



# Exploration07



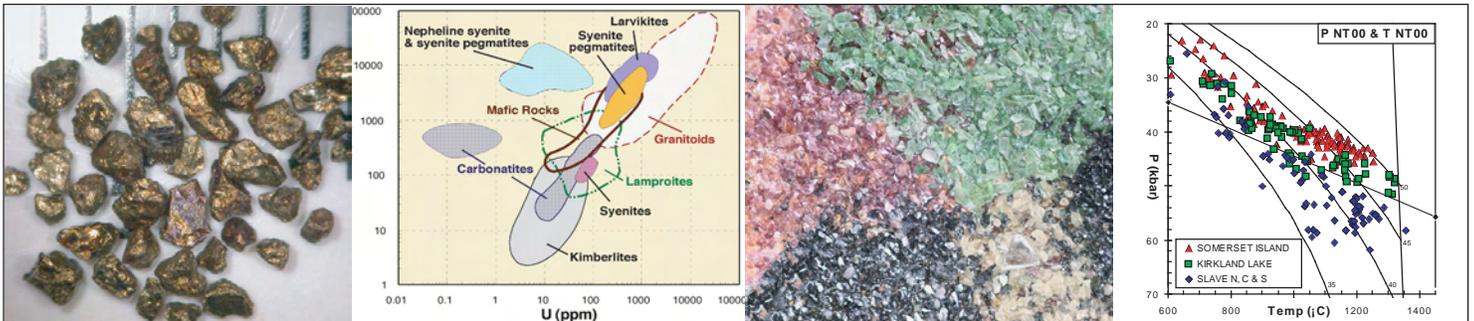
Exploration in the new Millennium  
5th Decennial International Conference on Mineral Exploration  
Toronto, Canada



## Workshop 3: Indicator Mineral Methods in Mineral Exploration

Convenors: L.H. Thorleifson and M.B. McClenaghan

September 9, 2007





# Workshop 3

## Indicator Mineral Methods in Mineral Exploration

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# Introduction to Exploration 07 Workshop 3: Indicator mineral methods in mineral exploration

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## ABSTRACT

In recent years, the application and effectiveness of indicator mineral methods in mineral exploration has been steadily increasing, and their scope has been expanding from well established procedures, such as those for gold grains and kimberlite indicator minerals, to new approaches for targets such as base metals. These methods, which rely on mineral grains suggestive of a possible mineral deposit in the rocks from which they were derived, are based on sampling of sediments such as glacial and stream sediments and detection of mineral deposit indicators dispersed by mechanical processes. These approaches are part of a spectrum of clastic-sediment-based methods ranging from boulder tracing to detection of detrital debris or their weathering products by chemical analysis of C-horizon soils and sediments. To summarize the current state of these methods, the *Exploration 07* workshop on indicator mineral methods in mineral exploration includes, in addition to this introduction, presentations on survey design, sample processing, mineral chemistry, QA/QC, indicator mineral methods in precious metal exploration, diamond exploration, and base metal exploration, a laboratory case study on sample representativity and integrity, an exploration case study, and a public sector case study dealing with an indicator mineral survey of the State of Minnesota.

## INTRODUCTION

Mineral exploration methods that are applied directly to the prospective rocks, at the preliminary stages of grass-roots prospecting and the advanced stages of property evaluation, include visual inspection, petrography, and litho-geochemistry. Intermediate between these phases of exploration are methods for remote detection of mineral deposits, achieved by geophysical or satellite observation methods, or by the detection of mineral deposit indicators that have been transported from their source. Application of exploration geophysical methods is directed at recognition of the mineral deposit and/or associated alteration, through detection of electrical, gravity, magnetic, seismic, or thermal properties. In contrast, geochemical and indicator mineral methods involve tracing of material dispersed from source. Whereas what might be regarded as purely geochemical methods rely on indicators that have been dispersed from source by aqueous and gaseous chemical processes, indicator mineral methods are those based on clastic indicators that have been dispersed from source by mechanical processes. Elemental analyses of, for example, soils may be used to detect a combination of chemical and clastic signals, although a strategy usually is directed at detecting one or the other. For example, the B horizon might be sampled and appropriate analytical procedures applied to seek a dominantly chemical signal, while the C horizon might be sampled and analyzed to seek primarily a clastic signal residing in mineral grains or their weathering products. In the case of visible indicator mineral grains, however, the signal is attributable to mechanical dispersal

processes alone. Hence indicator minerals methods can be regarded as part of a spectrum of clastic-sediment-based methods that range from boulder tracing, to pebble counts, to indicator minerals, to elemental methods designed to detect mechanically dispersed mineral grains and/or their weathering products. Recent syntheses have reviewed the application of indicator mineral methods in general (Thorleifson & McClenaghan 2003; McClenaghan 2005; Paulen & McMartin 2007) to both glacial sediments (Kujansuu & Saarnisto 1990; Kauranne *et al.* 1992; McClenaghan *et al.* 1997; McClenaghan & Kjarsgaard 2001) and stream sediments (Hale & Plant 1994; Fletcher 1997).

## CLASTIC SEDIMENTARY PROCESSES

Weathered and unweathered rocks exposed at the bedrock surface, as well as previously deposited sediments, may be eroded and transported by flowing water, wind, or ice, as well as by slope failure. Clastic sediments, as distinguished from dissolved load, are transported by suspension, saltation, and traction by water and wind, or by entrainment and shear by ice. These processes occur in colluvial, glacial, eolian, alluvial, deltaic, shoreline, shallow water, and deep water environments, and generate variably sorted deposits of clay, silt, sand, and gravel, which range in maturity from those containing minerals highly susceptible to weathering in earth surface environments, to those consisting only of resistate minerals concentrated as a result of both chemical weathering and abrasion. Sediment transport begins with a first cycle from source, followed by multiple cycles of reworking and mixing. An indicator and its source define

a vector or resultant vector of transport. During transport, some combination of comminution, weathering, and dilution change the character of the indicator and its host sediment, as discussed with respect to glacial sedimentary processes and sediment composition by, for example, Eyles *et al.* (1983), Klassen (1997), Boulton *et al.* (2001), as well as by Larson and Mooers (2004, 2005).

### Textural partitioning

Clastic mineral grains are partitioned into characteristic size fractions in which they preferentially occur by abrasion, crushing, and weathering. The most useful indicator minerals are those that preferentially occur in the readily visible medium to very coarse-sand size ranges, such as kimberlite indicator minerals. In glacial sediments, base metals preferentially occur in clay-sized (<0.002 mm) sediment (Shilts 1996), while gold and platinum group elements (PGE) tend to concentrate in the silt (DiLabio 1988; Nevalainen 1989). In the fine fraction of sediment (silt + clay, <0.06 mm), elemental variations may reflect glacial processes, provenance, or weathering (Shilts 1996). In addition, the proportion of clay in the fine fraction may strongly influence metal contents to the degree that elemental concentrations reflect textural changes (Lintinen 1995; Shilts 1996). Metal enrichment in the clay fraction in aerated sediments implies scavenging of metals liberated by oxidation of sulphides (Shilts & Kettles 1990). Shilts (1996), however, noted that clay from unweathered till also is enriched in metals, and that cations hosted by resistant minerals also are enriched in the clay fraction, indicating that, in addition to scavenging, rocks that produce clay must be metal rich at source, due to mineralizing processes and alteration. In contrast, Cr in chromite, W in scheelite, and Sn in cassiterite tend to occur in coarser sand-size (0.25–2.0 mm) fractions (Shilts & Kettles 1990).

### Postdepositional weathering

In even the most recent deposits, postdepositional weathering will have altered labile minerals. In recently deposited sediments, sulphide minerals will have almost entirely been destroyed by oxidation approximately above the water or permafrost table, commonly to a depth of several metres (Shilts & Kettles 1990; Shilts 1996). Weathering in these sediments may produce a colour change at several metres depth from brown to grey, and an abrupt change in sulphide content may be observed at this colour change (Thorleifson & Kristjansson 1993). Oxidized sediment will contain a few surviving sulphide grains, pseudomorphs after sulphides, or grains coated with hydroxides. In Canada, strongly calcareous sediments are leached of carbonate above 0.5 to 0.8 m depth (Thorleifson & Kristjansson 1993), whereas in slightly calcareous sediments leaching may extend to several metres depth at well drained sites (Shilts & Kettles 1990).

## CLASTIC INDICATORS

Mineral deposits may manifest themselves in clastic sediments as rock fragments, mineral grains, or elements disseminated by postdepositional weathering, and this debris may have been derived from the potential ore itself, or from associated rocks such as those affected by alteration.

In addition to mineral deposit indicators, clastic sediments may also indicate the provenance of the sediments, through lithological, mineralogical, or elemental variables that can be associated with regional geology, and therefore serve as indicators of transport distance and direction trends, as well as degree of reworking, that will facilitate interpretation of survey data. Provenance indicators useful in correlating sediments to their bedrock source include lithology of the gravel fraction, heavy mineral identification and yield, mineralogy of the fine fraction, and diagnostic elements. Sediments, or a fraction of the sediment, may be distinguished as exotic, derived from outside the area of interest, or local debris, derived from within the terrane being assessed.

Indicator plumes, known as dispersal trains (DiLabio 1990a), have total size and contrast with background governed by size of the source, concentration at source, dilution, regional background, visual distinctiveness of the debris, or character of sediment-transport processes. More sensitive analytical methods produce a stronger signal, hence a larger detectable plume, through enlargement of sample size, by amplification through concentration of the textural, density, and/or magnetic fraction of the sediment in which the indicator preferentially resides, or more sensitive analytical methods. Sample spacing must provide for the detectable portion of a plume to be sampled at least once. A higher signal-to-noise ratio will enlarge the detectable plume, permitting wider sample spacing. High sediment supply causes dilution of the signal, in areas of readily eroded bedrock or vigorous erosional processes. Background level is related to the number of mineral deposits in the area, as well as large, low-grade sources, which interfere with the desired signal. The train is much larger than its bedrock source, so commonly it is much easier to detect than the source. In glaciated terrain, a single erosional event produces a ribbon of enriched sediment as wide as the source. A change in sediment-transport trend will produce a fan-shaped train, and subsequent changes in ice flow direction will result in more complex train shapes. In other sedimentary environments, there is less opportunity for changes in sediment-transport trends.

## SURVEY DESIGN

Exploration surveys carried out by industry test for the presence of mineralization, in order to aid decisions regarding property acquisition and follow-up. Mapping surveys typically done by government agencies serve as a reference for exploration by defining trends in background, identifying problematic areas, and by providing examples of anomalies. Research surveys, carried out to enhance methods, understand processes, and to improve the effectiveness of both exploration and mapping, most commonly are done as case studies around known mineral deposits.

### Media

In a region, an indicator mineral sampling medium is chosen that presents the best combination of availability and suitability. Sampling of multiple media, such as stream sediment, glaciofluvial sediment, and till, should only be done if the media are distinguished in the field, in order to avoid

collecting multiple clear signals that together would be blurred. Stream sediments are available in most environments, while glacial, glaciofluvial, and fluvial sediments are obtained in glaciated terrain, and, in unglaciated environments, sampling of uppermost soil or loam sampling permits detection of deflation concentrates. Shoreline sediments may offer sampling opportunities on any of these deposits. Indicator mineral surveys tend to rely on sampling of active sediments or of C-horizon or deeper sediments in soil profiles, in order to obtain well preserved mineral grains for visual inspection, and also to obtain material for elemental analysis that will reveal a clastic rather than a chemical signal. In contrast, surveys directed at chemically dispersed signals tend to sample A or B soil horizons, or media such as vegetation, gases, organic lake sediments, or water.

### Site layout

Mapping surveys commonly use regular sample spacing from one to tens of kilometres to obtain an unbiased overview of an area. In exploration based on glacial sediments or other broadly distributed sediments, plumes are most likely to be intersected by a series of transects perpendicular to transport trend, with spacing along lines shorter than the space between lines. For stream sediments, samples might be obtained from a series of sites along the channel and at tributary mouths. Spacing will depend on the scale of terrane being tested, the size of the deposit or cluster being sought, the style of dispersal in the area, sampling medium, and sensitivity of the method. Research surveys typically simulate discovery by obtaining a case study at a known deposit.

### Sample size

Because visible indicator minerals commonly occur at an expected frequency of about one grain per litre of sediment, samples on the order of 10 litres or more of sediment are required (Clifton *et al.* 1969).

## SAMPLE COLLECTION AND PREPARATION

Active sediments such as stream sediments are sampled in the channel where accessible, taking into consideration textural trends and processes that will affect recovery of the desired indicators. In stabilized sediments, an excavation is required to remove soil or disturbed sediment prior to recovery of a large sample. Road construction may greatly aid sampling by stripping soil from ditches. If no exposure is available, a shovel may be used to reach depths up to approximately 1 m with limited environmental damage. A hand auger can be used to confirm the presence of the desired medium, prior to shovel excavation. Tools may be cleaned of coatings by sand blasting prior to a field season, and cleaned to an appropriate degree between sampling sites, and no contact with jewellery is permitted, to preserve the option of elemental analysis of the fine fraction. Where logistics permit, backhoe excavation to 3 to 5 m depth, or drilling to greater depths, enables sampling below stratified sediments or in thick sequences, and a vertical profile may be sampled to prevent dilution of or failure to detect thin dispersal trains (DiLabio 1990a). Sand may be screened in the field to remove the gravel, although

lithology should be noted, or a split retained. In the case of non-metallic indicator minerals, which tend to occur in the medium to very coarse sand fraction, sediment finer than medium sand may also be excluded by screening.

If sampling protocols are strict with respect to depth and medium, or if the medium is indicated by sample number, the only essential field data are locations plotted on a map. It is desirable, however, for additional observations that may aid interpretation to be made, including date of collection, name of collector, location name, GPS coordinates, sampling depth range, texture (sandy, silty, clayey), moisture (wet, moist, dry, frozen), structure (loose, soft, fissile, blocky, compact, hard), moist Munsell colour, reaction to 10% HCl (none, slight, moderate, strong), gravel fraction lithology, and site description. Indicator mineral samples are placed in pails or large bags, while a split for elemental analysis may be placed in a bag, or subsampled in the lab. Drilling is supervised by a geologist, and a log kept of all activity, including drilling rates. In the case of reverse circulation drilling, a stratigraphic and sedimentological analysis is made on site. During the processing of core, photographs of split core are taken and short segments of half core can be retained for reference prior to sampling of the entire remaining core for indicator mineral purposes. Randomization of samples prior to submission allows calibration drift or evolution in indicator mineral selection policies to be distinguished from subtle regional trends. Boulders that are mineralized or are provenance indicators may be recognized in the field by visual or other means, and features such as glacial striations can be recorded to aid interpretation.

At the laboratory, indicator mineral samples that may range broadly in size, but typically on the order of 10 litres, may be subsampled for a reference (~0.25 litre), as well as for fine fraction preparation and, in some case, moisture content (~0.25 litre). The remaining material is disaggregated, in some cases with the aid of agitation in a sodium hexametaphosphate (calgon) solution. Repeated washings may be required in carbonate or sulphate-rich samples to prevent flocculation. The gravel fraction (>2 mm) is then screened, and its lithology may be visually estimated. If quantitative lithological analysis is to be done, the gravel may be washed, dried, screened, e.g. at 4, 8, and 16 mm, and weighed prior to visual analysis.

## ANALYSIS FOR MINERALOGICAL INDICATORS

Several characteristics are required for a mineral to be ideally suited as an indicator. These characteristics include being coarse grained, derived almost exclusively from rocks being sought, visually distinctive, readily recovered, adequately abundant, and adequately resistant (Averill 2001). The mineral grains may be the native ore minerals that also represent the commodity itself, such as gold grains, may be ore minerals, or may be distinctive minerals associated with the commodity or source terrane of interest. Indicator minerals typically are reported as number of grains per sample. Reference to mass or volume of the sample or that of a fraction may in some cases be an effective alternative. Gold grains may be recovered and examined to determine their abundance, composition, size and shape

(Averill 1988; DiLabio 1990b; Grant *et al.* 1991). Useful indicators of kimberlite and lamproite and, in some cases in evaluation of diamond potential, include Cr-pyrope, Mg-ilmenite, Cr-spinel, eclogitic garnet, Cr-diopside, olivine, and, rarely, diamond. Kimberlite indicator minerals are recovered from the medium to very coarse sand-sized fraction of sediments, and analyzed by electron microprobe or similar instrument to determine concentrations of major oxides (Fipke 1989; Fipke *et al.* 1995; McKinlay *et al.* 1997; Morris *et al.* 1998; Lehtonen & Marmo 2002.), and in some cases also for trace elements using an instrument such as a proton microprobe or a laser ablation apparatus. Various other minerals indicative of mineral deposits and associated alteration include sulphides, cassiterite, chromite, scheelite, sulphides, apatite (Belousova *et al.* 2002), PGE-related minerals and minerals resulting from metamorphism of a deposit such as gahnite (Morris *et al.* 1997; Averill 2001; Karimzadeh Somarin 2004). The bulk composition of nonferromagnetic heavy mineral concentrates may also be used as an indication of source.

### Recovery

Indicator minerals are recovered from a sample using a series of laboratory procedures. Density preconcentration, by jig, table, spiral, dense media separator, or pan must be carefully designed and monitored, to ensure adequate recovery. Recognition of anomalous samples is advantageous, to prevent carry-over to subsequent samples. Density preconcentration may be combined with the use of an inexpensive heavy liquid, such as tetrabromoethane, prior to final preconcentration. If it is acceptable for gold grains to be lost, density preconcentration may be replaced by recovery by screening of the medium to very coarse sand, or rejection of nonparamagnetic minerals. Final density concentration is completed using methylene iodide (MI), commonly diluted with acetone to a density of 3.2, or a separating mechanism. The ferromagnetic fraction is then removed using a hand magnet or roll separator. With the exception of scheelite and zircon, which may be counted under short-wave ultraviolet light, and metallic minerals, such as gold and platinum group elements, which may be panned, indicator minerals are recovered from the concentrate by visual inspection by a knowledgeable person under a stereoscopic microscope. The 0.25 to 0.5 mm fraction is scanned separately from the 0.5 to 2.0 mm heavy minerals to prevent focus level and shadowing problems. Depending on regional mineralogy, paramagnetic sorting may be required, especially for the 0.25 to 0.50 fraction, in order to reduce number of grains to be scanned and to add information on the basis of degree of paramagnetism. The set of laboratory procedures being used will vary according to whether a single commodity or multiple commodities are being sought, and also the nature of the heavy mineral background. Quality assurance is monitored using spiked samples or repeat processing.

### Morphology

Gold grains may be intercepted for morphological analysis at two stages of processing. The grains may be panned,

counted, measured, and their morphology classified (e.g. Minter *et al.* 1993; Kinnunen 1996; Youngson 1998; Townley *et al.* 2003), to aid interpretation of distance and mode of transport, as pristine, modified, or reshaped (DiLabio 1990b) with the aid of optical microscopy after density preconcentration and prior to final density concentration, or concentrates may be panned after non-destructive INAA analysis and a few weeks to months of cool-down, using the Au analysis as a guide. Among the kimberlite indicator minerals, for example, Cr-pyrope is examined by optical methods or SEM are classified as those bearing remnant kelyphite, those with remnant sub-kelyphite pitted surfaces, angular grains, or grains that are rounded due to a morphology inherited from the kimberlite, or reworking from a sandstone (Mosig 1980; Afanasev *et al.* 1984; Garvie & Robinson 1984; McCandless 1990).

### Mineral chemistry

The chemistry of individual mineral grains, typically obtained at the percent level by electron microprobe analysis but also at the ppm level by proton microprobe (Griffin & Ryan 1995; Griffin *et al.* 1997), provides very significant information about the source. For example, gold grains may be characterized chemically (e.g. Chapman & Mortensen 2006), and in diamond exploration, peridotitic garnets are subdivided on the basis of Ca content into wehrlitic (high Ca), lherzolitic and harzburgitic (low Ca) affinities (Gurney 1984; Griffin *et al.* 1999; Grütter *et al.* 2004). Most peridotitic garnet inclusions in diamonds have low-Ca harzburgitic composition and thus these garnets are sought in diamond exploration, while similar largely empirical guidelines are also used to favour elevated Na<sub>2</sub>O levels in eclogitic garnet, as well as Cr-spinel with >60% Cr<sub>2</sub>O<sub>3</sub> and >12% MgO (Fipke *et al.* 1995). MgO and Cr<sub>2</sub>O<sub>3</sub> concentrations in ilmenites are used to determine probability of diamond preservation (McCallum and Vos 1993) and diopsides with >0.5% Cr<sub>2</sub>O<sub>3</sub> are classified as Cr-diopside (Fipke *et al.* 1995; Morris *et al.* 2002).

### ASSOCIATED LITHOLOGICAL INDICATORS

The effectiveness of an indicator mineral survey, and its interpretation, may be extended with the aid of recognition of mineralization or bedrock source provenance on the basis of examination of the gravel fraction of sediments, or as isolated boulders. These may be observed visually or by instrumental means, such as scintillometer in the field, or may be quantitatively determined by visual analysis of the gravel fraction. Lithological indicators in the gravel fraction may be reported as presence, visually estimated abundance, count percent, weight percent, or yield in mass per sediment mass. An appropriate split from one or more gravel textural fractions, such as 8 to 16 mm, 4 to 8 mm, or 2 to 4 mm, may be visually classified into lithological classes, and weighed or counted to obtain either percentage values or yield relative to the weight of sediment processed, ideally corrected for moisture content. The entire sample should be scanned for distinct indicator pebbles, and a representative split of 300 to 1000 clasts classified, depending on number of classes. A preclassification weight is used to check for post-classification data entry

errors. If only one size fraction is classified, a subset of the other gravel textural classes should be classified to demonstrate the correlation between fractions, such that data may be compared to other studies. Carbonate in the fine fraction of till may also be used as a provenance indicator. Inferences regarding regional provenance trends will significantly assist interpretation of indicator mineral results.

#### ASSOCIATED ELEMENTAL INDICATORS

A split of an indicator mineral sample may be processed for elemental analysis (Hall 1991; McClenaghan *et al.* 1997, 2002; McMartin & McClenaghan 2001), to extend recognition of clastic signals, and to help interpret provenance (e.g. McClenaghan 2001; McClenaghan & Kjarsgaard 2001; Woodruff *et al.* 2004; Lehtonen *et al.* 2005). As little as 0.1 litre of sediment or less may suffice, although collection of 1 litre or more is advisable if samples are being collected for elemental analysis only, to open analytical options, including analysis of a large aliquot for precious metals, or preparation of a clay or small heavy mineral concentrate. In the case of a program using elemental analysis only, a hand auger may be used to sample at about 1 m depth. Elemental analyses are used to detect the commodity of interest or elements that are associated with the target, whether that element is occurring in the minerals in which it occurs at source, or has been redistributed by postdepositional weathering. Elemental analyses are reported as %, ppm, or ppb in a specific textural, density, or magnetic fraction. Indicator elements commonly reside preferentially either in the clay fraction or in sand-sized heavy minerals. Analysis of whole sediment, to avoid missing a signal in a fraction other than the one analyzed, typically fails due to dilution of the signal, variable dilution causing false anomalies, the requirement for expensive crushing, and heterogeneity associated with the coarsest fractions. Analysis of the fraction in which the material of interest preferentially occurs produces the strongest signal to noise ratio, allowing detection of more subtle anomalies, and prevents errors caused by variations in the abundance of fractions poor in the material of interest. Analysis of the fine fraction (silt + clay), the least expensive option, in some cases will fail to generate an adequate signal. Metal concentrations in this fraction typically either represent diluted heavy minerals or diluted clay. It therefore commonly is necessary to resort to the more costly concentration of the clay or heavy mineral fractions, to obtain adequate signal to noise ratio. Elements concentrated in the sand fraction may be missed by analysis of the fine fraction. Although elemental analysis of the sand fraction is a possible solution, most sand-sized minerals of interest may be concentrated by density methods, so heavy mineral concentrates are more likely to be analyzed than sand fractions.

#### INTERPRETATION AND FOLLOW-UP

Data management begins with inspection of quality assurance monitoring, importation to a relational data structure, archiving of data, printing of tables and data displays, such as cumulative probability plots and proportional symbol maps, and exploratory data analysis, which brings into

play field observations and the geologist's experience and knowledge of the area. In exploration, various rules-of-thumb define the threshold between anomaly and background. Follow-up stimulated by positive results may involve better determination of sediment provenance, more detailed sampling, or a shift to other methods, such as geophysical surveys or diamond drilling.

#### SUMMARY

Methods for remote detection of mineral deposits include geophysics, geochemical methods based on aqueous and gaseous indicator dispersion, and clastic methods based on physical/mechanical dispersal of boulders, indicator minerals, and detrital debris or their weathering products detected by elemental analysis. Sediments bearing traceable clastic debris carried down the transport trend by glacial, fluvial, eolian, or colluvial processes, or in residuum, will be detected during exploration, mapping, or research, if sample spacing and signal to noise ratio are adequate. Plume size and contrast with background are governed by size of the source, concentration at source, dilution, background level, visual distinctiveness of the debris, and the nature of the processes of sediment transport. Detection method also affects signal-to-noise ratio in relation to sample size, the degree to which the textural, density, and/or magnetic fraction in which the target preferentially resides is concentrated, and analytical methods. Indicator minerals recovered for morphological and mineralogical analyses include those from kimberlite and lamproite, gold grains, sulphides, and other minerals indicative of mineral deposits and associated alteration. In addition to visual and mineralogical analysis of mineral grains, and to reduce cost or seek a signal not recoverable as mineral grains, elemental methods may be used to detect elements diagnostic of specific mineral grains or their weathering products, typically in a fraction defined by texture, density, and/or magnetic susceptibility. To avoid mixing clastic and chemical signals, C-horizon or deeper sediments in soil profiles, or active sediments, are sampled. The gravel, sand, and finer fractions may also be analyzed to trace provenance, in order to assist interpretation.

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# Design of indicator mineral surveys

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Survey design is the most important part of an indicator mineral survey; sample density, sample depth and sample medium must be chosen according to the needs of the exploration program. Survey design is critical because the actual field collection of samples is the most expensive part of an indicator mineral survey. The design should be optimized in order to cost effectively detect the signature of the deposit style that is being targeted. Although there are tremendous variations between indicator mineral surveys, the basic parameters are the same and are discussed below. There is an emphasis on survey design in glaciated terrains with some mention of survey design in arid terrains. Also there is an emphasis on survey design for kimberlite indicator minerals with some discussion for massive sulphide indicator minerals. Examples are drawn from work carried out by BHP Billiton.

Preliminary survey design should include identifying the sample medium that has a close relationship to the potential source. In glaciated terrains, this medium is till, which is a first-cycle sediment directly deposited by glacier ice. In temperate and arid terrains, the sample medium could be stream sediment from areas that have outcrop with potential for hosting the target mineralization. Covered terrains are more challenging and require an understanding of the type and thickness of the cover type so that an efficient sampling method can be determined.

Before undertaking the survey, a geological framework is needed and this should be in the form of a surficial geology map that shows the distribution, thickness and type of all surficial deposits. In many situations, this information is not available or only available at a large regional scale so consideration should be given to making custom maps from aerial photographs, topographic maps and digital elevation model (DEM) data, and/or remote sensing products, such as LANDSAT and global imagery available through viewers like Google Earth and Microsoft Virtual Earth. Figure 1 is an example of a surficial geology map constructed from aerial photographs that can be used to aid in the interpretation of results from an indicator mineral survey carried out in northern Canada (Stea 2006).

## Scale of Survey and Sample Density

### Glaciated terrains

The choice of the sampling scale is determined by the objective of the exploration program and can vary from large regional surveys at a density of 1 sample per 500 to 1000 km<sup>2</sup> to local surveys at 1 sample per 0.5 (in Table 1) to 5 km<sup>2</sup>. Table 1 summarizes typical sample spacing for surveys using till as the sample medium in northern Canada. The larger regional surveys can have a scale of one sample per 30 km<sup>2</sup> and a recent example is that of a heavy mineral sampling survey that covered the state of

Minnesota, USA (Thorleifson *et al.* 2007). Results from this scale of survey can be useful for environmental purposes, e.g. determining elevated levels of deleterious elements, as well as for helping to map regional bedrock geology and identifying mineral provinces.

For mineral exploration, sample spacing needs to be small and a first pass reconnaissance scale spacing is around 1 sample per 15 km<sup>2</sup>. In northern Canada, a typical first-pass approach for detecting kimberlites is based on a grid of 15 km x 15 km with a sample size of 30 kg. In northern Canada, the topography is subdued and has little influence on the design of the sampling survey, so a grid design is commonly adopted. A sampling grid that is regular in every direction is preferred where anomalies have no clearly defined length or where the character of sources is not well known. However, sometimes the sampling sites are constrained by extent and distribution of the surface till units and irregular grids are adopted. Occasionally in areas of elongate dispersal trains, the use of sampling lines can be more economical.

A reconnaissance sample spacing for indicator minerals for massive sulphides and other base metal styles of mineralization is typically much closer than that for kimberlite indicator minerals, at around 1 sample per 25 km<sup>2</sup>. The closer spacing is mainly due to the less resistant nature of some of the indicator minerals (e.g. sulphide minerals) and more restricted dispersal trains.

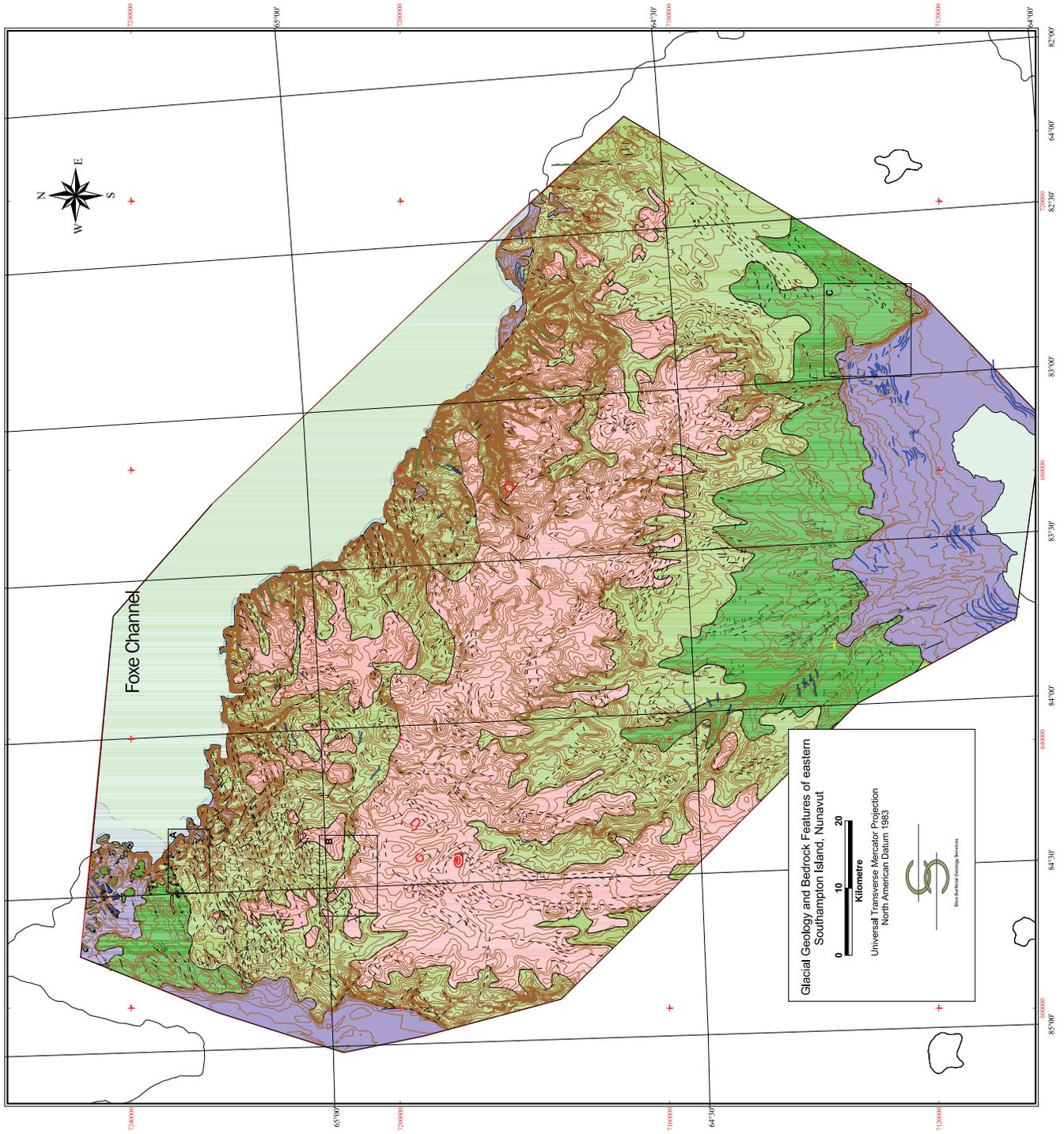
A regional scale 'follow up' to indicator minerals of interest from the reconnaissance-scale survey is carried out at a sample spacing of 1 sample per 25 km<sup>2</sup> with a sample size of 15 kg. Results from this scale of survey should define better the reconnaissance-scale signature and will provide clearer guidance for ground follow-up.

At a local scale, the sample spacing can be between 1 sample per 0.5 to 5 km<sup>2</sup>. Sample spacing as close as 250 m has been used in northern Canada in order to determine diamond potential of known kimberlites based on mineral chemistry. This approach can be very costly but can take a project to a key decision point very quickly. Slightly wider sample spacing of up to 1 km can be very useful in determining drill targets when they are used in conjunction with ground geophysics. Sample sizes at this scale of survey are usually about 15 kg of <2 mm material.

Where till is not available, alternative sample material in northern Canada can sometimes be stream sediment and paleobeach samples (reworked beach sand).

## TEMPERATE AND ARID TERRAINS

Stream sediments are the favoured sample medium in temperate and arid terrains and sample spacings are similar to those described in Table 1. However in many situations,



## Legend

### Pleistocene/Holocene glacial environment

Glaciomarine deposits: Sorted and unsorted deposits of stony clay and gravelly/sand deposited in a glacio-isostatic sea following the final retreat of major ice sheets.



Glaciomarine veneer: A; silty sand and stony silty clay generally forming a thin (<2m) and discontinuous veneer over bedrock (bedrock exposure 10-20%); B; discontinuous veneer over glacial till.



Glaciomarine blanket to veneer: Bouldery to pebbly gravel occurring as flights of beach ridges, grading downslope into massive and stratified sand, silt and clay. Overlying and grading into poorly-sorted stony, silty clay (glaciomarine diamicton) with shell fragments. Windows of thick unaltered or washed till deposits are found within marine limit. Mudboils are common; thick organic sequences near watercourses. On steep slopes, gelifluction processes produce slumps, solifluction lobes and stone stripes.



Till deposits: Unsorted deposits of silt/sand gravel/boulders, deposited directly by glacial ice.



Till veneer: Areas with a thin veneer of stony till over bedrock; 0.1 to 2m thick; 10-70% bedrock exposure; topography controlled by underlying bedrock; gradational to bouldery "regotill" consisting of angular to slightly abraded cobbles and boulders of largely local derivation.

Till blanket: Stony, sandy, clast to matrix-supported diamicton (till) forming featureless flat and rolling plain (till plain); 1-10m thick; extensively modified by gelifluction processes; till "boils" and frost fissures common.

### Pre-Pleistocene



Felsenmeer and regotill: Masses of angular boulders (felsenmeer) and angular cobbles and boulders in a sandy or muddy matrix residual from bedrock (regotill); material is largely in situ, with occasional erratic boulders; felsenmeer areas are found gradational to, and upslope of till and regotill regions.

### Symbols

	Drumlins; sense of flow not determined		U-shaped glaciated valleys
	Drumlins; direction of flow indicated by arrow		Meltwater channels
	Glacial striae; barb points in ice flow direction; data from Prest et al., 1968		Esker systems
	Crag and tail hills		Raised shorelines
	Stoss/lee bedrock hills		Topographic expression of geological structure, strike ridges, faults, joints etc.
	Glacial "megagrooves"		Circular bedrock structure
	Lineations of unknown origin		
	Morainal ridges		

### Map Notes

This map depicts the nature, distribution and thickness of Pleistocene/Holocene deposits on Southampton Island at the mouth of Hudsons Bay. These deposits were formed during and at the end of the last major glaciation (100-6 ka).

LANDSAT images as well as a compilation of data from previous regional scale maps were the sources of information used to create this map (references listed below). Satellite images were obtained from the GEOGRATIS (<http://geogratiss.cgdi.gc.ca/clf/en>) web site. This map is intended as a synoptic view of glacial deposits and glacial geology useful for determination of regional ice flow patterns. It should not be used for other purposes including detailed geological reconnaissance.

Prest, V. K., Grant, D. R., and Rampton, V. N., 1968. The Glacial Map of Canada, Geological Survey of Canada, Map 1253A Scale- 1:5,000,000.

Dredge, L. A., 2002. Quaternary Geology of Southern Melville Peninsula, Nunavut; Geological Survey of Canada, Bulletin 561, 109 p.

Boxes with letters refer to figures in report text: A- Figure 2; B-Figure 4; C-Figure 5

Fig. 1. Example of a surficial geological map, which can be used to aid in the interpretation of results from an indicator mineral survey.

**Table 1.** Sample spacing for indicator mineral surveys in northern Canada (till sampling).

Scale	Coverage	Spacing	Size of sample	Typical sample collection rate (helicopter supported)	Outcome
Regional	10000s km <sup>2</sup>	1 sample/500 km <sup>2</sup>	30 kg < 2 mm	10 samples/day	Produces regional information about geochemical provenances and dispersal trains over distinctive provenances.
Reconnaissance	1000s km <sup>2</sup>	1 sample/150 km <sup>2</sup>	30 kg < 2mm	30 samples/day	First step for mineral exploration in new province. Gives information about potential.
Regional	100s km <sup>2</sup>	1 sample/10-100 km <sup>2</sup>	15 kg < 2 mm	40 samples/day	May detect a mineral belt or large anomalies of till that have been transported 10 to 100s km
Local	10s km <sup>2</sup>	1 sample/0.5-5 km <sup>2</sup>	15 kg < 2 mm	50 samples/day	Outline mineralized ground or drill target definition

the density of the sampling is controlled by topography and there is often a tendency to take more samples than necessary at a regional scale. An example is given in Figure 2 of a survey for massive sulphide indicator minerals in the northwest United States where, although very effective, a higher than necessary density was used at the regional scale.

