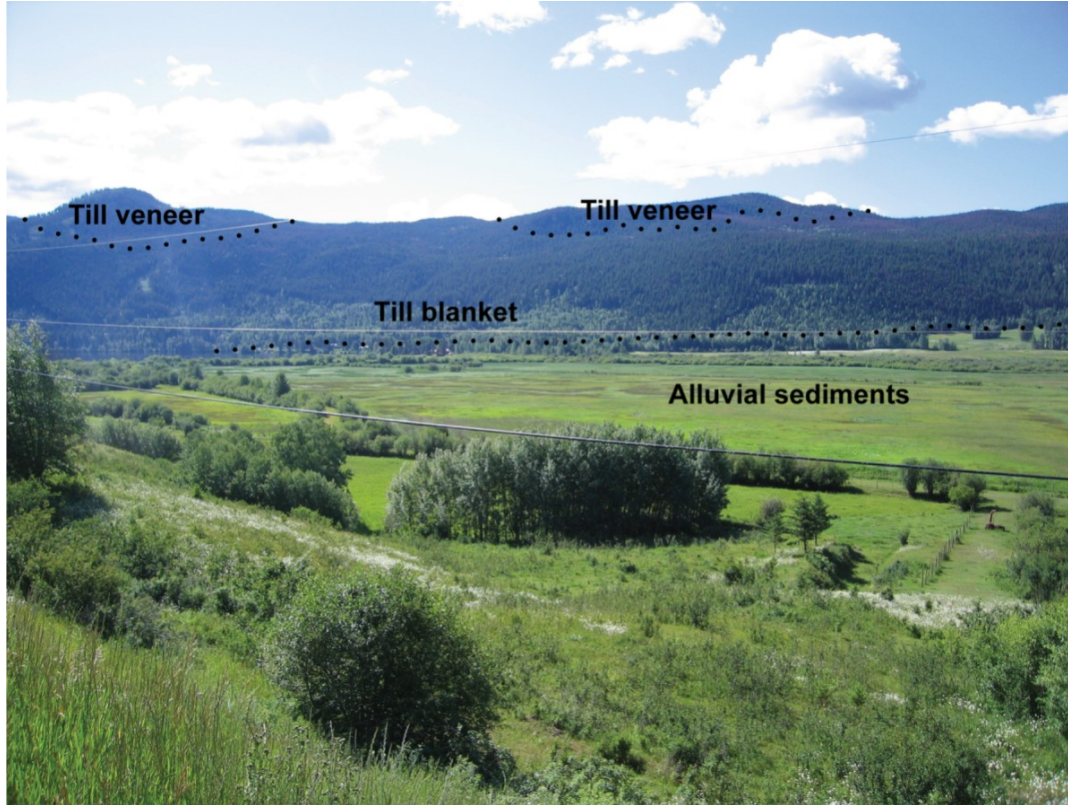


Figure 4. Valley filled with alluvial sediments where till sampling is possible by using overburden drills. Dotted line shows contact between sediment types.

4.2 Selecting Till Sites in Postglacial Marine and Glaciolacustrine Environments

- Postglacial marine and glaciolacustrine environments include glaciated regions once covered by a post-glacial sea or a proglacial lake following deglaciation, but that are no longer submerged by sea water or lake water;
- In areas formerly covered by post-glacial seas or proglacial lakes, the most useful information for selecting till sampling sites is the altitude of the marine or lacustrine limit (maximum elevation of submergence). Knowledge of the marine or lacustrine limit is helpful for determining the extent of inundation, hence the possibility of reworking of till by waves and currents, or incorporation of marine/lacustrine sediments from more distal sources, and for understanding marine or lacustrine sediment facies, distribution and thickness;
- Marine/lacustrine limits can either be clearly defined or show no obvious delineation in the same region. Common features related to marine/lacustrine limits are: 1) raised deltas; 2) ice-contact deltas; and 3) trimlines. Trimlines are defined by notches cut into till slopes or by sharp contacts between unmodified till and intensively wave-washed bedrock surfaces (Figure 5). Marine/lacustrine limits are commonly tilted and may

vary in elevation at a regional scale, as a result of differential isostatic rebound, late-glacial ice masses and/or opening of new outlets (lakes);



Figure 5: Oblique photo taken from the air showing a marine-limit trimline in till deposits in permafrost terrain near Baker Lake, Nunavut (red broken line; about 350 m across). The sharp contrast between unmodified till and littoral sediments marks a marine limit at 122 m a.s.l. Wave-washed till is found below the littoral sediments.

- Typically, till surfaces in topographically higher terrain near the limit of submergence have only been relatively weakly reworked by the postglacial sea or glacial lake, and therefore the till is of suitable quality for sampling. In contrast, till surfaces well below the marine/lacustrine limit may be significantly reworked and winnowed by waves and currents forming a bouldery mantle of unsorted debris (washed till) (Figure 6). Sampling in these areas must be done below the bouldery mantle;



Figure 6: Boulder lag and reworked stony till above unmodified till (grayish-brown colour). Till surface was covered by glacial Lake Agassiz following deglaciation in the central Shield area of Manitoba, west of Thompson.

- In areas that were temporarily submerged in a late glacial sea or lake, till can be completely reworked into coarse-grained flights of raised strandlines. Reasonable till samples can be taken from the occasional mudboils preserved in between large beach crests;

4.2.1 Sampling in thick marine/lacustrine blankets

- Thick offshore marine/lacustrine blankets 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 appreciation of the marine/lacustrine sediment thickness in order to evaluate the cost-effectiveness of hand sampling at depth below the cover, by trenching or overburden drilling. In areas of extensive blankets, acceptable sampling sites may be found on topographic highs where the offshore sediment cover is thinner, or at the base of sections along lake shores, stream banks or road cuts (Figure 7);

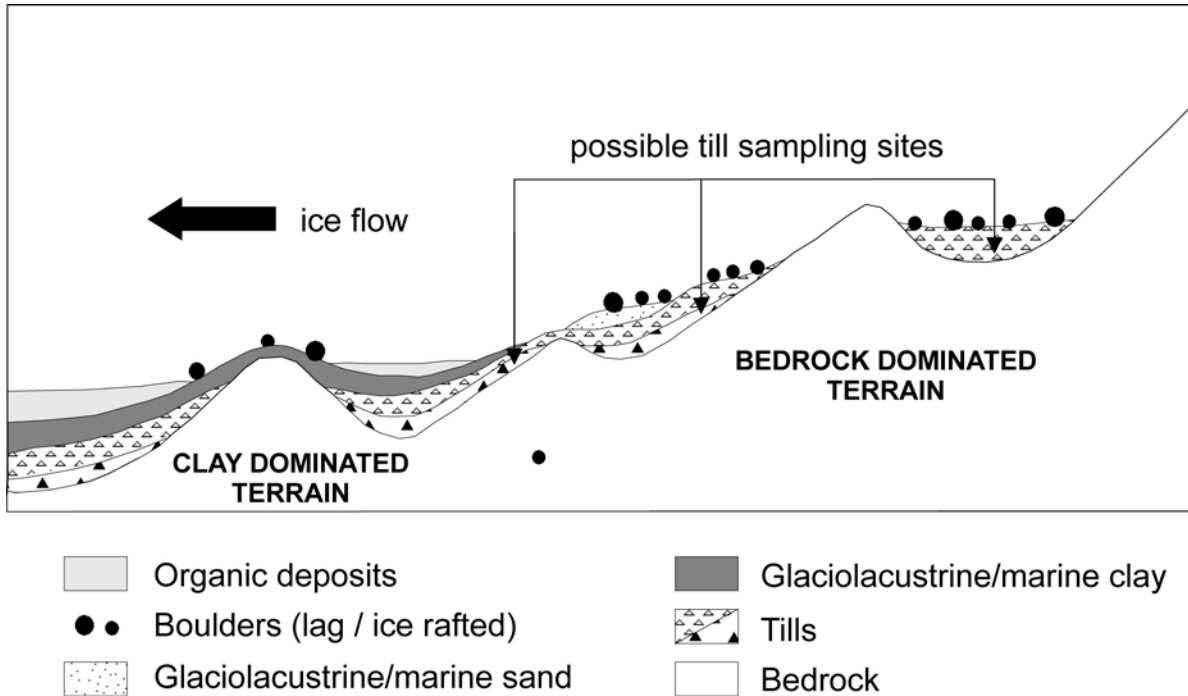


Figure 7: Sampling strategies in Shield terrain covered by relatively thin (1-5 m) drift: upland bedrock-dominated terrain and topographically lower areas dominated by thick glaciolacustrine clay deposits (after Henderson, 1995).

4.2.2 Sampling in marine/lacustrine veneers

- In areas covered by marine/lacustrine veneers, potential till sampling sites are usually easily accessed beneath the marine/lacustrine sediments. Till sampling sites may be found on the down-ice sides of outcrops (Figure 7) or streamlined landforms. On rocky uplands, till commonly forms a discontinuous veneer with thicker accumulations occurring in depressions from which it can be easily collected (Figure 7). Additional sites may be found in natural or man-made exposures along roads or trails, in natural sections along modern river and lake shorelines, or in borrow pits and open pits;

4.2.3 Sampling in permafrost areas

- Special considerations for till sampling in marine/lacustrine areas must be taken in areas underlain by permafrost. Mudboils, which are good sampling sites for till (Figure 8), may also form on silt- or clay-rich marine/lacustrine sediments (Figure 9). Therefore, it can be difficult to differentiate tills from fine-grained marine/lacustrine sediments from an aircraft or by air photo interpretation. As a general rule, mudboils in marine/lacustrine sediments generally have few or no rock fragments on the surface (Figure 9), except for exotic debris from offshore areas (iceberg dropstones). Mudboils in marine sediments may contain marine shells on the surface or within the sediment;



Figure 8: Mudboil developed in till (shovel is 1.2 m long).



Figure 9: Mudboil developed in silty marine sediments. Notice the lack of clasts on the surface of the marine sediments. Scale card is 9 cm long.

- The difficulty in selecting a till sampling site in areas covered by marine/lacustrine veneers (<1 m) can be significant in permafrost areas if the active layer extends down into the till sheet. In these areas, cryoturbation in the active layer may result in mixing of till with the overlying thin marine/lacustrine cover, and dilution of the glacial dispersal signal. In marine environments, the unusual reaction to HCl (10%) of a non-calcareous till may help to indicate the presence of mixing with calcareous marine sediments, or leaching and/or mixing of marine shells;
- Dilution of the local geochemical/indicator mineral signatures by marine/lacustrine sediments in mixed material is usually not significant if the till needs to be collected in between glacial landforms (depending on required sample spacing) because the fine-grained sediments are predominantly derived from reworked adjacent till landforms. However, in lower terrain which is typically more extensively covered by marine/lacustrine sediments, the marine/lacustrine influence can significantly dilute indicator mineral counts and potentially disguise dispersal trains. Therefore, sampling a till-marine/lacustrine mixture is not recommended;

4.3 Selecting Till Sites on Various Glacial Landforms

Not all glacial landforms are appropriate for surface till sampling. The suitability of a landform is related to its genesis as well as the mode and distance of transport of the glacial debris prior to deposition.

Till provenance and dispersal distance are linked to landform genesis and thickness. Some landforms are predominantly comprised of locally-derived material while others contain variable percentages of distally-derived material. Although all of these landforms are suitable for till sampling, it is preferable when possible, to sample locally-derived till which best reflects the underlying bedrock. To assist with the interpretation of the geochemical and indicator mineral results, it is important to note the landform sampled and any indicators at the sample site, such as clast/boulder lithologies, which would indicate the dispersal distance and provenance of the till.

Table 2 provides a summary of selected glacial landforms, their characteristics and preferred till sampling site, building on previous summaries by Aario and Peuraniemi (1992) and Proudfoot et al. (1995).

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Table 2: Rating Scheme for Selecting Sample Sites in Till on Various Glacial Landforms

Landform	Sample Medium Effectiveness	Transport	Depositional Environment	Provenance	Preferred Sampling Site	Comments
Till veneer (<2 m)	High to moderate	Mainly subglacial	Subglacial – lodgement, basal meltout	Predominantly local	- Well drained, top of slope, flat area - down-ice side of outcrops	Best sample sites for locally-derived debris
Till blanket or Till plain (>2 m)	Moderate to high depending on thickness	Subglacial and/or englacial	Subglacial	Variable; generally have a significant proportion of local debris	Well drained, top of slope, flat area	Proportion of local debris increases with depth to greatest percentage just above bedrock surface
Drumlin, streamlined ridge	Moderate to high	Subglacial and/or englacial	Subglacial	Variable; generally have a significant proportion of local debris	Top of landform, well drained, flat area	Multiple theories on genesis; till often more locally derived between drumlins where it is thinner. Internal composition of drumlins is variable.
Crag and tail landform	High to moderate	Subglacial and englacial	Subglacial	Variable; generally have a significant proportion of local debris	Stoss slope of crag but it may be difficult to find material therefore along top of tail	Caution: small scale crag and tails can resemble lee side deposits. See below.
Lee-side deposit	Low to moderate	Subglacial and englacial	Subglacial	Variable; generally have a significant proportion of local debris	Down-ice side of outcrop on top of slope	Cavity-fill deposits beside and down-ice of outcrops; sediments, or portions of, may have undergone some degree of meltwater sorting
Hummocky moraine “knob and kettle”	Low to moderate	Supraglacial /englacial/ subglacial	Ice marginal-stagnant ice	Variable with high proportion of distal debris	Between knobs but not in kettles (sorted sediments)	Variable sorting, flow and dump deposits; usually have a high proportion of distantly derived material; bouldery
Hummocky moraine – undulating, gentle relief	Moderate	Subglacial and englacial	Subglacial – meltout	Variable; generally have a significant proportion of local debris	Well drained, top of slope, flat area	Till composition similar to till plain

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Rogen moraine; ribbed moraine	Moderate to excellent	Subglacial	Subglacial	Surface till is local; core (lower till) has higher proportion of distal debris	Crest of ridge	Undetermined genesis; each type of ridged moraine may be linked to a specific depositional environment thus affect how the till results are interpreted; limited studies suggest the surface deposit is more locally derived.
De Geer moraine	Moderate to low	Subglacial and/or englacial	Ice-marginal/ subglacial	Variable, mixed provenances	Top of ridge; sample below reworked material	Subaqueous moraine ridges deposited by grounded ice akin to push moraine or basal crevasses fills, genesis is unclear; composition is variable – core can be glaciofluvial, glaciolacustrine or glaciomarine sediments with till carapace
Moraine ridge: end recessional, push, minor	Low to moderate	Englacial, supraglacial, subglacial	Ice-marginal	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
Lateral moraine	Moderate	Supraglacial valley wall-side of ice	Ice-marginal	Variable, mixed provenances	Top of ridge; where matrix is present	Restricted to valley glaciers/montane terrain; prospect following moraine up-ice; see comment for moraine ridge
Medial moraine	Moderate	Supraglacial	Ice marginal supraglacial	Variable with high proportion of distal debris	Top of ridge; where matrix is present	Rare; restricted to valley glaciers/ montane terrain; prospect following moraine up-ice; see comment for moraine ridge
Thrust moraine	Low	Subglacial and/or englacial/ supraglacial	Ice marginal	Variable, mixed provenances		Glaciotectonic ice thrust ridges; plucked, folded and/or stacked sequences; not recommended for sampling due to complexity
Crevasse fills - basal squeeze ridges	Moderate	Subglacial	Subglacial	Predominantly local debris	Top of ridge and between ridges	Basal debris squeezed up into crevasses
Crevasse fills	Low	Supraglacial / englacial	Ice marginal supraglacial	Variable with high proportion of distal debris		Variable meltwater sorting; not recommended for sampling

4.4 Selecting Till Sites in Areas of Anthropogenic Disturbance or Contamination

4.4.1 Definition

Anthropogenic deposits are defined as artificial materials, or geological materials so modified by human activities that their original physical properties (e.g., structure, cohesion, compaction) have been drastically altered (Howes and Kenk, 1997).

Such sediment should not be sampled for mineral exploration purposes for obvious reasons: composition is not related to geology and has been modified to various extents by anthropogenic material of various origins (e.g., scrap metal, tools, organic and inorganic contaminants, etc.).

Great care must be taken to avoid sampling till that has been contaminated by airborne particulates from smelters or refineries (e.g. McMartin et al., 1999; Bajc and Hall, 2000), railways and highways. Near-surface till that appears to be undisturbed can actually be highly contaminated in these areas.

4.4.2 Common localities of anthropogenic deposits and contamination

- Present and past producing mines, mills, smelters and refineries;
- Present and past producing granular aggregate and rock quarries;
- Construction sites (roads, bridges, dams, etc.);
- Regions of hydrocarbon infrastructure (pipelines, drill sites, etc.);
- Construction sites (roads, bridges, dams, etc.);
- Areas proximal to high vehicular traffic flow;
- Railway corridors;
- Populated regions;

4.4.3 Criteria to identify anthropogenic deposits

- Landforms (suspicious landforms not related to glacial processes);
- Porous sediment texture, poor compaction and chaotic structures;
- Sediment composition (visible contaminant or reworked material in the sediment; scrap metal, stumps, square logs, unusual odour, metal or glass fragments, etc.);
- Regional environment (proximity to regions defined above);
- Suspicious paleosol and/or organic matter (buried, truncated or homogenized modern soil profile/humus layers/wood debris overlain by anthropogenic deposits);

4.4.4 Sampling strategy

- 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;
- At mine sites, the walls of open pit mines can be sampled;
- Samples may be collected at the base of granular aggregate pits, if deposit has been mined down to the till subcrop (Figure 10);
- Avoid areas stained by oil or hydraulic fluid;
- Closely observe the soil profile;
- If you suspect area has been physically disturbed (e.g., bulldozed) look for an intact soil profile;
- Contamination around anthropogenic sites (e.g., mine site, smelter, etc.) can be airborne, exercise caution with exposed horizontal and vertical surfaces. Dig deep into the sediment before collecting a sample;



Figure 10: Till sampling at the bottom of an active gravel pit; a) general view of the pit with heavy machinery; b) till dug from the bottom of the pit and deposited by loader on the gravel surface; c) close-up of till.

5. Till Sampling

5.1 Sample Size

- 3 kg (1.5 L) is the optimal size for various till geochemical analyses, physical determinations (mineralogy, texture, etc.) and archiving (typically 800 g) (Figure 11). This small sample should be preferentially taken in the same hole as the large indicator mineral sample used for heavy mineral separation. Both samples should be collected in separate bags to facilitate shipping to different facilities. The small bag should be filled 2/3 to 3/4 full to ensure 3 kg has been collected;
- 10-15 kg (~5-8 L) of sandy till is sufficient for gold grains, heavy mineral counts, heavy mineral geochemistry and indicator mineral recovery (Figure 11);
- 20-30 kg (~10-20 L) of silt- or clay-rich till is needed for indicator mineral recovery (Figure 12);
- If the gravel fraction is needed for till provenance studies, pebbles can be collected from the pit or the surface of a sorted circle or a non-sorted circle (mudboil) and put in a separate bag, or set aside during sample preparation at the laboratory either from the small bag (for granules only) and/or from the large indicator mineral bag (granules and pebbles). Washed and sieved gravel fractions can also be obtained from the indicator mineral processing lab;



Figure 11: Two till samples from the same site: the large bag (10-15 kg) is for heavy mineral study and the small bag (ca. 3 kg) is for geochemical analyses, physical determinations and archiving (from Plouffe et al., 2009).



Figure 12: A 20-30 kg till sample in a 20 L pail lined with a plastic bag. Note that the sample number is recorded on the pail and on a tag (flagging tape) inserted in the sample.