### Sampling depth and sample media

#### *Glaciated terrains*

The proportion of far-travelled material to locally derived debris typically increases upwards in till deposits so that concentrations of indicator minerals derived from a buried bedrock source increase with depth towards its source. As the surface part of the till blanket represents a wider source area, sampling should be close (0.5-1 m) to the till surface in reconnaissance- and regional-scale surveys in order to intersect the tail of the dispersion trail (McMartin & McClenaghan 2001). In local and detailed surveys, till sampling close to the bedrock surface is most effective because the composition of the till most closely resembles the underlying bedrock.

Complications can occur in the near-surface environment with surface weathering and oxidation effects and this makes it important to have proper identification and descriptions of the overburden and/or soil profile. For trace element geochemistry, the B horizon is usually sampled because many metals become enriched in this ferruginous horizon. However for recovery of indicator minerals, the C horizon developed on till is preferred because the minerals are usually fresh or weakly oxidized and form dispersal trains as a result of mechanical processes.

In the permafrost areas of northern Canada (north of the tree line), physical weathering is the dominant process in the near-surface zone and soils are generally thin and immature. An ideal sample site for indicator minerals is the mudboil, which can be recognized by bare or lichen patches surrounded by low ridges of rocks. This is relatively unweathered till material extruded to the surface because of high water pressures built up in the active layer of the permafrost zone (McMartin & McClenaghan 2001).

Another key important sample material is lodgement till, which is glacial debris smeared onto the bedrock surface by the movement of the glacier. This process occurs when the frictional drag between the bed and debris is more than the shear stress created by the moving ice. This stress is enough to inhibit further movement of the till.

There is little or no reworking by water and, because they are mainly a direct result of the last glacial event, tracing back the indicator mineral anomalies to source can be relatively straight forward.

In areas of very thin cover or extensive till cover, the most effective procedure is to dig pits with a shovel or pick. Quite often it is possible to find lodgement till within 1m of the surface and good sample sites are usually found on the down-ice side of bedrock highs when the younger surficial deposits (e.g. marine sediments or other glacial sediments) are often thin.

#### *Temperate and arid terrains*

Preferred sample sites are natural sites of concentration of heavy minerals in streams and rivers, such as rock barriers and point bars. Pan concentrates are sometimes collected at each site, although, because of the relatively low specific gravities of some indicator minerals, a sieved sample is usually preferred so that a more careful controlled separation can be carried out in the laboratory.

#### *Covered terrains*

This is a major problem for indicator mineral surveys in all terrains and can often lead to other exploration methods being employed. The only reasonably effective method for indicator minerals is to drill (usually reverse circulation) in an attempt to get a representative sample of lodgement till or, if not lodgement, at least some sort of till in glaciated terrains or material from a paleoweathered surface in arid terrains.

### Control Samples

All surveys should have at least two types of control samples. The first is a field duplicate, which is a second sample taken about every 15 to 20 samples and is treated in the same way as the first sample. These results will provide insights into precision of the field sampling technique. The second type of control sample is a standard, which is a spiked sample with a known quantity of indicator minerals – typically laser-etched garnets, when kimberlite indicator minerals are being traced, and gahnites, in the case of indicators for Broken Hill-type base metal deposits.

### Field Data Capture

Careful collection of field data is extremely important for interpretation and understanding limitations of the sampling technique. Improvement in technology means that any survey design should include provision for the use of a

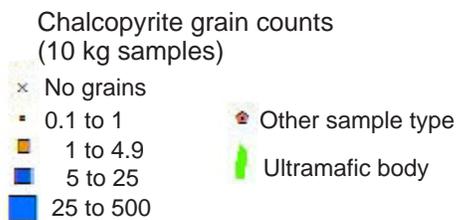
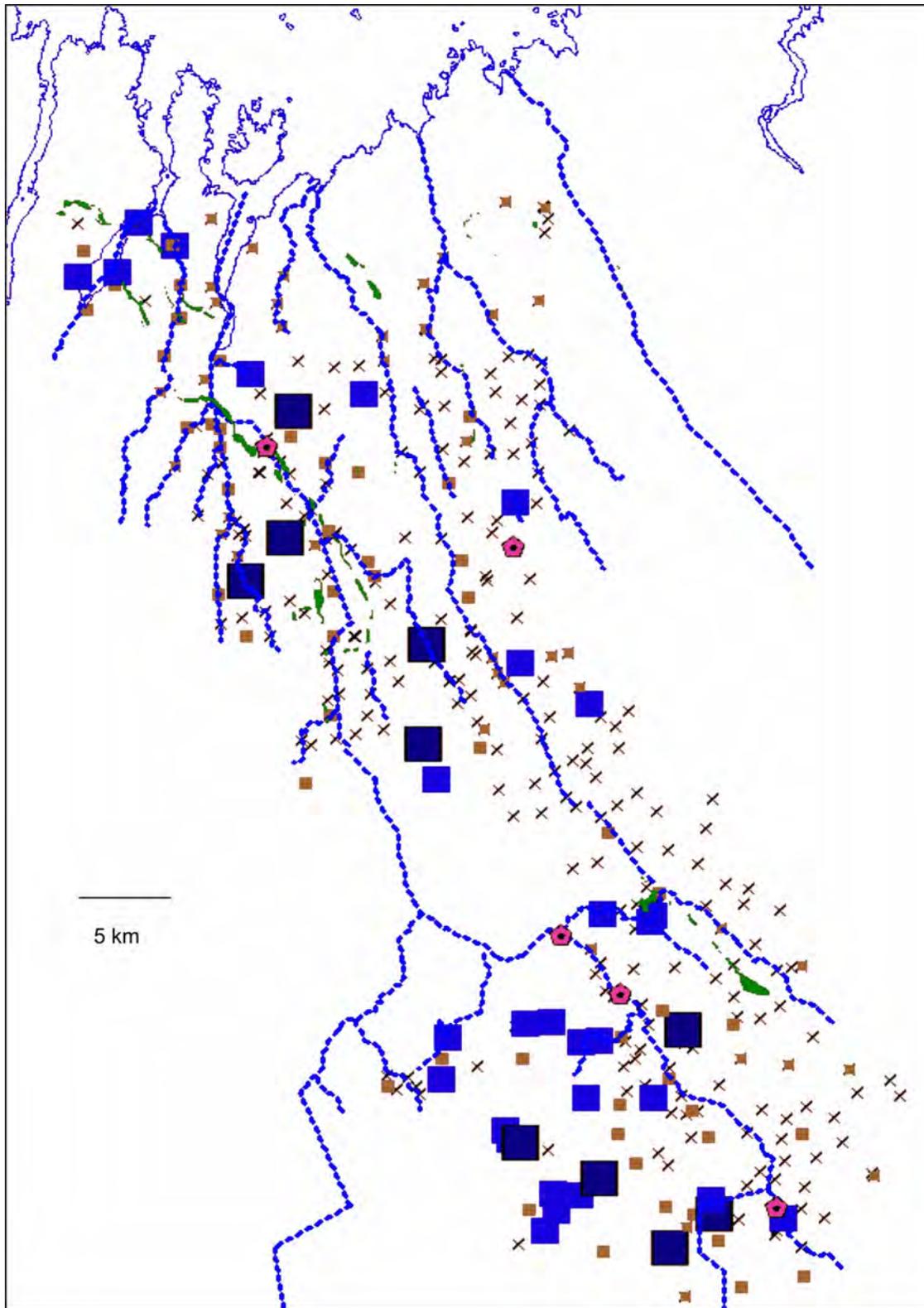


Fig. 2. Indicator mineral survey from northwestern United States showing an example of higher than necessary sample density.

**Table 2.** Recent costs for indicator mineral surveys for diamond and base metal exploration in northern Canada.

	Collection cost per sample	Analytical cost per sample	Overall cost per sample
Survey 1 (20 samples)	\$1,500.00	\$450.00	\$1,950.00
Survey 2 (280 samples)	\$1,200.00	\$310.00	\$1,510.00

Note: all costs are in Canadian dollars.

portable data capture device (PDAs). These devices provide more efficient, reliable and consistent descriptions of field samples. A key benefit is consistency of field descriptions, which leads to increased ability to map out sample parameters using a GIS.

There are several methods available and the Geological Survey of Canada have been successfully using an integrated field sample/data program called Ganfeld (Buller 2004). Simple entry methods can be made using software such as Microsoft Access or as part of a GIS package. Although this is a rapidly developing field and redundancy does occur rather quickly, the high cost of collecting sam-

ples and better interpretation of results easily justifies the extra cost of a field capture device.

### Cost of Surveys

The design of a survey is strongly influenced by the cost and Table 2 summarizes typical costs for recent helicopter-supported surveys in northern Canada. Costs in this region are particularly sensitive to the size of survey and sample density. In general, field collection costs are 70% of the total costs and this strongly reinforces the need for a carefully designed sampling survey.

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# Processing methods for recovery of indicator minerals from sediment and bedrock

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## INTRODUCTION

The application of indicator mineral methods to mineral exploration has grown and developed significantly over the past two decades. They are used around the world to explore for a broad spectrum of commodities. Heavy mineral suites now exist for detecting a variety of ore deposit types including diamond, gold, Ni-Cu, PGE, porphyry Cu, massive sulphide, uranium, and tungsten. Indicator minerals, including ore, accessory and alteration minerals, are usually sparsely distributed in their host rocks. They may be sparser in derived sediments, thus sediment samples must be concentrated in order to recover and examine them. Most indicator minerals have a moderate to high specific gravity, thus most processing techniques concentrate indicator minerals using some type of density separation, often in combination with sizing and/or magnetic separations. As few as one sand-sized grain of a particular indicator mineral in a sample may be significant. To recover such potentially small quantities (equivalent to ppb) of indicator minerals, samples are processed to reduce the volume of material that must be examined. In reducing the volume of material, processing techniques must be able to retain the indicator mineral(s) and do so without contaminating the sample, without losing indicator minerals, and at a reasonable cost.

Indicator minerals can be recovered from a variety of sample media, including stream, alluvial, glacial or eolian sediments and residual soils. They are also recovered from weathered and fresh bedrock as well as mineralized float. The combinations of processing techniques used by exploration companies or government agencies for recovering indicator minerals are quite variable (e.g. Gregory & White 1989; Peuraniemi 1990; Davison 1993; Towie & Seet 1995; Chernet *et al.* 1999; McClenaghan *et al.* 1999). This paper describes some of the common processing methods used to reduce sample weight, concentrate heavy minerals, and recover indicator minerals (Fig. 1), including those for deposits of diamond, precious and base metals, and uranium.

The methods used will depend on the commodities being sought as well as cost per sample. Most oxide and silicate indicator minerals (e.g. kimberlite, Ni-Cu-PGE, and metamorphosed massive sulphide indicator minerals; Averill 2001) are medium to coarse sand size (0.25 to 2.0 mm). Thus, concentration techniques that recover the sand-sized heavy minerals can be used. In contrast, approximately 90% of gold grains and platinum group minerals (PGMs) are silt sized (<0.063 mm), thus concentration of these indicators requires a preconcentration technique that

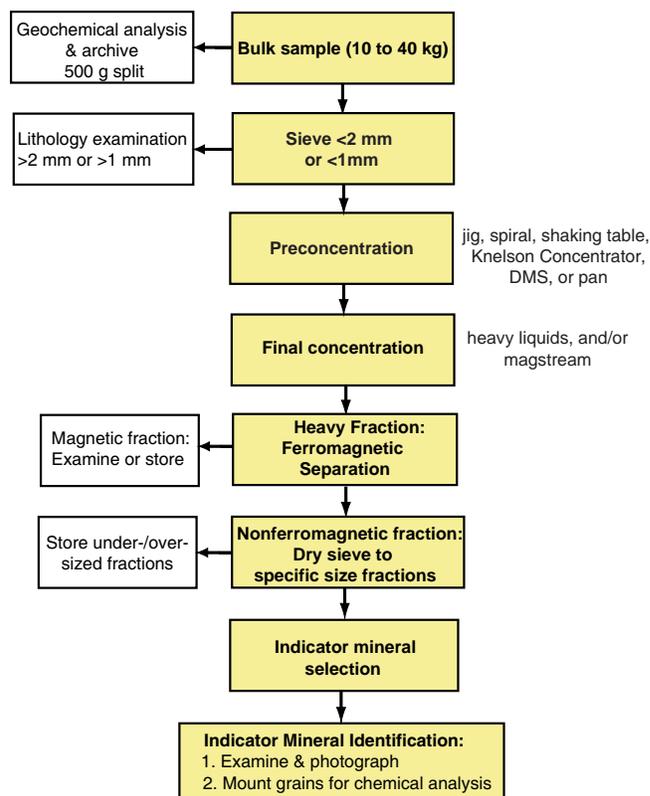


Fig. 1. Generalized flow sheet showing steps in sample processing used to reduce sample weight, concentrate heavy minerals, and recover indicator minerals.

includes recovery of the silt- as well as the sand-sized fractions.

## SAMPLE WEIGHT

The weights of material collected for indicator mineral studies will depend on the type of surficial sediment collected, the grain-size characteristics of the sample material, the commodity being sought and shipping costs (Table 1). For example, in glaciated terrain, clay-rich till samples may be up to 20 or 30 kg (or more) in order to recover a sufficient weight of sand-sized heavy minerals (Table 2, #5). Coarse-grained silty sand till typical of shield terrain requires smaller (10 to 15 kg) samples because it contains more sand-sized material in the matrix (Table 2, #1 to 4). Sediment samples collected for the recovery of porphyry Cu indicator minerals (PCIM) need only be approximately 0.5 kg because porphyry Cu alteration systems are large and rich in indicator minerals (Averill 2007). Bedrock and float samples usually vary from 1 to 10 kg.

**Table 1.** Examples of variation in sample weight and processing procedures with sample and target type at Overburden Drilling Management Ltd.'s heavy mineral processing lab (Averill & Huneault 2006).

Target	Typical Sample Weight (kg)	Required Separations				
		Table	Micropan	Heavy Liquid (Specific Gravity)	Ferro-magnetic	Para-magnetic
<b>A. Sediment Samples</b>						
Gold	10	Single	Yes	3.3	Yes	No
Kimberlite	10-30	Double	No	3.2	Yes	Yes
Massive sulphides (Ni-Cu-PGE, BHT, VMS, IOCG, MVT, skarn)	10	Single	Yes (PGE only)	3.2	Yes	Yes
Porphyry Cu	0.5	No	No	2.8, 3.2	Yes	Two
Uranium	10	Single	Yes	3.3	Yes	No
Heavy mineral sands (grade evaluation)	20	Triple	No	3.3	Yes	Optional
Tampering (investigation)	Variable	Optional	Yes	3.3	Yes	Optional
<b>B. Rock Samples</b>						
Gold, PGE, base metals	1	Optional	Yes	3.3	Yes	Optional
Kimberlite	1-10	Optional	No	3.2	Yes	Yes
Tampering (investigation)	1	No	Yes	3.3	Yes	Optional

### BEDROCK PREPARATION

Bedrock and float samples often need to be crushed prior to processing to recover heavy minerals. Crushing reduces rock fragment size to about 2 mm or the average size of mineral grains in the sample. Rock crushers can be difficult to clean. Barren quartz can be crushed as a blank between each sample to reduce contamination. To monitor heavy mineral carry-over, the blanks can then be processed and examined along with the actual sample concentrates.

### FIELD PRECONCENTRATION

If sample shipping costs are an issue, samples may be partly processed in the field to reduce the weight of material shipped to the lab. Samples may be sieved to remove the coarse (>1 or >2 mm) fraction, which may reduce weights from a few percent to 30% (e.g. Table 2, columns B-C). Preconcentrating, using a pan, jig, sluice box or Knelson concentrator, also may be carried out in the field to further reduce the weight of material to be shipped. Preconcentrates may be examined in the field, significantly

reducing the time to obtain results for follow up. However, preconcentrating in the field can itself be expensive and time consuming and the available methods may not provide optimal recovery of the indicator minerals of interest.

### LAB PRECONCENTRATION

Whether sieved off in the field or in the lab, the coarse >2 mm fraction may be examined (pebble counts) to provide additional information about sample provenance and transport distance. The <2 mm (or <1 mm) fraction is preconcentrated most commonly using sieving and/or density methods (e.g. jig, shaking table, spiral, dense media separator, pan, Knelson concentrator) to reduce the weight of material to be examined without losing indicator minerals. Some of the more common preconcentration techniques are described below.

### Tabling

Preconcentration using a shaking (Wilfley) table separates minerals on the basis of density. It recovers silt- to coarse

**Table 2.** Weight of each fraction generated by a combination of tabling and heavy liquid separation to reduce till sample weight, concentrate heavy minerals, and recover indicator minerals: A) initial sample weight; B) sieving off <2 mm; C) & D) tabling; E) heavy liquid separation; F) magnetic separation; G) final heavy mineral concentrate weight. Till samples are from 1) the South Pit of the Thompson Ni mine, Thompson, Manitoba; 2) Broken Hammer Cu-PGE deposit, Sudbury, Ontario; 3) Pamour mine, Timmins, Ontario; 4) Triple B kimberlite, Lake Timiskaming field, Ontario; and 5) Buffalo Head Hills, northern Alberta.

Location	Texture	A: total sample weight (kg)	B: weight 2 mm clasts (kg)	C: weight sample put across shaking table (kg)	D: weight shaking table concentrate produced (g)	E: weight heavy liquid light fraction (g)	F: weight magnetic fraction (g)	G: weight non-magnetic heavy mineral concentrate (g) 0.25-2.0 mm
1. Thompson Ni Belt	silty sand till	15.0	3.0	12.0	1015.9	104.5	36.4	47.9
2. Sudbury	very sandy till	15.0	5.6	9.4	1125.1	402.6	13.0	18.9
3. Timmins Gold camp	silty sand till	11.8	2.3	9.5	353.1	319.8	5.2	28.1
4. Triple B kimberlite	silty sand till	9.8	1.2	8.6	438.7	377.0	22.0	35.8
5. Northern Alberta	clay till	67.4	2.4	65.0	1,307.0	1,235.2	5.6	11.5

sand-sized heavy minerals for a broad spectrum of commodities including diamonds, precious and base metals, and uranium (Averill & Huneault 2006). A slurry of <2.0 mm sample material is put across a shaking table to prepare a preconcentrate. If kimberlite indicators are targeted, the sample is tabled twice to ensure higher recovery of the lowest density minerals (Cr-diopside and forsteritic olivine) and the coarsest grains. The advantages of this method are its moderate cost, ability to recover indicator minerals for a broad spectrum of commodities, and ability to recover silt- as well as sand-sized indicators. It is a well established method for the recovery of precious metal mineral grains as well as kimberlite indicator minerals (e.g. McClenaghan *et al.* 1998, 2004). The disadvantages of this method include the loss of some heavy minerals during tabling, the longer time required to process each sample, and that the tabling procedure is dependent on the skill of the operator.

### Dense media separator

A gravity method used to preconcentrate kimberlite indicator minerals is the micro-scale dense media separator (DMS) (Fig. 2). An overview of this method from Baumgartner (2006) is summarized below. Heavy mineral concentration is carried out using a gravity-fed high-pressure cyclone. The <1 mm fraction of a sample is mixed with a ferrosilicon (FeSi) slurry that has a controlled density and fed into the DMS where heavy and light minerals are separated. The heavy mineral concentrate is collected on a 0.25 mm screen and is then dried and screened to remove residual FeSi. A Tromp curve is used to define the efficiency and precision of the DMS separation. The cut-point or threshold spans a density range of 0.2 g/cm<sup>3</sup> at ~3.1 g/cm<sup>3</sup> and is calibrated to recover the common kimberlite indicator minerals that have a specific gravity >3.1 g/cm<sup>3</sup>: pyrope garnet, chrome-spinel, Mg-ilmenite, Cr-diopside, forsteritic olivine and diamond. The required cut point is established using the computerized control panel on the DMS and the Tromp curve is then tested using synthetic density tracers before proceeding with processing the samples. The density settings and cut points are checked once per day. The advantages of the micro DMS system are that it is fast, less susceptible to sample contamination than other heavy mineral concentrating techniques and not operator dependent. The method, however, is more expensive than other methods described here and it does not allow for the recovery of silt-sized precious and base metal indicator minerals.

### Knelson concentrator

The Knelson concentrator is a fluidized centrifugal separator that was originally designed for concentrating gold and platinum from placer and bedrock samples. Chernet *et al.* (1999) describe how they used a modified 3 Knelson concentrator to recover kimberlite indicator minerals and other slightly heavy minerals. The concentrator can handle particle sizes from >10 microns up to a maximum of 6 mm. The general processing procedure is summarized below from the Knelson Concentrator website (<http://www.knelsongravitysolutions.com>). Briefly, water



Fig. 2. Photograph showing a micro-scale dense media separator (DMS), which can be used to preconcentrate kimberlite indicator minerals.

is introduced into a concentrate cone through a series of holes in rings on the side of the cone. The sample slurry is then introduced into the concentrate cone from a tube at the top. When the slurry reaches the bottom of the cone, it is forced outward and up the cone wall by centrifugal force from the spinning cone. The slurry fills each ring on the inside of the cone wall to capacity to create a concentrating bed. High specific gravity particles are captured in the rings and retained in the concentrating cone. At the end of the concentrate cycle, concentrates are flushed from the cone into the sample collector. Chernet *et al.*'s (1999) procedure for kimberlite indicator minerals took 5 to 11 minutes. The advantages of the Knelson concentrator are that it is fast, inexpensive, and can be mobilized to the field to reduce the weight of material to be shipped to the lab (e.g. Lehtonen *et al.* 2005). However, recovery of kimberlite indicator minerals from silt-poor material, such as esker sand or stream sediments, is difficult due to the absence of fine-grained material to keep the slurry in suspension (Chernet *et al.* 1999).

### Magnetic separation and sieving

Kimberlite indicator minerals may also be preconcentrated using magnetic separation in combination with sieving, such as the lab procedures described by Le Couteur & McLeod (2006). Their methods include wet screening, typically at 0.86 mm and 0.25 mm. The 0.25 to 0.86 mm frac-

tion is dried, weighed and then treated to magnetic separation using a permanent Fe-Nd dry belt magnetic separator operating at 2.1 Tesla. The magnet divides the sample into three fractions: 1) non-magnetic and/or diamagnetic, 2) weakly paramagnetic, and 3) strongly paramagnetic. The weak (2) and strong (3) paramagnetic fractions are combined in one 'magnetic concentrate', which is then processed through heavy liquids. The advantages of this procedure are that it is fast and inexpensive. This method, however, does not allow for the recovery of silt-sized precious and base metal grains and does not recover coarse (> 1 mm) indicator minerals.

### FINAL CONCENTRATION

A preconcentrate is usually further refined using heavy liquids to further reduce the size of the concentrate prior to heavy mineral selection (Table 2, column E). Heavy liquid separation provides a sharp separation between heavy (sink) and light minerals (float) at an exact density. It is slow and expensive and therefore not economical for large volumes of sample material, hence the preconcentration procedures that come before this step. The most common heavy liquids used include methylene iodide (MI) with a specific gravity of 3.3 g/cm<sup>3</sup> and tetrabromoethane (TBE) with a specific gravity of 2.96 g/cm<sup>3</sup>. The density threshold will depend on the indicator minerals being sought. Some labs use a combination of both heavy liquids, separating first using TBE to reduce the volume of material to be further separated using MI (e.g. Le Couteur & McLeod 2006). The recovery of kimberlite and magmatic Ni-Cu-PGE indicator minerals requires heavy liquid separation at a specific gravity of 3.2 g/cm<sup>3</sup> (using dilute methylene iodide) to include the lowest density indicators Cr-diopside and forsteritic olivine. Recovery of porphyry Cu indicator minerals requires separation at a specific gravity of 2.8 to 3.2 to recover the mid-density indicators: tourmaline (dravite), alunite, and turquoise (Averill 2007).

Ferromagnetic minerals can comprise a considerable portion of the post heavy liquid concentrate (e.g. Table 2, column F) and thus removing them decreases concentrate size. The ferromagnetic minerals and any steel contaminants are removed using a magnetic separator and commonly set aside unless magnetite, pyrrhotite or magnetic Mg-ilmenite are important indicator minerals.

The nonferromagnetic fraction is commonly sieved into two or three (e.g. 0.25-0.5 mm, 0.5-1.0 mm, 1.0-2.0 mm) size fractions for picking of indicator minerals, however, the final size range will depend on the commodity sought. For example, kimberlite indicator minerals are most abundant in the 0.25 to 0.5 mm fraction (McClenaghan & Kjarsgaard 2007) and thus to maximize recovery and minimize counting time and cost, the finest size fraction is most commonly picked.

A specific size fraction of the nonferromagnetic heavy mineral fraction may be further separated electromagnetically into fractions with different paramagnetic characteristics to help reduce the volume of material to be examined for indicator minerals (Averill & Huneault 2006). Minerals, such as diamond, are nonparamagnetic, pyrope garnet, eclogitic garnet, Cr-diopside and forsteritic olivine

are nonparamagnetic to weakly paramagnetic, and Cr-spinel and Mg-ilmenite are moderately to strongly paramagnetic (see Table 1 in McClenaghan & Kjarsgaard 2007). If the non- or paramagnetic portion of the concentrate contains a significant amount of almandine garnet it may be processed through a magstream separator to separate the orange almandine from similar looking eclogitic or pyrope garnets. Magstream separation divides the concentrate into 1) a fraction containing most of the silicates (e.g. pyrope and eclogitic garnet) and no almandine, and 2) a fraction containing ilmenite, chromite and other moderately magnetic minerals, such as almandine (Baumgartner 2006).

### INDICATOR MINERAL SELECTION AND EXAMINATION

Indicator minerals are selected from nonferromagnetic heavy mineral concentrates during a visual scan, in most cases, of the finer size (e.g. 0.25-0.5 mm, or 0.3-0.5 mm, 0.25-0.86 mm) fractions using a binocular microscope. The grains are counted and a selection of grains is removed from the sample for analysis using an electron microprobe to confirm their identification. Methods for examining a sample for counting/picking vary from rolling conveyor belts to dishes and/or paper marked with lines or grids. If a concentrate is unusually large, then a split is examined and the indicator mineral counts are normalized to the total weight of the concentrate. If a split is picked, the weight of the split and the total weight should both be recorded. Usually, not all grains counted in a sample will be removed for microprobe analyses. If this is the case, the total number of grains counted and the number of grains removed should both be recorded.

Indicator minerals are visually identified in concentrates on the basis of colour, crystal habit and surface textures, which may include features such as kelyphite rims and orange peel textures on kimberlitic garnets (Garvie 2003; McClenaghan & Kjarsgaard 2007). Scheelite and zircon in a concentrate may be counted under short-wave ultraviolet light. Gold and PGM grains may be panned from concentrates that were prepared in such a way that the silt-sized fraction has been retained (e.g. tabling). The grain may be counted and classified with the aid of an optical or scanning electron microscopy. Commonly, gold grains are classified according to their shape and/or degree of wear (DiLabio 1990), which can provide information about relative transport distances.

### INDICATOR MINERAL CHEMISTRY

Mineral chemical analysis by electron microprobe, scanning electron microprobe (SEM), laser ablation-ICP-MS, or secondary ion mass spectrometry (SIMS) may be carried out to determine major, minor and trace element contents of specific indicator minerals because mineral chemistry is used to confirm identity, establish mineral paragenesis, and, in some cases, deposit grade (e.g. Ramsden *et al.* 1999; Belousova *et al.* 2002; Scott 2003; Heimann *et al.* 2005). For example, kimberlite indicator minerals are characterized by a specific range of compositions that reflect their mantle source and diamond grade (e.g. Fipke *et al.* 1995; Schulze 1997; Grütter *et al.* 2004, this volume; Wyatt *et al.*

2004). Gold, PGM and sulphide grains may be analyzed to determine their trace element chemistry or isotopic compositions (e.g. Grant *et al.* 1991).

### QUALITY CONTROL

Project geologists may use a combination of blank samples (no indicator minerals), spiked samples (known quantity of specific indicator mineral species), and field duplicates, as well as repicking of 10% of the heavy mineral concentrates to monitor a lab's potential for sample contamination and quality of mineral grain selection. In addition, heavy mineral processing and selection labs can be asked to report their own quality control monitoring procedures and test results.

### SUMMARY

This paper has described a few of the procedures available for processing surficial media and rocks to recover indicator minerals. The processing method used will depend on sample media, commodities being sought, budget, bedrock and surficial geology of the survey area, and processing methods used for previous batches. When reporting indicator mineral results in company assessment files, government reports, or scientific papers, it is helpful to report the lab name, processing methods used, and sample weights. Monitoring of quality control is essential at each stage in the processing, picking and analytical procedures described here and may be monitored both by the processing labs and clients. Geologists are encouraged to visit processing labs so that they have a clear understanding of the procedures being used and can discuss customizations needed for specific sample batches.

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# Mineral chemistry: Modern techniques and applications to exploration

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## INTRODUCTION: WHY IN SITU METHODS?

The routine application of mineral chemistry to mineral exploration only became realistic with the development of in situ microanalytical methods. Today, surrounded by a plethora of techniques and laboratories, it is easy to forget how recently this began. The first electron microprobes were built or installed in academic research institutions in the mid-1960s, making it possible for the first time to analyze small parts of individual mineral grains (not the cracks, the inclusions, the alteration....) for major- and minor-element contents, in a matter of minutes rather than weeks. This spatial resolution added enormously to the interpretation of geochemical data, allowing studies of zoning, chemical equilibrium, inclusions and reactions between minerals. However, the robust instrumentation and reliable data-reduction methods that we now take for granted took another 15 years to evolve.

In situ trace-element analysis came along even more recently, with the development of the ion microprobe and the proton microprobe in the mid-1980s. The first practical laser-ablation ICPMS microprobe dates from the mid-1990s, and in situ isotope-ratio analysis by laser-ablation multi-collector ICPMS did not really become practical until ca. 1998. As with the electron microprobe, the in situ trace-element and isotope techniques have brought the interpretative benefits of high spatial resolution, and have eliminated the time and costs associated with mineral separation and the chemical separation of isotopes. This talk will provide a brief overview of some of the more widespread in situ microanalytical techniques, focusing mainly on the types of data they can provide to the mineral exploration industry, their advantages and drawbacks, and some examples of applications.

## ANALYTICAL METHODS — AN OVERVIEW

### Major and minor elements: The electron microprobe

The electron microprobe (EMP) generates a stream of electrons by heating a metallic filament. Electrostatic lenses focus this beam to a spot (typically 1-3 microns in diameter) on the sample; interaction between the electron beam and the inner electron shells of atoms generate characteristic X-rays from the upper 3 to 5 microns of the sample. These can be analyzed by two types of spectrometers. Wave-length dispersive (WDS) spectrometers measure the intensity of one X-ray wavelength at a time, and must be moved during the analysis to detect and measure each element of interest. Energy-dispersive (EDS) spectrometers are solid-state (Si(Li); lithium-drifted silicon) devices that collect the entire X-ray spectrum simultaneously and use computer software to deconvolute it into the intensities of individual X-ray lines. In both types of analysis, the broad

continuum X-ray background produced by the deceleration of the electrons results in relatively high detection limits, especially for lighter elements or the lower energy lines of heavy elements.

WDS analysis is typically more precise and has lower limits of detection, especially for minor elements, whereas EDS is faster; it is extremely useful for the identification of target grains, and can provide good major-element analyses. Most modern EMPs are equipped with both WDS and EDS spectrometers. Each can be used for mapping the element distribution within a selected area, which can be as large as the range of the stage travel (approximately 80 x 45 mm on a Cameca EMP). The number of elements that can be mapped simultaneously by WDS is limited to the number of spectrometers, whereas in most modern EDS systems maps of 30 to 35 elements can be acquired at the same time. WDS-EMP analysis of major elements and minor elements (at levels down to a few hundred ppm, or 0.02 to 0.05 wt.%) is now routine. The minimum detection limit (MDL) for some elements can be driven below 100 ppm, but only with very long counting times or high beam currents; this is seldom cost-effective and can result in damage to the sample.

EMP is a truly mature technology, and in routine analysis the user will have little interaction with the intricacies of data reduction. Quantitative analysis is a comparative technique and involves the ratio of raw counts on the sample to those on a standard, so the accuracy of the analyses depends on the quality of the standards and the calibration. In both types of analysis, analytical precision is a function of the total number of counts collected, and thus is controlled by counting times versus concentration. A typical WDS analysis of a common mineral will take several minutes, with counting times of 10 to 30 seconds on individual peaks and backgrounds.

Some laboratories try to reduce costs by using much shorter counting times (2-5 seconds) and fewer background measurements; the resulting analyses will be of poor quality and of questionable usefulness even in reconnaissance applications. In our experience, attempts to cut costs on WDS-EMP analysis in this way may produce large errors in individual (major) elements (e.g. Cr and Fe in chromite) that can seriously skew the interpretation, and hence exploration outcomes. If simple mineral identification is the aim, an EDS analysis is likely to provide more usable information at lower cost.

### Trace elements

#### *Ion microprobe*

The ion microprobe (Secondary Ion Mass Spectrometer, or SIMS) accelerates a beam of metal ions (e.g. Cs) onto the

target, sputtering off a cloud of ionized sample material. These secondary ions are in turn accelerated into a mass spectrometer, which directs beams corresponding to single masses (single or multiple-element ions) into a single collector or an array of collectors. Typical sputtering pits are on the order of 30 microns across, and a few microns deep; this spatial resolution is the major strength of the technique. Drawbacks are the long analysis times required to collect statistically useful numbers of counts; coupled with the high initial cost of the instrument, this usually leads to relatively high cost per analysis.

The major applications of most ion microprobes lie in isotopic analysis (see below), but they can be used for trace-element analysis in situations where the larger pits produced by laser-ablation microprobes (see below) are a drawback. Typical minimum detection limits (MDL) are in the low ppm range. However, the technique is quite sensitive to matrix effects, and requires well characterized standards similar in composition to the target grain. As a result, most applications have focused on a small range of elements (e.g. rare earth elements).

#### *Proton microprobe*

A proton microprobe uses a particle accelerator to shoot a beam of high-energy (typically 3 million electron volts (MeV)) protons into the sample. Interactions between protons and the inner electron shells produce characteristic X-rays (Proton Induced X-ray Emission, or PIXE); interactions of the protons with light atomic nuclei produce characteristic gamma rays (Proton Induced Gamma Emission, or PIGE). Typical spot sizes are from 1 to 30 microns, but the penetrating power of the protons means that the analyzed volume extends tens of microns below the surface. In PIXE, X-rays are collected by EDS detectors; MDLs vary depending on the element, and can range from 0.2 to 0.3 ppm to several ppm for long counting times (Ryan *et al.* 1990). Although the PIXE is similar to the EMP in that an X-ray spectrum is produced, the MDLs are one or two orders of magnitude lower. This is because the protons 'die' deep in the sample, and the continuum X-rays are mostly absorbed before reaching the surface; thus background is lower and peak to background ratio higher than in the EMP. The PIXE technique is non-destructive, and does not require standards. The major disadvantages are in the cost of the infrastructure and the limited number of available laboratories.

The proton microprobe was the first in situ trace-element analysis technique with practical applications to the minerals industry, because it could rapidly analyze a range of elements in a single spot, providing data that could be used for discriminants. The first and most highly visible application was in diamond exploration (see review by Griffin & Ryan 1995); applications also have included other types of resistate indicator minerals and sulphide mineralogy.

The most spectacular recent development in PIXE analysis has been the development of the scanning nuclear microprobe, which combines a fine beam with a rastering technique, and allows high-resolution imaging of trace-element distributions in areas from a few tens of microns to

mm<sup>2</sup> in size (Ryan *et al.* 2001). More importantly, because each pixel contains the complete X-ray spectrum, quantitative analyses can be extracted from any portion of the image.

#### *LAM-ICPMS*

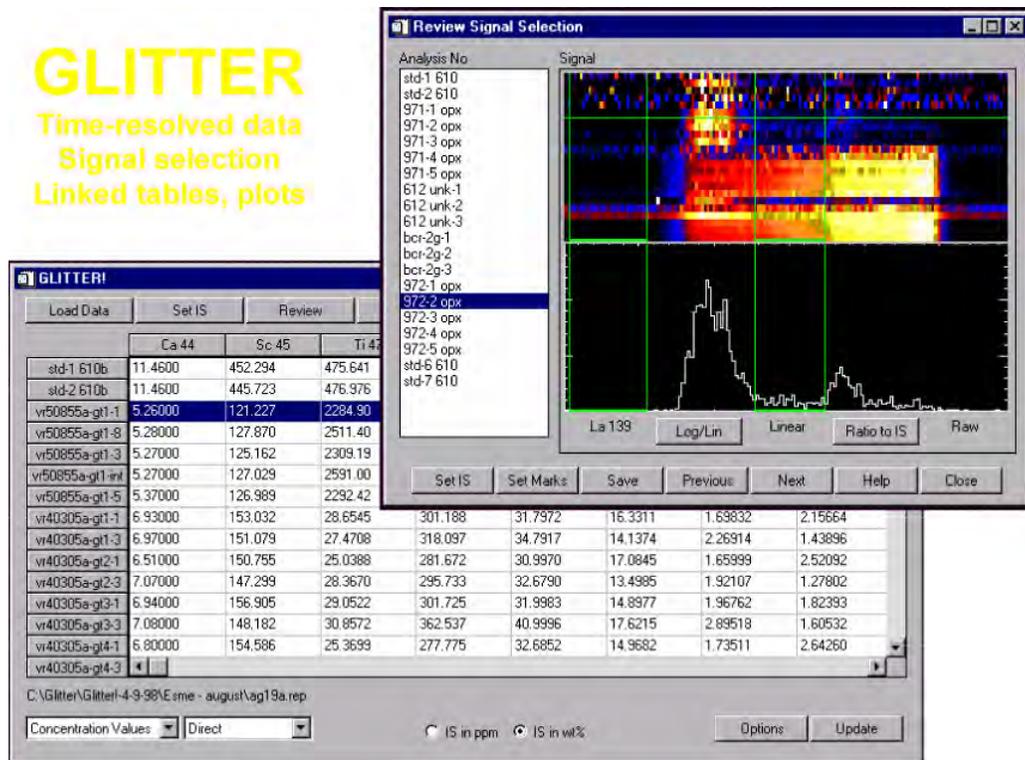
The development of inexpensive solid-state laser-ablation microprobes (LAM) and their coupling with Inductively Coupled Plasma Mass Spectrometers (ICPMS) has revolutionized the analysis of trace elements in minerals. Compared to other technologies, LAM-ICPMS offers relatively low capital costs (quadrupole instruments; see below), high spatial resolution (typical pits 20-50  $\mu\text{m}$  in diameter, 20-50  $\mu\text{m}$  deep), high sensitivity (low ppb range for most elements of interest) and high sample throughput (low cost/analysis). Matrix effects are small, making it possible to analyze a wide range of materials with a small range of multi-element standards. Each grain requires at least one known element (from EMP, or assumed from stoichiometry) to serve as an 'internal standard'.