5.2 Sampling for QA/QC

- One sample per block of 20 samples should be collected as a field duplicate to test site variability, and taken randomly within each block (i.e. not every 20th);
- A field duplicate is collected from a second hole or sediment exposure up to 10 m away from the original site. The amount collected should be larger (2x) than the normal sample size because it will be split into a blind duplicate after the entire field duplicate sample is sieved. The field duplicate sample must not be homogenized with the original sample. Both the original and duplicate samples are collected into two separate bags with different sample numbers. A number consecutive to the original sample can be given to the field duplicate sample;
- In addition, one sample number per block of 20 samples should be set aside for a standard (either primary or secondary), and one for a blind duplicate (i.e. split from the field duplicate in the lab after sieving the entire field duplicate sample). Refer to QA/QC section for details about the [sample numbering system](#);

5.3 Sample Depth

- Samples are commonly collected in the upper part of a till unit (0.5-1.0 m below the natural land surface) in reconnaissance and regional scale surveys, as the surface part of the till blanket generally represents the composition of a wider source area;
- In detailed surveys designed to locate buried ore bodies with the greatest possible precision (e.g., prior to drilling), samples are collected closer to the bedrock surface (<1 m above the bedrock surface), as concentrations of indicator minerals derived from a buried source rock increase with increasing depth downward towards the source;
- If possible, samples should also be collected at different depths (0.2-0.5 m intervals) at a small number of sites in thicker (>2 m) drift areas exposed in sections or trenches, to characterize compositional variations related to till stratigraphy and/or weathering;
- In areas of continuous permafrost, the depth of till sampling is often restricted by the thickness of the active layer. The active layer commonly extends to 1-2 m depth in till during the maximum summer thaw period, but depending on local climate conditions, frozen material can be found at 0.25 m depth (or shallower) early in the summer;

5.4 Sampling in Different Terrains

5.4.1 Forested areas

- Chemical weathering is the dominant factor controlling the nature of weathering products in sediments above the zone of oxidation in forested areas. Brunisolic (Figure 13), Luvisolic and Podzolic soils developed on till dominate the well-drained upland areas of the boreal and deciduous forest where till samples are usually collected;
- Interpretation of geochemical data from B-horizon soil developed on till in glaciated terrain is complicated by the difficulty in distinguishing clastic (glacial) dispersal from soil formation and weathering processes, or from anthropogenic input. C-horizon soil developed on till is the optimal medium because the material most closely reflects the primary composition of the bedrock from a source area;
- Till samples from the C soil horizon should be collected between depths of 0.5 m and 1 m, preferably above the water table, but definitely below the B-horizon which is commonly enriched in amorphous material (Al- and/or Fe-oxyhydroxides, organic matter, clay), and/or shows a change in color due to oxidation of Fe (orange to brown), or leaching of carbonates;

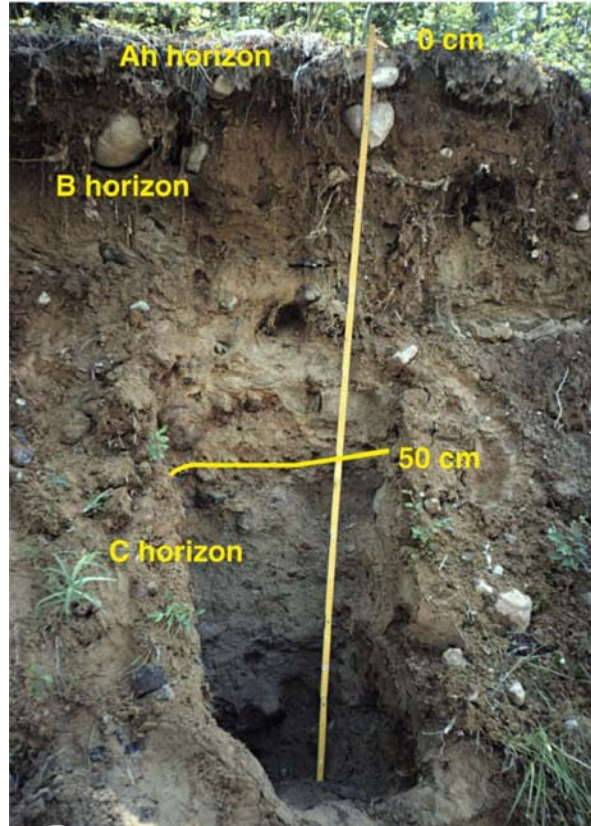


Figure 13: Orthic brunisol developed on sandy-silty till near Flin Flon, Manitoba (from McMartin and McClenaghan, 2001).

5.4.2 Permafrost areas

- Physical weathering is by far the dominant process in soils underlain by permafrost. Turbic cryosols are the predominant soil type developed on glacial sediments in these areas;
- Cryoturbation results in mixing of soil horizons and is indicated by patterned ground features such as sorted and unsorted nets, circles, polygons, and stripes. Mudboils, or non-sorted circles, are the preferred till sampling sites (Shilts, 1977). They are easily recognized from air-photo interpretation (depending on scale), or directly from the aircraft or on the ground by bare to lichen-covered, round to oval patches, commonly surrounded by ridges of vegetation. Sorted circles also develop in till; these have a border of stones surrounding finer material in which till can also be collected;
- Strategies for sampling till in permafrost areas are summarized below:
 - the material in mudboils and sorted circles is normally well homogenized and relatively unweathered, hence representative samples may be collected with a shovel at shallow depths (minimum 0.30 m) at the centre of the circle;
 - the depth of till sampling is often restricted by the thickness of the active layer which commonly extends to a depth of 1-2 m in till during the maximum summer thaw period (Shilts, 1978);

- vertical mixing of the soil can create a redistribution of surface organic-rich horizons at depth in the active layer. Therefore, special care must be taken 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 the pit has too many cryoturbated organics or oxidized material, dig another hole nearby (it's easy!). Sometimes several attempts are necessary;
- in areas where mudboils are poorly developed or scarce, such as in coarse-grained bouldery till or where surface till is covered by a thin organic mat, till samples can be collected below the thin soil profile;

5.5 Till Sampling Methods

Please consult Coker and DiLabio (1989), Coker (1991), Plouffe (1995c), McMartin and McClenaghan (2001), Levson (2001b), McMartin and Campbell (2009) and Paulen (2009) for detailed method descriptions or for further references on the following till sampling methods.

After sampling using any of the methods described below, it is important to restore the site.

5.5.1 Hand excavation



Figure 14: Till samples collected from a road cut in south central British Columbia (from Plouffe et al., 2010).

- The most cost-effective procedure to collect samples at shallow depth is from pits dug with a shovel;

- 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/below a large boulder and into the sides of the hole. 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 secateurs (pruning shears) are useful to cut roots. Till samples can also be collected with a shovel from road cuts (Figure 14) and borrow pits (Figure 15), or from natural sections along rivers or lake shorelines;



Figure 15. Till sampling in a borrow pit dug into till, northwest Alberta.

- In the boreal forest, black (and white) spruce trees interspersed by a thick moss cover typically grow on till; pine trees grow on well-drained material (sand and gravel); poplars and birch trees grow on clay. However, local conditions (drainage, till texture, stratigraphy, etc.) can completely disrupt this typical association;
- Depending on accessibility and spacing between sample sites, about 6 to 8 holes can be hand dug in a 8 hour-day by a two-person crew in forested areas. The pits should be filled with excavated material after the till sampling is completed in order to avoid leaving any hazards for people or wildlife;
- In permafrost terrain, as many as 20 samples per day can be collected by a two-person crew with helicopter support. In addition to mudboils, river sections also provide excellent opportunities for sampling by hand excavation in permafrost terrain. Undisturbed till, i.e. till that has not been slumped or soliflucted, should be collected (look for hoodoos). A hand-dug hole in a mudboil fills back in by itself after a few days (or a few minutes!) due to solifluction;

- It is essential that the shovel be thoroughly cleaned between each sample site, preferably with a water rinse, but if this is not possible, then wipe the shovel clean with a clean rag or moist moss;

5.5.2 Dutch Auger

- The Dutch Auger is a light, portable hand tool that can be used before digging with a shovel in order to identify the proper sampling material and to facilitate the choice of the sampling site;
- Dutch Augers save time in forested areas as they can be used to determine if the thickness of overlying glaciomarine/glaciolacustrine clays are thin enough (<0.5 m) to dig a hole and reach the till layer below. Dutch Augers are rarely used in permafrost areas;
- Dutch Augers can also be used to collect small-sized till samples (1-2 kg) in fine-grained surface tills (e.g., Prairies); or where the water table is elevated; and/or to collect high quality geochemistry samples at depths unreachable with a hand shovel;

5.5.3 Trenching

- The use of a wheeled or tracked excavator for digging trenches can be economical in areas of thicker (>3 m) drift where terrain and access permit. A typical backhoe machine can dig trenches from 3 to 5 m deep;
- The use of an excavator is particularly helpful for detailed sampling in a small region;
- 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 along a profile at regular intervals;
- Environmental impact can be important particularly if trails have to be cut in areas of larger trees. Hazards of working in a trench are significant and walls should be stepped or reinforced (see [links](#) in section 3.2.2 for trenching safety). The pits must be back filled with excavated material after the till sampling is completed in order to avoid any hazards to people or wildlife;

5.5.4 Portable drills (Wacker Neuson, Pionjär, Cobra®, Sipre)

- Portable drilling equipment is an alternative for till sampling where drift is moderately thick (<30 m), or where accessibility, costs or environmental impact restrictions prohibit the use of a backhoe;
- The stratigraphy of the 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 to 300 g) and can be used for fine fraction till geochemistry, but are not large enough for heavy mineral or gold grain studies;
- The advantages of portable drills are that they are portable and inexpensive to operate. For a sampling survey using portable drills to be efficient, experienced, strong, and physically fit operators are a must;

5.5.5 Power auger drilling (semi-portable)

- The heavier, solid stem auger drill can be used in 1 to >100 m of unconsolidated sediments if the near surface stratigraphy is not overly complex and cobble/boulder content is low (e.g., Prairies). Till samples are collected on the auger stem flights. Some sidewall contamination is unavoidable, but usually material close to the drill stem is reliable enough for crude stratigraphy and sampling. Once the groundwater table is penetrated, mixing of sediment on the auger stems is unavoidable, recovery is poor and control on sample depth is unreliable;
- The hollow stem auger drill can be used to collect continuous core and sampling media for indicator minerals samples. Typically, truck-mounted drilling rigs are used. They are equipped with a 6 inch hollow stem auger with a fitted split core barrel designed to retrieve core in segments up to 1.5 m long and 7.6 cm diameter. Within the Interior Plains, this is probably the most economic means of obtaining shallow sedimentary core large enough for sampling purposes. As with solid stem auger drills, indurated beds, large cobbles and boulders are impediments;
- In areas of continuous and discontinuous permafrost, semi-portable auger drills have been used in the past for mineral soil sampling;

5.5.6 Reverse circulation rotary drilling

- Reverse circulation rotary drilling can be used in areas of extremely thick glacial sediments (10 m to >125 m), and where till is stony and bouldery;
- 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 to a tricone bit 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 4.0 mm screen and then into a two bucket system (Figure 16) to allow the sediment to settle and excess water to flow off;
- A 10 kg till sample is collected from material in both buckets for approximately every 1 to 2 m drilled. Most clay-sized material, and approximately 30% of the silt-sized material in till, is lost by this drilling method. Also, till samples can become cross-contaminated by the recirculating water;
- Recovery is generally good in 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 chips and the geologist has only one chance to describe and sample the material as the drill rapidly penetrates the ground;

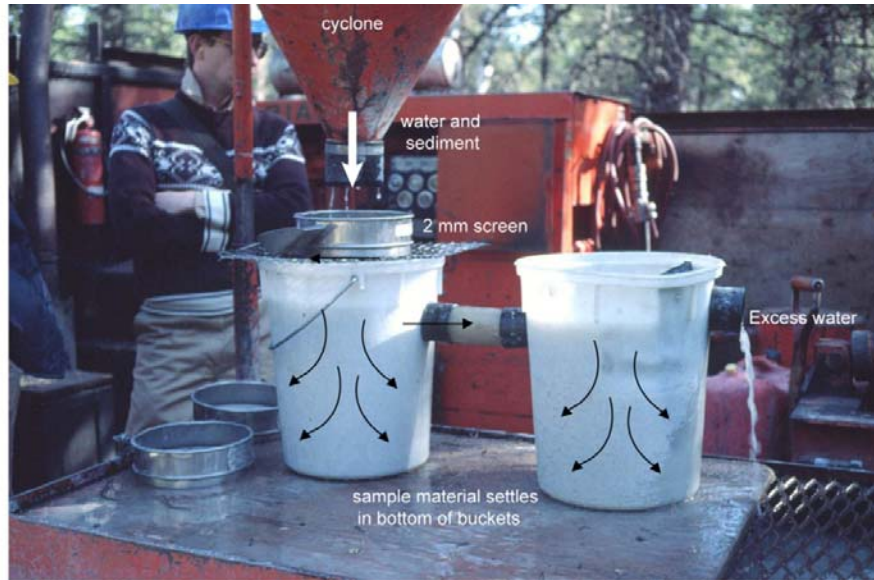


Figure 16: Reverse circulation drill and cyclone used to decrease velocity of the slurry returned from the drill and the two-bucket system used to allow excess water to flow off. A till sample is collected from the bottom of both buckets for a specific depth interval (from McMartin and McClenaghan 2001).

5.5.7 Mud rotary drilling

- Mud rotary drills are typically used for water wells and groundwater investigations in areas of extremely thick glacial sediments (10 m to \pm 300 m), particularly where the tills are fine-grained and matrix-rich (Figure 17);
- A suspended bentonite slurry is used as circulation fluid which 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 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. Compacted samples (i.e. clay, till) are collected using screens while 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 minerals;
- Recovery is generally very good in all sediment types. Loose, unconsolidated sediments have the poorest recovery. The quality of the sample and stratigraphic interpretation is variable depending on the drilling conditions which affect the return rate, size and abundance of cuttings;
- Continuous core can be collected using a 10 ft (3 m) long, 2.85" (7.3 cm) inside diameter split barrel insert, or with a wire-line sidewall sampler;

- Geophysical logs, which measure the single-point resistance and spontaneous potential (SP) of the materials, are critical for the identification and correlation of lithologic units;



Figure 17: a) Mud rotary drilling rigs are a cost effective method for subsurface sampling of fine-grained, matrix-rich tills; b) Cleaned samples collected from a mud rotary drilling rig range from clays and silts (cuttings), fine sands, sands and gravels, to fine-grained till (cuttings; extreme back).

5.5.8 Rotasonic drilling

- Rotasonic drilling is an optimal drilling method for areas of extremely thick glacial sediments (10 m 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 finer fraction are significant;
- Rotasonic drilling uses a combination of high frequency resonant vibration and rotation to drill through glacial sediments, boulders and bedrock with minimal compaction or disturbance to recover a continuous core 9 cm in diameter. 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. Core is resonated from the core barrels into plastic sleeves usually in 1.5 m increments and then the rods and core barrels are replaced down the hole;

- The major advantage of rotasonic drilling over reverse circulation drilling is that it recovers high quality drill core. However, it should be noted that in gravelly units recovery is variable. The major 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 casing, pull core barrels and rods to recover core and reenter the hole reduces the productivity of rotasonic drilling;

6. Field Notes

Field notes can be recorded in field notebooks, on field cards or using a handheld digital device such as GanFeld (Shimamura et al., 2008). If using a digital device, also record the location of each sample site on an air photo or topographic map. At each sample site, take at least one photo of the site.

The minimum field data requirements for till samples collected for heavy mineral and geochemical analyses are listed below:

- Site number
- Sample number
- Location: UTM zone, easting, northing, or latitude and longitude
- Datum: NAD 83 or 27
- NTS sheet (1:250 000 scale)
- Province/territory
- Date collected
- Collector (person who took the sample)
- Material type: diamicton (very likely to be a diamicton if till is being sampled; otherwise, describe)
- 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, mudboil, etc.
- Purpose of sample: 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, heavy
- Sample moisture: dry, moist, wet
- Sample texture: silty sand, sandy, clay-rich, etc.
- Sample color: grey-brown, reddish, orange-brown, etc.
- Clast comments: lithologies observed, size, roundness, abundance
- General notes if required

This list represents the minimum field data required for till collected for heavy mineral and geochemical analyses. More extensive field notes can be collected.

7. Sample Preparation

- Confer with GSC Sedimentology Lab as to time frame for preparation, and use commercial lab only if backlog in GSC Sedimentology Lab will severely hinder project delivery;
- 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 Lab. This allows for more control over the preparation stage, minimizing the possibility of contamination;
- If some samples are suspected to be metal-rich, then specify processing order to the lab, with the most metal-rich samples processed last. This will minimize the possibility of cross-contamination;
- If nothing is specified, the default processing order is numerical order; confirm that the GSC Sedimentology Lab or commercial lab will record the processing order used;

7.1 Shipping

- Ship samples to GSC Sedimentology Lab (go to [7.2](#)) or commercial lab (go to [7.3](#)) for preparation for geochemical analysis;

7.2 Sample preparation at GSC Sedimentology Lab

Sample preparation protocols for geochemical analysis of 3 kg till sample are detailed below and summarized in Figure 18.

7.2.1 Archiving

- Set aside ~1 kg unprocessed split prior to preparation of sample;
- Split will be air dried and 600-800 g will be archived at GSC Sedimentology Lab;
- Remainder of sample will be used for other tests conducted by the GSC Sedimentology Lab;

7.2.2 Sample preparation

- Air dry sample in plastic disposal dish at <40°C;
- Disaggregate sample in agate (not porcelain) mortar & pestle or inside unused plastic sample bag using rubber mallet- new bag for each sample;
- Prepare one blind duplicate from large field duplicate (sieve and then split the field duplicate), to be submitted for geochemical analysis in every block of 20 samples (see also [QA/QC](#) section);
- Freeze dry samples with high clay content if clay sticking to sand sized grains;

7.2.3 Sieving to <0.063 mm

- The <0.063 mm fraction is the most commonly used size fraction for till geochemical analysis (e.g., Tarvainen, 1995), because ore minerals are easily comminuted to this size range over short distances (Nevalainen, 1989; Shilts, 1995) and it contains phyllosilicates that will scavenge cations released during weathering (Shilts, 1993; 1995). The other major reason for using the <0.063 mm fraction is that it can be prepared for analysis rapidly and inexpensively (Lett, 1995; Levson 2001b).
- For case studies around mineralized sites, arrange field samples in a list from least metal-rich to most metal-rich, and sieve them in this order to minimize the possibility of cross-contamination;
- To monitor possible cross-contamination during sample preparation, silicic acid blanks should be used (provided by the GSC Sedimentology Lab). The blanks must be sieved at regular intervals within the batch of routine samples and using the same equipment as for the routine samples;
 - Sieve approximately 400 g of silicic acid to create a 60 g lab preparation blank. If a problem is suspected, this amount will allow for replicate analyses by methods using a 30 g aliquot;
 - As a minimum, sieve the blanks at the beginning, middle and end of each batch. If more blanks are used, they can be sieved at the beginning and end of the batch as well as regularly (every ~25 samples, for example) within the batch. The silicic acid blanks should be sieved more frequently if samples are suspected to be metal-rich;
 - Assign sample numbers to the silicic acid blanks (see section [9.4.1](#)). Submit the routine samples and the blanks, in the order they were sieved, for geochemical analysis to monitor possible cross-contamination.
 - If you choose to randomize the samples after sample preparation and before geochemical analysis, ensure that the blanks are not all analysed in one group, but are interspersed within the batch;
- For routine samples, sieve sufficient sample material (~2 kg Shield derived till) to generate at least 62 g of <0.063 mm fraction material. This amount will allow for replicate analyses by methods using a 30 g aliquot if a problem is suspected;
- Sieve sample aliquot to completion, to avoid the artificial concentration of gold in the sample;
- Request that the GSC Sedimentology Lab sieves to completion the oversize field duplicate samples. This will ensure that there is sufficient material to create a [blind duplicate](#) from the field duplicate;
- Use stainless steel sieves with epoxy or silicon seal covering solder on both upper and underside edges of the screen;
- Sieve into a stainless steel pan, not onto paper or a plastic sheet;

- Pour the sieved fraction back into the sample dish that was used to air dry the sample; then, transfer this sieved fraction to the sample envelope ;
- Sieve and pan are to be cleaned with a brush and air hose between each sample;
- Pan must be wiped clean with distilled water between each sample;
- Sieve is to be cleaned with ultrasonic cleaner before the first sample and then after every 10th sample;

7.2.4 Clay separation of the <0.002 mm fraction (optional)

- Analysis of the <0.02 mm fraction is an optional, add-on procedure;
- Although costly and time consuming to recover, the <0.002 mm fraction of till may also be recovered and analysed for specific survey areas or commodities, such as uranium, because of its greater capacity to retain elements released during weathering and to avoid textural bias on geochemistry. Examples of previous GSC surveys that have analysed the clay-sized fraction include Kettles (1992), Kaszycki et al. (1996), and Plouffe et al. (2011);
- Clay separations should be completed following the procedures outlined in GSC Open File 4823 (Girard et al., 2004);
- The samples should be mixed with distilled water during clay separation and not with a defloculant such as sodium hexametaphosphate if the phosphorous concentrations are important to the project, or if selective extractions are to be carried out on the samples (Plouffe et al., 2001a);
- Centrifuge bottles and decanting equipment must be rinsed and cleaned with distilled water in between samples;
- Check with the GSC Sedimentology Lab for the number of times the bottle is used before new bottles are used - this may require adjustment;
- Desired aliquot for geochemical analysis is 2 g - usually this will require 500 g of till to be processed - this will have to be adjusted based on the clay content of till sample;
- Request that the GSC Sedimentology Lab prepares the entire oversize field duplicate sample. This will ensure that there is sufficient material to create a [blind duplicate](#) from the field duplicate;