The plasma torch of the ICPMS operates at 6000 to 8000°C (equivalent to the Sun's surface); it ionizes most fine particles introduced into it, providing a stream of ions to be sorted into a mass spectrum by the spectrometer. The most widespread ICPMS instruments come in two general types, quadrupole and sector.

In a *quadrupole ICPMS*, the mass analyzer consists of four parallel cylindrical rods (~20 cm long and 1.5 cm in diameter). Ion lenses preceding the quadrupole focus the ions that have been extracted from the plasma on a trajectory down the centre of the four rods. The mass analyzer generates a mass spectrum by linearly varying RF and DC voltage amplitudes on the quadrupole rods. At a given RF/DC voltage ratio and voltage amplitude, only ions of a given mass/charge ratio can traverse the length of the rods, with all other ions being deflected and lost. The advantages of quadrupole instruments are relatively low cost, very rapid scanning of the entire mass spectrum, and high sensitivity. Some versions are equipped with a gas-collision cell that can eliminate some isobaric and molecular interferences.

*Double-focussing magnetic-sector ICPMS* instruments have higher mass resolution than quadrupole instruments, but this feature is rarely a significant benefit for trace-element analysis, especially for elements heavier than Sr. They also have higher intrinsic sensitivity (counts/second/ppm) than quadrupole instruments. However, to analyze more than a narrow range of masses in a single ablation, the instrument must switch its magnet several times in each scan, and each switch requires time for the magnet to settle, thereby losing counting time. As a result, the intrinsic advantage in sensitivity is lost; in most applications there is little difference in the typical MDLs between quadrupole and sector instruments. The sector instruments are significantly more expensive (by 1.5 to 2 times) than quadrupole instruments; this investment may or may not be passed on to the user.

In a LAM-ICPMS, ablation is carried out inside a sample cell through which a carrier gas (Ar or He) flows, carrying the cloud of ablated particles to the plasma torch. The lasers most commonly used in mineralogical applications



**Fig. 1.** Window dumps from GLITTER, showing analytical table and signal review window. In the signal review window, the bottom panel shows the time-resolved signal for a single mass (La); each row of pixels in the upper panel records the time-resolved signal for an individual mass, making it simple to recognize an inclusion. Green lines mark the selected background counts and the portion of the signal that has been selected to avoid two La-rich inclusions. The element concentrations for the selected interval are displayed in the linked analysis table and/or a plotting window. This type of data collection/visualization is essential for LAM-ICPMS analysis.

are either solid-state (Nd:YAG) or gas-phase (excimer) lasers. The Nd:YAG lasers have a fundamental wavelength of 1064 nm (IR); harmonic-generator crystals are used to produce shorter wavelengths ranging from visible (532 nm) to UV (266 nm, 213 nm and most recently 193 nm). For mineral analysis, most laboratories use the 266 nm and/or 213 nm models, which cause less damage to the sample, and produce a more stable signal than the longer wavelengths. Excimer lasers used in mineralogical work typically operate at 193 nm and less commonly at 157 nm; these are significantly more expensive than the solid-state lasers. In principle, the shorter wavelengths should produce better ablation characteristics in more transparent minerals. However, in our own laboratory, we have found little difference in performance between an excimer laser and a 213 nm Nd:YAG laser for applications such as zircon analysis. For the analysis of sulphides and coloured silicates and oxides, the 266 nm Nd:YAG laser is more efficient.

External users of LAM-ICPMS labs should be aware of a game played among such labs: “mine makes nicer pits than theirs does”. There is no relationship between the shape of the ablation pit and the quality of the analytical data, so pin-ups of LAM holes are not a good criterion for choosing a laboratory.

The laser drills into the sample through time, ultimately producing a pit up to 100  $\mu\text{m}$  deep, and this pit may intersect cracks, inclusions or other extraneous material. It is essential that the data for each analyzed element are

recorded as a function of time (“time-resolved analysis”), and that the analytical software allows both the visualization of these time-resolved data and the on-line selection of a specific ablation integral for analysis, to avoid inclusions, etc. We have developed a package of this type (GLITTER; Fig. 1), which is available through GEMOC, and is now used at more than 100 sites around the world.

Compared to SIMS or PIXE, LAM-ICPMS offers much lower capital costs, more rapid analysis of a larger range of elements, lower detection limits and the advantages of time-resolved analysis. SIMS offers better spatial resolution (similar beam size, but shallower pits) but is slow, expensive, and requires intensive work on standardization. For these reasons, LAM-ICPMS is the technique of choice for industrial applications, as for most academic research.

### Isotopic analysis

The use of isotopic signatures in potential indicator minerals is still in its infancy, with the exception of U-Pb and Hf-isotope analysis of zircon. However, there is a wide range of possibilities that could be explored.

#### *Ion probe (SIMS)*

Most exploration groups would be aware of the application of the ion microprobe (especially the Australian-designed SHRIMP instruments) to U/Pb dating of zircon. The same technique is now being applied to other minerals (monazite, xenotime). Typical precision is 1 to 2% (though ‘reconnaissance ages’ of considerably lower precision are

produced for some applications). This is lower than can be attained by single-crystal TIMS (Thermal Ionization Mass Spectrometry) analysis. However, TIMS is a 'bulk' technique that is not usable for complex grains; more importantly, it cannot identify them. As for trace-element analysis, the advantage of the SIMS technique lies in its spatial resolution. This is offset by the lack of time-resolved analysis, but ion-probe ablation pits are shallow, making this less of a concern in most cases.

SIMS instruments are now being used to measure the isotopic composition of light elements, including C, O, N and S, and many other applications are being developed that may eventually have relevance to mineral exploration.

#### LAM-ICPMS

Applications of LAM-ICPMS to U-Pb dating of zircons began in the mid-1990s (e.g. Hirata & Nesbitt 1995) and have advanced rapidly to become routine in many laboratories worldwide. The analytical precision and accuracy are equivalent to that provided by SIMS (see review by Jackson *et al.* 2004; Griffin *et al.* this conference), with the advantages of speed and lower cost/analysis. A typical working day (8 hours) will produce ages for between 40 and 50 zircons; equivalent numbers of standards are run to monitor instrument drift and improve data quality. The larger volume sampled by the LAM-ICPMS analysis compared to the ion probe is offset by the use of time-resolved analysis; if zones of different age are intersected by the laser during the analysis, these can be recognized, and pulled out for separate analysis.

The relative insensitivity of the LAM-ICPMS technique to matrix effects allows its application to many other U-Th-bearing minerals, working from well characterized zircon standards. In the GEMOC laboratory, we have successfully dated perovskite (in kimberlite groundmass), titanite, rutile, apatite, baddeleyite and monazite by U-Th-Pb, and experimented with Pb-isotope analysis of K-feldspar.

#### LAM-multicollector (MC)-ICPMS

The 1 to 2% precision on isotope ratios provided by the quadrupole ICPMS is adequate for U-Pb dating, but the application of most other isotopic systems requires 100 to 10000 times better precision to be geologically useful. The MC-ICPMS is a double-focusing instrument; an electrostatic analyzer sorts ions by their energy, then a sector magnet separates ions by mass and/or charge. Ion beams are directed into an array of detectors (Faraday cups, ion counters) for static collection. The simultaneous detection of isotopes provides a significant improvement in precision on the isotope ratios compared to single-collector instruments. There have been three generations of MC-ICPMS; most laboratories now use either the Nu Plasma (fixed collector array, electrostatic zoom lens) or Thermo-Finnigan Neptune (movable collector array).

LAM-MC-ICPMS analysis requires corrections for isobaric overlaps, and such corrections rapidly lead to magnified errors. This limits applications to minerals where the isotopes of interest are relatively abundant, and overlaps on them are relatively small. For instance,  $^{87}\text{Rb}$  overlaps

its daughter product,  $^{87}\text{Sr}$ . Thus it is relatively straightforward to analyze  $^{87}\text{Sr}/^{86}\text{Sr}$  in a plagioclase or a carbonate (high Sr, low Rb/Sr), but difficult to obtain meaningful data from a biotite (high Rb/Sr, low Sr).

The applications of LAM-MC-ICPMS to radiogenic systems therefore are focused on measuring the initial isotopic ratios of elements of interest, rather than on geochronology. The most widespread application at the moment is the analysis of  $^{176}\text{Hf}/^{177}\text{Hf}$  in zircon, which can be done to typical  $2\sigma$  precisions of  $\pm 0.00002$  (Griffin *et al.* 2000). The Hf-isotope data improve the interpretation of U-Pb age data, and provide information on magma sources and crustal evolution (Griffin *et al.* this conference). At GEMOC we have had some success in generating Sm-Nd isochron ages using titanite grains in granites.

LAM-MC-ICPMS also can be used to measure stable-isotope ratios of a wide range of elements (e.g. Mg, Cu, Zn, Fe and Ni) with applications to sulphide minerals.

### EXAMPLES OF APPLICATIONS — THE 'INDICATOR MINERAL' APPROACH

In principle, almost any mineral might serve an 'indicator' in an exploration situation: you must know your system, decide what you need to look for, and then use your imagination and the best technology. Empirical 'fingerprints' can work, or not; if you do not understand your target, they may become misleading.

#### Trace element applications: The diamond exploration example

The application of EMP to garnets in kimberlites and diamonds in the early 1970s led to the recognition of subcalcic ('G10') garnets as a significant trace component of diamondiferous kimberlites in the Kaapvaal and Siberian cratons (Sobolev 1971; Gurney & Switzer 1973). This became a key discriminant, leading to the establishment of EMP labs by most of the larger exploration companies. However, this purely empirical discriminant soon showed problems, producing 'false positives' in some cases (e.g. Kuruman field, RSA; Shee *et al.* 1999) and 'false negatives' in others (e.g. Orapa; Gurney & Zweistra 1995).

One of the first applications of trace-element analysis to diamond indicator minerals (using the proton microprobe) improved this situation by adding information on such critical genetic factors as temperature (Ni in garnet: Griffin *et al.* 1989; Ryan *et al.* 1996) and metasomatic processes that might produce or destroy diamonds (Griffin & Ryan 1995). This use of *genetically linked* signatures led to improved discrimination between good and bad targets, independent of the G10 criterion (and led to the establishment of LAM-ICPMS labs by some explorers). By the beginning of exploration in the Slave Craton, these techniques were able to show that in this particular area G10 garnets tend to be counter-indicative (coming mainly from the graphite stability field), and that the most reliable indicators may be specific types of eclogitic garnet and Cr-diopside (Griffin *et al.* 1999).

#### Trace elements: examples of other potential indicators

Zircon is widespread and resistant, and shows wide chemical variability. Kimberlitic zircon is easily distinguished

from crustal zircon by its trace-element patterns (Fig. 2), and zircons from hydrothermal systems also may have distinctive patterns, including large positive Ce anomalies (Fig. 3).

Apatite also shows wide chemical variability, and apatite grains from different types of ore-forming systems can be distinctive against the general background of grains from common rock types (Fig. 4).

Tourmaline is commonly associated with several types of ore deposits, and easily recovered from HMC samples. It shows considerable variation in major-element and trace-element composition and potentially is useful as an indicator mineral (Griffin *et al.* 1996).

Clinopyroxene (skarns, ultramafic rocks, gabbros) also show a wide range of composition and can be good indicators in specific situations (Thorleifson *et al.* 2007).

Chromite is widely used in diamond exploration, but can also be valuable in the search for NiS deposits. For example, Yao (2000) demonstrated that chromites from mineralized and barren komatiites in the Yilgarn Craton have markedly different trace-element signatures, reflecting the interaction of mineralized magmas with S-bearing black shales.

In each of these more specific situations, a ground-truthing exercise would be advisable to understand the specific signature of the type of orebody or host rocks being sought.

Finally, a mention should be made of diamond as an indicator mineral. Recent developments in the quantitative trace-element analysis of diamond by LAM-ICPMS (Rege *et al.* 2005) indicate that it may be possible to fingerprint diamonds from individual sources. Trace-element patterns potentially could be used to recognize different populations within alluvial diamond deposits, link them to their possible sources, and identify the presence of sources that have not been found yet.

### Isotopic indicators

The integrated analysis of U-Pb age, Hf-isotope composition and trace elements in detrital zircon from modern drainages (the *TerraneChron*<sup>®</sup> methodology; Griffin *et al.* this conference) has become a powerful tool for mineral exploration. It typically is applied to establish a basic geological and/or tectonic framework in a new exploration area. However, it also can be deployed to search for specific rock types or associations; examples include exploration for young Cu porphyries in the Andes and Tibet, where a few grains of distinctive morphology, age and composition have identified the presence of these rocks at distances of tens to hundreds of kilometres downstream.

Other radiogenic-isotope applications with potential use in mineral exploration, based on LAM-MC-ICPMS, include

- Sr isotopes in feldspar, carbonate, clinopyroxene, amphibole, apatite, and titanite;
- Nd isotopes in titanite, apatite; Os isotopes in sulphides (usually mantle-derived) and alloys;
- Pb isotopes in feldspars and several other phases.

The in situ analysis of heavy stable isotopes to exploration is in its infancy, but has large potential. The analy-

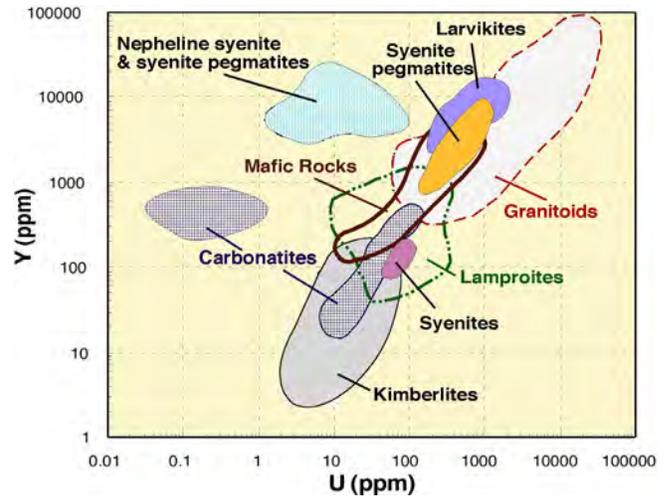


Fig. 2. Trace-element discrimination of zircons from different rock types; this is one slice of a multivariate analysis (after Belousova *et al.* 2002a).

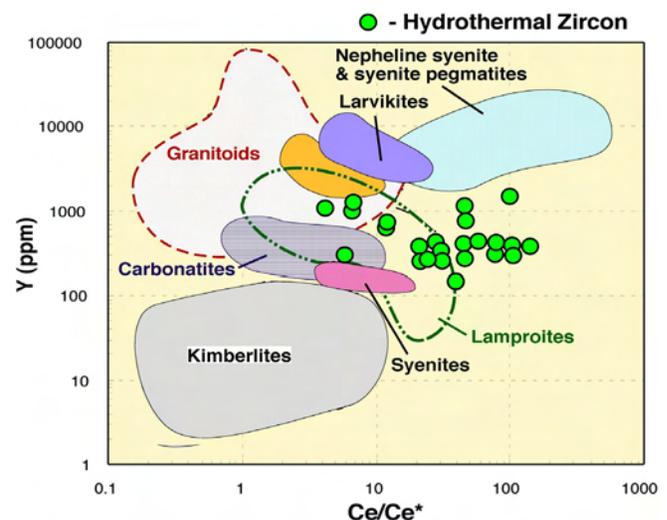


Fig. 3. Y contents and positive Ce anomalies in hydrothermal zircons, compared with fields of zircons from magmatic rocks (after Belousova *et al.* 2002a).

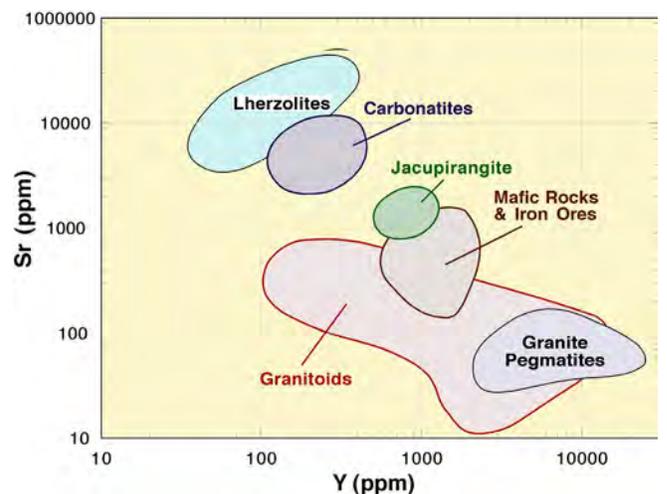


Fig. 4. Trace-element discrimination of apatite from different rock types; this is one slice of a multivariate analysis (after Belousova *et al.* 2002b).

sis of Cu-Fe isotopes in sulphides minerals is especially promising, though thus far it has been used primarily for ore-genesis studies. It has been possible to establish vectors toward orebodies and to recognize distal components of specific orebodies (Graham *et al.* 2004); both have applications to brown-fields exploration. In a given area, it is also possible to fingerprint sulphides related to low-grade versus high-grade mineralization.

### SUMMARY

In situ microanalysis of major and trace elements is a key to the successful application of resistate indicator minerals techniques to exploration. The combination of EMP and LAM-ICPMS has brought down the cost per sample over the last few years. The introduction of LAM-MC-ICPMS is producing a similar revolution in the rapid, high-precision analysis of isotope ratios (Sr, Nd, Hf, Os; Cu, Zn, Fe, etc.). The technology is now remarkably powerful; the possible applications to exploration problems are limited only by your vision.

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# Application of new-age clinopyroxene and garnet thermobarometry techniques in Arctic diamond exploration

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## INTRODUCTION

Modern-day diamond exploration projects are complex and expensive undertakings conducted in a range of settings (e.g. Arctic, deserts, savannah, tropical), jurisdictions (e.g. Canada, Greenland, SADEC countries, Brazil, India, Australia) and commonly involving several geoscientific disciplines (e.g. geology, geochemistry, geophysics, geostatistics). Over the past 30 years, considerable effort has been directed at retrieving kimberlitic indicator minerals (KIMs) and matching their chemical compositions with those of known inclusions in diamonds, in order to focus the effort of discovery teams on potentially economic primary diamond deposits. Due in part to public disclosure of exploration results, Canadian explorers have over the past decade assured rapid expansion of this field of study, primarily by embracing application of new-age thermobarometry techniques to kimberlite indicator minerals recovered in Arctic settings. These advances in kimberlite indicator mineral-based search techniques are reviewed in this talk by discussing the interplay of composition, pressure and temperature for open-file clinopyroxene and gar-

net data from exploration projects at Safartok (West Greenland), Attawapiskat and Kyle Lake (northern Ontario, Canada).

## APPLIED SINGLE-GRAIN THERMOBAROMETRY FOR CLINOPYROXENE AND GARNET

Due to their preservation in cold Arctic climates, mantle-derived Cr-diopside grains are commonly recovered from till samples. Varieties with moderate Cr/Al ratios are suitable for thermobarometry using the single-clinopyroxene technique of Nimis and Taylor (2000), provided the electron microprobe analyses obtained are of research quality. The clinopyroxene pressure-temperature technique shows slightly more scatter and produces pressure-temperature arrays with lower pressure/temperature ratios than conventional (xenolith-based) thermobarometry, but importantly retains the capacity to clearly discriminate cold, 'normal' and hot geotherms from known settings in Canada (Fig. 1). Data from known settings should always be used as a pressure-temperature benchmark for clinopyroxene data derived from exploration projects.

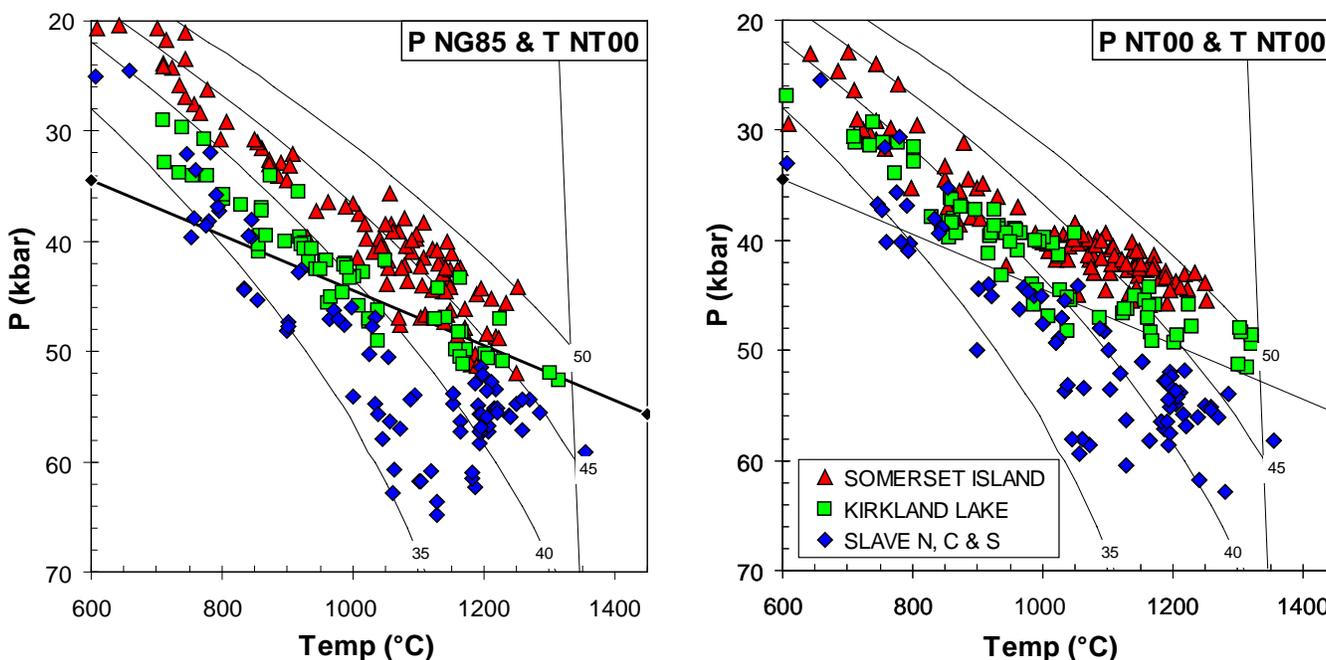


Fig. 1. Conventional (at left) and clinopyroxene single-grain (at right) pressure-temperature results for garnet-lherzolite xenoliths from Canadian kimberlite provinces. Either technique defines clearly separated cold, 'normal' and elevated geothermal arrays (blue, green and red symbols, respectively). Although prone to more scatter and somewhat larger errors, the single-grain clinopyroxene technique of Nimis and Taylor (2000) is suitable for application to exploration data sets. Localities represented are JP, Ham, Elwin Bay, Amayersuk, Nanorluk, Batty Bay and Nikos (Somerset Island kimberlites), C14, A1, A4 and B30 (Kirkland Lake kimberlites) and Jericho, Torrie, Diavik and 5034 (Slave Craton kimberlites). See Grütter and Moore (2003) for further detail.

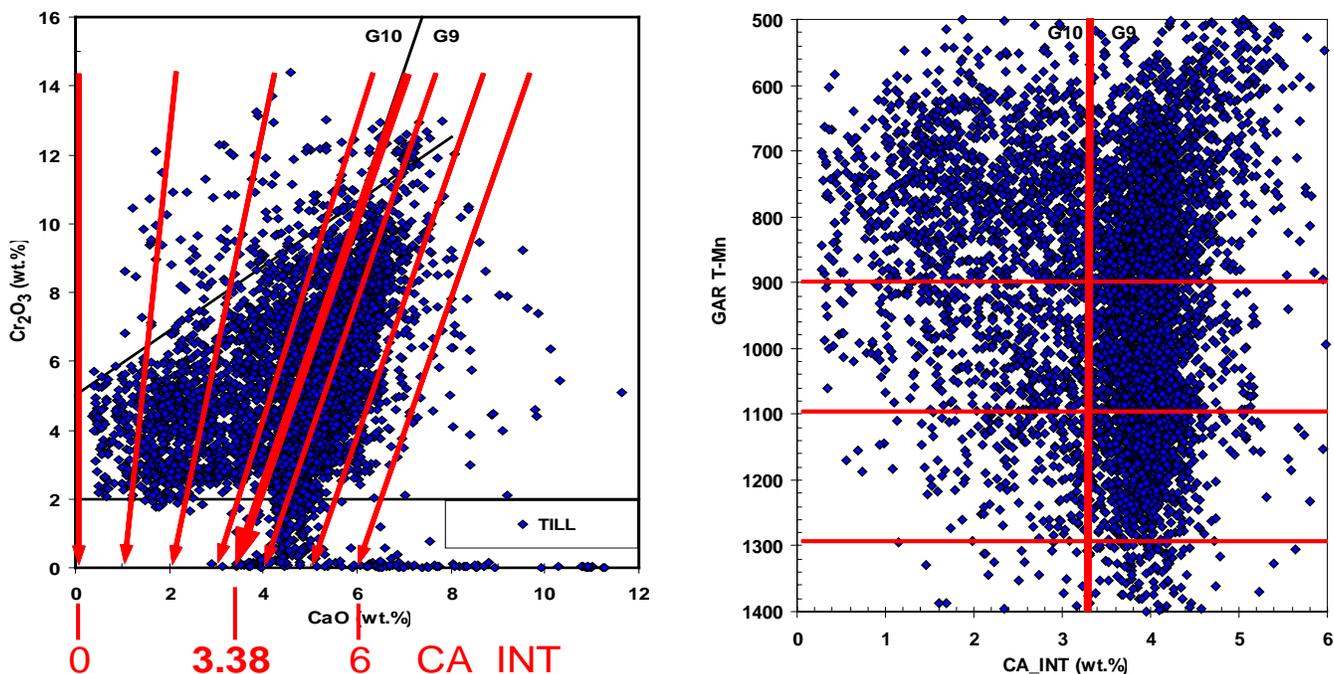


Fig. 2.  $\text{Cr}_2\text{O}_3$  vs.  $\text{CaO}$  diagram for 5921 garnet analyses from 1568 till samples from the Safartog area (West Greenland), displaying the presence of abundant G10 garnets (at left, data from Jensen *et al.* 2004). The  $\text{CA\_INT}$  projection of Grütter *et al.* (2004, red arrows) transforms the conventional  $\text{Cr}_2\text{O}_3$  versus  $\text{CaO}$  diagram to one variable, but retains the important G10/G9 boundary at  $\text{CA\_INT} = 3.375$ . Plotting garnet Mn-temperature (after Grütter *et al.* 1999) against  $\text{CA\_INT}$  for each grain yields a mantle section (at right) in which roughly 60% of the G10 garnets are located at temperatures of less than  $900^\circ\text{C}$ , inside the graphite stability field. Shallow and deep diamond-facies conditions are delimited at  $900$  to  $1100^\circ\text{C}$  and  $1100$  to  $1300^\circ\text{C}$ , respectively.

Mantle-derived garnets with  $\text{Cr}_2\text{O}_3 > 0.94 \cdot \text{CaO} + 5.0$  (in wt.%) are derived from within the diamond stability field. Grütter *et al.* (2006) expanded this simple relationship to calibrate the P38 *minimum*-pressure barometer that requires only the  $\text{Cr}_2\text{O}_3$  and  $\text{CaO}$  content of common G10 or G9 garnets as input variables, plus an assumed geotherm. The exchange of Fe, Mg, Ni and Mn between forsteritic olivine and Cr-pyrope garnet forms the basis of very useful mantle thermometers (O'Neill & Wood 1979; Ryan *et al.* 1996; Grütter *et al.* 1999). Since the open-file data sets discussed below lack trace-element analyses for garnet Ni content (as do most diamond exploration data sets), an updated version of the garnet-Mn thermometer (after Grütter *et al.* 1999) has been applied to construct mantle thermal profiles for western Greenland (Figs. 2 and 3) and northern Ontario (Figs. 4 and 5).

#### SAFARTOQ (WESTERN GREENLAND)

Jensen *et al.* (2004) have compiled an exploration-style database from assessment reports filed for a number of areas in western Greenland. I discuss here the microprobe data for approximately 13,400 clinopyroxene and approximately 7,800 garnet grains derived from approximately 1,600 till and stream samples collected in the Safartog area near Kangerlussuaq Fjord. Over 95% of the clinopyroxene analyses are from non-kimberlite indicator mineral (i.e. crustal) varieties, but the minority of mantle-derived grains define a cold geotherm that enters the diamond stability field at a temperature greater than  $900^\circ\text{C}$ , similar to clinopyroxene in Slave Craton kimberlites (see Fig. 1). As shown in Figure 2, the garnet  $\text{CA\_INT}$  projection of

Grütter *et al.* (2004) can be combined with garnet Mn-thermometry results to construct a mantle thermal profile. The majority of G10 garnets from Safartog are seen to be derived from temperatures of less than  $900^\circ\text{C}$ , i.e. from inside the graphite stability field. Separating garnets by T-Mn into shallow ( $900$ - $1100^\circ\text{C}$ ) and deep ( $1100$ - $1300^\circ\text{C}$ ) diamond-facies classes (Fig. 2) allows representation of deep mantle tenure at the scale of individual sample results (Fig. 3). Entrainment of deep diamond-facies G10-bearing mantle is uniquely characteristic of samples in the Garnet Lake area where Hudson Resources are executing a bulk sample program on significantly diamondiferous kimberlite (see [www.hudsonresources.ca](http://www.hudsonresources.ca)). Application of clinopyroxene and garnet thermobarometry techniques has highlighted analogous high-interest results elsewhere in the Safartog area.

#### ATTAWAPISKAT (NORTHERN ONTARIO)

The Attawapiskat kimberlites are 156 to 180 Ma in age and intrude Ordovician limestones overlying basement rocks of the Archean Superior Craton. The kimberlite province was discovered by conventional kimberlite indicator mineral-based and geophysical exploration (Kong *et al.* 1999), and contains the Victor kimberlite complex, soon to be a producing mine (see [www.debeerscanada.com](http://www.debeerscanada.com)). Here I briefly interpret the electron microprobe data provided by Sage (2000) for a comprehensive suite of kimberlite indicator minerals derived from samples of kimberlite. Cr-diopside from Attawapiskat define a normal cratonic geotherm that falls inside the diamond stability field over the temperature range of  $1000$  to  $1250^\circ\text{C}$  (Fig. 4). The garnet

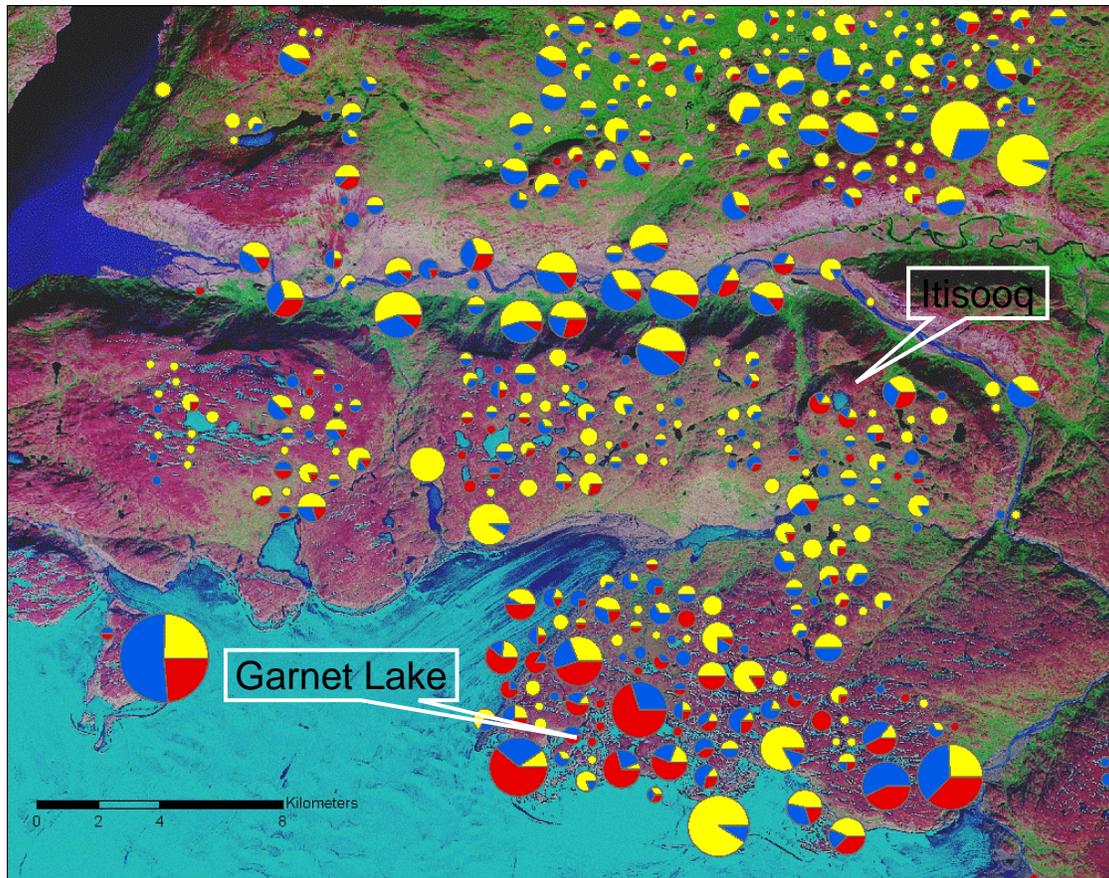


Fig. 3. Variation of garnet T-Mn classes by sample in an area around Safartoq, west Greenland. Following classification as in Fig. 2, the pie diagrams illustrate relative abundance of grains classed as graphite-facies (yellow, T-Mn < 900°C), shallow diamond-facies (blue, T-Mn 900-1100°C) or deep diamond-facies (red, T-Mn 1100-1300°C). Note dominance of red and blue colours, relative to yellow, in the immediate vicinity of the Garnet Lake locality. Hudson Resources has targeted and drilled kimberlite in the lake labelled “Itisooq”.

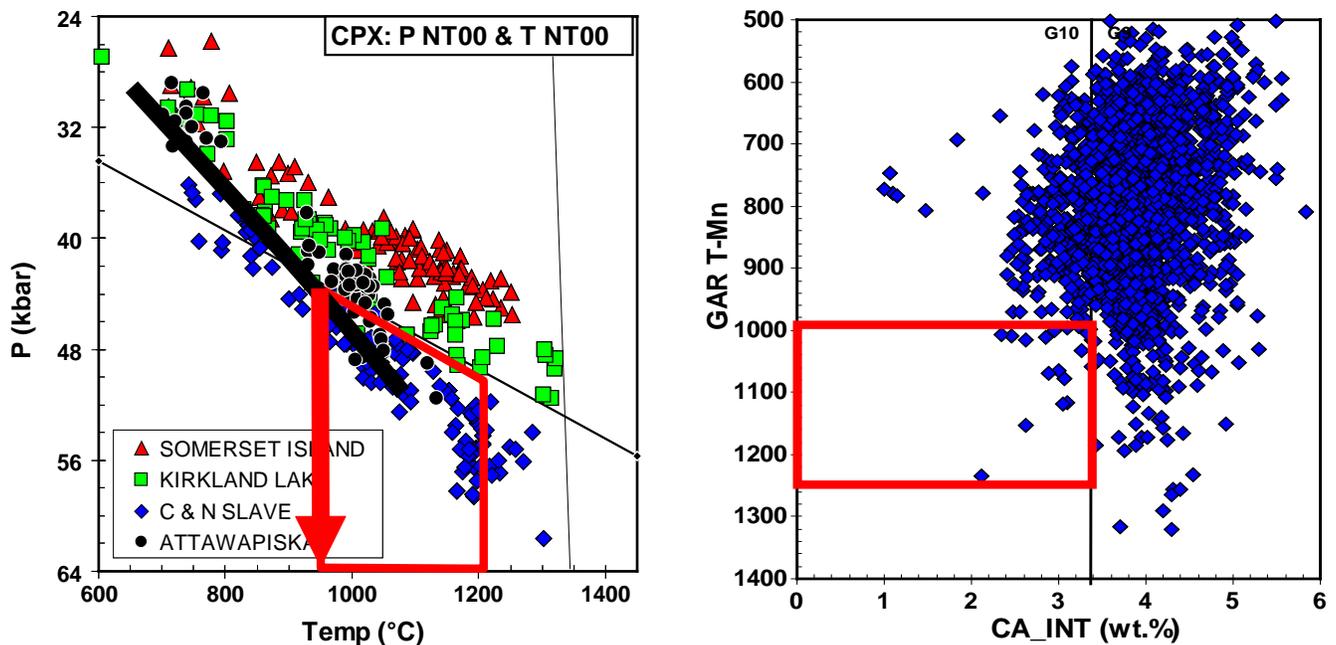


Fig. 4. The clinopyroxene geotherm (black dots and thick black line, at left) for the Attawapiskat province kimberlites defines a diamond window over the temperature range of 1000 to 1250°C (red box). A very small proportion of the Attawapiskat G9 and G10 garnets were entrained from within the diamond window (at right), implicating non-peridotitic lithologies as possible source(s) for diamond. All data from Sage (2000).

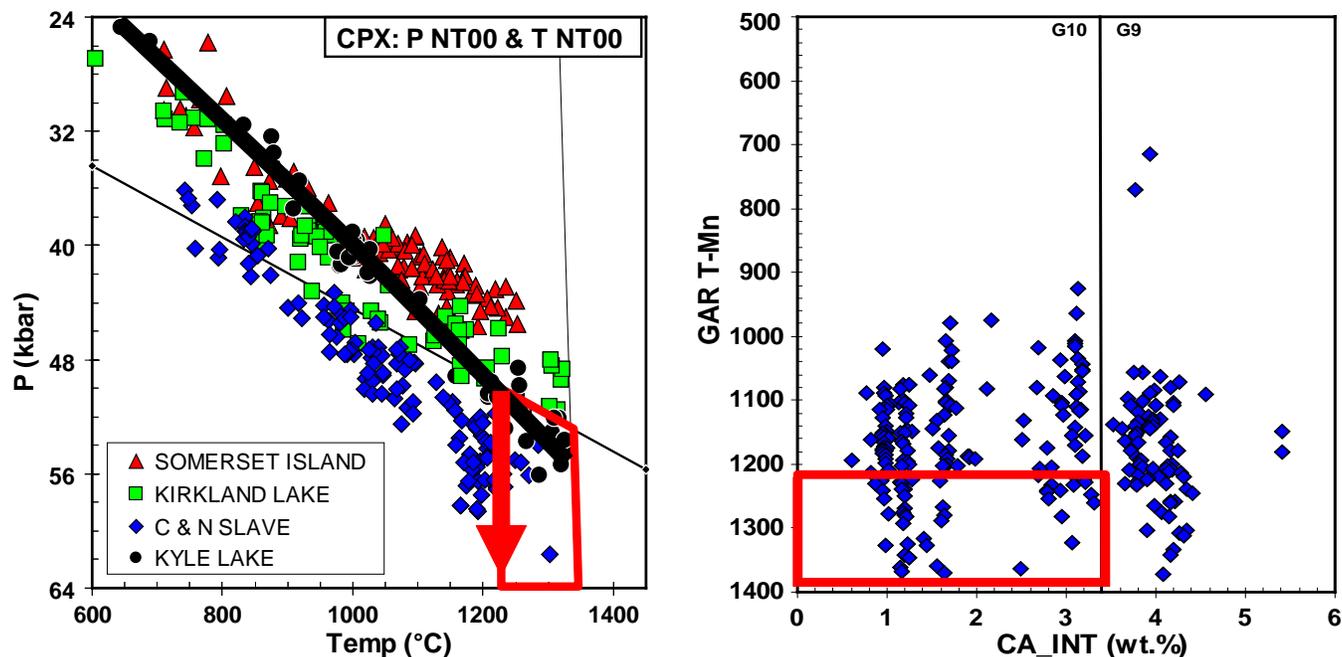


Fig. 5. The clinopyroxene geotherm (black dots and thick black line, at left) for the Kyle Lake-1 and -2 kimberlites is elevated and defines a restricted diamond window over the temperature range of 1220 to 1350°C (red box). Although most Kyle Lake garnets are derived from moderate to high temperatures, only a minority occur within the diamond window. All data from Sage (2000).