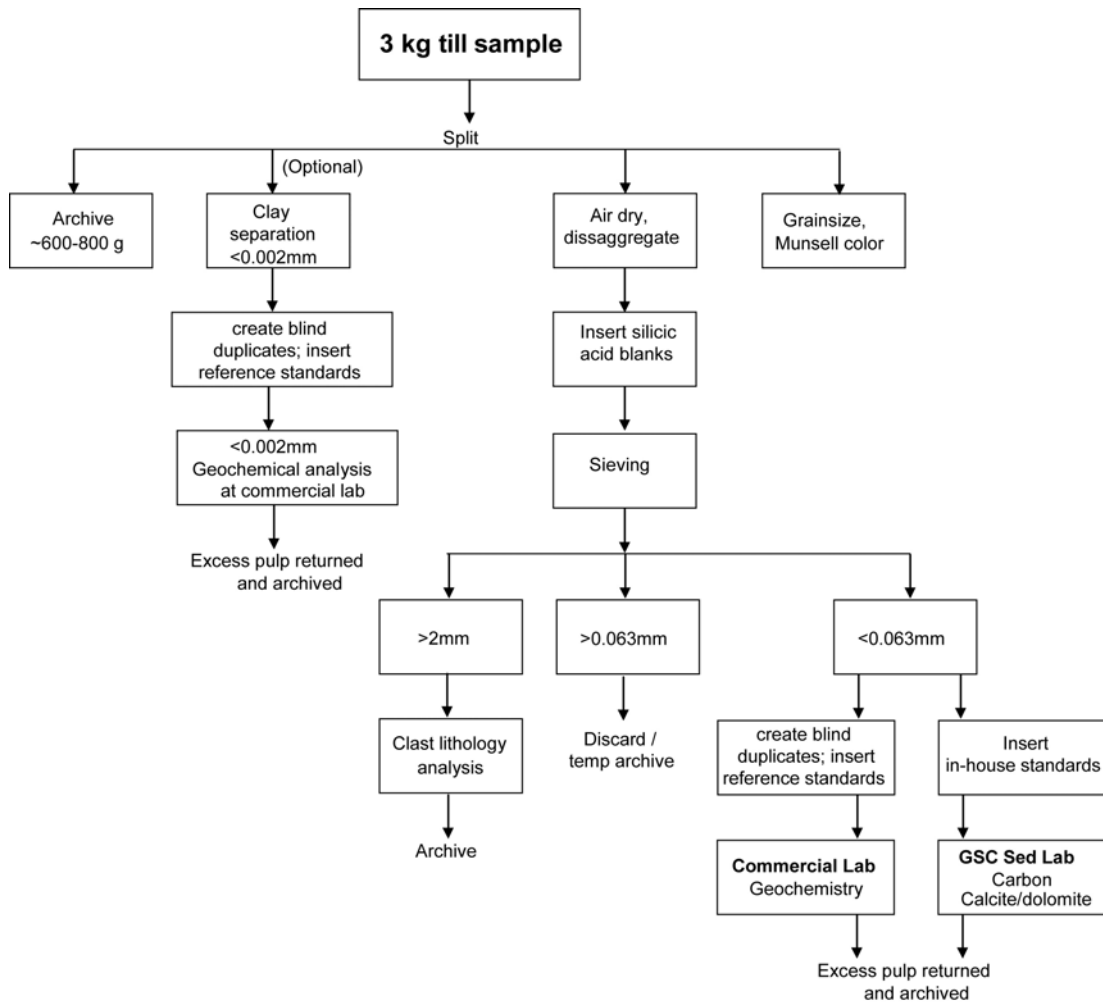


Figure 18: Flowchart outlining sample preparation steps at GSC Sedimentology Lab.

7.3 Sample Preparation at Commercial Lab

7.3.1 Silicic acid blank

- To monitor possible cross-contamination during sample preparation, silicic acid blanks should be used (provided by the GSC Sedimentology Lab). The blanks must be sieved at regular intervals within the batch of routine samples and using the same equipment as for the routine samples;
- If the samples are being shipped from the field to a commercial for preparation, there are two options for inserting the silicic acid blank into your batch:
 - i) Insert 400 g aliquots of unsieved blank regularly in the sample batch and label each one 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. Possible contamination can occur if the silicic acid aliquots are not prepared in the GSC Sedimentology Lab;

- ii) Ship the required number of 400 g aliquots of unsieved silicic acid blank to the commercial lab with instructions of how they are to be sieved within the sample batch of routine samples; before shipping to the commercial lab, label each aliquot using the sample numbering scheme already being used for the routine samples;
- Instruct the commercial lab to sieve approximately 400 g of silicic acid to create a 60 g lab preparation blank. If a problem is suspected, this amount will allow for replicate analyses by methods using a 30 g aliquot;
- As a minimum, have the commercial lab sieve the blanks at the beginning, middle and end of each batch of routine samples. If more blanks are used, they can be sieved at the beginning and end of the batch as well as regularly (every ~25 samples, for example) within the batch. The silicic acid blanks should be sieved more frequently within the batch if samples are suspected to be metal-rich;
- After sieving following the instructions in section [7.3.4](#) or [7.3.5](#), have the commercial lab ship the sieved routine and blank samples to the GSC Sedimentology Lab;
- The GSC Sedimentology Lab will submit the routine samples and the blanks, in the order they were sieved, for geochemical analysis to monitor possible cross-contamination.
- If you choose to randomize the samples after sample preparation and before geochemical analysis, the GSC Sedimentology Lab will ensure that the blanks are not all analysed in one group, but are interspersed within the batch;

7.3.2 Archiving

- Instruct commercial lab to set aside ~1 kg split prior to preparation of sample for geochemical analysis and ship the split directly to GSC Sedimentology Lab;
- At the GSC Sedimentology Lab, complete the last two steps in section [7.2.1](#);

7.3.3 Sample preparation

- Air dry sample at <40°C;
- Disaggregate sample in agate (not porcelain) mortar & pestle or inside unused plastic sample bag using rubber mallet - new bag for each sample;

7.3.4 Sieving to <0.063 mm

- Sieve 2 kg sample to completion, to avoid the artificial concentration of gold in sample;
- Request that the commercial lab sieves to completion the oversize field duplicate samples. This will ensure that there is sufficient material to create a [blind duplicate](#) from the field duplicate;
- Use stainless steel sieves with epoxy or silicon seal covering solder on both upper and underside edges of the screen;

- Sieve into stainless steel pan, not onto paper or plastic sheet;
- Pour sieved fraction into sample envelope or vial;
- Sieve & pan to be cleaned with brush and air hose between each sample;
- Pan must be wiped clean with distilled water between each sample;
- Sieve to be cleaned with ultrasonic cleaner prior to sieving any samples in batch, and at end of every day;
- Ship samples back to GSC Sedimentology Lab for insertion of reference standards and blind duplicates (see [Table 4](#)); note, the blind duplicate will be created by the GSC Sedimentology Lab by splitting the sieved oversize field duplicate sample;

7.3.5 Clay separation of the <0.002 mm fraction

- Use centrifuge to collect <0.002 mm fraction according to methods described in GSC Open File 4823 (Girard et al., 2004);
- Separate a 2 g aliquot for geochemical analysis;
- If clay-rich till, process approximately 500 g of sample material; if sand-rich till, process 1-2 kg;
- Request that the commercial lab prepares the entire oversize field duplicate samples. This will ensure that there is sufficient material to create a [blind duplicate](#) from the field duplicate;
- Centrifuge bottles and decanting equipment must be rinsed and cleaned in the sonic bath with distilled water between each sample;
- Determine the commercial lab protocols for replacing the centrifuge bottles; request that bottles that do not come clean after being washed in the sonic bath are not used for GSC samples;
- Ship samples back to GSC Sedimentology Lab for insertion of reference standards and blind duplicates (see [Table 4](#)); note, the blind duplicate will be created by the GSC Sedimentology Lab by splitting the prepared oversize field duplicate sample;

8. Geochemical and Other Analyses of the <0.063 mm Till Fraction

8.1 Recommended Minimum Requirements for GEM Projects

Having all GEM projects use the same analytical protocols and reference standards will allow direct comparison of analytical datasets between projects over the long term. Therefore, the same suite of standards should be used for each batch and over multiple years.

Two digestions are recommended as a minimum for analysis of the <0.063 mm till fraction: aqua regia and borate fusion total digestion. The use of aqua regia is recommended because the GSC has a long history of using this digestion to analyse till in mineral exploration focused surveys and its continued use allows for comparison with past survey results. Borate fusion total digestion is recommended to characterize total till geochemical signatures that are related to bedrock lithology.

8.1.1 Aqua regia digestion

Digest 30 g split in aqua regia, analyse by ICP-ES and ICP-MS for 65 elements, including the rare earth elements (REEs) suite.

8.1.2 Borate fusion total digestion

Major oxides by ICP-ES (lithium borate fusion), LOI at 1000°C and LECO® total carbon and sulphur analysis plus trace element analysis by ICP-MS (lithium borate fusion) including Cu, Mo, Ni, Pb and Zn.

This method is ideal for major oxides and trace elements including REE and other high field strength elements. This method has higher detection limits for some elements as compared to a 4-acid digestion.

Note: These analytical packages may also be applied to the <0.002 mm fraction.

8.2 Additional Geochemical Methods

8.2.1 Four acid digestion

Digest 0.25 g split in 4-acid digest (HF-HClO₄-HNO₃-HCl) with ultra-trace analysis by ICP-ES and ICP-MS for 55 elements including REEs.

This method is NOT suitable for rare earth elements. This digestion is used for Cu, Ni, Co, Pb, Zn, Mo and Ag assays. Note: the digestion is only partial for some Cr and Ba minerals and some oxides of Al, Fe, Hf, Mn, Sn, Ta, Zr. Volatilization during fuming may result in some loss of As, Sb and Au.