T-Mn versus CA\_INT diagram (Fig. 4) shows entrainment of abundant graphite-facies material by the Attawapiskat kimberlites, with minor diamond-facies material present. The range of T-Mn obtained here (dominantly 550-1100°C) is substantially lower than that found by Ni-thermometry for 24 Cr-pyrope garnets in the AT-56 kimberlite (dominantly 1000-1260°C; see Armstrong *et al.* 2004). The latter authors identified websteritic rock types containing Na-enriched low-Cr garnets as a likely source for diamonds in the Attawapiskat setting. No thermobarometers are currently available for low-Cr garnet compositions, but the graphite-dominant mantle sampling profile shown in Figure 4 indicates that any diamond-bearing lithology must be situated in the deepest portion of the Attawapiskat mantle. Failure by a magma to entrain such a specific diamond-associated lithology would likely result in an effectively barren kimberlite, consistent with extreme grade variability noted across kimberlite contacts within the Victor kimberlite complex (Fowler *et al.* 2001; Webb *et al.* 2004).

#### KYLE LAKE (NORTHERN ONTARIO)

Five intrusions fall within the Kyle Lake kimberlite cluster, located some 100 kilometres to the west of the Attawapiskat kimberlites. Radiometric dating indicates intrusion ages of 1123 to 1076 Ma, and the kimberlites are covered by up to 200 metres of Ordovician limestone. Garnet and clinopyroxene compositions for samples from Kyle Lake-1 and -2 are provided by Sage (2000), and are briefly discussed here. The available garnet compositions are dominated by low-Ca G10 types and most grains are derived from moderate to high mantle temperatures in the range 1000 to 1380°C (Fig. 5). Such temperatures would all fall within a 'normal' diamond window on a 'normal' mantle geotherm and would constitute strong support for a

high tenure of G10-associated peridotitic diamonds. However, the pressure-temperature data for Kyle Lake Cr-diopside grains bear witness to a distinctly elevated geotherm, thereby restricting the diamond window for Kyle Lake to high temperatures (1220-1380°C, red box in Fig. 5) and substantially reducing the peridotitic diamond potential. It is not known whether low-Cr eclogitic or websteritic lithologies occur at Kyle Lake, but the markedly different composition of peridotitic garnets indicates little communality with the mantle present at Attawapiskat.

#### CONCLUSIONS

The examples covered in this talk are of necessity selective, but nevertheless demonstrate that the application of new-age clinopyroxene and garnet thermobarometry techniques

- depend on high-quality analytical data for kimberlite indicator minerals,
- permit prioritization of indicator source(s) early during the exploration cycle,
- bring into sharp focus the mantle sampling profile of kimberlite indicator mineral source(s) and thus the likely presence and relative abundance of diamond,
- are particularly well suited to constrain the diamond tenure related to diamond-facies G10 garnets (G10D garnet types in Grütter *et al.* 2004).

Future thermochemical investigations are likely to target quantification of diamond tenure related to low-Cr eclogitic and websteritic garnet compositions (G3, G4 and G5 garnet types in Grütter *et al.* 2004).

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# Viability indicators in surficial sediments for two major base metal deposit types: Ni-Cu-PGE and porphyry Cu

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## INTRODUCTION

In the last ten years, increasing use has been made of indicator minerals from surficial sediments in the search for magmatic or metamorphosed base metal sulphide deposits (Averill 2001, 2007a,b). Many of these indicator minerals were first recognized during kimberlite indicator mineral surveys. In fact, at least four are 'crossover' minerals that are also used in kimberlite exploration but differ in their physical and chemical details (Averill 2007b). Two of the most important base metal indicator mineral suites are those associated with Ni-Cu-PGE and porphyry Cu deposits.

By definition, indicator minerals are source specific and have certain properties that facilitate their extraction and identification at very low levels in surficial sediments (Averill 2001). In general, they are (1) sufficiently heavy that they can be concentrated effectively; (2) mostly coarse grained (i.e. readily identifiable once concentrated), although silt-sized grains of gold and PGE-bearing minerals (PGMs) can also be used effectively due to their very high specific gravity; and (3) sufficiently stable that they remain intact and available for sampling in weathered sediments. The stability requirement excludes most sulphide minerals. However, chalcopyrite is somewhat resistant to degradation (Averill 2001, 2007a,b) and numerous resistant silicate and oxide indicator minerals have proven useful. For example, nearly twenty minerals have been used in Ni-Cu-PGE exploration (Averill 2007b). Many porphyry Cu indicator minerals are also available (Averill 2007a) because each primary alteration zone (potassic, phyllic, argillic, propylitic) associated with porphyry deposits supplies useful minerals. Moreover, many porphyry deposits occur in arid areas where oxidation has produced secondary minerals that are more stable than primary sulphides. This paper describes the various Ni-Cu-PGE and porphyry Cu indicator minerals and explains how they are currently being



Fig. 1. Large (~40 cm) upper mantle xenolith of garnet peridotite from the Premier kimberlite pipe, South Africa. Specimen 116686, National Museum of Natural History, Smithsonian Institution.

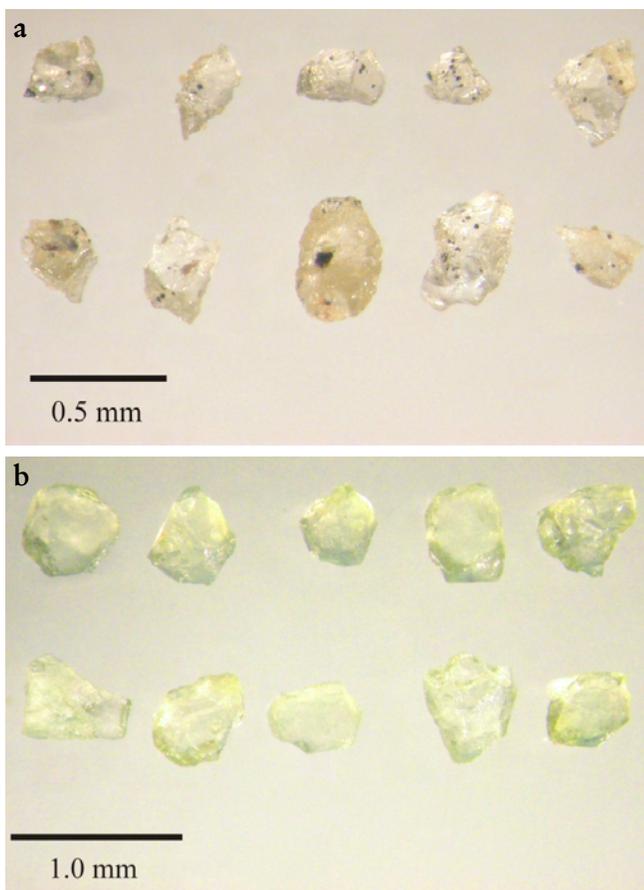
used in mineral exploration. Most of the examples shown employed either 10 to 15 kg (Ni-Cu-PGE) or 0.5 to 1 kg (porphyry Cu) samples.

## Ni-Cu-PGE INDICATOR MINERALS

Four types of indicator minerals are potentially present in the vicinity of Ni-Cu-PGE deposits (Table 1; Averill 2007b). Each type is inexorably linked to one or more of the conditions of formation of such deposits because these conditions involve chemical factors that influence mineral compositions. The two principal conditions of deposit for-

Table 1. Proven Ni-Cu-PGE indicator minerals. The cumulus indicators are identical to the melt fertility indicators but occur in higher, more localized concentrations in bedrock and produce stronger, more constricted dispersal anomalies.

Melt fertility indicators		Cumulus indicators		Hybrid indicators		Indicators of mineralization	
Mineral	Composition	Mineral	Composition	Mineral	Composition	Mineral	Composition
Enstatite	(Mg,Fe) <sub>2</sub> Si <sub>2</sub> O <sub>6</sub>	Enstatite	(Mg,Fe) <sub>2</sub> Si <sub>2</sub> O <sub>6</sub>	Ruby corundum	(Al,Cr) <sub>2</sub> O <sub>3</sub>	Chalcopyrite	CuFeS <sub>2</sub>
Forsterite	(Mg,Fe)SiO <sub>4</sub>	Forsterite	(Mg,Fe)SiO <sub>4</sub>	Hercynite	FeAl <sub>2</sub> O <sub>4</sub>	Isoferroplatinum	Pt <sub>3</sub> Fe
Low-Cr diopside	Ca(Mg,Cr)Si <sub>2</sub> O <sub>6</sub>	Low-Cr diopside	Ca(Mg,Cr)Si <sub>2</sub> O <sub>6</sub>	Cr-andradite	Ca <sub>3</sub> (Fe,Cr) <sub>2</sub> (SiO <sub>4</sub> ) <sub>3</sub>	Native palladium	Pd
Chromite	(Fe,Mg)(Cr,Al) <sub>2</sub> O <sub>4</sub>	Chromite	(Fe,Mg)(Cr,Al) <sub>2</sub> O <sub>4</sub>	Cr-grossular	Ca <sub>3</sub> (Al,Cr) <sub>2</sub> (SiO <sub>4</sub> ) <sub>3</sub>	Native gold	Au
				Uvarovite	Ca <sub>3</sub> Cr <sub>2</sub> (SiO <sub>4</sub> ) <sub>3</sub>	Sperrylite	PtAs <sub>2</sub>
						Stillwaterite	Pd <sub>8</sub> As <sub>3</sub>
						Loellingite	(Fe,Ni)As <sub>2</sub>
						Stibiopalladinite	Pd <sub>5</sub> Sb <sub>2</sub>



**Fig. 2.** Examples of (a) cumulus non-kimberlitic and (b) xenocrystal kimberlitic forsterite grains from till. Note the colourless character of the non-kimberlitic grains and the presence of microscopic Cr-magnetite inclusions in some of these grains. Source: Overburden Drilling Management client files.

mation have been elucidated most recently by Mungall (2005). The first condition is that the parental melt must be enriched in Ni-Cu-PGE, i.e. partial melting of the source rocks, normally garnet peridotite in the upper mantle (Fig. 1), must progress to a sufficient degree to produce a fertile melt. Such high-degree melts tend to be komatiitic; they are enriched in Mg and Cr in addition to Ni-Cu-PGE.

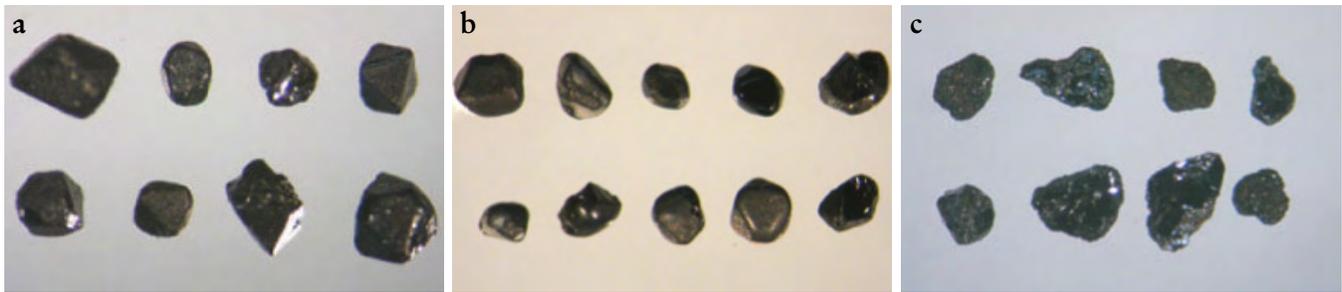
Therefore, upon emplacement in the crust, they tend to crystallize Mg- and Cr-bearing minerals that can be used as indicator minerals, including orthopyroxene (especially enstatite), olivine (especially forsterite), Cr-diopside and chromite. These indicators of melt fertility are all crossover minerals, as they are also used in kimberlite exploration. Indeed, both suites are derived from the same or similar garnet peridotite horizons in the upper mantle. Rather than being produced by melting and later recrystallization of the peridotite, however, the kimberlite indicators are solid xenocrysts captured from this peridotite by a compositionally different kimberlitic melt that originates at a greater depth; they are simply passengers in the melt.

The crossover minerals are differentiable by colour, surface texture, grain size and, in some cases, mineral chemistry. Non-kimberlitic forsterite is colourless like quartz (Fig. 2a) and often contains Cr-magnetite inclusions that render the grains paramagnetic, whereas kimberlitic forsterite is tinted green (Fig. 2b), inclusion free and non-paramagnetic. The non-kimberlitic grains are also fine biased (Table 2); the ratio of 0.25-0.5 to 0.5-1.0 mm grains is generally greater than 20:1 compared to less than 5:1 for kimberlitic forsterite. Non-kimberlitic chromite is similarly fine biased and the crystals are sharply angular to rough textured (Fig. 3a), whereas chromite xenocrysts from kimberlite are smoothed and rounded by resorption (Fig. 3b). In lateritic terrains, however, differentiation on the basis of texture may be impeded by the development of secondary corrosion textures (Fig. 3c). Non-kimberlitic Cr-diopside contains less chromium than the kimberlitic variety, reducing the amount of emerald green pigment (Fig. 4). A 1.25% Cr<sub>2</sub>O<sub>3</sub> boundary is helpful for distinguishing this 'low-Cr' diopside from xenocrystal kimberlitic Cr-diopside grains but visual identification is generally more reliable due to significant Cr<sub>2</sub>O<sub>3</sub> overlap (Averill 2007b).

Melt fertility indicators are the most abundant of the four types of Ni-Cu-PGE indicators and tend to give very large dispersal trains. For example, a 400 km long low-Cr diopside train has been defined in till down-ice from the Thompson Ni Belt in Manitoba, Canada (Fig. 5; Thorleifson & Garrett 1993) and a chromite anomaly of

**Table 2.** Typical ratios of 0.25-0.5 to 0.5-1.0 mm grains for non-kimberlitic (Project A) and kimberlitic (Project B) forsterite in till samples. From Overburden Drilling Management client files.

Sample no.	Project A			Sample no.	Project B		
	Number of forsterite grains 0.25-0.5 mm	0.5-1.0 mm	Ratio of 0.25-0.5 to 0.5-1.0 mm grains		Number of forsterite grains 0.25-0.5 mm	0.5-1.0 mm	Ratio of 0.25-0.5 to 0.5-1.0 mm grains
3169	~ 500	1	500	24-01	3	3	1
3170	~ 300	14	21	24-02	0	0	1
3171	~ 50	0	> 50	24-03	8	4	2
3172	~ 200	8	25	24-04	26	35	1
3173	~ 500	19	26	25-01	~ 100	20	5
3174	~ 80	7	11	25-02	~ 150	60	3
3175	~ 300	3	100	25-03	~ 200	88	2
3176	~ 200	2	100	25-04	~ 200	49	4
3177	~ 200	15	40	25-05	~ 40	48	1
3178	~ 600	10	60	25-06	~ 60	18	3



**Fig. 3.** Examples of transported chromite grains derived from specific bedrock lithologies. All grains are 0.5 to 1.0 mm. (a) Sharp to ragged crystals derived from peridotite (b) Resorbed crystals derived from kimberlite. (c) Crystals corroded by lateritic weathering, masking their primary form and paragenesis.

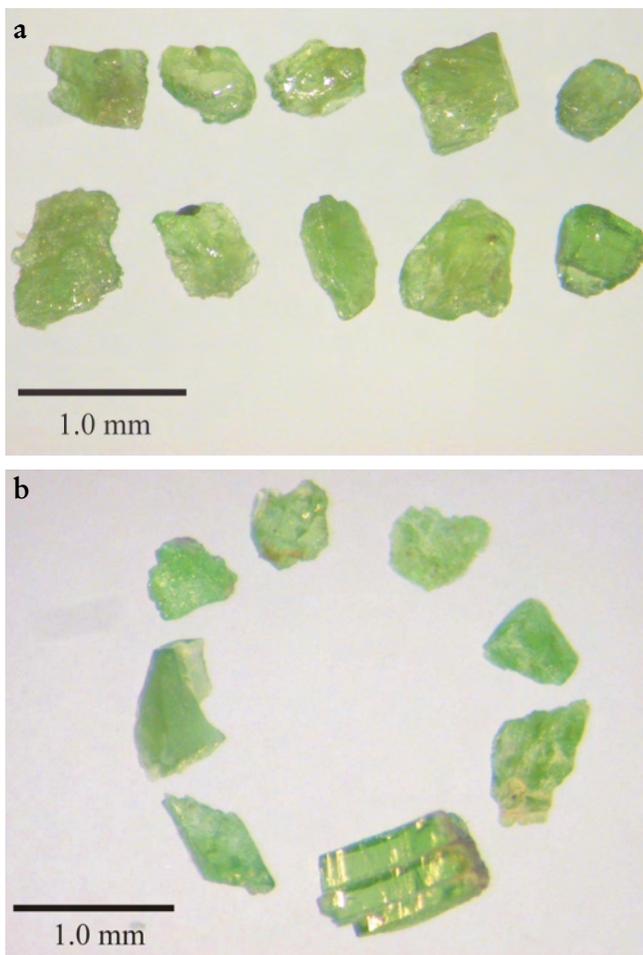
similar length extends downstream from a fertile intrusion near the head of the Attawapiskat River, Ontario (Crabtree 2003). However, a fertile mantle melt does not in itself signify a mineralized intrusion or komatiitic belt. A second critical condition must be fulfilled when this melt is emplaced in the crust – the melt must become saturated in sulphur (e.g. Mungall 2005; Naldrett 2005), inducing the

separation of an immiscible sulphide liquid from the silicate phase. This sulphide liquid collects Ni-Cu-PGE from the melt and, being denser, settles in pools or layers, further concentrating the metals.

Sulphide saturation can be achieved passively, for example by slow cooling of the melt following emplacement, but dynamic processes are much more efficient. The two main dynamic mechanisms are (1) copious fractionation of cumulus minerals at a particular time or site during emplacement or extrusion of the melt, as appears to have occurred at the mouth of the feeder conduit of the Reid Brook Intrusion at Voisey's Bay, Newfoundland (e.g. Naldrett 2005); and (2) assimilation of felsic country rocks, especially sulphide-bearing metasediments, as also occurred at Voisey's Bay (Li & Naldrett 2000). Each process potentially generates useful indicator minerals.

The main cumulus indicator minerals are the same as the melt fertility indicators (Table 1), i.e. enstatite, forsterite, low-Cr diopside and chromite. However, the anomalies that they produce in surficial sediments are stronger and more constricted because major cumulus mineral segregation is concentrated at specific sites, whereas melt fertility is a feature of the entire intrusion or lava flow. This is well illustrated by a chromite-in-till anomaly associated with the mineralized portion of the Lac des Iles Intrusive Complex, Ontario (Fig. 6).

The indicator minerals produced by assimilation of felsic country rocks by a komatiitic melt are hybrid alteration minerals containing both felsic elements, such as Si and Al, and mafic elements, such as Mg, Cr and Fe (Table 1). Examples are hercynite, ruby corundum and green Cr-garnet. In some cases the minerals are hydrated. For example, the till at Lac des Iles, Ontario is very anomalous in hydrated Cr-andradite garnet, defining a dispersal train roughly coincident with but up to 100 times stronger than the chromite train (Fig. 6). The hydrated grains are cryptocrystalline and drusy (Fig. 7a), bearing little resemblance to ordinary crystalline garnet. Similar grains have not been identified in till elsewhere but hydrated Cr-grossular garnet locally sheaths chromitite bands (Fig. 7b) in the layered Bushveld Complex, South Africa. More Cr-rich uvarovite garnet of a normal crystalline form is very abundant at Outokumpu, Finland, both in the calc-silicate alteration shells bordering the fertile serpentinites (Kontinen 1998) and the till down-ice from these serpentinites (Aumo & Salonen 1986).



**Fig. 4.** Examples of (a) cumulus non-kimberlitic low-Cr diopside grains from till; and (b) xenocrystal kimberlitic Cr-diopside grains from till. Note the paler emerald green colour of the non-kimberlitic grains, reflecting their lower  $\text{Cr}_2\text{O}_3$  content. Source: Overburden Drilling Management client files.

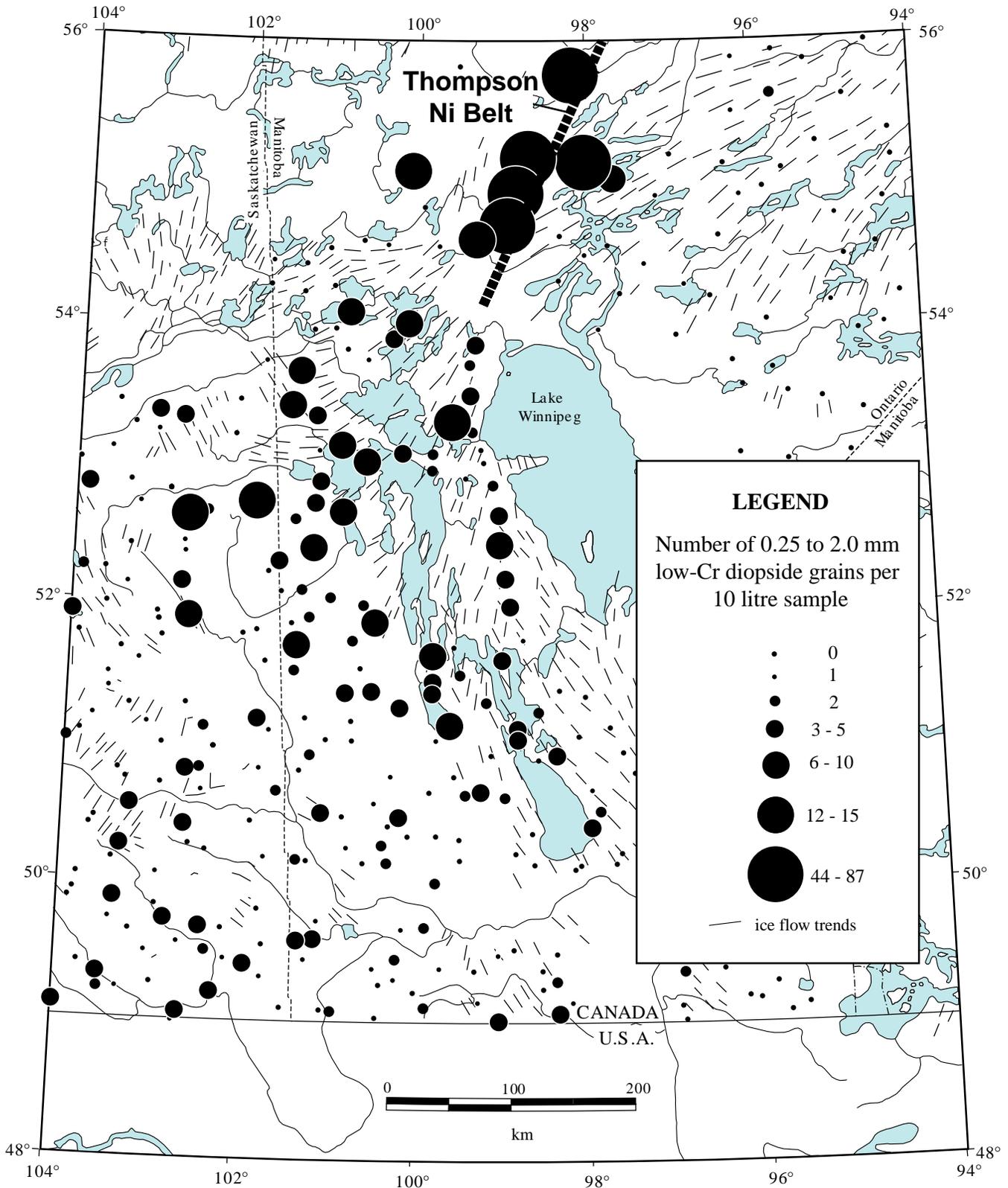


Fig. 5. Regional distribution of low-Cr diopside in till in western Manitoba and eastern Saskatchewan. Note the broad dispersal train extending 400 km southwest from the Thompson Nickel Belt. Modified from Thorleifson & Garrett 1993.

The fourth group of Ni-Cu-PGE indicators is derived directly from sulphide-bearing mineralized zones (Table 1) and is limited by the inherent instability of most sulphide minerals in weathered surficial sediments. Pyrrhotite,

pyrite and all Ni and PGE sulphides appear to be completely unstable, whereas chalcopyrite is marginally stable (Averill 2001, 2007b). Significant chalcopyrite anomalies are identified by comparing the number of chalcopyrite

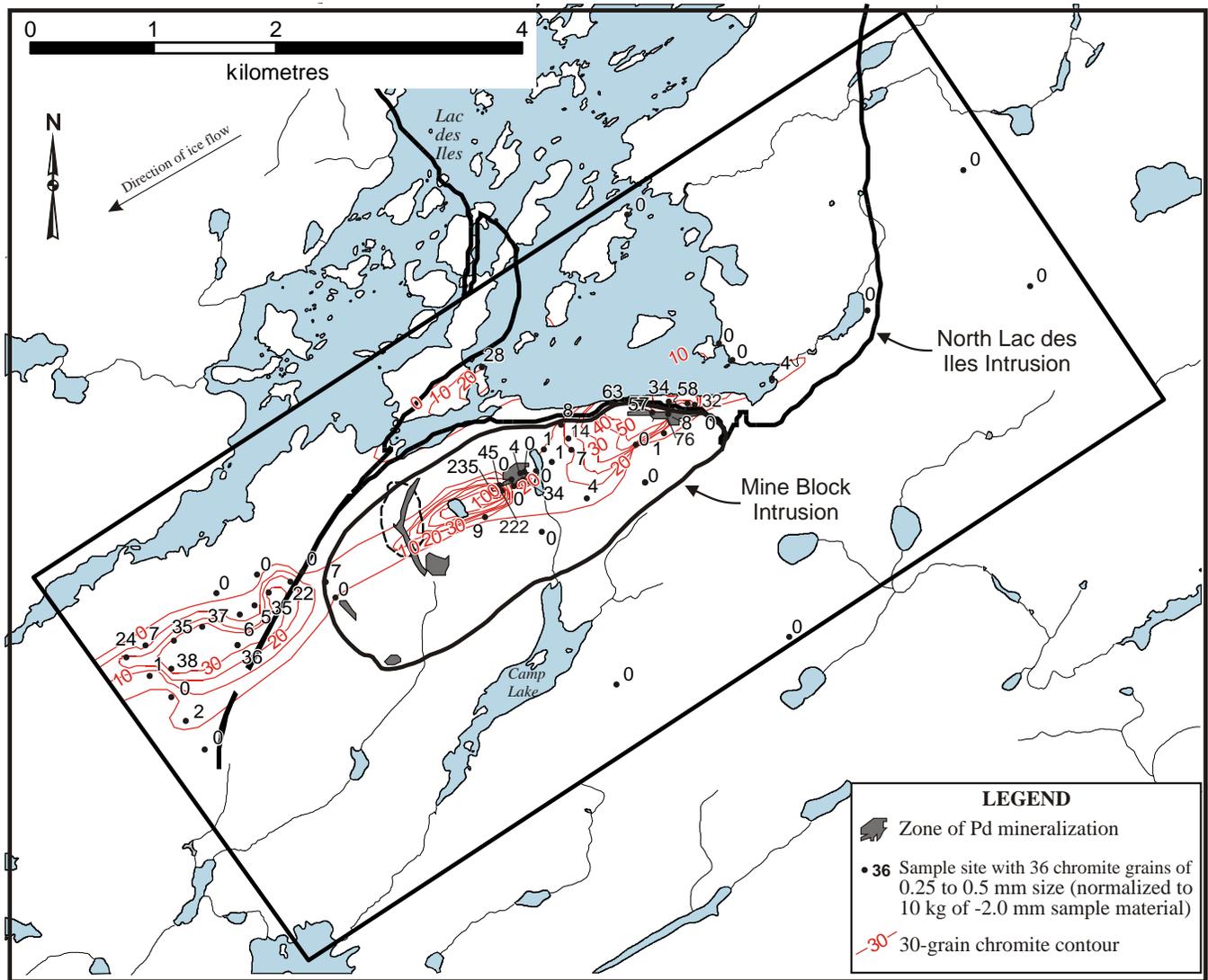


Fig. 6. Distribution of chromite in till near the Lac des Iles Pd mine, Ontario. From Barnett & Averill, in press.

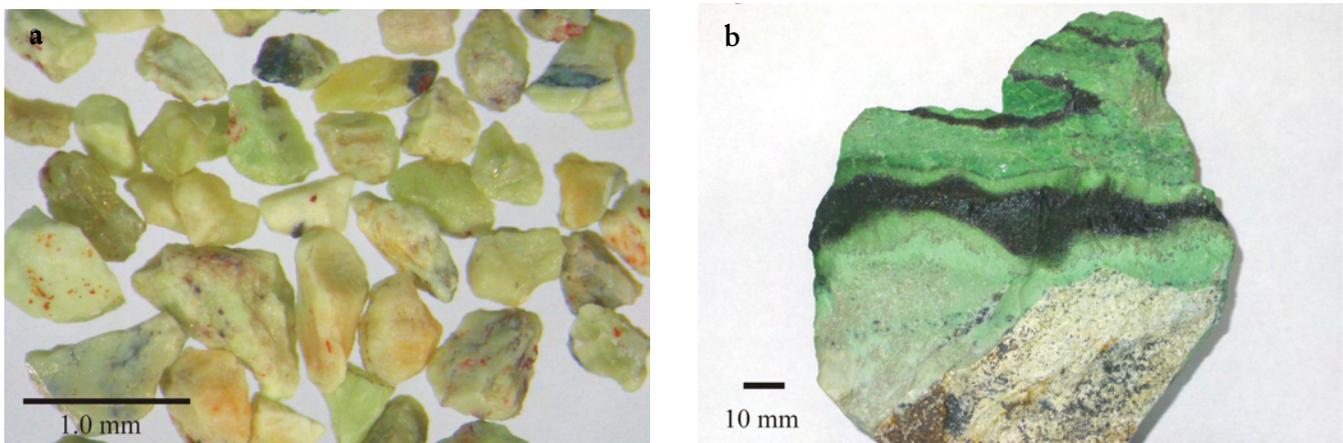
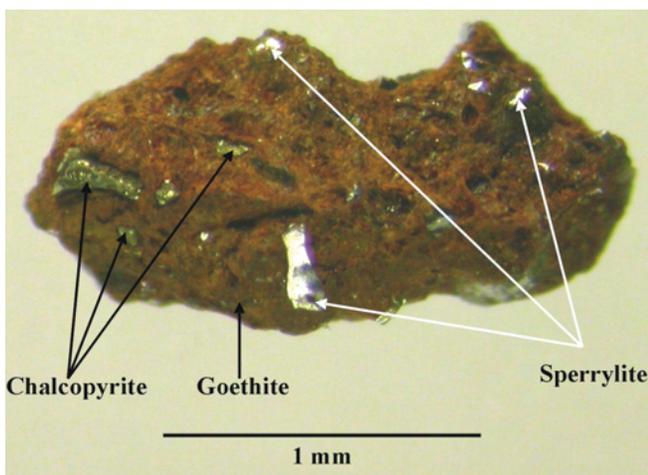


Fig. 7. Examples of (a) Cr-andradite garnet grains from till at the Lac des Iles Pd mine, Ontario and (b) Cr-grossular garnet associated with chromitite bands in the Bushveld Complex, South Africa. Note the cryptocrystalline (hydrated) form of the garnet at both localities and the variation in colour from white to green with increasing  $\text{Cr}_2\text{O}_3$  content and with decreasing distance from the chromitite bands. From Barnett & Averill, in press.

**Table 3.** Examples of relative abundances of chalcopyrite and pyrite grains in 10 to 15 kg sediment samples. The samples in each batch are consecutive (no gaps). The till samples contain normal background concentrations of sulphides whereas several of the alluvial gravel samples are anomalous in chalcopyrite. The fresh till samples were obtained by reverse-circulation drilling. They retain all of their original sulphide grains whereas in the weathered samples most sulphide grains have been degraded, with chalcopyrite degradation lagging pyrite degradation. Source: Overburden Drilling Management client files.

Number of sulphide grains by sample type and location											
Unweathered till Quebec			Weathered till				Weathered alluvium Peru				
Sample no.	Cpy	Py	Sample no.	Cpy	Py	Sample no.	Cpy	Py	Sample no.	Cpy	Py
24-01	0	~6000	003	1	5	042	1	~40	251	~200	~40
24-02	10	~4500	004	0	1	043	9	~50	252	11	3
24-03	8	~4000	005	1	8	044	3	7	253	0	1
24-04	12	~8000	006	2	7	045	4	5	254	~300	~100
25-01	10	~4500	007	13	~30	046	6	5	255	82	~80
25-02	6	~5000	008	10	~25	047	11	~50	256	10	~150
25-03	8	~6500	009	11	~100	048	3	15			
25-04	13	~10,500	010	3	7	049	4	10			
25-05	10	~7000				050	7	~400			
25-06	8	~4500				051	1	~50			

and pyrite grains present in the sample, as shown for the four sample batches in Table 3. In most rocks, pyrite is at least 1000 times more abundant than chalcopyrite. This high ratio is maintained in fresh till, as shown by the Quebec samples that were obtained by deep drilling. In shallower till at the C-horizon soil level, pyrite degrades quickly but chalcopyrite partially survives. Due to this lag effect, the chalcopyrite background can effectively become as high as the pyrite background, as shown by the Ontario and Nunavut till samples. Anomalous samples generally contain tens to hundreds of surviving chalcopyrite grains, as shown by the batch of alluvial gravel samples from Peru where chalcopyrite has remained sufficiently stable thus enabling its use as an indicator mineral even though weathering is more severe than in Canada.



**Fig. 8.** Small gossan fragment from Broken Hammer occurrence, Sudbury, illustrating variable resistance to weathering of sulphide and arsenide minerals. All pyrrhotite/pyrite has degraded to goethite but chalcopyrite and sperrylite remain fresh. Photograph courtesy of Beth McClenaghan and Doreen Ames, Geological Survey of Canada.

Till sampling near eight widely scattered PGE occurrences in Ontario, including Lac des Iles in the northwest and Broken Hammer near Sudbury (Ames *et al.* 2006), has consistently shown a total absence of PGE-bearing sulphide and telluride minerals even where these are the main PGMs in the mineralized zones. All PGM grains recovered from the till, other than rare grains of native Pt or Pd, have been of PGE-bearing arsenides and antimonides such as sperrylite and stibiopalladinite, clearly demonstrating that these minerals are much more stable than PGE sulphides and tellurides. Loellingite, a Fe-arsenide mineral that commonly contains significant Ni, is similarly stable. Gossan fragments from Broken Hammer (Fig. 8) contain fresh sperrylite and chalcopyrite grains, whereas all pyrrhotite and pyrite has been oxidized to goethite. The regional PGE arsenide/antimonide background for till throughout Ontario is zero grains per 10 kg till sample and the presence of even one grain has invariably indicated proximity to mineralized bedrock.

#### PORPHYRY Cu INDICATOR MINERALS

Porphyry Cu indicator mineralogy (PCIM®)<sup>1</sup> has emerged as a significant exploration tool only in the last five years (Averill 2007a). The initial focus was on porphyry deposits in arid regions; for example in the Atacama Desert in Chile, the climate has led to the stabilization of primary sulphide mineralization by conversion to chemically resistant (to ongoing arid weathering) supergene minerals, such as jarosite, alunite, atacamite and turquoise. The original test samples were primarily of *chusca* (powdery soil) developed on alluvium and were typically collected at a depth of 0.2 to 0.3 m, taking care to avoid surface sediment potentially containing wind-blown mineral contamination from mining or drilling activity. Samples of fresher alluvium from deep reverse-circulation holes drilled to test bedrock have also been used effectively to extend the exploration

1. PCIM is a registered trademark of Overburden Drilling Management Limited.

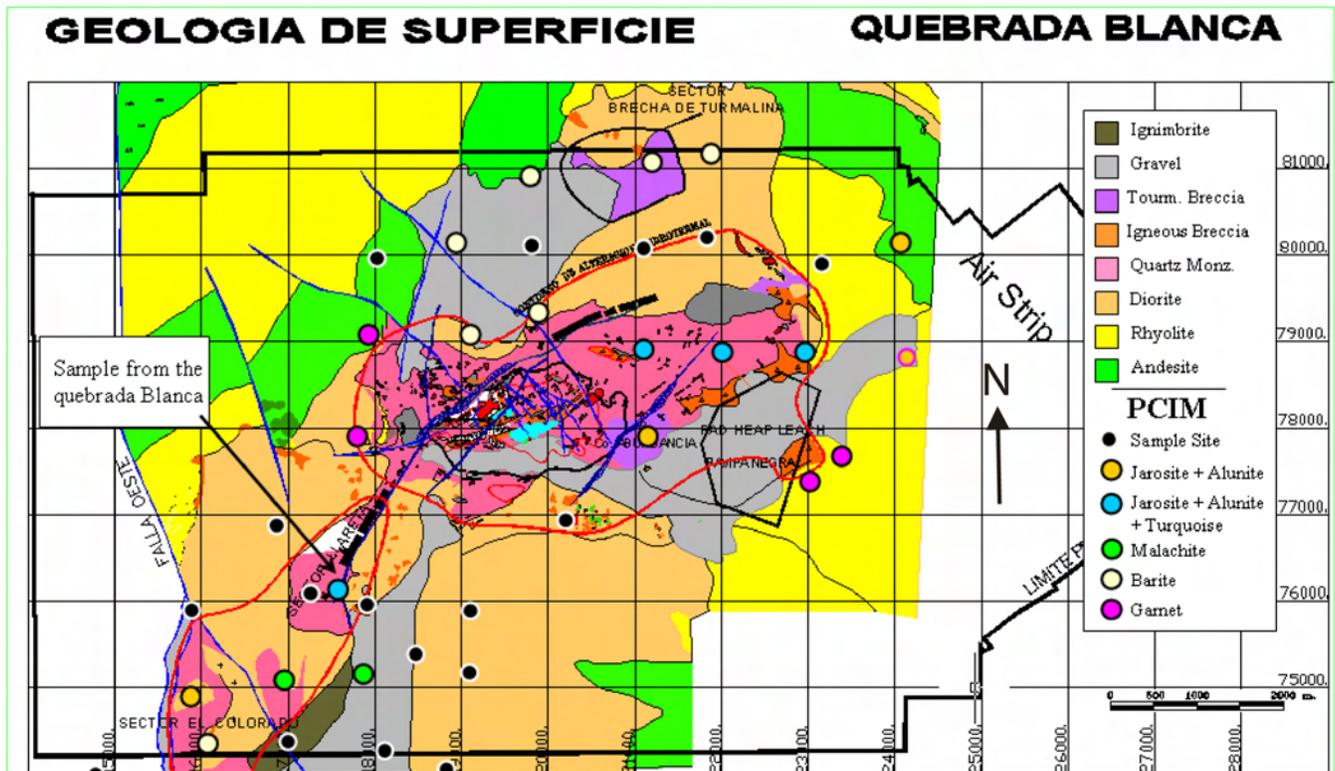


Fig. 9. Distribution of porphyry Cu indicator minerals (PCIMs) in weathered alluvium near the Quebrada Blanca deposit, Chile. Courtesy of Aur Resources Inc.

coverage of the holes at minimal added cost. More humid regions have recently been tested with appropriate changes to the sampling medium and indicator mineral suite. In addition, PCIM technology has been used to explore for epithermal Au deposits peripheral to porphyry Cu deposits.

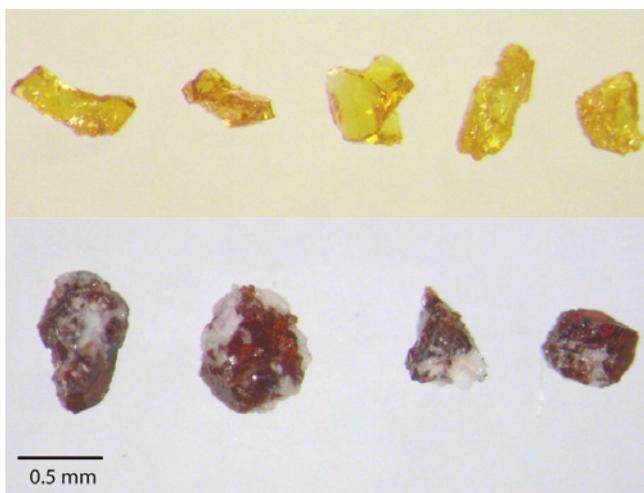
PCIMs are of the same grain size as kimberlite and Ni-Cu-PGE indicator minerals (0.25 to 2.0 mm) and tend to produce much stronger anomalies in surficial sediments due to the extreme size and alteration intensity of porphyry systems. Consequently, only 0.5 to 1 kg rather than 10 to 15 kg samples are required, facilitating sample collec-

tion and shipping, and just one sample/km<sup>2</sup> has proven sufficient to identify and outline significant porphyry Cu and epithermal Au systems (Fig. 9). However, processing costs are similar to those incurred on kimberlite and Ni-Cu-PGE indicator mineral surveys because PCIMs have a wider specific gravity range, requiring the extraction and examination of both a mid-density (specific gravity of 2.8 to 3.2) and heavy (specific gravity > 3.2) mineral concentrate.

Ten PCIMs have proven useful to date in arid regions (Table 4). These include five hypogene alteration minerals – diaspore, Mg-tourmaline (dravite), FeCaMn-garnet (primarily andradite but variably grossular or spessartine), pri-

Table 4. Proven porphyry Cu indicator minerals in surficial sediments in arid regions.

Mineral	Composition	Principal provenance (alteration zone)				
		Potassic	Argillic	Phyllic	Propylitic	Epithermal Au
Hypogene suite						
Diaspore	AlO(OH)					
Alunite	KAl <sub>3</sub> (SO <sub>4</sub> ) <sub>2</sub> (OH) <sub>6</sub>					
Dravite	NaMg <sub>3</sub> Al <sub>6</sub> (BO <sub>3</sub> ) <sub>3</sub> (Si <sub>6</sub> O <sub>18</sub> )(OH) <sub>4</sub>					
Andradite	Ca <sub>3</sub> Fe <sub>2</sub> (SiO <sub>4</sub> ) <sub>3</sub>					
Barite	BaSO <sub>4</sub>					
Supergene suite						
Alunite	KAl <sub>3</sub> (SO <sub>4</sub> ) <sub>2</sub> (OH) <sub>6</sub>					
Jarosite	KFe <sub>3</sub> (SO <sub>4</sub> ) <sub>2</sub> (OH) <sub>6</sub>					
Atacamite	Cu <sub>2</sub> Cl(OH) <sub>3</sub>					
Turquoise	CuAl <sub>6</sub> (PO <sub>4</sub> ) <sub>4</sub> (OH) <sub>8.5</sub> H <sub>2</sub> O					
Malachite	Cu <sub>2</sub> CO <sub>3</sub> (OH) <sub>2</sub>					



**Fig. 10.** Examples of andradite garnet grains from weathered alluvium near a porphyry Cu deposit. Grain colour may vary from yellow-orange (upper row) to red-orange (lower row; garnet is intergrown with fine-grained quartz alteration) or orange-brown.

mary alunite and barite – plus two supergene alteration minerals – jarosite and secondary alunite – and three ‘oxide’ Cu minerals, turquoise, atacamite and malachite. Other minerals showing significant promise are red rutile, rose zircon, blond titanite, sapphire corundum, apatite and possibly epidote and biotite. Together these minerals fingerprint the overall porphyry Cu system; some also define individual alteration and mineralization zones within the system. For example, the presence of diaspore, tourmaline or primary alunite indicates advanced argillic or potassic alteration, FeCaMn-garnet indicates propylitic alteration and barite suggests a transition from porphyry Cu to epithermal Au mineralization. This indicator mineral zoning was clearly demonstrated in one of the earliest PCIM surveys, which was performed in 2003 by Aur Resources Inc. at the company’s Quebrada Blanca mine in Chile. The terrain at Quebrada Blanca is steeply sloping and the thickness of the alluvial cover ranges from <1 to ~20 m. Aur collected 38 samples at ~1 km intervals. These samples were processed blindly by the author’s company with no knowledge of the sample locations or deposit geology, yet the indicator minerals obtained (Fig. 9) clearly outline the outer propylitic (andradite garnet) and more central advanced argillic/potassic (jarosite, alunite, turquoise) alteration zones and, at higher elevations, suggest a change to epithermal (barite) alteration.

Andradite garnet, together with its grossular and spessartine variants, appears to be the ‘holy grail’ of PCIMs. Anomalous concentrations of this mineral have been encountered in the surficial sediments at every porphyry Cu deposit tested to date even though garnet alteration has only been reported in bedrock at a few deposits (e.g. Escondida; Padilla Garza *et al.* 2001). Andradite forms the largest dispersal anomaly at most porphyry deposits, including Quebrada Blanca (Fig. 9), because it is closely associated with propylitic alteration, which is the weakest, outermost, uppermost and generally most extensive type

of porphyry-related hypogene alteration and may be the only alteration zone exposed at unroofed deposits. Its chemical composition ( $\text{Ca}_3\text{Fe}_2(\text{SiO}_4)_3$ ) closely reflects the Fe (pyrite) + Ca (calcite, epidote) metasomatism that characterizes propylitic alteration. It is easily identified in surficial sediment samples because porphyry deposits typically occur in unmetamorphosed terrains lacking almandine and other types of garnet. The andradite grains are generally of a distinctive yellow-orange, red-orange or orange-brown colour (Fig. 10) and may have adhering silica (quartz) alteration. They are also very stable in surficial sediments, whether in arid or humid regions.

## DISCUSSION AND CONCLUSION

Over the last ten years, following on the heels of many successful kimberlite indicator mineral surveys in Canada, increasing use has been made of base metal indicator minerals in surficial sediments. Two of the most promising types are Ni-Cu-PGE and porphyry Cu indicator minerals because both tend to give large, distinctive dispersal anomalies.

The Ni-Cu-PGE indicator minerals are of four main types. In order of decreasing anomaly size and increasing affinity with actual Ni-Cu-PGE mineralization, these are (1) the Mg- and Cr-rich minerals enstatite, forsterite, low-Cr diopside and chromite, which indicate a fertile, Ni-Cu-PGE-rich komatiitic melt and can produce dispersal anomalies hundreds of kilometres long; (2) the same four minerals but at higher concentrations in a much more restricted area indicating copious, localized cumulus mineral fractionation from the melt, a dynamic process capable of rapidly inducing sulphide saturation in the residual melt and the separation and pooling of an immiscible, Ni-Cu-PGE-rich sulphide liquid; (3) hybrid felsic/mafic (Si, Al/Mg, Cr, Fe) alteration minerals, such as ruby corundum, hercynite and green Cr-garnet, indicating contamination of the komatiitic melt by felsic crustal rocks, another dynamic process that can rapidly induce sulphide saturation and pooling; and (4) grains of actual mineralization that are sufficiently stable to survive in weathered surficial sediments, especially chalcopyrite and PGE-arsenides and antimonides. The presence of even one PGE-arsenide or antimonide grain has invariably indicated close proximity to mineralized bedrock.

In the case of a mantle-derived melt, the above four mineral groups can be used in sequence, progressively tightening the sample spacing from a few kilometres to a few hundred metres in order to first identify a fertile intrusion or komatiitic belt, then focus in on those parts of the intrusion or belt where sulphide saturation may have occurred and, finally, confirm the presence of pooled sulphides. Care must be taken to distinguish the melt fertility and cumulus indicators from their kimberlitic counterparts. In the special case of the Sudbury Igneous Complex, which appears to have been generated by wholesale melting of mostly felsic crust following asteroid impact rather than by partial melting of mantle peridotite (e.g. Naldrett 2005), minerals of the first three groups are either absent or less Mg rich (e.g. orthopyroxene occurs as bronzite rather than enstatite and olivine is fayalitic; Morris *et al.* 1995) due to

the inherently low Mg and Cr content of felsic rocks. Therefore more reliance must be placed on chalcopyrite and PGE-arsenide and antimonide grains in indicator mineral surveys at Sudbury, and a tighter sample spacing is required.

Porphyry Cu indicators have been used for only five years but appear to be particularly effective, possibly even more so than kimberlite indicators, due to the very large scale of porphyry systems and the extreme alteration associated with these systems. Just one small, 0.5 to 1 kg sample per square kilometre appears to provide very effective exploration coverage. The ten indicator mineral species presently proven for arid regions fingerprint both the overall porphyry system and, if the cover is not excessive, the individual zones of alteration and mineralization within this system. Andradite garnet is a particularly useful indicator mineral. It appears to be derived from the propylitic zone, which is the weakest, outermost, uppermost and generally largest alteration zone and may be the only exposed zone present. It is very stable, easily identified in surficial sediment samples and forms significant dispersal anomalies at all tested porphyry deposits in both arid and humid regions.

Other useful PCIMs will undoubtedly be identified as more surveys are done. Meanwhile several questions have arisen concerning the chemistry of some of the proven or promising minerals. Which, if any, of the three garnet species (andradite, grossular, spessartine) associated with propylitic alteration zones is the best indicator of Cu fertility? Is red colouration in rutile due to Cu or Cr? What imparts the distinctive rose colour to some zircons in otherwise colourless populations? Does blondness in titanite have any chemical significance? Can we discriminate chemically between primary and secondary alunite grains? To what extent are weak Cu anomalies in partial extraction geochemical surveys due to traces of Cu-bearing mineral grains in the sediment versus some mechanism of aqueous, gaseous or electrochemical migration of Cu from bedrock to surface? Recent record Cu prices suggest that the answers to such questions are of more than academic interest.

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# Field sampling for indicator minerals: How to choose and locate the correct medium and avoid anthropogenic contamination

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## INTRODUCTION

Quality controls in heavy mineral sampling programs typically concentrate on sample processing, labelling and tracking, and accidental contamination from personnel and equipment. All the money and effort invested in these quality controls may be squandered if the proper overburden medium is not sampled. Sampling overburden material that is not suitable for specific indicator minerals can yield results that are as unreliable and potentially misleading as for those that have been contaminated by anthropogenic processes.

## SAMPLE MEDIUM DETERMINATION AND COLLECTION

Heavy indicator mineral surveys typically target commodities such as gold, diamonds and base metals. The physical properties of the desired indicator mineral(s) must be examined to determine which overburden medium is ideally suited for the survey. Three basic criteria should be used to when making this determination: (1) the indicators sought; (2) the physical properties of the indicators (i.e. grain size); and (3) the character of the dispersal train generated. Once this information is known the most suitable overburden media type can be determined.

Heavy mineral surveys for gold deposits typically use only one indicator, gold grains. The majority of gold grains in a primary gold deposit are silt-sized ( $< 63 \mu\text{m}$ ), therefore, the gold that is liberated by glacial-, chemical- or other mechanical-weathering processes would also be silt sized (Averill 2001). Gold-dispersal trains are also typically short (average 500 m). Glacial till is the ideal sampling medium because it usually has a large silt-sized component and can be of local provenance. Although gold has a very high specific gravity, the small silt size of natural gold grains negates the high specific gravity and they tend to be eliminated from high-energy eskers and modern rivers, therefore making these overburden media unsuitable for gold exploration.

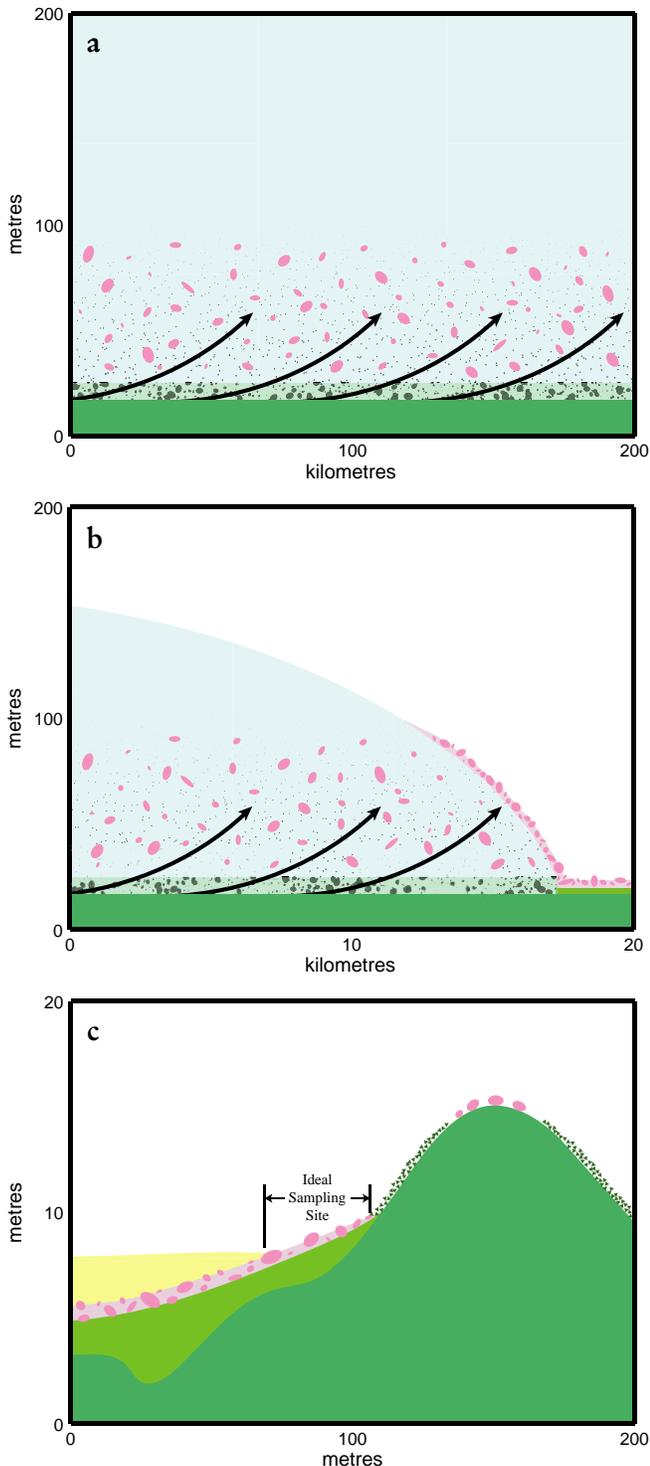
Not all till is created equal. At Rainy River, Ontario for example, two separate ice advances deposited two distinctly different till sheets (Bajc 1991). The older Labradorian till is a prime sampling medium for gold grains as it consists of stony, silt-sand till of local provenance and is invariably in contact with the subcropping Archean bedrock. The overlying Keewatin till is of very limited exploration value as it consists of more than 90% recycled Lake Agassiz clay and 1% distal pebble-sized clasts, and has had limited contact with the local bedrock. The thick Keewatin till and associated glaciolacustrine and

glaciofluvial deposits at Rainy River make sampling the desirable Labradorian till possible only by reverse circulation drilling.

Surface pit sampling is the cheapest and most common method of sampling till in areas of limited cover. Although relatively thin till may make sampling appear straight forward, the till's suitability may be suspect due to depositional and post-depositional modification.

During glaciation, the underlying bedrock is continually eroded by two processes: 1) plucking along fractures, which produces large blocks, especially of jointed granitic rocks, and 2) grinding, which produces silty rock flour (Dreimanis 1976). Most of this debris accumulates in the lower few metres of the ice sheet (Michaud & Averill 2006; Hozjan & Averill, 2007; Fig. 1a). Once the ice thins through meltdown, distal debris begins to accumulate on the ice surface where its fine matrix is susceptible to winnowing by meltwater. Eventually, when ice meltdown is complete, the coarse residue or lag from the distal debris is deposited as supraglacial till directly upon undisturbed subglacial till of more proximal provenance (Fig. 1b). The underlying subglacial till is typically 0.5 to 3 m thick with the thickest sections occurring in bedrock depressions. Sections exceeding 5 m are generally restricted to regions where the till was derived from a soft, easily eroded substrate, as in young sedimentary basins or large meltwater lakes, and was molded into drumlins.

Although armoured by the bouldery supraglacial till, the subglacial till is susceptible to erosion until a protective forest cover is established. In greenstone belts that were not flooded by glacial lakes, most bedrock highs eventually become rock outcrops topped by only a few large, perched lag boulders, mainly of rounded, distal granite from the supraglacial till (Fig. 1c). Minor hollows in the outcrops and any steep slopes adjoining these outcrops become mantled by stony, angular rubble, which is partly lag from eroded subglacial till but mostly bedrock dislodged by frost action. Texturally this rubble is matrix deficient and compositionally it reflects only the immediately subadjacent bedrock rather than that for a significant distance up-ice. It is therefore unsuitable for exploration sampling. Further downhill where the slope moderates, the distal granitic boulders tend to be partly submerged rather than perched, indicating that the supraglacial till has not been totally eroded and the underlying subglacial till layer is fully intact. Large supraglacial till boulders normally settle approximately 0.1 m into the compact subglacial till. Therefore a scattering of 1 m granitic boulders with 0.4 m exposed and 0.6 m submerged will normally signify the



**Fig. 1.** Schematic sections showing (a) pre-meltdown generation; (b) syn-meltdown deposition; and (c) post-meltdown modification of supraglacial and subglacial till over a greenstone belt. Note the difference in scale between sections and the location of ideal sample sites in 1c.

presence underfoot of up to 0.5 m of supraglacial till and at least 0.5 m of subglacial till — conditions ideal for sampling with a 1 m deep shovel pit. Further downhill, on the edge of adjoining flats, both the supraglacial and subglacial till are normally well preserved but may be completely cov-

ered by sand and silt winnowed from the up-slope till, by glaciofluvial outwash sand if an esker occurs nearby, or by glaciolacustrine clays if the flats were flooded by a glacial lake. This sand or clay cover will be apparent from a complete absence of exposed granitic boulders.

One case study that clearly illustrates the differences between sampling supra- and subglacial till started in 2004 with a till sampling program targeting gold deposits conducted by the Minnesota Department of Natural Resources (MDNR) at Mud Creek within the Vermilion greenstone belt in the northeastern part of the state. Dahl (2005) reported two types of till were present in the area: (1) an uppermost till containing large, well rounded, granitic boulders of distal provenance with a loose, sandy, moderately water-sorted, silt-depleted matrix (i.e. supraglacial till; Fig. 2a); and (2) a lower till texturally and compositionally characterized by small, more angular clasts of primarily local greenstone lithology with a compact, unsorted matrix rich in silty rock flour (i.e. subglacial till; Fig. 2b). The MDNR survey yielded normalized gold-grain background levels ranging from 0 to 3 grains and anomalies between 88 and 1282 grains.

Subsequent acquisition of the property and sampling by Rainy River Resources Ltd. (RRR) in 2006 confirmed that some of the MDNR samples were unsuitable, being either of supraglacial till or bedrock rubble (Michaud & Averill 2006). Of the 117 samples collected by RRR during two phases of exploration, all but one was of high-quality subglacial till. RRR's survey showed that background gold-grain values in high-quality subglacial till are approximately 10 grains per sample, significantly higher than the 0 to 3 grains found in the silt-depleted, supraglacial till sampled by the MDNR. Supraglacial till samples taken by the MDNR and one intentionally taken by RRR to further test gold-grain response in this type of till, yielded consistently lower values (0 to 2 grains) than those of neighbouring subglacial till samples that were not depleted in silt (typically 8 to 24 grains). A rubble sample, inadvertently taken directly overtop of a weakly mineralized shear zone produced the MDNR 1282 grain anomaly; a RRR subglacial till sample taken 100 m down-ice yielded only 21 grains.

The effect of sampling different media for coarser grained indicators can be just as striking. Kimberlite indicator minerals (KIMs) and base metal indicators are naturally medium- to coarse-grained (0.25–1.0 mm) rather than silt sized like gold grains (Averill 2001). KIM dispersal trains are also much longer than gold trains, in some cases more than 100 km. Therefore, in a regional kimberlite exploration program, it is advantageous to initially target eskers and large modern fluvial systems to search for the kimberlite field and subsequently sample subglacial till and lower order drainages to locate individual kimberlite bodies within the field.

Figure 3 shows the results of a surface sampling survey for kimberlite in Quebec. Four sediment types were sampled over the course of the exploration program: (1) till; (2) glaciofluvial gravel and sand (includes some glaciolacustrine sand); (3) modern alluvial gravel and sand; and (4) beach gravel and sand. For all samples only the 0.25 to



**Fig. 2.** Till pits. (a) Numerous  $<0.2$  m boulders and cobbles removed from the supraglacial till have been placed to the left of the pit while two large,  $>0.3$  m, partially submerged boulders with covering moss partially stripped off are visible just beyond the pit. (b) Road-cut till exposure with grey, unoxidized subglacial till and angular, locally derived, greenstone clasts.

2.0 mm fraction was processed for KIMs. Glaciofluvial gravel samples in the central portion of the property yielded very high KIM counts (often  $>100$  grains). The immediately neighbouring till in the central area yielded low KIM counts (2 to 11 grains). Fine-sand ( $<0.25$  mm) samples invariably yielded very low KIM counts; these samples should not have been collected due to the potential of producing artificially low KIM counts. Interpreting the data on the basis of overburden type appears to indicate that the source of the kimberlite is not in the central portion of the map area as suggested by the glaciofluvial KIM counts but some distance up ice; the higher KIM counts in the north-central till samples would seem to confirm this. The variability in KIM results illustrates how data interpretation must be based on a rigorous sampling protocol.

#### ANTHROPOGENIC CONTAMINATION

Present day mineral exploration not only takes place in remote areas but also in areas of current and historical mining and industrial activity. Cultural features (roads, railways, bridges, tailings ponds, etc.) may introduce anthropogenic contamination that, dependent on contaminant type, may be a mere nuisance or, more seriously, produce false anomalies.

Several indicator mineral sampling programs undertaken by the Ontario Geological Survey (OGS) have clearly demonstrated the impact of contamination. Morris

*et al.* (2002) documented a heavy mineral sampling survey in the area of the closed Winston Lake and Zenmac zinc mines near Schreiber, Ontario. Stream sediment samples taken in drainages were highly anomalous in the metamorphosed massive sulphide indicator minerals (MMSIM<sup>®</sup>s) sphalerite, chalcopyrite, pyrite, galena and gahnite (a Zn-bearing spinel that occurs in the alteration haloes of metamorphosed volcanogenic massive sulphide deposits; Morris *et al.* 2002; Fig. 4a). The entire populations of chalcopyrite and pyrite were angular, had milling cracks and were very fresh, which was unusual because sulphides are extremely susceptible to weathering in the surface environment. Some of these grains also had freshly leached surfaces. The gahnite was also very fresh and angular and always attached to quartz, which is again characteristic of milling. The results of this survey were properly interpreted as contamination because the authors were aware of the presence of the mines as well as the fact that mine haulage roads crossed active streams and were maintained with mining equipment.

Stream sediment samples taken near the town of Kapuskasing, Ontario yielded hundreds to thousands of grains of slag (Fig. 4b), a byproduct of metal smelting, as well as lesser amounts of aluminum oxide (synthetic corundum; Fig. 4b), an abrasive used in sand blasting. The source of the contamination was determined to be from railroad

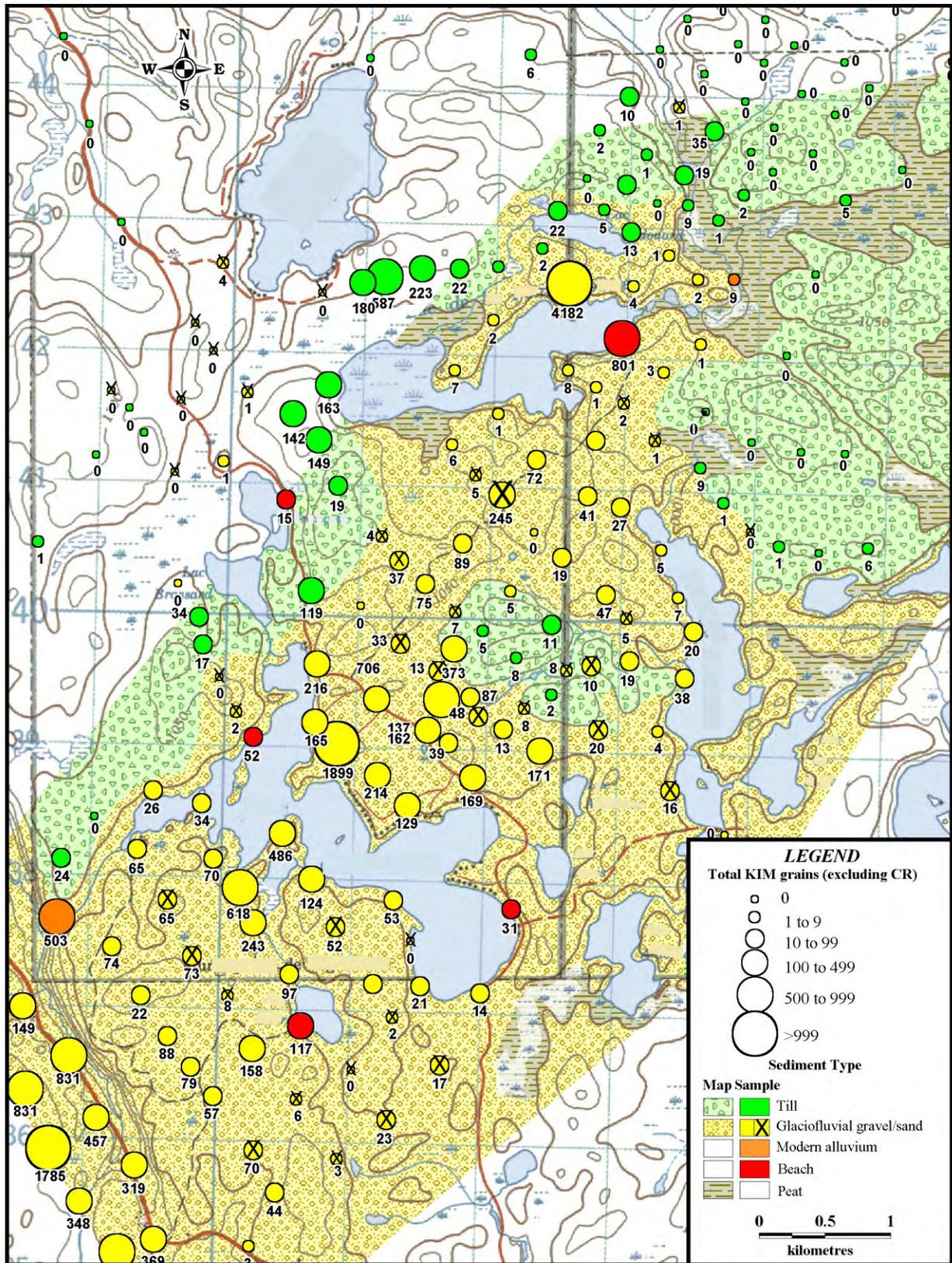
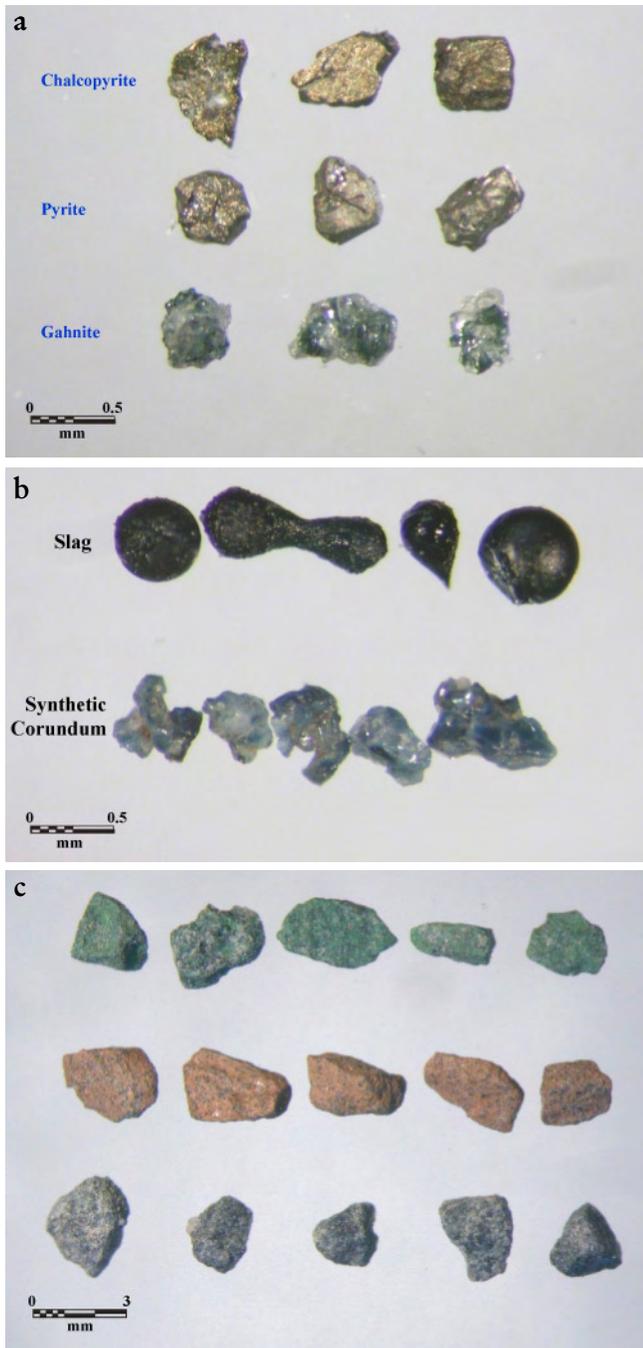


Fig. 3. Example of the distribution and strength of kimberlite indicator mineral anomalies in surface samples by sediment type.

beds and bridges 2 to 5 km upstream from the sample sites (Ontario Geological Survey 2001).

Felix *et al.* (2006) reported on a heavy mineral survey near Tweed, Ontario, a populated area that has seen his-

torical production of a variety of industrial minerals and precious and base metals dating back to the early 1900s. Results of the survey showed 97 of 250 samples were contaminated. The contaminants included paint-coated epi-



**Fig. 4.** Heavy mineral contamination in the form of (a) chalcopyrite, pyrite and gahnite contamination from Schreiber, Ontario, (b) slag and synthetic corundum contamination from Kapuskasing, Ontario and (c) paint-coated epidote grains from Tweed, Ontario.

dote (roofing granules used in the production of asphalt shingles; Fig. 4c), smelter slag, synthetic corundum, lead and tin solder, brass and glass (Pb crystal). Also noted was milled rock containing diverse metallic mineral grains, including primary chalcopyrite and arsenopyrite and secondary arsenolite, pharmacolite and zaratite probably derived from the site of the former Deloro gold mine (Hozjan & Averill 2007).

Airfall contamination is a rare modern phenomenon that can affect heavy mineral surveys in areas where extensive mining or smelting operations have been in effect. For example, Averill (1989) demonstrated that a property-wide gold-in-humus anomaly near Kirkland Lake, Ontario was due to silt-sized gold particles dispersed nearly 5 km from an abandoned Au-tailings pond. Similarly, a large Pd-in-humus anomaly identified by Hattori & Cameron (2004) near the Lac des Iles Pd mine in northwestern Ontario was subsequently shown by Barnett & Dyer (2005) to be due to wind-blown dust from the near-by open-pit mine operation. Although humus is not sampled in indicator mineral surveys, contamination of underlying till may result if the till is exposed or if humus is accidentally sampled.

### CONCLUSION

Sample quality and reliability of data from a heavy indicator mineral survey can be enhanced by field crews who are informed about the targeted indicator mineral(s) and also the desirable overburden sampling medium. Sampling predetermined sites that do not contain the preferred sampling medium may have a detrimental impact on the results of a survey. Foreknowledge of potential anthropogenic influence may also provide insight into anomalies that may be amplified or false.

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# Public sector case study: Indicator mineral survey of Minnesota

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## ABSTRACT

As a cooperative project of the Minnesota Geological Survey and industry, the entire State of Minnesota and immediately adjacent regions were sampled for indicator minerals and till geochemistry at a 30 km spacing during the autumn of 2004 (Thorleifson *et al.* 2007). Within target cells, each measuring a quarter-degree latitude by a half-degree longitude, till from between about 1 and 2 m depth was sampled by filling a 15 litre plastic pail. At a few sites, vertical profiles were collected. In addition, three transects to the north were sampled, to help identify sediments derived by long-distance glacial transport, to obtain reference samples from the Thompson nickel belt, and also to extend sampling to the limit of Hudson Bay-derived carbonate-bearing sediments, in order to permit comparison to Minnesota carbonate-bearing sediments. Three control samples from Kirkland Lake, Ontario, which are anomalous in kimberlite indicator minerals, were also obtained. The resulting batch consisted of 250 samples covering Minnesota and immediately adjacent areas, 20 samples from the northern transects, and the three control samples. Upon completion of the sampling, the samples were randomized, given numeric laboratory identifications, and shipped to the processing lab, where four quarter-litre splits, two for fine-fraction geochemistry, one for texture, and one for an archive, were removed. The remaining 14 litres were disaggregated, screened at 2 mm, and the gravel was retained for lithological analysis. The <2 mm fraction was then processed for gold grains, a ferromagnetic heavy mineral concentrate, and a non-ferromagnetic heavy mineral concentrate that supported subsequent analysis for precious metal, base metal, and gemstone indicator mineral counts, indicator mineral chemistry, bulk mineralogy counts, and heavy mineral geochemistry. The resulting data are now a significant new information resource with respect to environmental geochemistry topics, such as understanding the distribution of deleterious elements in food and water, while providing insights into the transport history and the composition of the sediments that make up soil parent materials. Many variables provide insights into regional geology and reflect known mineral deposits. Some of the data provide insights into what may be mineralization that was not previously recognized, such as various base metal- and precious metal-related elements that show patterns of varying clarity over portions of the state. With respect to kimberlite indicator minerals, there are two noteworthy patterns, including Cr-pyrope garnets in an area from the Twin Cities to southwestern Minnesota, as well as Mg-ilmenites and high-chrome Cr-diopsides in the far north-central part of the state. Sample spacing in the thin sediments of northeastern Minnesota was not adequate to fully test for the presence of sources, such as potential single kimberlite pipes, so samples at 10 km spacing were collected in this region as a follow-up. Elsewhere, drilling may be required to adequately test areas of thick sediment cover. The kimberlite indicator mineral results may indicate sources within the state, or quite possibly could be manifestations of long-distance glacial-sediment transport, possibly from known or unknown sources in neighbouring states or in Canada. In summary, the results are a highly significant step forward in mapping the state geochemical landscape, in clarifying mineral potential, in provision of reference data useful to environmental protection, public health, and exploration, and in supporting follow-up with respect to potential mineralization.

## INTRODUCTION

Minnesota is a region with a high potential for discoveries of economic base metal, precious metal, and gemstone deposits. The Minnesota Geological Survey (MGS) therefore maintains public knowledge that supports mineral exploration and mineral development, in cooperation with

Minnesota Department of Natural Resources (DNR) and Natural Resources Research Institute (NRRI).

Given the rapid progress that has been made in indicator mineral methods (e.g. Paulen & McMartin 2007), new information is required to help adapt these methods to Minnesota geology, to map regional background trends

and to map characteristics of the sediments that govern the manner in which the methods may be applied. Concurrently, efforts to obtain this regional information were recognized as an opportunity to obtain enhanced information on regional soil chemistry, a key factor in many topics, including considerations related to public health.

In mid-2004, plans to do so at MGS coincided with similar plans being developed by WMC Resources Ltd. (WMC). By entering into an agreement to cooperate on a survey, both parties were able to increase their effectiveness and likelihood of success. The agreement between MGS and WMC specified a survey design that satisfied the objectives of both parties, outlined a cost-sharing agreement in which about 5% of the costs were borne by MGS, and specified that all resulting data would be made public by early 2007, allowing WMC a reasonable period to conduct their follow-up.

The State of Minnesota and immediately adjacent regions were sampled at a spacing of about 30 km. Target cells across Minnesota were designated at quarter-degree latitude and half-degree longitude spacing. Within each target cell, till from between 1 and 2 m depth was sampled at an arbitrarily chosen site by filling a 15 litre plastic pail. At a few sites, vertical profiles were collected.