8.2.2 For magmatic Ni-Cu-PGE exploration, determination of Au+ PGE

- Use 30 g aliquot;
- Pb fire assay fusion for total decomposition, digestion of the Ag doré bead and ICP-MS analysis;

8.2.3 INAA for non-destructive analysis of <0.25 mm heavy mineral concentrate

- Note: lower detection limits are elevated compared to methods 8.1.1 and 8.1.2;
- Note: Au, As, Sb, Mo, W, and U are best determined using epithermal neutrons whereas the REEs are best determined using thermal neutrons;

8.2.4 For uranium exploration and speciation studies

- <0.063 mm fraction should be analysed;
- If additional funds are available, analyse the <0.002 mm fraction for greater contrast between background and anomalous values;
- Use a partial digestion using HNO₃ and HCl that is weaker than conventional aqua regia (3 HCl: HNO₃), in order to capture uranium tied up mainly in uranium oxides and loosely bound in weathered material; useful for speciation studies. Use either ICP-MS or Fluorimetry analytical methods to obtain low detection limits;
- Borate fusion/ICP-MS recommended in order to digest all minerals including REEs that often accompany uranium, to determine total uranium in the sample matrix;

8.3 GSC Sedimentology Lab Testing

8.3.1 Munsell colour

The GSC Sedimentology Laboratory is developing a new application in order to determine Munsell Color of soil samples. An SP64 X-RITE Spectrophotometer linked to IQC color software will be used for the measurement. Although Munsell notations can be calculated via the IQC software, the values computed represent the entire Munsell color chart and can't be restricted solely to the Munsell Soil Color sub-group. The laboratory has built its own Munsell Soil Color database by scanning each Munsell Soil Color chip contained in the handbook. Unknown samples are compared to the database and matched to the nearest color.

Preliminary results on dried samples revealed good agreement between the instrumental and the visual method. Determinations of Munsell Color on moist samples are proving to be more difficult. Note: this method is not described in the GSC Sedimentology Lab Manual, Open File 4823 (Girard et al., 2004).

8.3.2 Grain size analysis

- Minimum determination, % sand, silt, clay;
- Determined using Lecotrac LT100™ for the <0.063 mm fraction and the Camsizer® for the >0.063 mm fraction;
- QA/QC - duplicates are run for ±5% of each batch;

8.3.3 Total carbon, organic carbon and LOI

- Determined using LECO® and Chittick apparatus;
- QA/QC for Chittick – 10 to 20% standards of known CaCO₃ content; 5% duplicates;
- QA/QC for LECO® – 5 to 10% duplicates; 5% replicates; LECO® 12% standard (501-034) used for calibration of machine; CANMET standard SO-3;

For additional tests available, see GSC Sedimentology Lab Manual- GSC [Open File 4823](#)

9. Quality Assurance/Quality Control

Geochemical surveys are made up of many stages and it is important to control the quality at all stages. Having a well thought out quality assurance/quality control program for all steps, from sample collection to analysis, to data management and archiving ensures that no systematic bias is introduced into the project. The data can then be relied upon and are consistent for comparison with other datasets. A robust QA/QC program also saves time and money as it eliminates the need for repeat sampling and or analyses.

9.1 Precision and Accuracy

The Geological Survey of Canada has always been responsible for publishing large volumes of geochemical data and for ensuring its quality. Precision and accuracy are used to describe the data quality.

Accuracy reflects how close the analytical result is to the true value. Accuracy is evaluated by using reference materials, or standards, that have been inserted among the samples sent to the laboratory.

Both internationally certified reference materials (ICRM), also known as primary standards, and internal control reference samples (secondary standards) should be used. ICRMs are homogeneous materials that have been analysed by a large number of laboratories by various techniques. Internal control reference samples are prepared to be homogeneous and are chosen because they have a relatively consistent distribution of elements. The internal control reference samples must have been established as being suitable by analysing them multiple times with ICRMs. If the ICRM results agree with published data, then the internal reference can be used as a standard in future projects. As such, they should be prepared and analysed in the same manner as the sample material from the survey with which they are being analysed (Garrett, 1991). The choice of reference materials depends on the anticipated concentration levels. For example, is your survey area highly mineralized?; are you aware of what commodities might be present? Whenever possible, the matrix of reference samples should match the survey samples. Considerations are sample media, e.g. till vs. lake sediment, and carbonate content.

Precision is a measure of the reproducibility of the results. Precision can be evaluated by analysing duplicate samples, either collected at the same site, or prepared from the same field sample (Garrett, 1991). Precision can also be estimated from replicate analyses of the same sample.

The desired result is for the data to be both precise and accurate, but one does not automatically imply the other. For example, it is possible for data to be precise, but inaccurate.

9.2 Quality Control in the Field

A significant source of variability in the geochemical 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 is not contaminated. These include the following:

- Remove jewellery, especially rings, before sampling; or wear gloves;
- Ensure that your samplers and assistants are well-trained;
- Make sure sample bags or buckets are accurately and permanently labelled;
- Use a standardized field collection form so that the same minimum level of information will be collected by everyone; e.g., GanFeld. See also [Field Notes](#) section;
- Ensure accurate site location using GPS; make sure everyone uses the same datum;
- Review field notes and sample locations each night to be sure there are no obvious errors;
- Use sampling tools that are free from coatings or paint and that are cleaned between sample sites;
- Collect field duplicates to measure sample site variability; collect the duplicates from different holes (or from the same hole if it is large enough) but do not homogenize. One of the field duplicates can then be split during sample preparation and used as a blind duplicate which will measure analytical variability. Collect one field duplicate in each block of 20 samples. Duplicates can be flagged as such in GanFeld. See also [Till Sampling](#) section;
- When possible, check sample numbers on the bags against the sample list on a daily basis; check the master sample list against samples before shipping; (Note: checking sample numbers on a daily basis increases the chance of correcting sample numbering mistakes or noticing that samples are missing);

9.3 Quality Control in the Preparation Laboratory

9.3.1 Silicic acid blanks for till matrix geochemistry

- These blanks are used to monitor potential cross-contamination during sample sieving prior to geochemical analysis;
- The silicic acid blanks are provided and inserted into sample batches by the GSC Sedimentology Lab;
- Consists of silicic acid n-hydrate powder (not sand); composition in Table 3 below;
- Supplied by J.T. Baker® in 2.5 kg pail;
- Each pail has a unique ID number which the GSC Sedimentology Lab will track;
- Approximately 400 g of silicic acid is sieved to create each 60 g lab preparation blank. If a problem is suspected, this amount will allow for replicate analyses by methods using a 30 g aliquot;
- Amount needed for analytical aliquot (usually 30 g) is then placed in sample container and labelled using the sample numbering scheme already being used for the routine samples (see section [9.4.1](#));
- Analytical results for each blank will be reported by GSC Sedimentology Lab as part of their routine QA-QC reporting system;

Table 3: Approximate reported composition of silicic acid

Assay (as SiO ₂)	100%
Non-volatile with HF	0.07%
Chloride (Cl)	<0.01%
Sulfate (SO ₄)	<0.005%
Heavy Minerals (as Pb)	<0.002%
Iron (Fe)	<0.003%
Loss on Ignition (as H ₂ O)	12%

9.3.2 If using the GSC Sedimentology Lab for sample preparation

1. Enter your samples into the ESS Sample Management System (SMS);
2. Fill out a GSC Sedimentology Lab request form; check off “Archive” on the requisition to ensure the archive split is taken before the samples are prepared;
3. Flag your field duplicates on the request form; communicate to the lab how you want the numbering of your QA/QC samples handled;
4. Insert silicic acid blanks prior to sample preparation;
5. Always indicate a preparation order to the GSC Sedimentology Lab and the lab will keep track of this order - the default preparation order 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 [Sample Preparation](#) section;
7. Before submitting samples to the analytical lab, have the GSC Sedimentology Lab insert standards and blind duplicates as indicated in Table 4 below. Note that silicic acid blanks, the number of times the sieves are cleaned and the archiving of samples returned from the lab must be specifically requested. See also [Sample Preparation](#) section ;
8. If the samples are metal-rich, indicate an analytical order to the analytical lab, from least to most metal-rich;
9. When the pulps are returned to the GSC Sedimentology Lab from the analytical lab, they will be archived at Tunney’s Pasture;

9.3.3 If using an outside lab for sample preparation

Using an outside sample preparation lab is recommended only if the GSC Sedimentology Lab is unable to process your samples because of large volume.

1. Enter your samples into the ESS Sample Management System (SMS);
2. Insert silicic acid blanks prior to sample preparation;

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3. If the samples are suspected to be metal-rich, indicate a preparation order to the commercial laboratory, from least to most metal-rich to minimize the possibility of cross-contamination;
4. Samples must be physically prepared as indicated in the [Sample Preparation](#) section;
5. Indicate to the commercial laboratory the amount of material needed for geochemical analysis (e.g., 30 g of <0.063 mm and 2 g of <0.002 mm material) for each sample;
6. Request that the commercial lab sieves to completion the oversize field duplicate sample. This will ensure the GSC Sedimentology Lab has sufficient material to create a blind duplicate from the field duplicate;
7. After the samples are prepared at the commercial laboratory, they should be shipped back to the GSC Sedimentology laboratory to insert standards and blind duplicates as indicated in Table 4; note that the field duplicate will have to be split to make a blind duplicate and renumbered;
8. If you have metal-rich samples, indicate an analytical order to the analytical lab, from least to most metal-rich;
9. Request that the analytical lab returns the pulps;
10. After the pulps are returned from the analytical lab, ask the GSC Sedimentology Lab to archive them at Tunney's; they will find your samples in SMS and flag them as being archived;

Table 4: Notes about Archiving, Standards, Duplicates, Silicic Acid Blanks, Cleaning

Preparation Step	Details	Recommendations
Archive a split from the unprocessed sample (the GSC Sedimentology Lab fills a 1 pint container)	Must be requested on initial GSC Sedimentology Lab requisition.	800 g or the equivalent of filling up a fixed-size (1 pint) archive container.
Cleaning of the sieves	Air hose/brush done automatically after each sample, but ultrasonic cleaning is done at noon and at the end of every day. Must be specifically requested if different.	Air hose and brush the sieves after every sample. Ultrasonically clean the sieves at the beginning of each batch and at the end of every day.
Field duplicates (make sure they are flagged in your GSC Sedimentology Lab request form for QA/QC)	The duplicates will be prepared as routine samples and are part of the numbered field sequence.	1 per block of 20 samples
Primary standards	* TILL-4	1 per block of 20 samples at random intervals.
* see http://www.nrcan.gc.ca/mms-smm/tect-tech/ccrmp/cer-cer/till-1-4-eng.pdf for published, accepted values		
Secondary standards note: more secondary standards	No secondary standards are available at this time.	1 per block of 20 samples, at random intervals;