In addition, three transects were sampled to the north in Canada, to assess the background inherited by long-distance transport of glacial sediment, to obtain reference samples from the Thompson nickel belt, and also to extend sampling to the limit of Hudson Bay-derived carbonate-bearing sediments in order to better constrain regional trends in sediment carbonate content. Three control samples from Kirkland Lake, Ontario, known to be anomalous with respect to kimberlite indicator minerals, also were added to the batch. The resulting sample set thus consisted of 250 samples covering Minnesota and immediately adjacent regions, 20 samples from Manitoba and northwestern Ontario, and three Kirkland Lake standards. Upon completion of the survey, the samples were randomized so that subtle geographic trends would be distinguishable from potential lab-induced trends, given numeric laboratory identifications, and were then shipped to the sample processing contractor.

The resulting open file (Thorleifson *et al.* 2007), included data for sample location, description of the sampled sediments, lithological analyses that included texture, matrix carbonate content, and pebble lithology based on the 8 to 16 mm fraction, mineralogy of the heavy mineral fraction, geochemistry of the <63 micron fraction, geochemistry of the heavy mineral fraction, visible gold-grain morphology and count, mineral chemistry, and indicator mineral counts.

### REGIONAL GEOLOGY

The Quaternary geology of Minnesota (Hobbs & Goebel 1982) is dominated by till related to Late Wisconsinan glaciation. In the central and northeastern regions of the state, the till is sandier with sand and gravel deposits more common. In addition, older tills occur, particularly in the subsurface, with sand and gravel extensive in the central

part of the State, and the silty clay deposits of Lake Agassiz dominate in the northwest. The Late Wisconsinan tills are readily divisible into the carbonate- and shale-bearing sediments of the Des Moines Lobe and associated St. Louis sublobe that were derived from the Red River valley/Lake Winnipeg region, the red-volcanic-rock-bearing sediments derived from the Lake Superior basin and deposited by the Superior lobe, and intervening sediments of the Rainy Lobe that were derived from the northeast and that are dominated by debris, such as granite clasts. The Rainy lobe split as it diverged around the bedrock high of the Mesabi Iron Range, resulting in the Wadena lobe to the north, which deposited sediment in west-central Minnesota that was moderately rich in carbonate, and the Brainerd lobe to the south, which deposited carbonate-poor sediment in east-central Minnesota. Beyond the limit of Late Wisconsinan glaciation in the southeastern and southwestern corners of the state, older tills occur that are dominantly derived from the northwest, and thus bear a moderate level of carbonate and a low level of shale.

Minnesota bedrock geology (Morey & Meints 2000) ranges from thin and discontinuous Mesozoic sedimentary rocks in the southwest, to Paleozoic carbonates and sandstones in the southeast, to a diverse array of Precambrian rocks that intersect the bedrock surface from parts of southwestern Minnesota to the entire northern portion of the State. The youngest of these Precambrian rocks include Mesoproterozoic sandstones extending southwest from Lake Superior, as well as volcanic rocks and Duluth Complex gabbros and associated rocks along the north shore of Lake Superior. Paleoproterozoic rocks include the Sioux Quartzite of the southwest, and a broad array of rocks in central Minnesota, including the widely mined iron formation of the Mesabi Range. The oldest rocks exposed at the bedrock surface, of Archean age, occupy roughly the northwestern third of the State, and consist of greenstone belts and intervening intrusions and metamorphic rocks ranging in metamorphic grade up to gneiss and migmatite.

### FIELD METHODS

Bearing in mind the need to conduct basic compositional mapping of sediments for multiple applications, till was chosen as the sampling medium for the regional survey due to the role that till plays as the source of other Quaternary sediments in the region, while also being the sampling medium likely having the clearest pattern of regional derivation. In addition, till is the principal control on geochemical trends in media such as soil. Till also permits accompanying lithological analysis of the gravel fraction and the silt and clay fine fraction, which may be used to assess the provenance of the sediments in relation to bedrock sources. In contrast, fluvial or glaciofluvial sand is irregular in distribution, highly variable in composition, and lacks consistently accompanying coarse and fine fractions. Till thus provided by far the most consistent and comprehensive sampling medium available. In addition to satisfying basic mapping objectives, the field procedures were equally designed to ensure collection and processing of a set of till samples that would, to the extent possible in

a manageable and appropriate first campaign, detect indicator mineral and elemental plumes derived from potential economic mineralization, accompanied by an enhanced interpretation of glacial sediment provenance that would support interpretation and follow-up. A 30 km spacing statewide was considered adequate to consistently quantify known major features in the drift composition, to identify broad indicator mineral plumes in areas of thick sediments, and to map regional trends in background for geochemical and indicator mineral variables. Furthermore, this spacing was considered adequate for determination of what spacing would be required to carry out a follow-up survey to confidently detect individual mineral deposits, such as isolated kimberlite pipes that would be missed, if present, by the low-density survey in areas of thin and discontinuous sediments in the northeastern part of the state.

In addition, till was collected along three northern transects at a spacing of 75 to 100 km. These transects were designed to obtain data that would provide insight into the nature of sediments in Minnesota that were inherited by long-distance glacial-sediment transport from Canada. These results were regarded as needed for survey interpretation, given the likelihood that something would be found in Minnesota that could possibly be attributed either to derivation within the state, or to long distance transport from Canada. It was anticipated that something found within the state that was lacking in the Canadian samples would more readily be attributed to derivation within the state, while a compositional constituent in Minnesota sediments that could be shown to be abundant in the Canadian samples would be more likely to be attributed to long-distance glacial-sediment transport. In addition, extension of the Manitoba transect through the Thompson nickel belt resulted in collection of two samples that could be considered controls, as anomalous numbers of Cr-diopside indicator minerals were anticipated, based on previous surveys (Matile & Thorleifson 1997). Furthermore, all three transects extended to within the contiguous limit of calcareous till derived from the Hudson Bay Lowland, allowing comparison of carbonate in Minnesota sediments to carbonate near Hudson Bay, given the possibility that some calcareous debris in Minnesota could possibly have been derived from the Hudson Bay Lowland.

Sampled sites were to be considered an example of till in the target cell, collected at a depth that would, to the extent possible, minimize the effects of carbonate leaching and pedogenic alteration. It was anticipated that a portion of each till unit resting on bedrock in any given region would have been reworked into overlying tills, resulting in a progressively fainter signal several tills up from initial dispersal from source. With each succeeding till, the signal was anticipated to be fainter, so in some cases it was accepted that the sampling would fail to detect clastic dispersion due to dilution of the signal to a level indistinguishable from background. Lower tills therefore were favoured, such as sampling a stream cut rather than a nearby road cut, so long as this consideration did not significantly slow progress. Lower tills were also anticipated to possibly have a preserved sulphide component that would be of great interest to mineral exploration. It was, however, antici-



Fig. 1. Typical field sampling conditions.

pated that an exception to this consideration would be cases where it was the judgment of the sampler that the lower till was entirely derived from an area smaller than the region between the site and the next sample up-ice. In this case, the lower till would have been considered too locally derived, and thus not able to provide the required information, so an upper till was to be favoured. In most cases, however, whatever till was available at a readily accessible road cut, stream bank, or shovel hole was sampled, and rarely was there an easily accessed site where there was a choice between till stratigraphic units. In a few cases where an accessible exposure provided the opportunity to sample two or more till units, however, multiple samples were collected.

Plans were made for one till sample to be collected within each cell covering 0.25 degree of latitude and 0.5 degree of longitude; resulting in a spacing of approximately 30 km. Rows at 0.25 degree latitude spacing were labeled from A to Y, from A in Iowa to Y in Manitoba. The columns at 0.5 degree longitude spacing were numbered from 1 to 16, from 1 in the Dakotas to 16 in Lake Superior. Samples were given a field identification according to their row and column, such as F5 or M12. If two or more samples were collected at a site, the format used was, e.g. F5-a and F5-b.

Where till was not readily accessible within a cell, no sample was collected. Effort was made to sample every cell located at least in part in Minnesota, as well as adjacent cells where the edge of a cell coincided or nearly coincided with the state boundary. In the case of cells located partially outside Minnesota, the Minnesota portion of the cell was not favoured, and till was sampled wherever it was readily available within the cell.

Field equipment utilized by the sampling crews included a road atlas, 1:250,000 sample location maps, surficial geology maps, 15 litre plastic sample pails, shovel, GPS unit, Munsell colour book, acid bottle, permanent markers, datasheets, labels for inside pail, spare batteries for GPS, cell phone, and a first aid kit. Vehicles utilized for the survey were two wheel drive trucks and vans suitable for all-weather roads. A typical increment in sampling was for an MGS Quaternary geologist with an assistant, when possi-

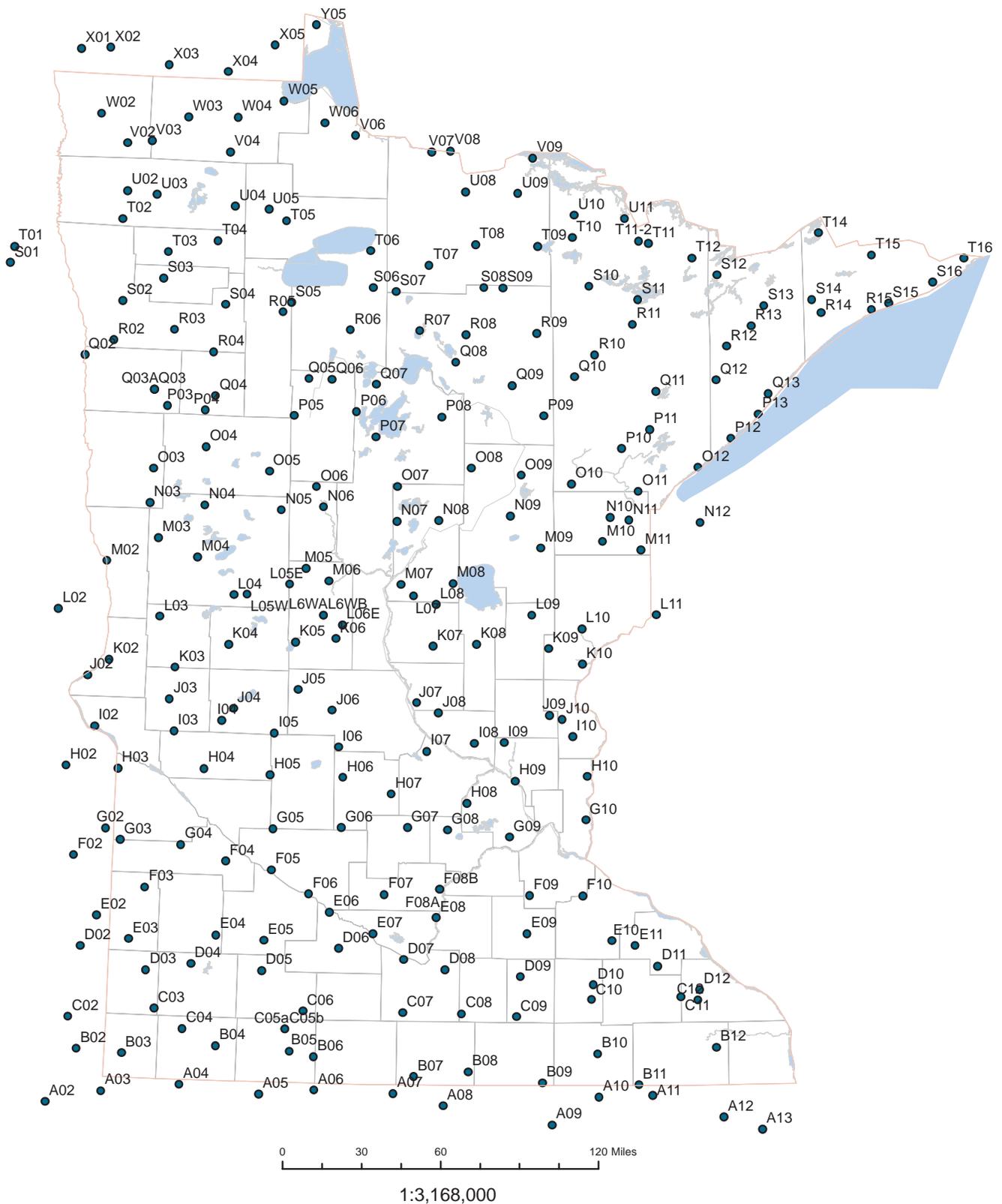


Fig. 2. Location of Minnesota survey sample sites (from Thorleifson *et al.* 2007).

ble, to travel to the field on a Monday morning, spend four days sampling, collect as many as 8 to 12 samples per day, and return to unload samples in St. Paul, Minnesota on a Friday afternoon.

All field activity followed guidelines mutually agreed upon by MGS and WMC relating to safety, environmental protection, and community relations. At the sites, a 15 litre till sample was collected from below the B-horizon, wher-



Fig. 3. Location of northern transect sample sites (from Thorleifson *et al.* 2007).

ever possible clearly on public lands, along roadsides at an existing exposure such as a road cut or riverbank, or from a shovel hole (Fig. 1). Sampling depth was commonly between 0.6 and 2 m, but greater depths were considered fully acceptable. Where discontinuous stratified sediments cover till, an auger was in some cases used to test for the presence of till prior to digging with a shovel. Clasts larger than about 3 cm were rejected by hand. No attempt to homogenize the sediment was made, and the processing laboratory was advised that subsamples should be taken with this in mind. No contact with jewelry was permitted, and the use of tools with coatings was minimized. Caution

was exercised to avoid any disturbance of buried cables. Sample containers were 4 US gallon plastic pails. A permanent marker was used to label the upper and lower side of the lid and opposite sides of the pail. A water-resistant tag was also labelled and placed in the pail on top of the sampled sediment. Tools were cleaned between sites, while bearing in mind that initial excavation at the subsequent site would reduce the chance of carryover. All fieldwork was completed between September 14, 2004 and October 29, 2004.

Locations were marked by hand on 1:250,000 topographic maps, and data sheets were completed in the field.



Fig. 4. Sample shipment, Minnesota and Ontario samples.

The field data included site identification, WGS84 latitude, WGS84 longitude, date, collector, topographic map name, site name based on a nearby geographic feature, upper limit of the sampling depth range, lower depth limit, moist Munsell colour as hue, value, and chroma, reaction to dilute hydrochloric acid (none, weak, moderate, strong), texture (clayey, silty, sandy, loam, gravelly), consistency (soft, firm, hard), gravel fraction lithology (Riding Mountain for shale-bearing sediments, Winnipeg for carbonate-bearing and shale-free sediments, Rainy for sediments lacking rock types derived from the Red River valley or Superior Basin, and Superior for sediments bearing rocks of Lake Superior provenance), presence of carbonate

pebbles, presence of secondary carbonate, and assessment of likely stratigraphic unit being sampled. Uncategorized notes were also taken on features such as texture, structure, mottling, pedogenesis, stratigraphy, and position relative to regional undissected landscape.

The resulting sample set consisted of 250 samples in the Minnesota survey (Fig. 2), and 20 samples in the three northern transects, which extended to Gillam, Manitoba, Pickle Lake, Ontario, and Geraldton, Ontario (Fig. 3). In order to obtain samples that were known to be anomalous and therefore would be a check on sample processing, three samples from the Kirkland Lake kimberlite field in Ontario were added to the batch. These sediments were collected by Beth McClenaghan of the Geological Survey of Canada (GSC), and processed under the supervision of Harvey Thorleifson, then of the GSC, in 1996, from two sites previously found to be anomalous with respect to kimberlite indicator minerals. One of the sites was more anomalous in oxide indicator minerals, while the other site was more anomalous in silicate indicator minerals. The three control samples used for the Minnesota survey were from the more silicate-indicator-mineral-rich reference material. In 1996, to prepare these reference samples, several large pails of sediment were collected at each site and the material was carefully homogenized. A representative test sample was analyzed to confirm indicator mineral abundance. Upon confirmation of the approximate indicator mineral abundance, the bulk samples were split in

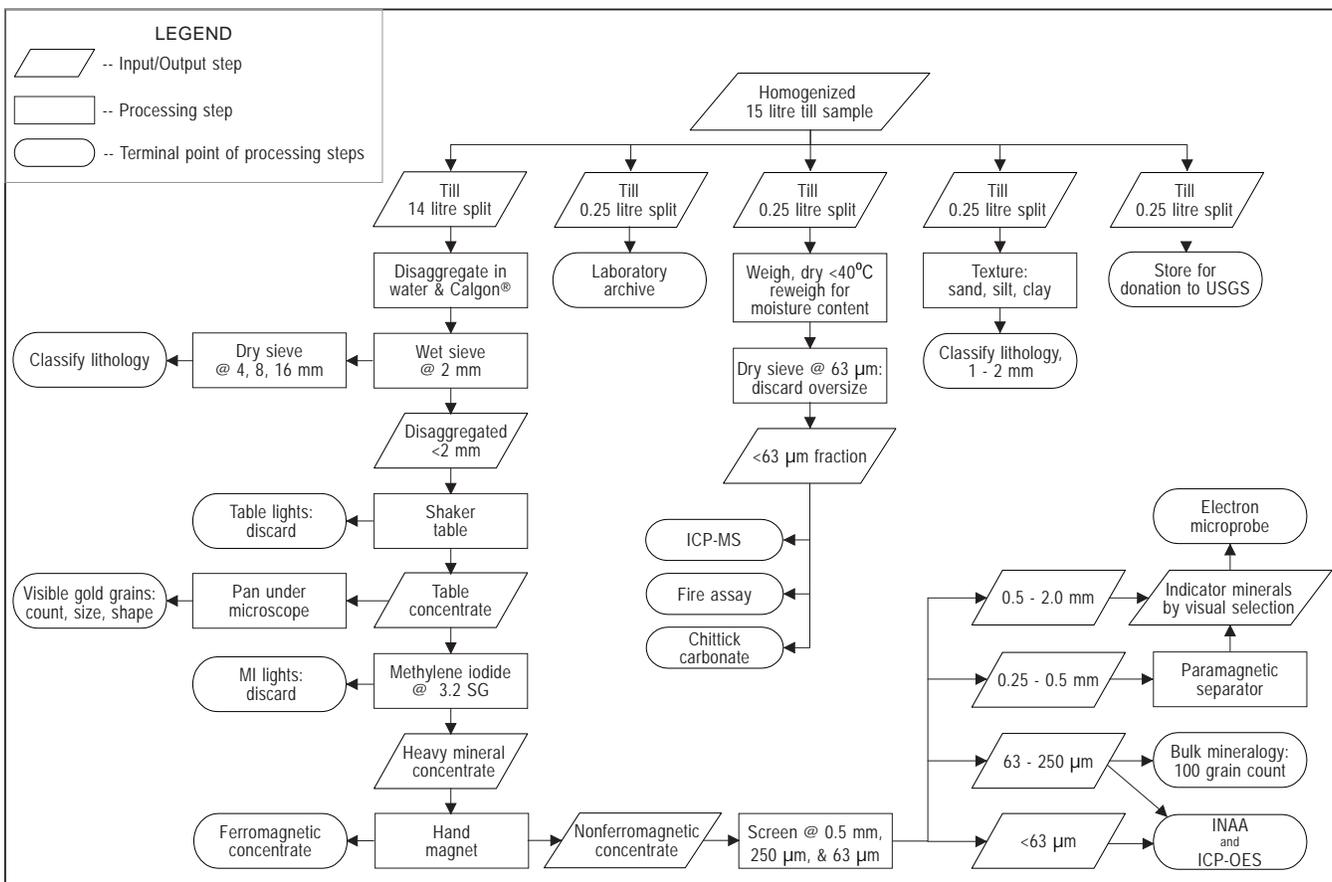


Fig. 5. Sample processing flow sheet (from Thorleifson *et al.* 2007).

order to obtain an expected indicator mineral frequency of about 10 kimberlite indicator minerals in the 0.25 to 2.0 mm fraction. The oxide reference is stored in Ottawa in vials, while the silicate reference is stored in bags. The control samples used in the Minnesota batch were further prepared in 1996 by mixing the silicate reference material with till from near the north end of Lake Winnipeg, as samples labelled with the prefix 96TCA-HT. The silicate reference material has previously been analyzed by the GSC and found to contain about twenty Cr-pyrope grains and ten Mg-ilmenite grains in each subsample.

Following completion of all sampling, samples were relabelled with a laboratory identification number in random order relative to location. This was done to ensure that trends in the data due to gradual regional compositional change could be confidently distinguished from any possible analytical drift in the laboratory, including subjective personal criteria for visual selection of indicator minerals. With the addition of 3 Kirkland Lake control samples, the total number of samples was 273.

Samples collected in Manitoba were shipped directly to the sample preparation laboratory in Ottawa, Ontario. Laboratory number labels were sent to Ottawa for these samples. Samples collected in Minnesota and adjacent states, as well as from Ontario, were accumulated at the MGS, prior to reordering, relabelling, and shipment by truck in November 2004 (Fig. 4). Permission for export of material that could potentially be perceived as soil was obtained in advance from the Government of Canada. Samples collected in Ontario had been transported to St. Paul on the basis of acceptance by border-crossing agents that the materials can be considered not to be soil, due to lack of admixed humus, so no formal arrangements had to be made for export to Minnesota in this case.

### LABORATORY METHODS

Upon arrival of the Minnesota and Ontario samples in Ottawa in November 2004, this sample batch was merged with the shipment of samples from Manitoba, as well as the control samples that had been taken from storage in Ottawa after agreement was obtained from the GSC for provision of these materials. Priority in initial indicator mineral recovery was placed on sites north of 47°, while second priority was placed on those south of 45°, due to WMC priorities. Highest priority samples were processed first, although some crossover between the areas was arranged for, to maintain as much geographic randomness as possible in the initial processing. In subsequent analyses, all batches were fully randomized.

At the sample-processing laboratory, Overburden Drilling Management Ltd. (ODM), four quarter-litre subsamples were removed from each 15 litre till sample (Fig. 5). The splits were weighed before and after air drying, at less than 40°C to avoid loss of volatile Hg, in order to determine moisture content as a basis for correcting weight of the larger sample. One split was retained as a laboratory archive, for example as a reference should an investigation of possible laboratory contamination be required. A second was screened using a stainless steel 230 mesh sieve, in order to recover about 50 g of the <63 micron fraction for

geochemical and mineralogical analyses, as well as a replicate for 5% of the samples. The oversize fraction in this preparatory step was discarded. The third split was shipped to the MGS, to be processed for textural analysis of percent sand, silt, and clay by sieve and hydrometer analysis, as well as recovery of the 1 to 2 mm fraction for future lithological analysis. The fourth set of splits was shipped to the MGS for temporary storage prior to donation to the United States Geological Survey for geochemical analysis.

The remaining material, approximately 14 litres, was disaggregated with the aid of gentle mechanical agitation and suspension in a solution of water and sodium hexametaphosphate (Calgon<sup>®</sup>), and screened at 2 mm. The >2 mm fraction was washed, dried, screened at 4, 8, and 16 mm, weighed, and shipped to the MGS for lithological analysis. The <2 mm fraction was pre-concentrated with respect to density using a shaker table, using a multiple-pass protocol designed to maximize recovery of coarse silicate heavy minerals. Table reject was discarded, except in the case of 5% of the samples, to permit an audit of table recovery. Visible gold grains were recovered at the table and by panning of the table concentrate under a stereoscopic binocular microscope, and subsequently counted and classified with respect to morphology. Final density concentrates were prepared using a heavy liquid, methylene iodide diluted with acetone to a specific gravity of 3.2. The ferromagnetic fraction that largely consists of magnetite was then removed, weighed and retained, while the methylene iodide light fraction was discarded. The nonferromagnetic concentrates were then screened at 0.25 mm, and the 0.25 to 2.0 mm fraction was visually scanned under a stereoscopic microscope for possible and probable mineral deposit indicator minerals. While the 0.5 to 2.0 mm fraction was examined without further treatment, the 0.25 to 0.5 mm fraction was processed into multiple magnetic susceptibility fractions, to reduce the amount of material examined and to add information to guide visual selection. An approximately 2000 grain split of the 0.063 to 0.25 mm nonferromagnetic heavy minerals was prepared for later visual mineralogical analysis of the nonferromagnetic concentrate as a whole at another lab.

In preparing for submission of the fine fraction, the randomized batch of 273 samples was supplemented by 14 duplicates. A total of 16 reference materials also were added. These standards consisted of 7 splits of WMC reference WM5, 7 splits of WMC reference HL5, and 2 splits of GSC reference TCA8010, a gold standard originally prepared by Harvey Thorleifson (Thorleifson & Kristjansson 1993). In the case of the <0.25 mm nonferromagnetic heavy mineral concentrate, 14 samples were divided approximately in half to prepare duplicates, which were designated samples 276 to 289. No reference materials were added to this batch, due to the unavailability of appropriate materials.

The fine (<63 micron) fraction was analyzed geochemically at ALS Chemex in Vancouver, based on approximately 1 gram of sediment processed using their procedure ME-MS61. In procedure ME-MS61, a four-acid, near-total digestion was used, including an HF-HNO<sub>3</sub>-HClO<sub>4</sub> acid digestion, and an HCl leach. This preparation dissolves

nearly all elements in the majority of geological materials. Only the most resistant minerals, such as zircons, would be partially dissolved using this procedure. Analysis for 47 elements by a combination of Inductively Coupled Plasma Mass Spectrometry (ICP-MS) and Inductively Coupled Plasma - Atomic Emission Spectrometry (ICP-AES) followed. The elements analyzed and ranges in ppm unless otherwise noted are as follows: Ag (0.02-100), Al (0.01-25%), As (0.2-10,000), Ba (0.5-10,000), Be (0.05-1000), Bi (0.01-10,000), Ca (0.01-25%), Cd (0.02-500), Ce (0.01-500), Co (0.1-10,000), Cr (1-10,000), Cs (0.05-500), Cu (0.2-10,000), Fe (0.01-25%), Ga (0.05-500), Ge (0.05-500), Hf (0.1-500), In (0.005-500), K (0.01-10%), La (0.5-500), Li (0.2-500), Mg (0.01-15%), Mn (5-10,000), Mo (0.05-10,000), Na (0.01-10%), Nb (0.1-500), Ni (0.2-10,000), P (10-10,000), Pb (0.5-10,000), Rb (0.1-500), Re (0.002-50), S (0.01-10%), Sb (0.05-1,000), Se (1-1,000), Sn (0.2-500), Sr (0.2-10,000), Ta (0.05-100), Te (0.05-500), Th (0.2-500), Ti (0.01-10%), Tl (0.02-500), U (0.1-500), V (1-10,000), W (0.1-10,000), Y (0.1-500), Zn (2-10,000), Zr (0.5-500).

The fine (<63 micron) fraction also was analyzed at ALS Chemex in Vancouver using approximately 30 grams by fire assay using procedure PGM-ICP23, in which a 30 g nominal sample weight is analyzed for Pt, Pd and Au by fire assay and ICP, at ppm ranges of Pt (0.005-10), Pd (0.001-10), and Au (0.001-10).

The <0.25 mm nonferromagnetic heavy mineral concentrates were analysed by the non-destructive Instrumental Neutron Activation Analysis (INAA) technique, using ALS Chemex procedure ME-NAA01, which is carried out by subcontractor Actlabs of Ancaster, Ontario, using their procedure 3A. Elements analyzed and lower detection limits, in ppm unless otherwise indicated are as follows: Au 5 ppb, Ag 5, Mo 20, Ni 200, Zn 200, Hg 5, As 2, Ba 200, Br 5, Ca 1%, Ce 3, Co 5, Cr 10, Cs 2, Eu 0.2, Fe 0.02%, Hf 1, Ir 50 ppb, La 1, Lu 0.05, Na 0.05%, Nd 10, Rb 50, Sb 0.2, Sc 0.1, Se 20, Sm 0.1, Sr 0.2%, Ta 1, Tb 2, Th 0.5, U 0.5, W 4, and Yb 0.2. The irradiated concentrates were then stored at ODM to permit follow-up mineralogical analysis following several months of cool-down. In addition, a 0.5 gram split of the concentrates was analyzed by Actlabs procedure code 3C, which is designed to test for base metals and associated elements in heavy mineral concentrates, such as those known to contain sulphide minerals or to possibly contain at least remnant sulphide minerals and associated minerals that could be indicative of base metal mineralization. The Actlabs Code 3C procedure uses an aqua regia extraction Inductively Coupled Plasma Optical Emission Spectrometry (ICP/OES) package, providing results for the following elements, with an indication of the lower detection limit: Ag 0.2 ppm, Cu 1 ppm, Cd 0.5 ppm, Mn 2 ppm, Mo 2 ppm, Ni 1 ppm, Pb 2 ppm, Zn 1 ppm, and S 0.01%.

An estimate of calcite and dolomite content in the fine fraction was obtained using the Chittick gasometric method (Dreimanis 1962) at the GSC in Ottawa.

Visually selected possible and probable indicator mineral grains were mounted in epoxy disks, polished, and analysed for Al<sub>2</sub>O<sub>3</sub>, CaO, Cr<sub>2</sub>O<sub>3</sub>, FeO, K<sub>2</sub>O, MgO, MnO, Na<sub>2</sub>O, SiO<sub>2</sub>, TiO<sub>2</sub>, and ZnO at the electron microprobe

facilities of the Gemoc Labs in New South Wales, Australia, followed by additional trace element analyses where warranted. These data served as the basis for categorization of the indicator minerals in some cases.

At MGS, the 8 to 16 mm gravel fraction was visually classified with respect to lithology, and the classes were weighed in order to obtain weight percentages. In addition, textural analysis of percent sand, silt, and clay was completed by sieve and hydrometer analysis. At Consorminex in Gatineau, Quebec, a portion of the 2000 grain split of the 63 to 250 micron nonferromagnetic heavy minerals was mounted in araldite on a glass slide, and 300 grains were visually identified by a highly experienced operator using a stereoscopic binocular microscope fitted for crossed polarized light.

## RESULTS

The Open File (Thorleifson *et al.* 2007) presented tables and maps for the results. Location and descriptive data were presented in the first appendix, including field number, lab number, WGS84 latitude and longitude, date of collection, name of collector, topographic map sheet name, site name based on nearby geographic feature, mean sampling depth, minimum sampling depth, maximum sampling depth, Munsell colour, reaction to hydrochloric acid, field assessment of texture, degree of consolidation, provenance, presence of carbonate pebbles and secondary carbonate, and identification of the likely stratigraphic unit being sampled. From this table, maps followed for reaction to HCl, texture, consistency, provenance, as well as presence of carbonate pebbles and secondary carbonate. In all appendices, the maps depict data for the Minnesota samples, including samples in adjacent jurisdictions along the border only. Data from the northern transects in Canada were only presented as tables.

Lithological analyses were presented in the next appendix (Thorleifson *et al.* 2007). The first table presents data related to initial processing of the full-pail sample at Overburden Drilling Management in Ottawa, and presents total moist weight, weight after removal of splits, total gravel weight, weight of finer material processed for indicator minerals, moisture content determinations based on drying of the four subsamples, and weight of the gravel-size fractions. Results of textural analyses completed on one of the subsamples at Minnesota Geological Survey were then presented, followed by matrix carbonate analyses completed at the Geological Survey of Canada in Ottawa. Data for visual categorization of the 8 to 16 mm pebbles completed by the authors at Minnesota Geological Survey were then presented, first as weight, and then as weight percent. These data were then presented in the form of maps for >2 mm gravel yield, percent sand, silt, and clay in the <2 mm fraction, calcite, dolomite, and total carbonate in the <63 micron matrix, followed by maps for the pebble lithology results.

Mineralogy of the nonferromagnetic 63 to 250 micron heavy mineral fraction data were presented in the next appendix (Thorleifson *et al.* 2007), first at the highest level of subdivision, followed by comments from the person doing the visual identification, and then data combined in

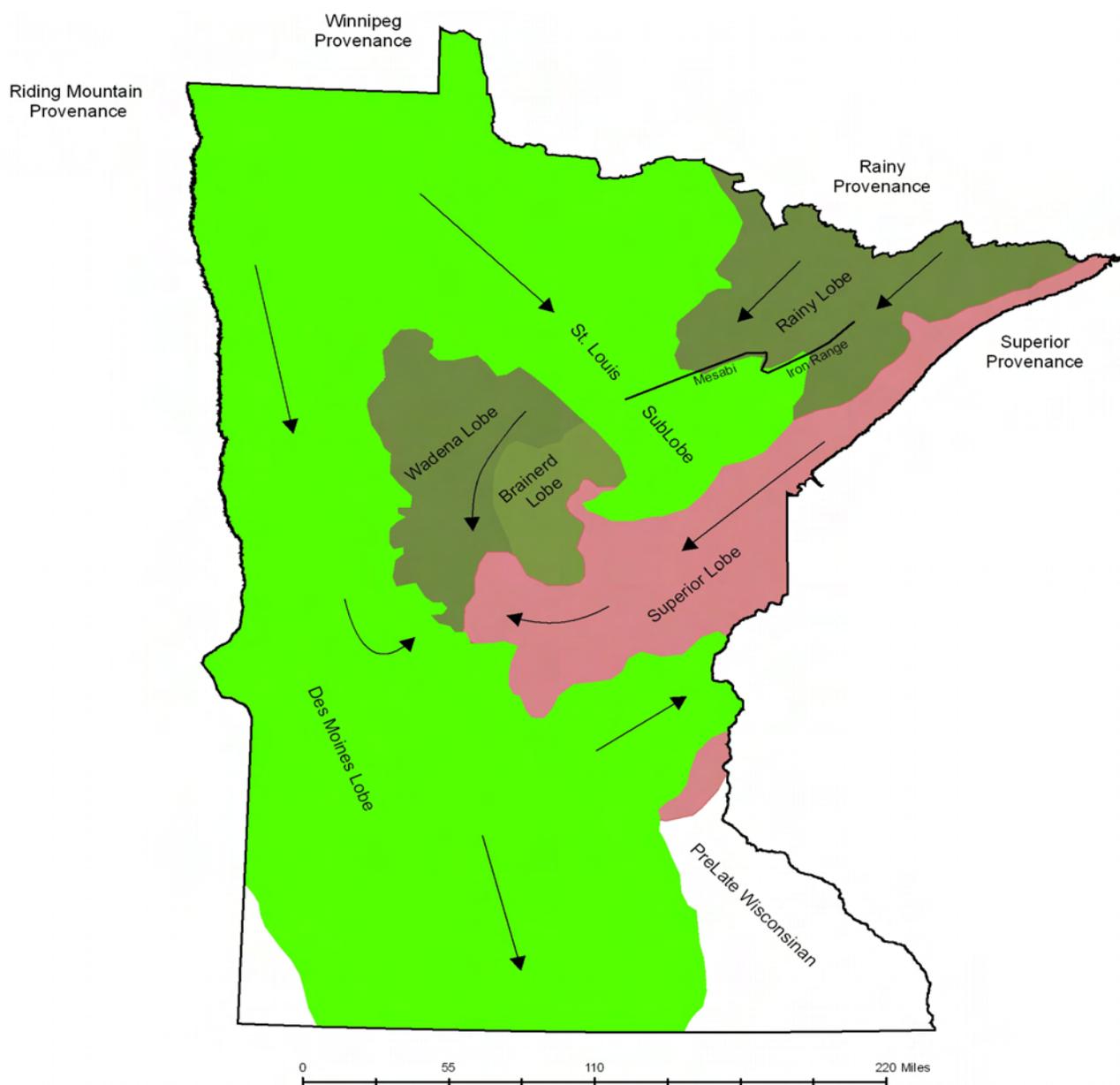


Fig. 6. Overview of till provenance trends (from Thorleifson *et al.* 2007).

fewer groups. The following maps then presented the heavy mineral count results, followed by the grouped data.

Geochemistry of the <63 micron fraction data were presented in the next appendix, including data for duplicates and standards. A similar appendix for the geochemistry of the nonferromagnetic, <250 micron heavy mineral fraction followed.

Indicator mineral data were then presented in the final data appendix, with the first several tables being derived from analyses at Overburden Drilling Management in Ottawa. The first table presents gold grain counts, as well as predicted assay for the heavy mineral concentrates, as a guide to interpreting the geochemical analysis of these fractions. This was followed by a table that presents detailed measurement of observed gold grains and remarks made at this stage of processing, in order of lab number following the prefix NA-01. The following table presented data

related to preparation of the heavy mineral concentrates, followed by kimberlite indicator mineral counts based on visual identification supported by checks on a scanning electron microscope (SEM). Comments made at this stage of processing followed. The next table presented observations made during a scan of the 0.25 to 2 mm nonferromagnetic heavy mineral concentrates for the full range of mineral deposit indicators, having previously examined the concentrates for gold grains and kimberlite indicator minerals. Indicator mineral counts other than gold grains were then summarized in the following table. Mineral chemistry analyses completed at GEMOC in Australia were then presented, beginning with clinopyroxene chemistry, which shows that the clinopyroxenes from the Thompson nickel belt in Manitoba differ from the grains scattered across Minnesota, for example in their Li concentration. Ilmenite data followed, and these data were used to refine the tenta-

tive identifications done in Ottawa, thereby confirming several Mg-ilmenite occurrences in Minnesota. Garnet chemistry followed, including major element chemistry, and both preliminary and final rare earth analyses. The Cr-pyrope grains were given categories based on the scheme of Thorleifson & Garrett (2000), with additional distinction of favourable mineral chemistry based on factors such as favourable Sc/Y ratios (Griffin & Ryan 1995). Finally, chromite mineral chemistry was presented including an assignment to kimberlitic, lamproitic, ultramafic lamprophyre, or greenstone affinities, based on the classification scheme of Griffin *et al.* (1997). The following maps then showed the size of the heavy mineral concentrates, gold-grain counts, kimberlite indicator mineral counts, and other indicator mineral occurrences.