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Preparation Step	Details	Recommendations
than primary should be used		use extra primary standards until a secondary standards becomes available
Blind duplicates	The blind duplicate is created by splitting the sieved field duplicate in half. The appropriate aliquot size for geochemical analysis of the blind and field duplicate is then taken from each sieved split.	1 per block of 20 samples; <u>must be geochemically analysed away from the field duplicate from which it was split (suggest analysing it first in each analytical block of twenty samples; see Notes about Sample Numbering)</u> .
Silicic acid blanks	** Must be requested on the lab requisition; these blanks should be prepared with the regular samples and then geochemically analysed with the regular samples..	As many as you would like, but at minimum, 1 at the beginning, middle and end of each batch. Use more if you have metal-rich samples and insert them regularly throughout the batch. Silicic acid blanks can be obtained from the GSC Sedimentology Laboratory if using a commercial lab for sample preparation.
Commercial laboratory standards (if applicable)		Insertion rate determined by the commercial laboratory.
Archiving of pulps	The return of pulps must be requested on the analytical lab requisition. Archiving at Tunney's is done automatically if samples are prepared by the GSC Sedimentology Lab; archiving must be specifically requested of Alain Grenier if sample preparation is done by an outside laboratory.	Archive the pulps returned from the analytical laboratory at Tunney's Pasture warehouse. Inform Alain Grenier that the pulps will be shipped to Tunney's or bring him the pulps if they have been shipped back to you.

** Silicic acid blanks – when analysed, these blanks are important for monitoring cross-contamination between samples that may have occurred during preparation. Inserting blanks in the preparation sequence will also help purge after metal-rich samples but they should not be considered as a cleaning step. Once a sample preparation order has been established, based on the metal content of the samples, the number of silicic acid blanks to be inserted can be determined. The more metal-rich samples you have, the more silicic acid blanks you should use.

9.4 Notes about Sample Numbering

It is up to the geologist to choose a sample numbering scheme. This scheme must allow for inserting standards, duplicates and silicic acid blanks in the preparation stage and therefore sample numbers might need to be reserved for them while in the field. Pre-planning this scheme before you go in the field is encouraged.

If your numbering scheme does not allow for easy insertion of samples in the numerical suite after you return from the field, you will need to leave 3 sample numbers free per block of 20 samples while you are in the field. This allows for a field duplicate, a [blind duplicate](#) and a

standard (Friske and Hornbrook, 1991) to be inserted by the preparation lab. In addition, you will need to leave a few numbers blank (not necessarily one in every block of 20) for the insertion of silicic acid blanks, based on your needs.

For the field duplicate which measures site variability, using the successive sample number after the original sample number is recommended. Randomize their location between blocks, so that, for example, if you collect a field duplicate at sample number 8 (which is a duplicate of sample number 7) in the first block of twenty, do not collect the routine sample and its duplicate at sample numbers 27 and 28 in the next block (i.e. positions 7 and 8 of the second block). When the location of the field duplicate within each block of twenty is unpredictable, its relationship to the blind duplicate in the first position of every block is effectively random (Garrett, 2011).

Always reserve the first number in the block of twenty for the [blind duplicate](#) which is used to measure analytical variability. The result is that numbers 1, 21, 41 etc. are left blank/unused when you are in the field.

Another number in the block of twenty is reserved for the standard. Randomize the location of the standard between blocks so that it falls in a different location in each block of twenty. It is important that the standards are not grouped together, for example at the end of the batch you are sending to the analytical lab. Leave samples numbers blank at regular intervals for the silicic acid blanks.

Note: as no secondary standards are available at this time, only primary standards are being used. When the secondary standards are available, the insertion rate and numbering instructions will be updated.

If GanFeld's alpha numeric system is used, it is not as critical to reserve numbers while in the field because the standards, duplicates and silicic acid blanks can be added as an extra sample number at a station (e.g. 07-PMA-201-1 and 07-PMA-201-2 could be a routine and duplicate sample respectively and 07-PMA-230-1 and 07-PMA-230-2 could be a routine and a standard sample respectively). As long as the standards are inserted randomly and the blind duplicate and silicic acid blanks are inserted at regular intervals, this method will work.

Regardless of the numbering system chosen, it is critical to communicate with the preparation lab and to indicate how sample numbers for duplicates and standards are handled.

9.4.1 How to handle numbering the silicic acid blanks

As a minimum, the silicic acid blanks should be inserted at the beginning, middle and the end of the sample batch. If samples are metal-rich, it is advisable to insert them more often, regularly throughout the batch. The decision of how many silicic acid blanks to insert might not be determined until after you return from the field, therefore it is more difficult to decide how many numbers to reserve ahead of time. In this case, assign the silicic acid blanks 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 as the preparation lab will know they are blanks, but it is critical to ensure they occur throughout the batch and not all at the end.

9.5 Quality Control Once the Analyses are Completed

Once the geochemical results are received from the analytical laboratory, they should be sent to the GSC Sedimentology Lab to be entered in LIMS (Laboratory Information Management System). This will ensure that the geochemical results become part of the Northern Canada Division geochemical database. Also it greatly facilitates the generation of QA/QC reports. If standards and duplicates have been identified properly, a QA/QC report can be prepared using the QA/QC Add-on in MS-Excel written by Richard Laframboise (GSC). Different filters can be applied. Contact the GSC Sedimentology Lab for more details on how to install and use this functionality.

1. Evaluate the report. The GSC Sedimentology Lab will assist in identifying analytical problems, but it is essential that the reports are examined by the geologist;
2. Note: currently, calculations on and graphs of field duplicates are not available as a standard option, but can be done on a one-by-one basis. This functionality will be added to the module.
3. Plot historical results for primary standard TILL-4 vs. results obtained from your batch, as well as any secondary standards, once available. It is important to ensure that your standards are compared to standards that have been treated the same way (digestion and technique). LIMS currently plots 10% acceptance levels (default) and the percentage value can be changed by the user. LIMS is now being updated to also use standard deviations to define the acceptance level.

9.5.1 What if you suspect errors?

If you suspect that the analytical data are incorrect, see the manager of the GSC Sedimentology Lab. The manager will review the report with you and contact the analytical laboratory to have the samples in question re-analysed. More of the original sample can be prepared and analysed if there is a suspected problem with sample preparation.

9.5.2 Other QA/QC

A QA/QC analysis can be also be carried out using other statistical packages (e.g. SPSS, SYSTAT), but it is often more time consuming to set up and complete than using the QA/QC module in LIMS (described in Girard et al., 2004). An explanation of how to use analysis of variance to determine both sampling and analytical variability is outlined in Reimann et al. (2008) and software to undertake the calculations is given in Garrett and Chen (2007).

9.6 Metadata

If you are storing the field and analytical data in your own database or spreadsheet as well as in LIMS, it is important to ensure that the necessary metadata (field, sample preparation and analytical metadata) are also recorded. In addition to the sample number, geographic location and collected field parameters (see [Field Notes](#)), as a minimum, the following should also be stored in your database or outlined in your publication:

9.6.1 Sample and Project Metadata

- Sampling protocol –provide enough detail to allow someone to reproduce what you did;
- Sample medium and the number of samples of each type in a summary table, for example; useful to summarize number of samples of each type with respect to NTS sheet as well;
- Set your work in the context of the project or program it lies within;
- Funding source;
- If the data are published in places other than the current publication or if you are referring to background work reported in other publications, these should be clearly identified;

9.6.2 Sample Preparation Metadata

- Laboratory name;
- Methodology – describe in as much detail as possible;
- Number of samples prepared (i.e. was every collected sample prepared?);
- Include a reference to the techniques, if possible (e.g. Girard et al., 2004; Percival and Lindsay, 1997);

9.6.3 Analytical Metadata (bulk geochemistry)

- Laboratory name;
- Date the samples were analysed;
- Size fraction analysed;
- Aliquot weight;
- Digestion (if applicable) – be specific about ratio and type of acid(s);
- Analytical method;
- Upper and lower detection limits;
- Lab package name- use the package name the laboratory advertises in its catalogue;
- Lab package description - reproduce the description from the catalogue in your Open File (the catalogues are often hard to find at a later date; when labs are bought out, the new owner usually does not preserve the old catalogues on the web);

If you are collecting samples for indicator mineral (IM) processing, please also see the list of required [IM Metadata](#) in Chapter 10.

9.7 A Note about Publishing your Data

Publishing your data provides a permanent archive. Databases (corporate and personal), spreadsheets as well as backups (CD, DVD, network server, external hard drive) are ways to archive your data, but they are not necessarily permanent. Publishing your data ensures that there is a permanent record of your work which will be available even after you leave.

Open Files should publish all of the laboratory data, ideally as received from the lab, unless received with errors. The publishing of derived data should not be at the expense of publishing raw data – the raw data should be published in addition to any derived data.

10. Sampling and Processing of Glacial and Fluvial Sediments for Recovery of Indicator Minerals

These notes are applicable for till, glaciofluvial sediment and fluvial sediment samples. Refinements of this methodology may be necessary for detailed studies of indicator minerals from glacial sediment samples collected near mineralized zones. These suggested and recommended procedures are written for GSC staff. If the notes are consulted by provincial and territorial researchers, some specifics are not applicable (e.g., requirement of filing information in GSC heavy mineral database).

In the text of Chapter 10, the term laboratory refers to the heavy mineral processing laboratory unless otherwise noted.

10.1 Field Sample Collection

10.1.1 Quality control measures in the field

Basic quality control measures for field work outlined in the [QA/QC](#) section should be followed.

10.1.2 Tool maintenance

Ensure that sampling tools are thoroughly cleaned between each sample site to avoid cross contamination. This can be accomplished by rinsing the tools in any standing water; otherwise, a steel brush or a hard bristle brush and clean cloth could be used to ensure that no material adheres to the shovel between sample sites.

10.1.3 Sample size

Recommended sample size is dependent largely on glacial sediment texture, but also may be dictated by the range and type of analyses to be performed. In the field, consistent sample size is collected based on volume (e.g., full pail, full sample bag, etc.). The weight of the sediment will vary according to moisture content, sediment compaction and composition. As a general rule, a full 22 litre rock pail (if the pail is metallic, it should be lined with a thick plastic bag), equivalent to ca. 20-40 kg, might be required in regions where the till contains small amounts of sand-sized material (e.g., Western Canada Sedimentary Basin). In regions underlain by intrusive and metamorphic rocks (e.g., Canadian Shield), a 10 litre pail or large sample bag (10-20 kg) may suffice. Similarly, a 10 litre pail (approximately 12-25 kg) should be collected for glaciofluvial sediment samples. For the same volume, glaciofluvial sediment samples are usually heavier than till. Preferably, glaciofluvial sediment samples should be collected with a sieve to remove the coarse fraction (i.e. >2.5 cm). The best facies to sample for recovering indicator minerals from glaciofluvial sediments remains to be tested. Pebbles from the till or glaciofluvial sediments can be retained for subsequent pebble counting.

10.1.4 Duplicate samples

It is recommended that approximately two percent of the samples be field duplicates. For duplicate samples, select a site easily accessible and easy to sample. Field duplicates can be collected for two reasons:

- i) Measuring laboratory precision. To measure laboratory precision, the following procedure is recommended: the routine sample and the field duplicate are collected from the same hole or sediment exposure, mixed together in the field and then divided into two separate containers with different sample numbers. Mixing the samples together will reduce sediment heterogeneity such that the laboratory precision at recovering and identifying indicator minerals is better assessed. In some cases, mixing the two samples might not be feasible because of sediment compaction or wetness. In this case, the duplicate samples will measure a combination of sediment heterogeneity and laboratory precision (see below). If no duplicate samples were collected in the field, one larger sample could be well-mixed and split into two smaller samples in the field or in the office prior to sending the samples to the laboratory.
- ii) Measuring field variability. To measure field variability, the following procedure is recommended: the routine sample and the field duplicate are collected from different holes or sediment exposures within 10 m of each other, not mixed but placed into two separate containers with different sample numbers.

The procedure followed (i or ii) when collecting each field duplicate should be recorded.

10.1.5 Labelling the samples

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

10.2 Preparing to Send Samples for Heavy Mineral Separation and Identification

10.2.1 Blank samples

Contact the GSC Sedimentology Laboratory to obtain a blank sample collected from Bathurst New Brunswick: 09-PTA-001. Insert the first blank sample at the beginning of each sample batch. This blank sample should be labelled with a sample code similar to the rest of the samples. This first blank sample will be used to monitor cross-contamination from previously processed batches in the laboratory. The blank cannot 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 50 samples in order to assess the potential for cross contamination in between samples. If less than 50 samples are submitted in a batch, it is

recommended that one blank should be inserted at the beginning and a second one part way through the batch. A blank till sample (09-PTA-001) should not be inserted immediately after a spiked sample.

10.2.2 Spiked samples

Insert one spiked till sample per group of 50 samples or equivalent to two percent. The spiked mineral species used should be the ones expected to be encountered in the survey area. It is recommended that mineral grains recovered from glacial sediments are used to spike the samples. Otherwise, mineral grains obtained from crushed bedrock can be used but those are generally easy to recognize because they have fresh angular surfaces. Ideally, spiked mineral grains should be etched with a laser and/or well imaged using an SEM so that each spiked grain can be identified once recovered. Also, density beads or cubes can be used for spiking. Density beads should be picked out of heavy mineral concentrates and recovered for re-use.

Staff members of GSC Northern Division are developing a GSC mineral grain library for spiking samples. Please let any of the working group members know if you have grains that you can donate to this library (e.g., KIMs, gold grains, sulphides, etc.). To develop the grain library and keep it active, please ask the laboratory to pick indicator minerals whenever possible. Grains which do not have to be further analysed and which have no more scientific value, could be placed in the grain library. Any spiked grains recovered during sample processing should be returned to the mineral grain library.

A till standard (till sample with a known indicator mineral content) can be spiked. Two sites in the Ottawa area have been tested and one (09-PTA-002) has been chosen for a till standard that will be used for spiking. This work is in progress.

The spiked mineral grains should be inserted in the middle of the till sample and not at the top of the pail or the bag. Inserting in the middle will ensure that the spiked grains are not removed when the laboratory collects an initial 500 g of material for archiving from the heavy mineral sample. Spiked grains should be added to the standard till sample in a clean environment in the field or in the office prior to shipping the material to the laboratory. Part of the sample may need to be removed from the pail to insert the spiked grains at depth in the pail.

The spiked and blank samples can be inserted in a sample batch in the field or after the field season depending on logistics. Preferably, the spiked and blank samples should be sent to the laboratory with other samples and not separately so that they cannot be identified by the laboratory's staff.

10.2.3 Numbering system and order of analysis

The numbering system on the blank and spiked samples should be the same as the rest of the sample set. The order in which all 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 to allow calibration drift or evolution in indicator mineral selection criteria to be distinguished from subtle regional trends.

10.3 Recommended Laboratory Procedures

Sample processing and indicator mineral picking are usually not done at the GSC Sedimentology Laboratory but instead at a commercial laboratory. In recent years, standing offers with a commercial laboratory have been established. Please consult with the GSC Sedimentology Laboratory to find out the status and details of the active standing offer for heavy mineral separation and analyses.

The heavy mineral concentrates from the indicator mineral samples should be prepared using the shaking table and heavy liquids on the <2.0 mm sized-fraction. Using a heavy liquid separation at a 3.2 specific gravity (SG) is recommended. However, separations at 3.3, 2.9 and other SGs are also available and can be used depending on project objectives.

The laboratory can be instructed to process the entire sample or only a portion of it. Arrangements can be made for archiving or disposing of unused material.

The heavy mineral processing laboratory can collect an 800 g representative sample of material prior to the heavy mineral separation. This procedure is not necessary if a representative sample was already collected by the geologist in the field or by the GSC Sedimentology Laboratory.

During the tabling phase of the heavy mineral separation procedure, the pebble fraction can be obtained. A specific pebble size fraction (e.g., 8-16 mm) can be requested from the laboratory for future pebble lithological examination depending on the geological terranes that need to be recognized in that size fraction. An oxalic acid bath may be required to wash the pebbles obtained from oxidized sediment.

Visual indicator mineral identifications are commonly carried out on three size fractions: the 2-1 mm, 1-0.5 mm and 0.5-0.25 mm nonferromagnetic heavy mineral fractions. Heavy mineral identifications on smaller size fractions (e.g., 0.25-0.18 mm) can be carried out in consultation with the laboratory. The <0.25 mm table concentrate should be processed to completion using heavy liquids and ferromagnetic separation to produce a <0.25 mm nonferromagnetic heavy mineral fraction for further picking, mineral liberation analysis (MLA) and/or geochemical analysis. Visual identification of pyrrhotite may also be completed on the 0.25-2.0 mm ferromagnetic fraction at special request.

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 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 data files.

Gold, platinum group mineral (PGM), sulphide and uranium mineral grain counts can be conducted on the heavy mineral samples by panning. This procedure can be completed on the entire shaking table concentrate prior to heavy liquid separation, or on the <0.25 mm fraction after the complete heavy mineral separation and geochemical analyses (non-destructive instrumental neutron activation) are done. Gold grain shape should be characterized and size should be estimated. Panned indicator mineral grains may be returned to the concentrate before

it is processed further or set aside in a small plastic vial. Each geologist is responsible for communicating to the processing lab which option they prefer.

Kimberlite, metamorphosed/magmatic massive sulphide (MMSIM®) or other indicator minerals can be identified in the heavy mineral concentrates. It is recommended that expected heavy minerals and the general geological setting from which the samples were collected be communicated to the laboratory. The specific requested indicator mineral identification (e.g., MMSIM®, KIM or others) is project dependent.

10.4 Laboratory Data Reporting

The processing laboratory must report:

- A sample processing flow sheet for every sample batch which should include any modification to the standard heavy mineral separation method;
- The magnetic separations performed and equipment used, with its settings;
- The sample processing order;
- Internal quality assurance/quality control procedures followed and their results;

As indicated in section 10.3, if only a split of the heavy mineral picking fraction was examined, the laboratory report should include:

- The weight of the split picked;
- Grain abundances for the split;

10.5 After Receiving Data from the Laboratory

10.5.1 Replicate mineral counts

To verify the reproducibility of indicator mineral grain counts, heavy mineral concentrates should be re-submitted for picking. If this procedure is followed, the sample numbers should be changed so that the mineralogy lab does not know which samples are being re-submitted. At least 5 % of carefully selected samples that reflect a range of concentrations of the indicator minerals sought should be resubmitted for re-picking.

10.5.2 Data archiving

Once indicator mineral results are published by GSC, the [raw](#) heavy mineral lab data files should be submitted for entry into the GSC indicator mineral database.

10.5.3 Sample archiving

Once picking and analytical work is completed, project or activity leaders are responsible for properly archiving heavy mineral concentrates, picked indicator minerals, and other fractions at the GSC. The GSC Sedimentology Laboratory should be contacted in this matter. Metadata information on all archived material should be entered in the Sample Management System (SMS).

10.5.4 Metadata for Indicator Mineral Data

In addition to the metadata indicated in the [Field Notes](#) section and the sample, project and preparation metadata indicated in [Section 9.6](#), open file reports and other publications reporting indicator mineral data should include the following metadata about the samples:

- Sample medium: till, glaciofluvial sediments, stream sediments, or indicate if it is another sample medium;
- Name of processing laboratory, and name of picking laboratory if different;
- Weight of material processed for recovery of indicator minerals (e.g. table feed);
- Pre-concentration method: panning, hydro-separator, shaking table, dense media separator, Knelson concentrator, jig, rotary spinal concentrator, other;
- Method for disaggregation and name of laboratory: crusher, Electric pulse disaggregator (EPD), other method;
- Heavy liquid separation: type and density;
- Magnetic separation: hand magnet, Frantz, roll magnet, other;
- List all size fractions prepared and their weights;
- Weight and size range of fraction(s) picked for indicator minerals : % of concentrate examined for each sample;
- Mineral identification method: binocular microscope, mineral liberation analysis (MLA), SEM, or other methods;
- Mineral chemistry determination method and lab name: electron microprobe (EMP), SEM, LA-ICP-MS, other;
- Raw mineral count data as reported by the picking laboratory;
- Mineral count data as confirmed by EMP, SEM or other methods;
- Report mineral count data as values normalized to total sediment weight processed: (e.g. number of grains per 10 kg table feed). Normalization should be based on the average weight of samples;

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