### SUMMARY

Several maps are presented here as examples of the results. To demonstrate the comparison between the new lithological data and previous knowledge of till provenance trends (Fig. 6), data are presented for carbonate pebbles (Fig. 7), which show a clear pattern of sediment derivation from the northwest, granite and similar felsic intrusive and high-grade metamorphic pebbles (Fig. 8), as an indication of sediment derivation from the shield terrane to the north and northeast, reddish volcanic pebbles (Fig. 9) as clear indicators of sediment derivation from the Superior Basin, and shale pebbles (Fig. 10), as an illustration of compositional zonation within the extent of calcareous sediments. Epidote (Fig. 11) is an example of heavy mineral results that are elevated in the central part of the state, in the zone between derivation from the Red River Valley/Lake Winnipeg region and derivation from the Superior Basin, while goethite (Fig. 12) is elevated in the old tills of the southeast, ilmenite (Fig. 13), at least in the size fraction analyzed, is elevated in a zone of the Superior sediments, while siderite (Fig. 14) is an example of a zone along the Minnesota River that perhaps is related to shale. With respect to geochemistry, cadmium (Fig. 15) in the silt and clay fraction is first presented as an element that is elevated in the southwest, and that perhaps is shale-related, having a pattern similar to elements such as arsenic and molybdenum in this fraction. Barium (Fig. 16) in the silt and clay is an element elevated along and beyond the upper Minnesota River in a manner similar to siderite, while bismuth (Fig. 17) in this fraction is elevated both in the southwest and the north-central region, presumably for varying reasons. Chromium (Fig. 18) in the silt and clay is clearly elevated in the northeast, while chromium in the heavy mineral fraction (Fig. 19), as analyzed by instrumental neutron activation analysis, varies significantly in relation to the sediment fraction that was analyzed, presumably for various textural and mineralogical reasons - for example, chromium in sand-sized material may reside in magnetite in this region. Zinc (Fig. 20) as indicated by partial acid extraction from the heavy mineral concentrate, shows elevated results in southeastern Minnesota and northeastern Iowa in an area of known Mississippi Valley-type mineralization. Gold-grain results (Fig. 21) show a clear pattern of elevated regional background in the central part of the

state, although it should be noted that these are microscopic gold grains that would not be seen by field panning except for rare exceptions. Other gold analyses presented in the open file (Thorleifson *et al.* 2007) show anomalies in northern Minnesota near known mineralization. The Cr-pyrope map (Fig. 22) illustrates results for the principal kimberlite indicator mineral, and is one of the most noteworthy kimberlite indicator mineral results in this survey, followed by the kimberlite indicator minerals Mg-ilmenite (Fig. 23), and Cr-diopside (Fig. 24), which are progressively less specific to kimberlite than Cr-pyrope.

These results as a whole are now a significant new information resource with respect to environmental geochemistry topics, such as understanding the distribution of deleterious elements in food and water, while providing insights into composition and transport history of the sediments that make up soil parent materials. Many variables provide insights into regional geology, and reflect known mineral deposits. Some of the data seem to provide insights into what may be mineralization that was not previously recognized, such as various base-metal- and precious-metal-related elements that show patterns of varying clarity over the eastern portion of the state.

With respect to kimberlite indicator minerals, there are two noteworthy patterns, including a few Cr-pyrope garnets in an area from the Twin Cities to southwestern Minnesota, as well as Mg-ilmenites and Cr-diopsides in the far north-central part of the state. Sample spacing in the thin sediments of northeastern Minnesota was not adequate to fully test for the presence of sources such as potential single kimberlite pipes, although samples at a closer spacing are presently being processed by the Natural Resources Research Institute to address this point. The current results, consisting of one grain per sample in a few samples, are similar to the results found in areas of thick multiple tills in, for example, southern Saskatchewan, where kimberlites are known to occur (Garrett & Thorleifson 1995). These results are faint but clear indications of kimberlite indicator mineral sources, which may be deeply weathered or otherwise at source, that are not unlike several of the patterns that have been found, for example, in Canada, where some of such patterns have eventually resulted in kimberlite discoveries. In the case of the Minnesota results, the data may indicate sources within the state, or quite possibly could be manifestations of long-distance glacial-sediment transport, possibly from known or unknown sources in neighbouring states or in Canada. In the case of the Cr-pyrope occurrences, the samples tend to have been obtained from calcareous till derived from the northwest, but it is noteworthy that three of the grains are in Superior Basin-derived till, and the frequency of occurrences abruptly diminishes to the northwest. It therefore is suggested that the southern Minnesota Cr-pyrope occurrences may have been dispersed southwestward by an old Superior Basin ice lobe, and the mineral grains have subsequently been reworked into younger tills, an observation that is compatible with lithological data. In the case of Mg-ilmenites and Cr-diopsides in far north-central Minnesota, a source close to the Canadian border, or in Canada, is implied.

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Carbonate

Year of collection: 2004  
 Material sampled: C horizon till  
 Number of samples: 250  
 Analytical method: Visual identification  
 Analyzed fraction: 8-16 mm

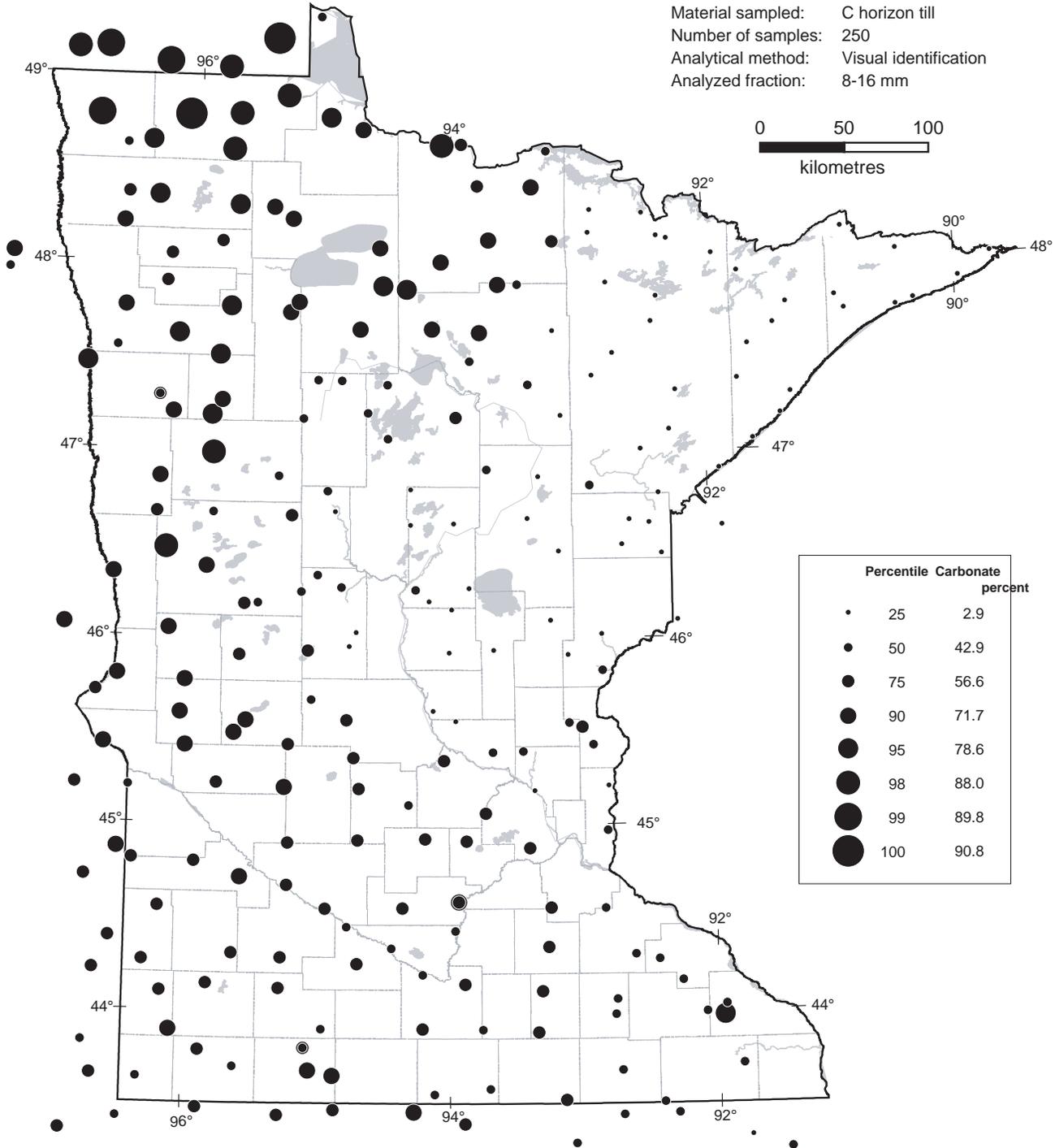
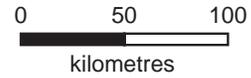
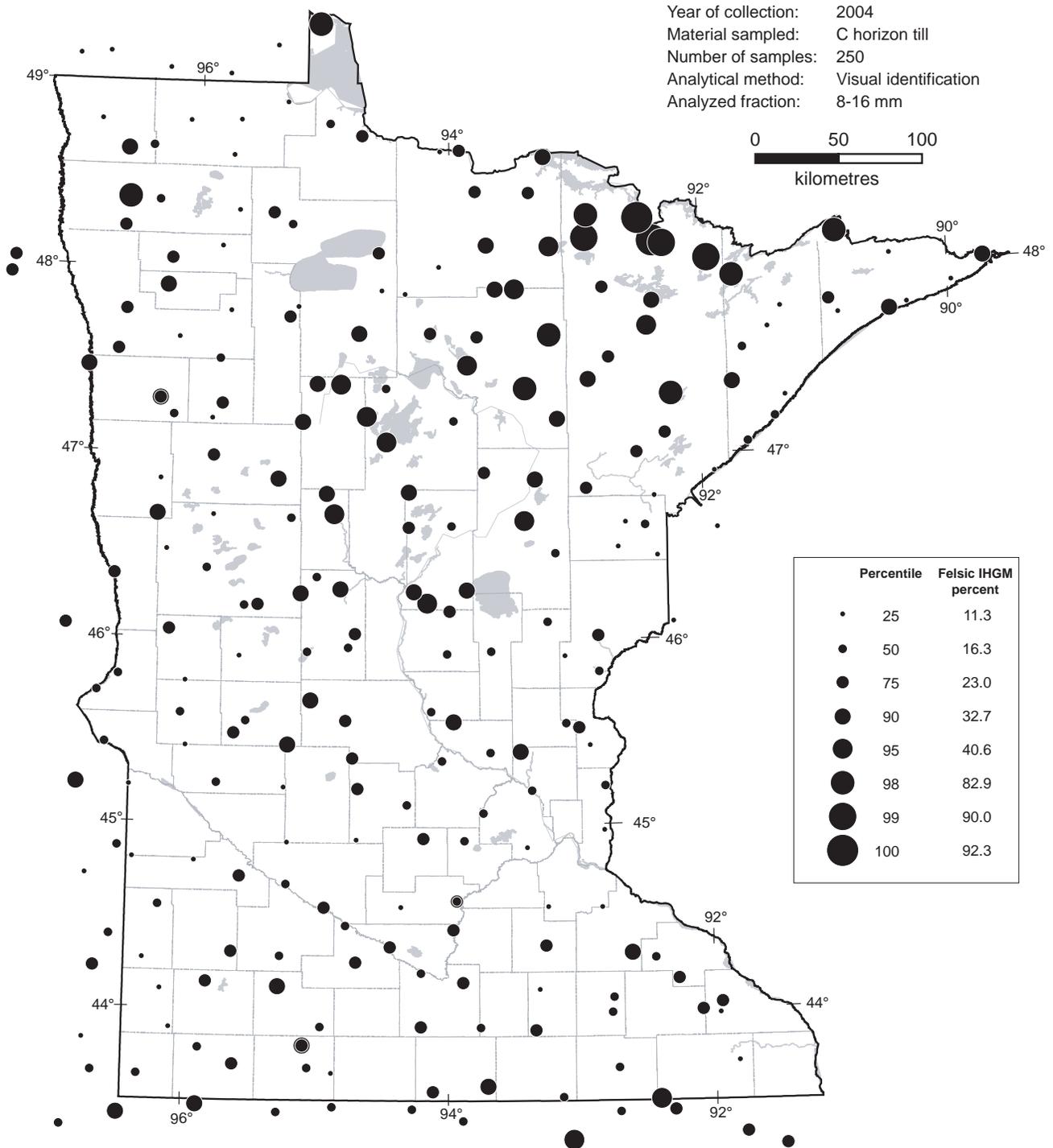


Fig. 7. Carbonate in the 8 to 16 mm gravel fraction (from Thorleifson *et al.* 2007).

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Survey of Till in Minnesota

Felsic intrusive and



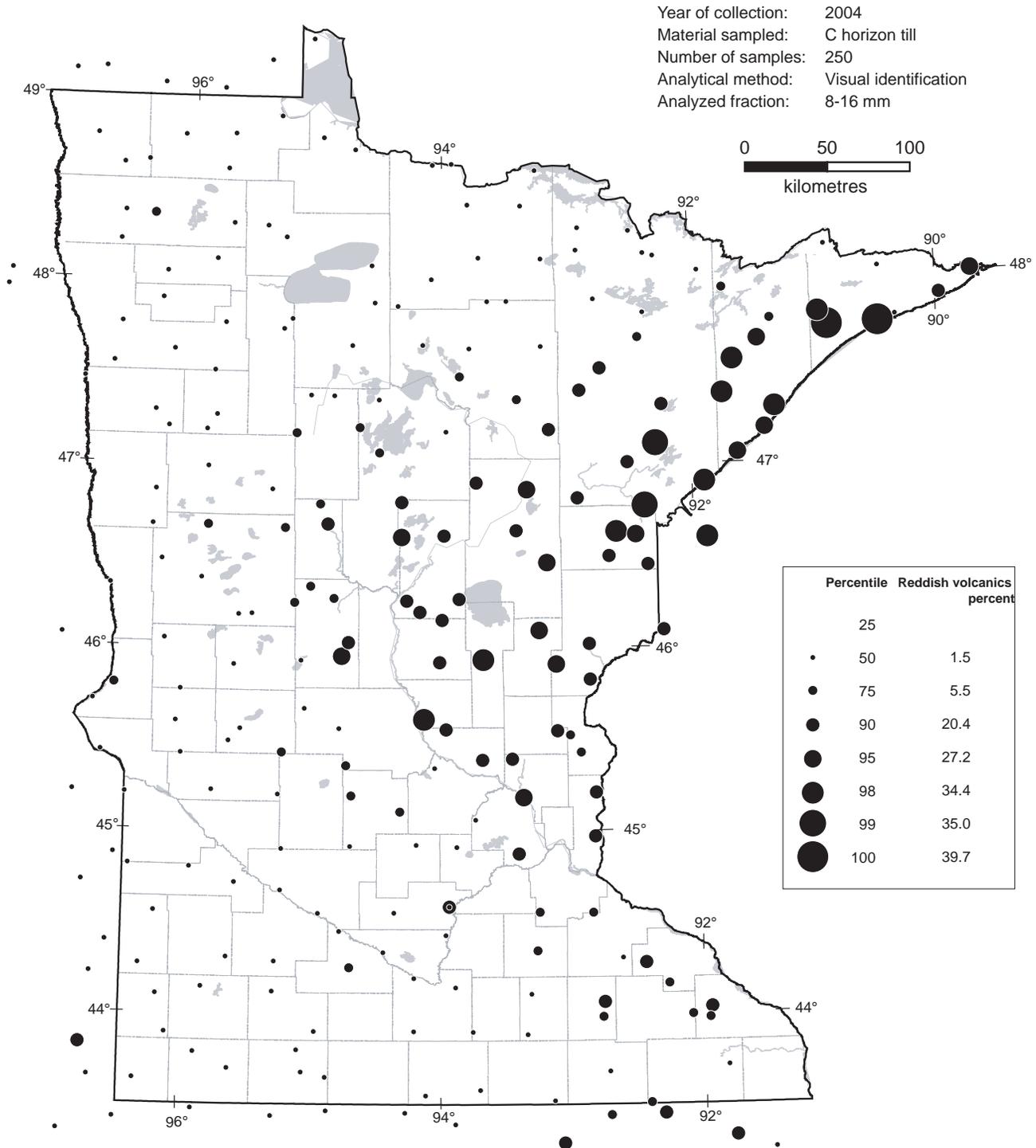
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Fig. 8. Granite and similar rocks in the 8 to 16 mm gravel fraction (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Reddish Volcanics



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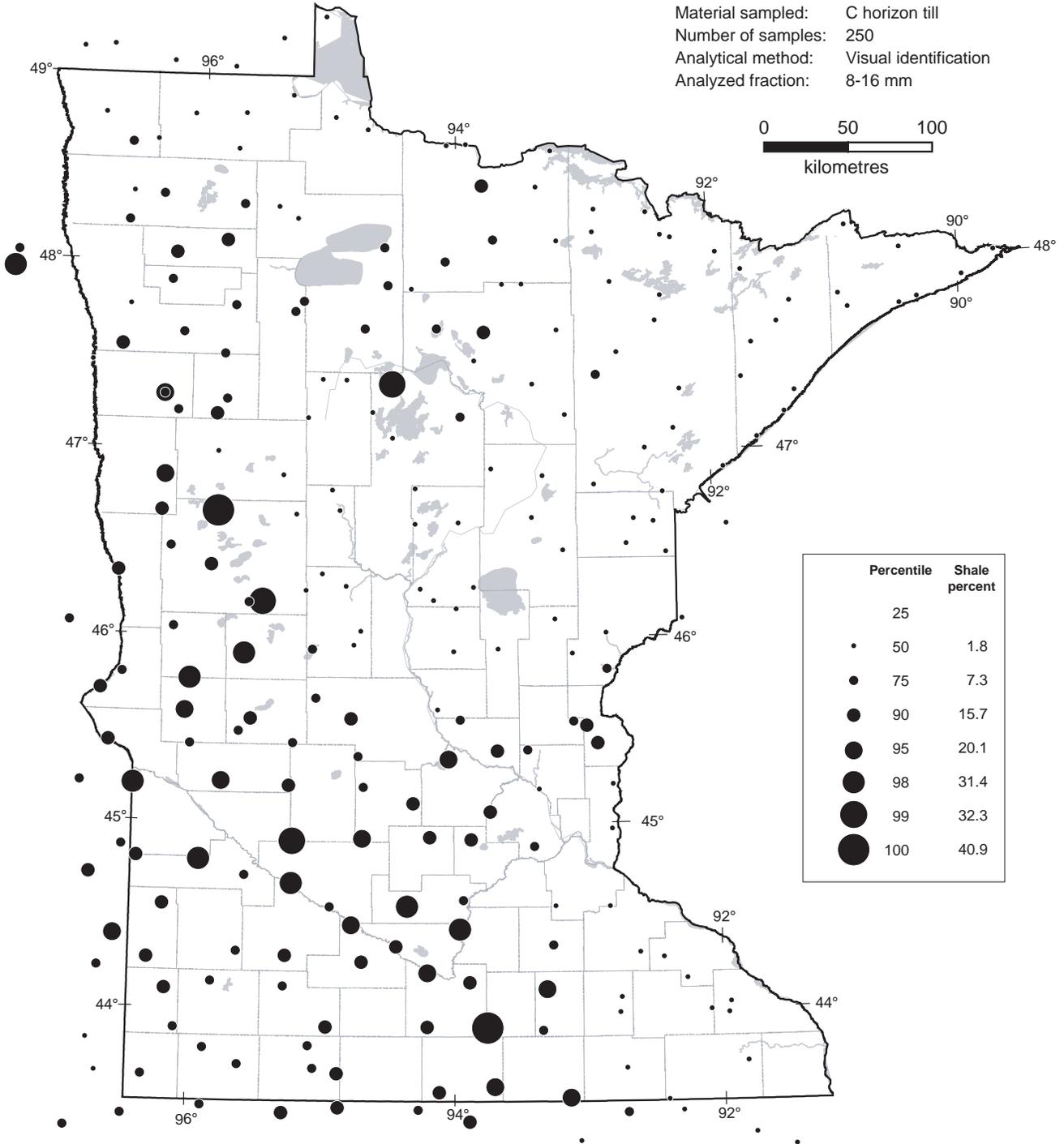
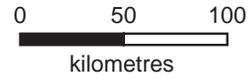


Fig. 9. Reddish volcanics in the 8 to 16 mm gravel fraction (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Shale

Year of collection: 2004  
Material sampled: C horizon till  
Number of samples: 250  
Analytical method: Visual identification  
Analyzed fraction: 8-16 mm



Percentile	Shale percent
25	
• 50	1.8
• 75	7.3
• 90	15.7
• 95	20.1
• 98	31.4
• 99	32.3
• 100	40.9

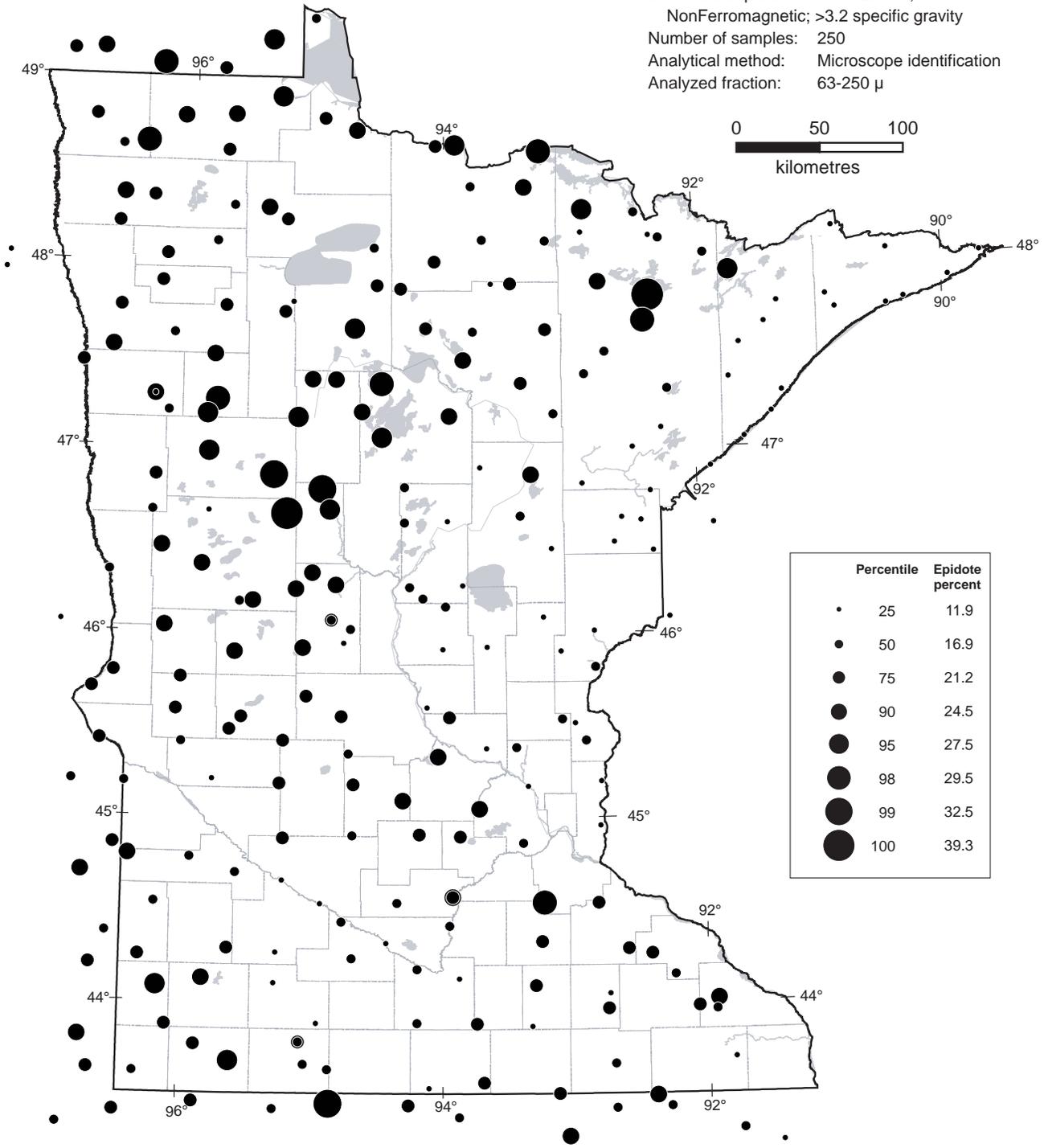


Fig. 10. Shale in the 8 to 16 mm gravel fraction (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Epidote

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: Microscope identification  
 Analyzed fraction: 63-250  $\mu$



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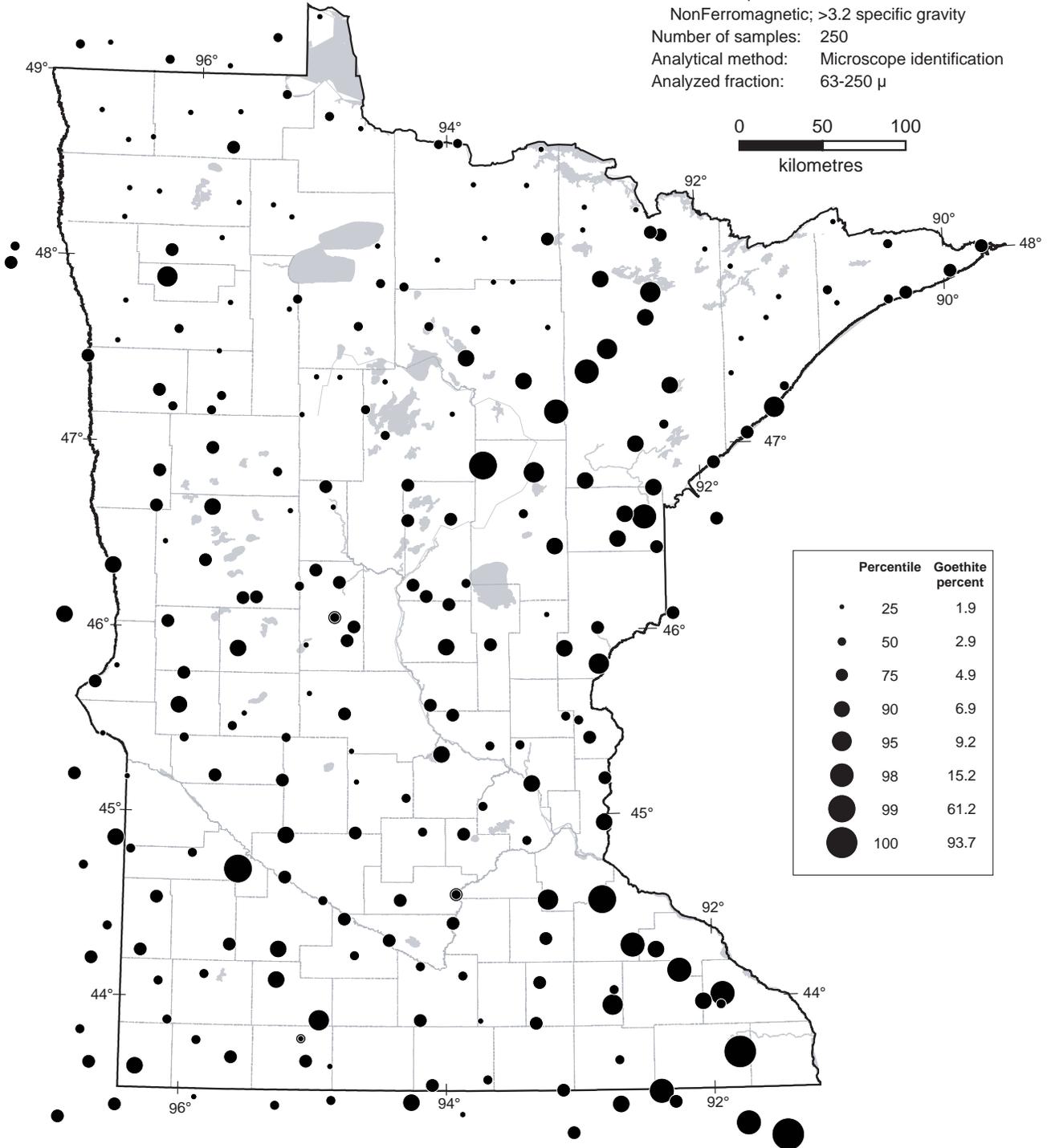


Fig. 11. Epidote in the 63 to 250 micron nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Goethite

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: Microscope identification  
 Analyzed fraction: 63-250  $\mu$



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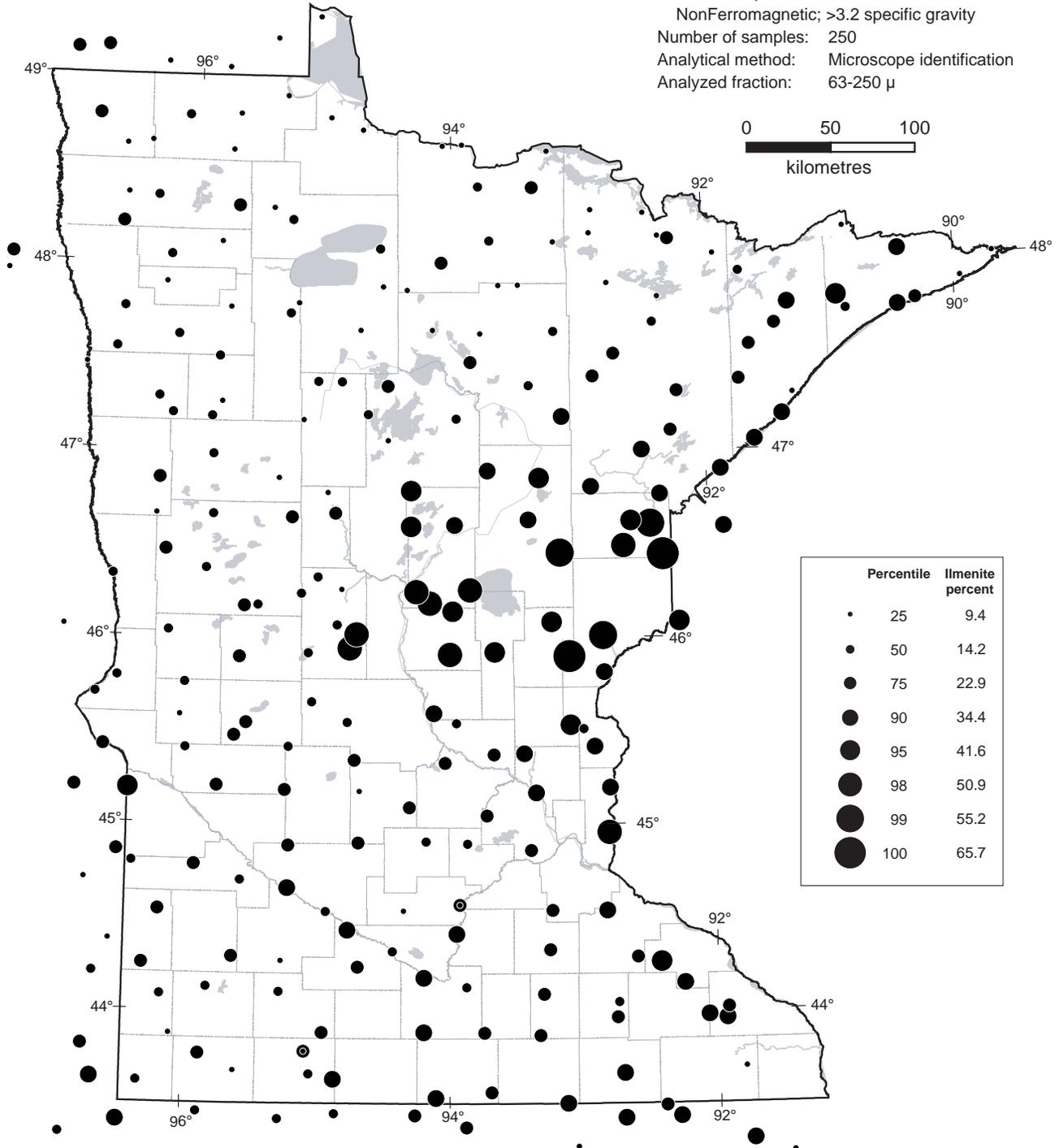


Fig. 12. Goethite in the 63 to 250 micron nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Ilmenite

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: Microscope identification  
 Analyzed fraction: 63-250  $\mu$



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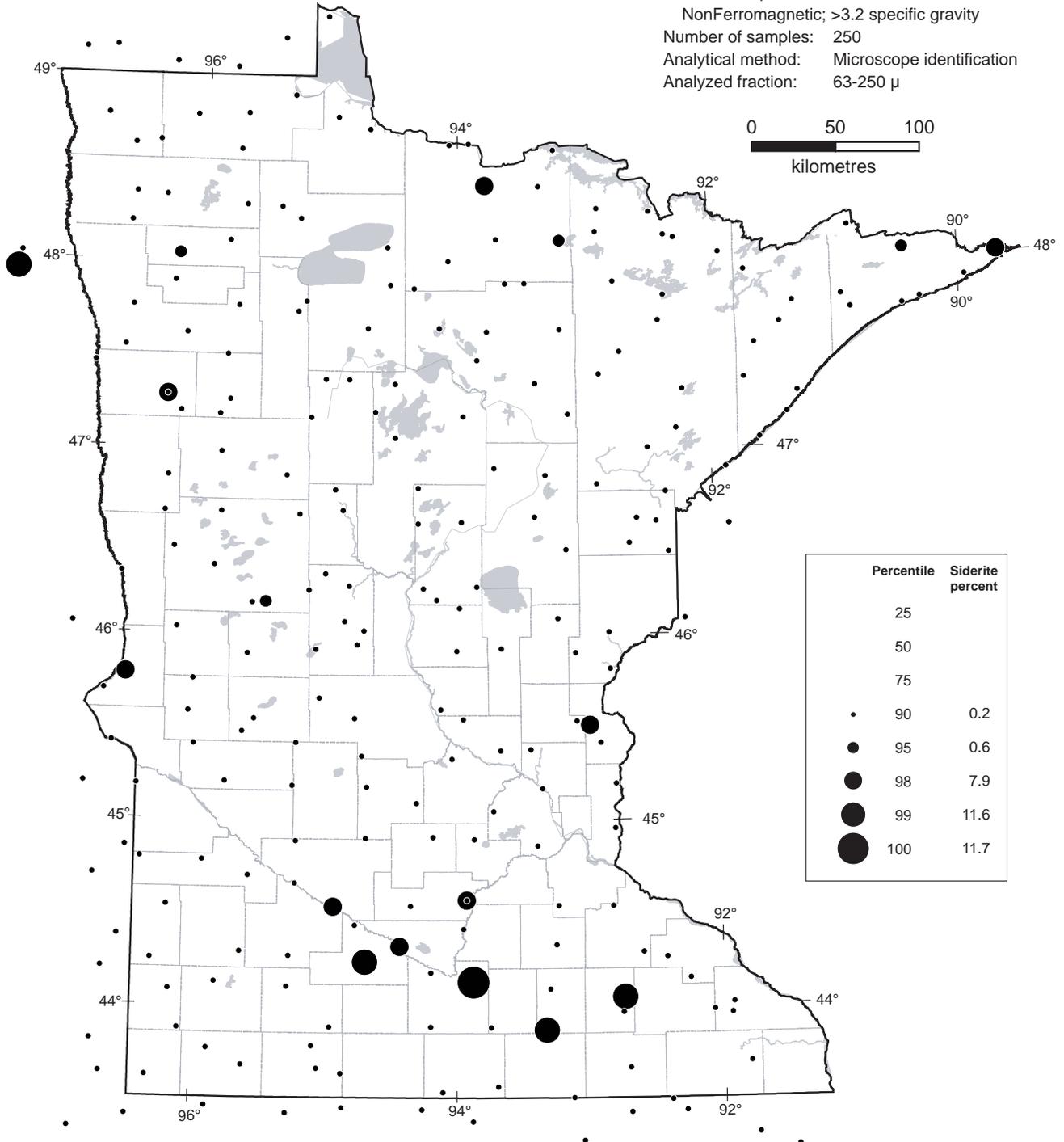


Fig. 13. Ilmenite in the 63 to 250 micron nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Siderite

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: Microscope identification  
 Analyzed fraction: 63-250  $\mu$



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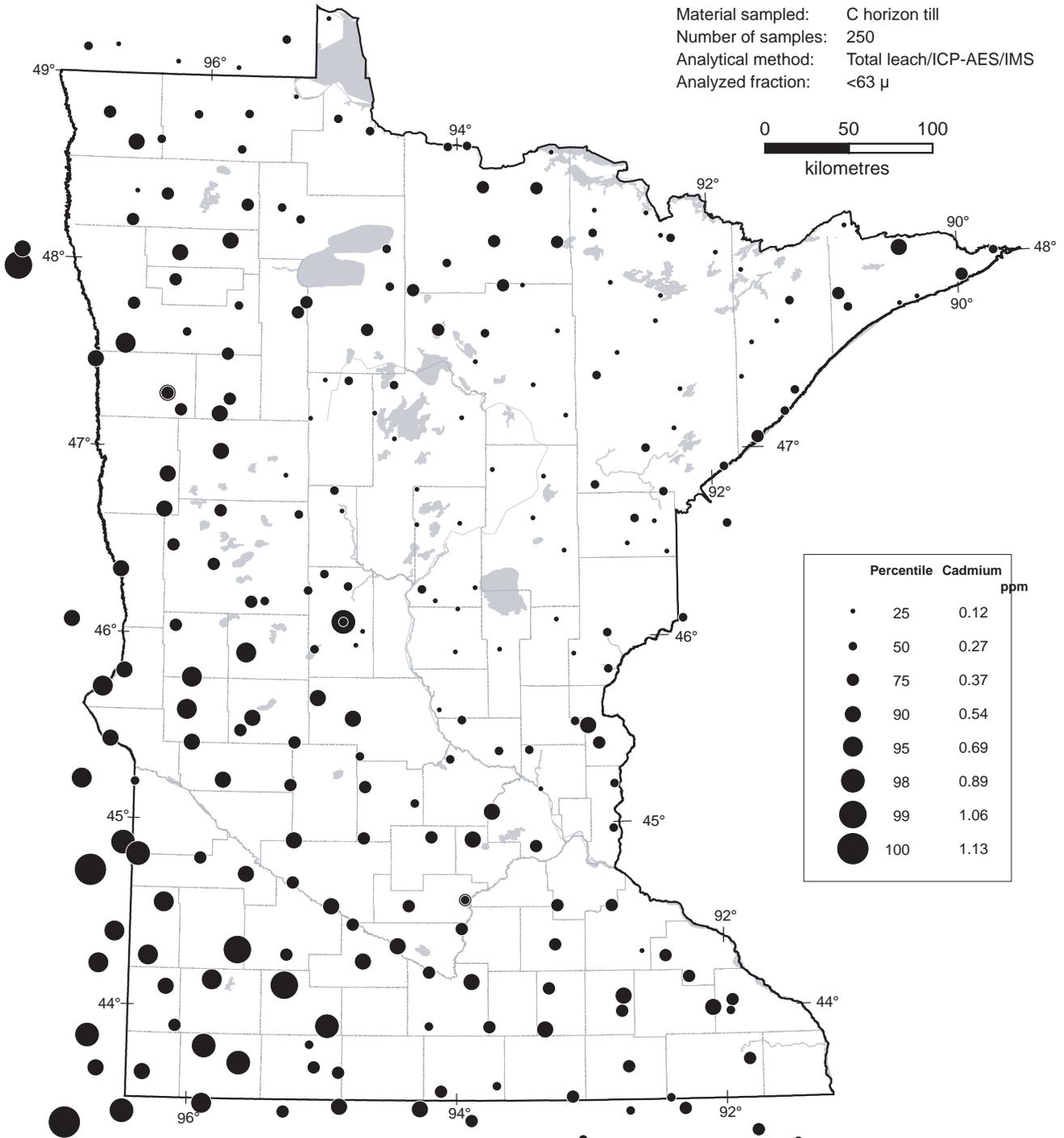
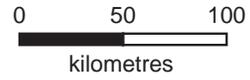


Fig. 14. Siderite in the 63 to 250 micron nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Cadmium

Year of collection: 2004  
 Material sampled: C horizon till  
 Number of samples: 250  
 Analytical method: Total leach/ICP-AES/IMS  
 Analyzed fraction: <63  $\mu$



Percentile	Cadmium ppm
•	25 0.12
•	50 0.27
•	75 0.37
•	90 0.54
•	95 0.69
•	98 0.89
•	99 1.06
•	100 1.13

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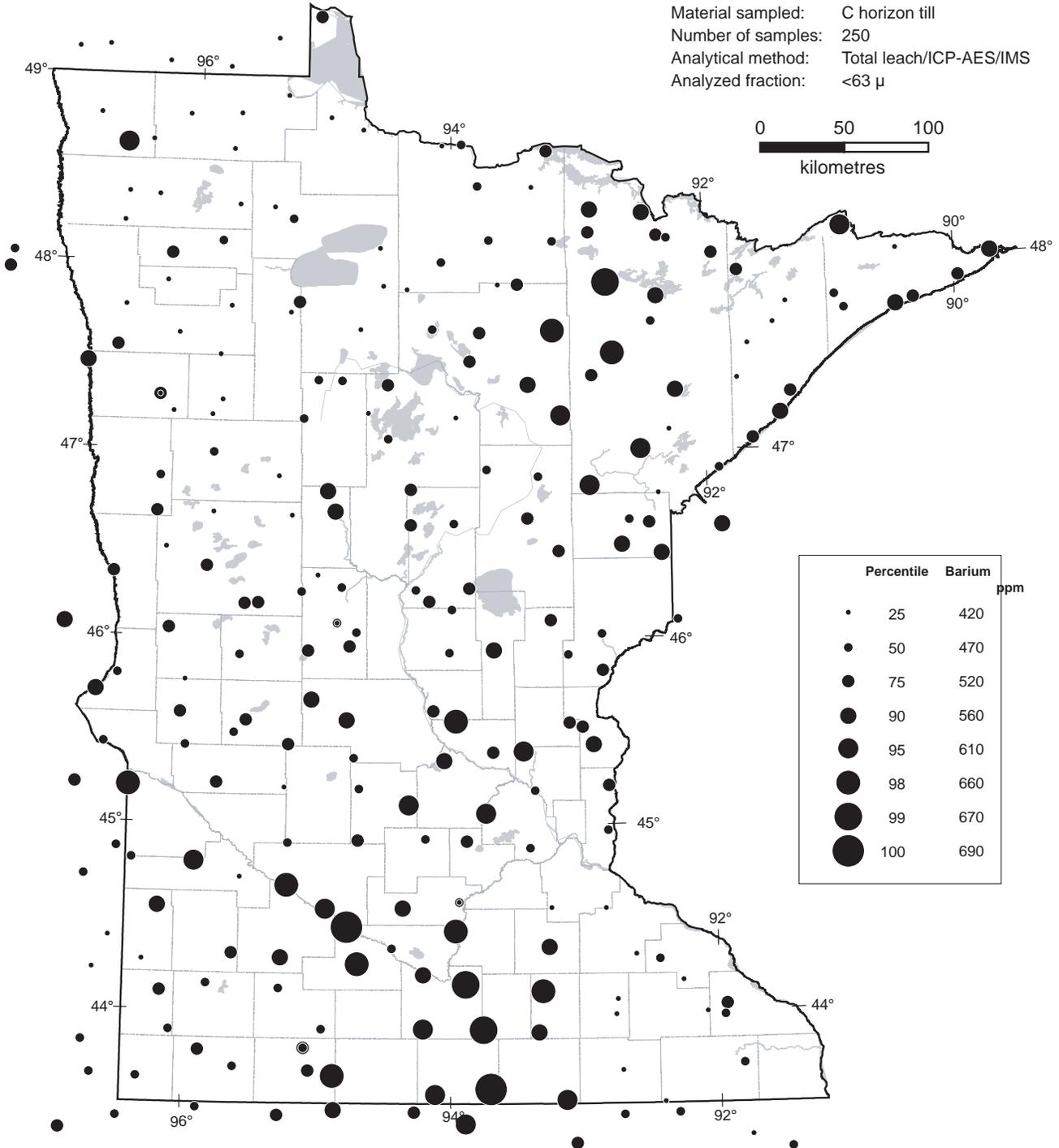
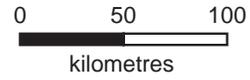


Fig. 15. Cadmium in the <63 micron fraction (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Barium

Year of collection: 2004  
 Material sampled: C horizon till  
 Number of samples: 250  
 Analytical method: Total leach/ICP-AES/IMS  
 Analyzed fraction: <63  $\mu$



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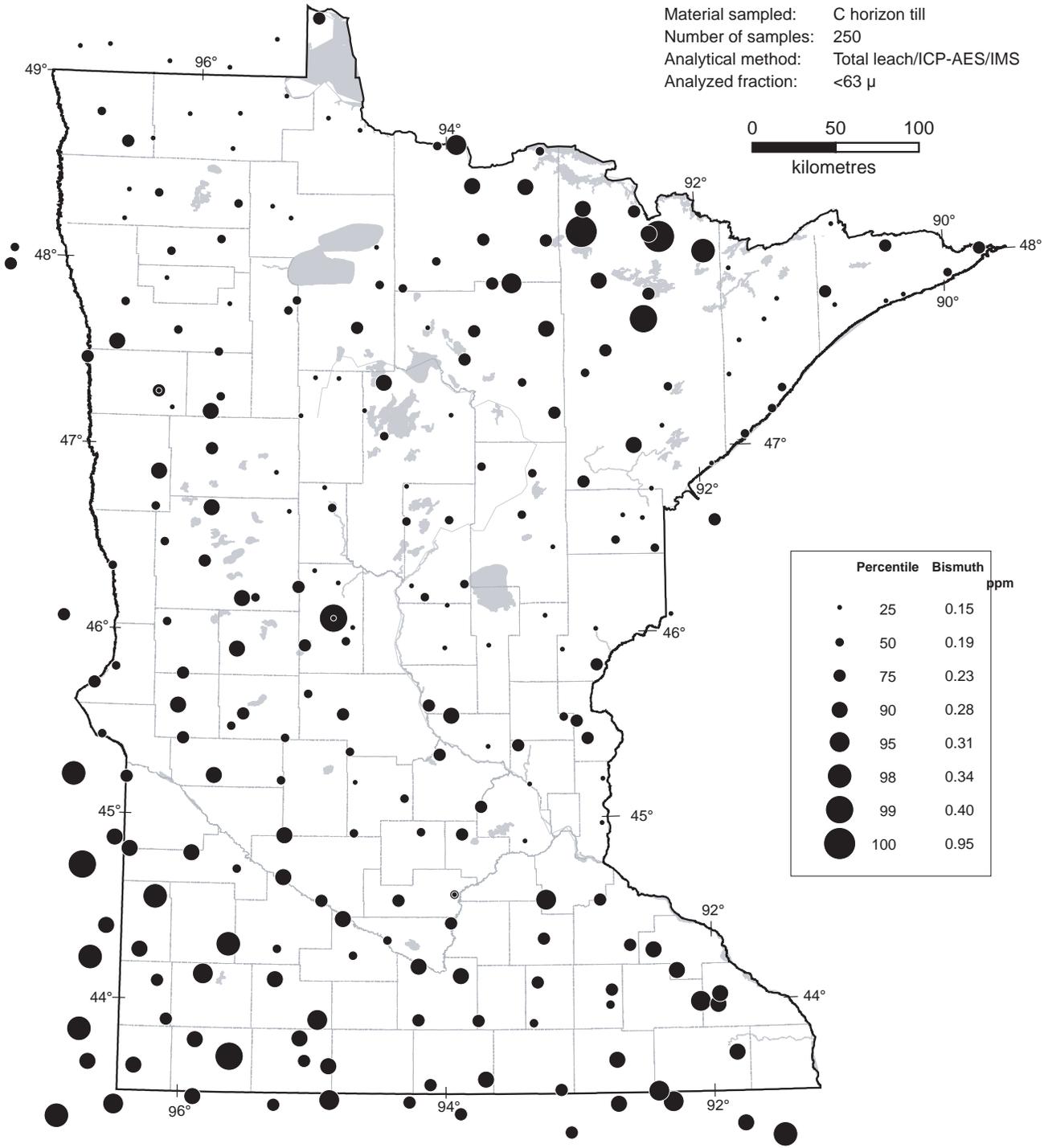
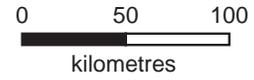


Fig. 16. Barium in the <63 micron fraction (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Bismuth

Year of collection: 2004  
Material sampled: C horizon till  
Number of samples: 250  
Analytical method: Total leach/ICP-AES/IMS  
Analyzed fraction: <63  $\mu$



Percentile	Bismuth ppm
•	25 0.15
•	50 0.19
•	75 0.23
•	90 0.28
•	95 0.31
•	98 0.34
•	99 0.40
•	100 0.95

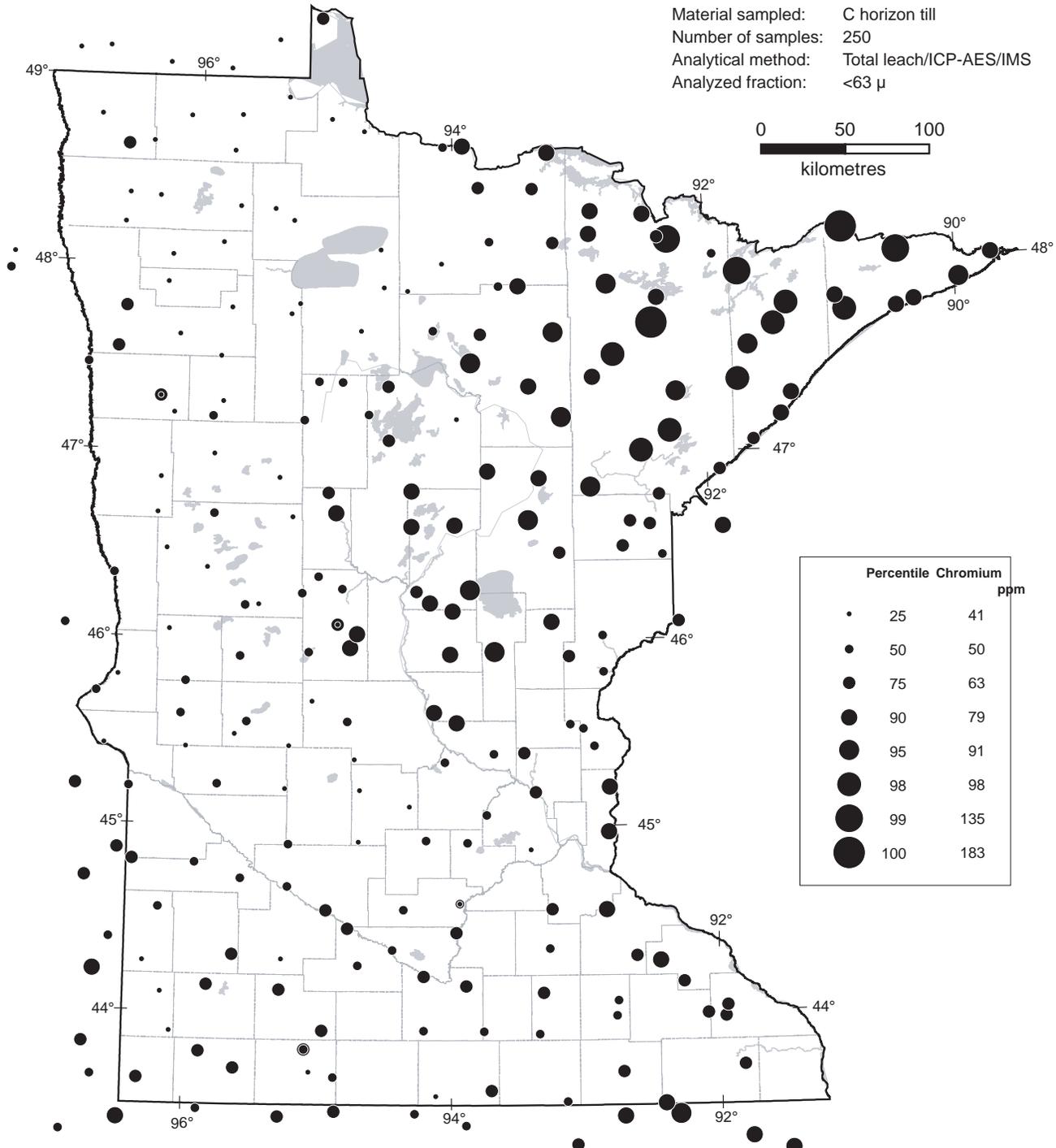


Fig. 17. Bismuth in the <63 micron fraction (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Chromium

Year of collection: 2004  
 Material sampled: C horizon till  
 Number of samples: 250  
 Analytical method: Total leach/ICP-AES/IMS  
 Analyzed fraction: <63  $\mu$



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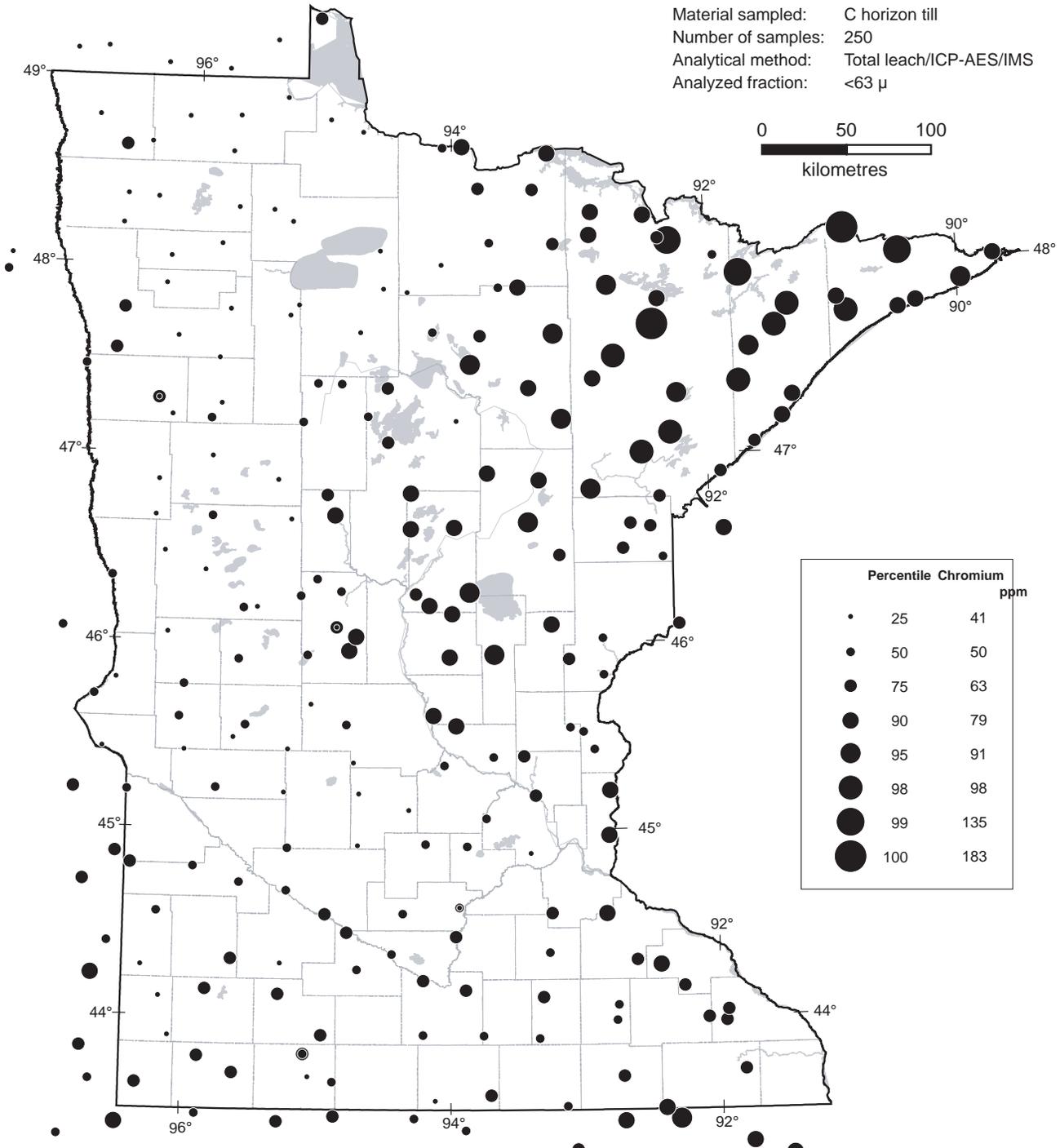
Fig. 18. Chromium in the <63 micron fraction (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Chromium

Year of collection: 2004  
Material sampled: C horizon till  
Number of samples: 250  
Analytical method: Total leach/ICP-AES/IMS  
Analyzed fraction: <63  $\mu$

0 50 100  
kilometres



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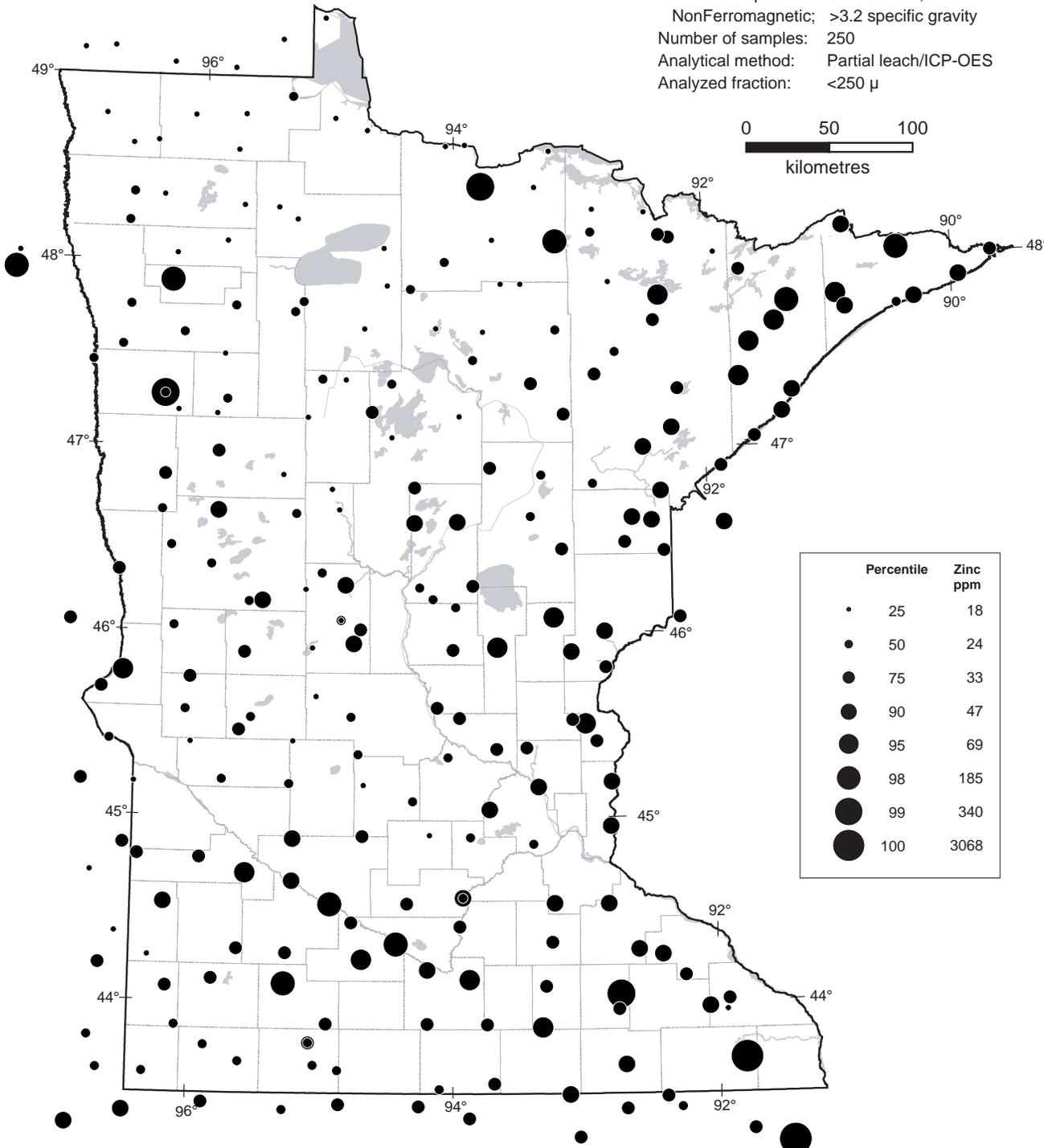
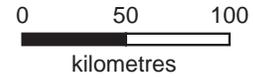


Fig. 19. Chromium in the <0.25 mm nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Zinc

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: Partial leach/ICP-OES  
 Analyzed fraction: <250 μ



Percentile	Zinc ppm
•	25
•	50
•	75
•	90
•	95
•	98
•	99
•	100
	18
	24
	33
	47
	69
	185
	340
	3068

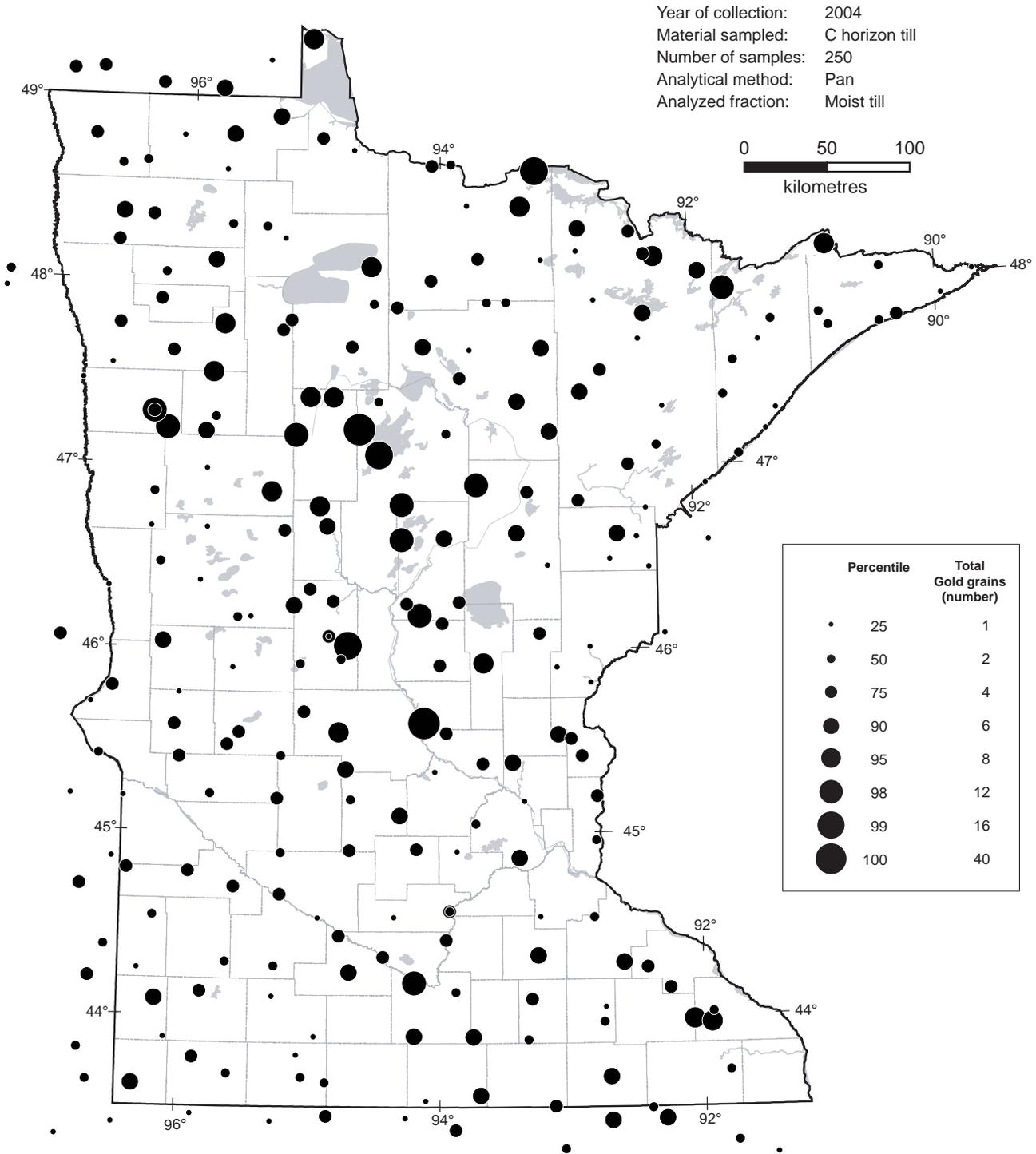
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Fig. 20. Partial zinc in the <0.25 mm nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Total Gold Grains



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Fig. 21. Total gold grain count (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Cr-pyrope garnet

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: Microscope identification  
 with SEM checks  
 Analyzed fraction: 0.25-2.0 mm

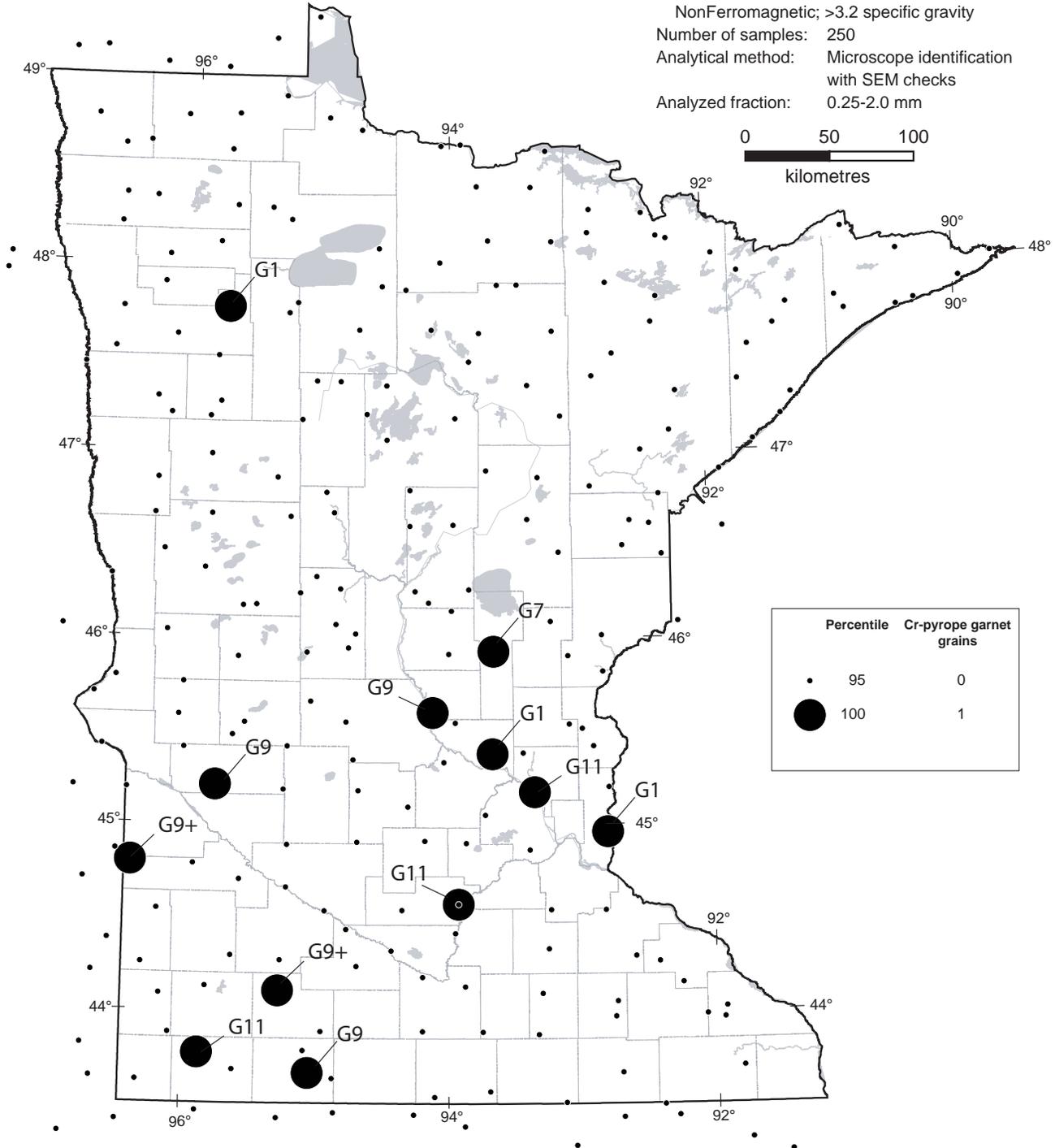
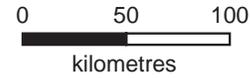
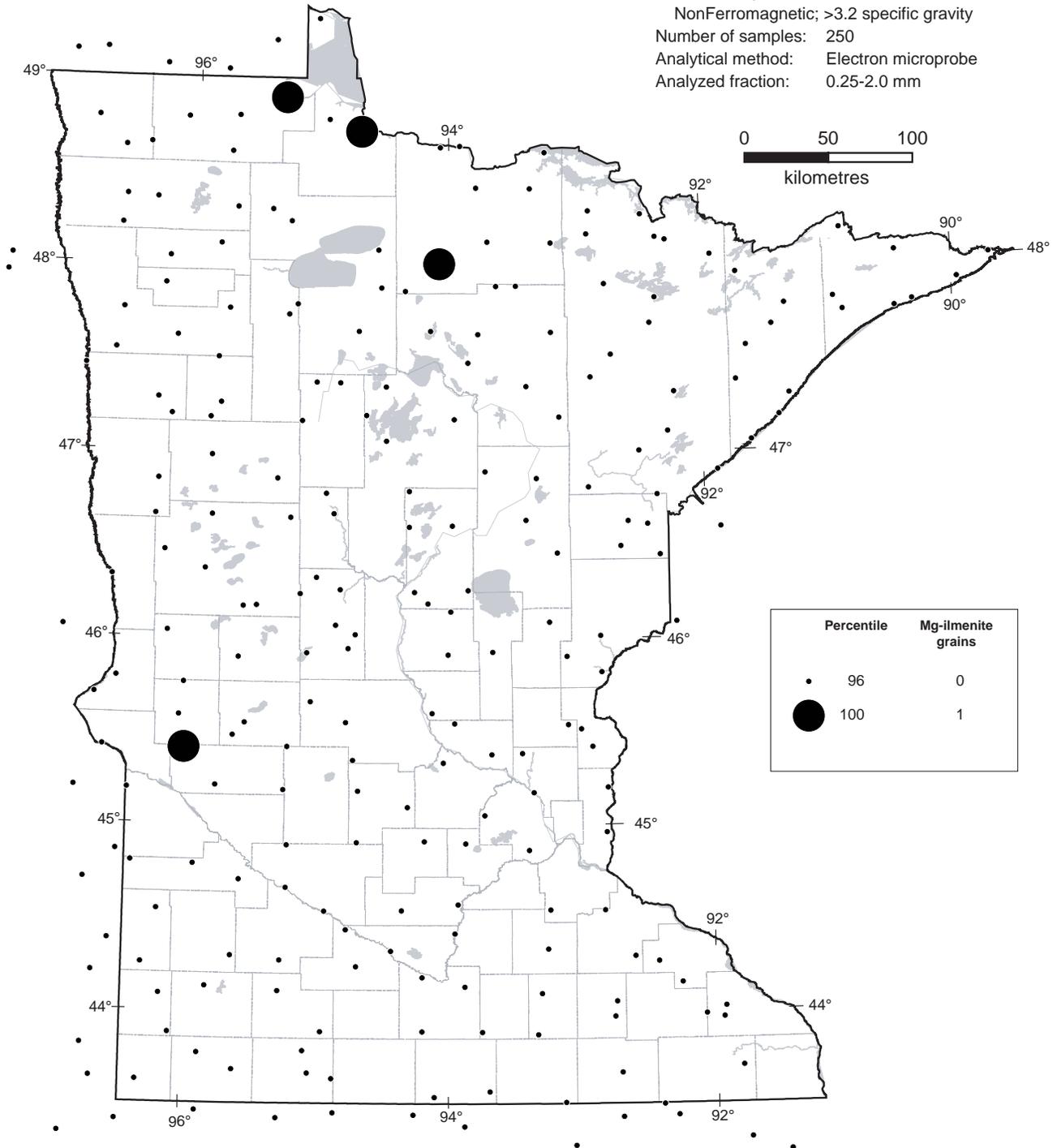


Fig. 22. Cr-pyrope in the 0.25 to 2 mm fraction (from Thorleifson *et al.* 2007).

# Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

## Mg-ilmenite

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: Electron microprobe  
 Analyzed fraction: 0.25-2.0 mm



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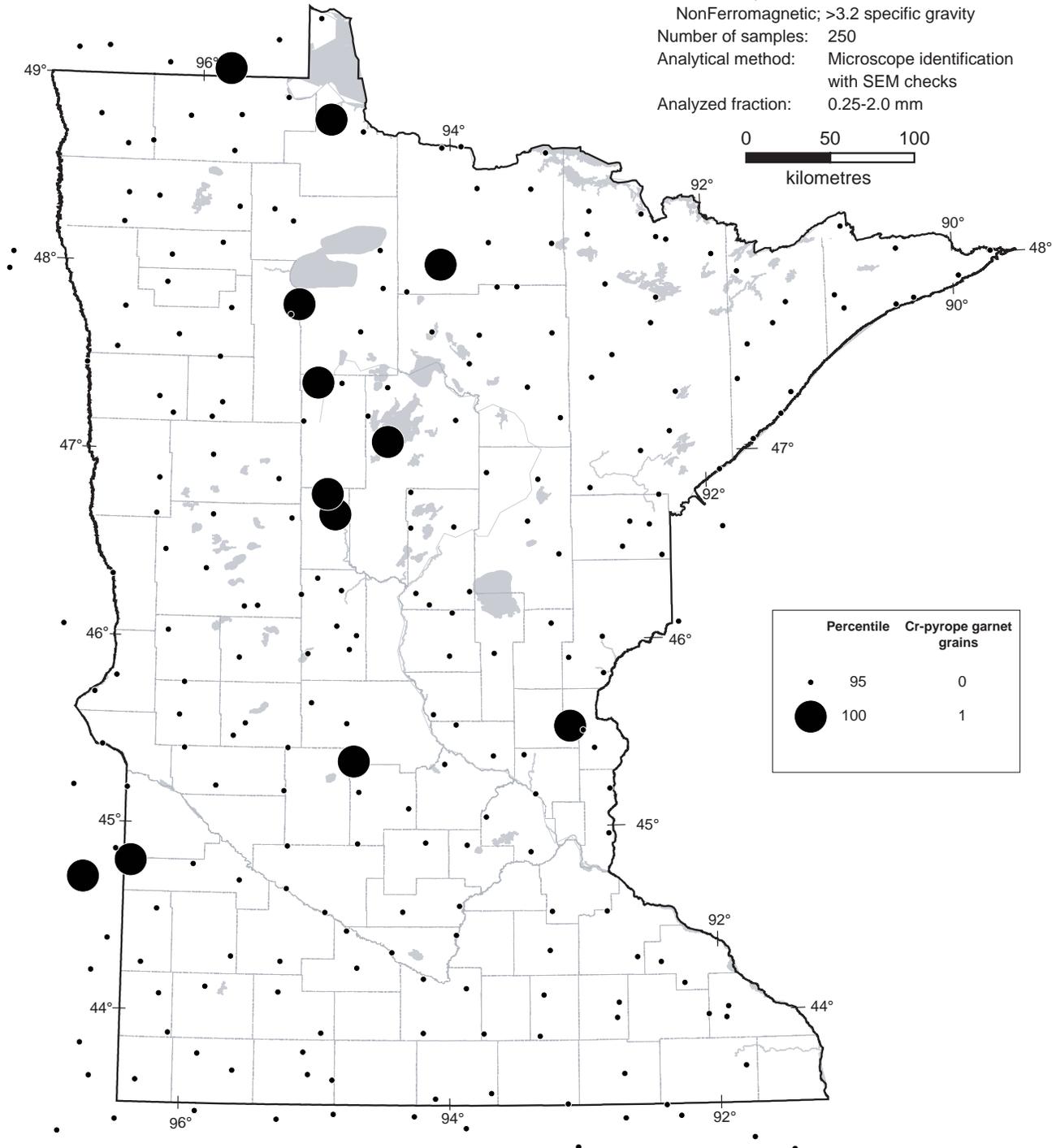


Fig. 23. Mg-ilmenite in the 0.25 to 2 mm fraction (from Thorleifson *et al.* 2007).

## Soil Geochemical and Indicator Mineral Reconnaissance Survey of Till in Minnesota

### High-Cr-diopside

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: Microscope identification  
 with SEM checks  
 Analyzed fraction: 0.25-2.0 mm



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Fig. 24. High-chrome Cr-diopside in the 0.25 to 2 mm fraction (from Thorleifson *et al.* 2007).

In summary, the results are a highly significant step forward in mapping our geochemical landscape, in clarifying mineral potential, and in provision of reference data useful to activities related to environmental protection, public health, and exploration, as well as in supporting follow-up with respect to potential mineralization.

#### ACKNOWLEDGMENTS

This work was carried out as a highly efficient, rewarding, and enjoyable team effort with Ken Harris, Howard Hobbs, Carrie Jennings, Al Knaeble, Rich Lively, Barb Lusardi, and Gary Meyer of the Minnesota Geological Survey. The highly productive partnership between Minnesota Geological Survey and WMC Resources Ltd. is acknowledged with much appreciation, as is the excellent service provided by Overburden Drilling Management in Ottawa, ALS Chemex in Vancouver, Actlabs of Ancaster, Ontario, the Geological Survey of Canada in Ottawa, GEMOC Labs in New South Wales, Australia, Consorminex in Gatineau, Quebec, and the Minnesota Geological Survey sediment lab. The generous assistance of the Manitoba Geological Survey in obtaining samples in Manitoba also is acknowledged with sincere appreciation.

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