



# 24th International Applied Geochemistry Symposium

## Workshop B: Indicator Mineral Methods in Mineral Exploration

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

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## **WORKSHOP OVERVIEW**

This one-day workshop reviews principles, methods, and developments in indicator mineral methods in mineral exploration. Since playing a key role in the discovery of the Lac de Gras diamond field in northern Canada, indicator mineral methods have risen in prominence. These exploration methods rely on sampling of sediments such as glacial and stream sediments, and detection of mineral deposit indicators dispersed by mechanical processes. The workshop addresses how indicator mineral methods are part of a spectrum of clastic sediment-based methods that ranges from boulder tracing to detection of detrital debris or their weathering products by chemical analysis of C-horizon soils and sediments. Whereas the panning for gold grains has long been known, the search for kimberlite indicator minerals has only become widely familiar over the past two decades. The scope of indicator mineral methods has now broadened to a full array of mineral deposits, including base metals and uranium. The workshop consists of presentations by some of the most experienced practitioners in the field. Topics include 1) principles of clastic sediment transport, 2) indicator mineral survey design, 3) sampling and sample processing, 4) mineral chemistry methods, 5) quality assurance, 6) regional, multiple commodity surveys, and 7) presentations on specific indicator minerals and commodities, including Au, Ni-Cu-PGE, kimberlite, gahnite, IOCG, and porphyry Cu. Authors/speakers have prepared informative workshop notes that provide an overview of their topics as well as selected references for further reading. Layout of this workshop volume was completed by E. Ambrose.

Beth McClenaghan and Harvey Thorleifson  
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# Workshop B:

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# Indicator mineral methods in mineral exploration: Introduction

L. H. Thorleifson

*Minnesota Geological Survey, 2642 University Avenue West, St Paul, Minnesota 55114*

*(e-mail: thorleif@umn.edu)*

## 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.

## INTRODUCTION

Mineral exploration methods that are applied directly to the prospective rocks, at the preliminary stages of grassroots 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 mineral 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 resistant 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 mineral-

izing 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; Scott & Radford 2007; Ghosh & Praveen 2008). The bulk composition of non-ferromagnetic 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 wehrilitic (high Ca), lherzolititic 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 for mineral exploration

Chris Benn

*BHP Billiton World Exploration Inc., Suite 800, Four Bentall Centre,  
1055 Dunsmuir Street, Vancouver, British Columbia V7X 1L2  
(e-mail:Chris.Benn@bhpbilliton.com)*

## INTRODUCTION

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 - scale 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 deleteri-

ous 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 approximately 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.

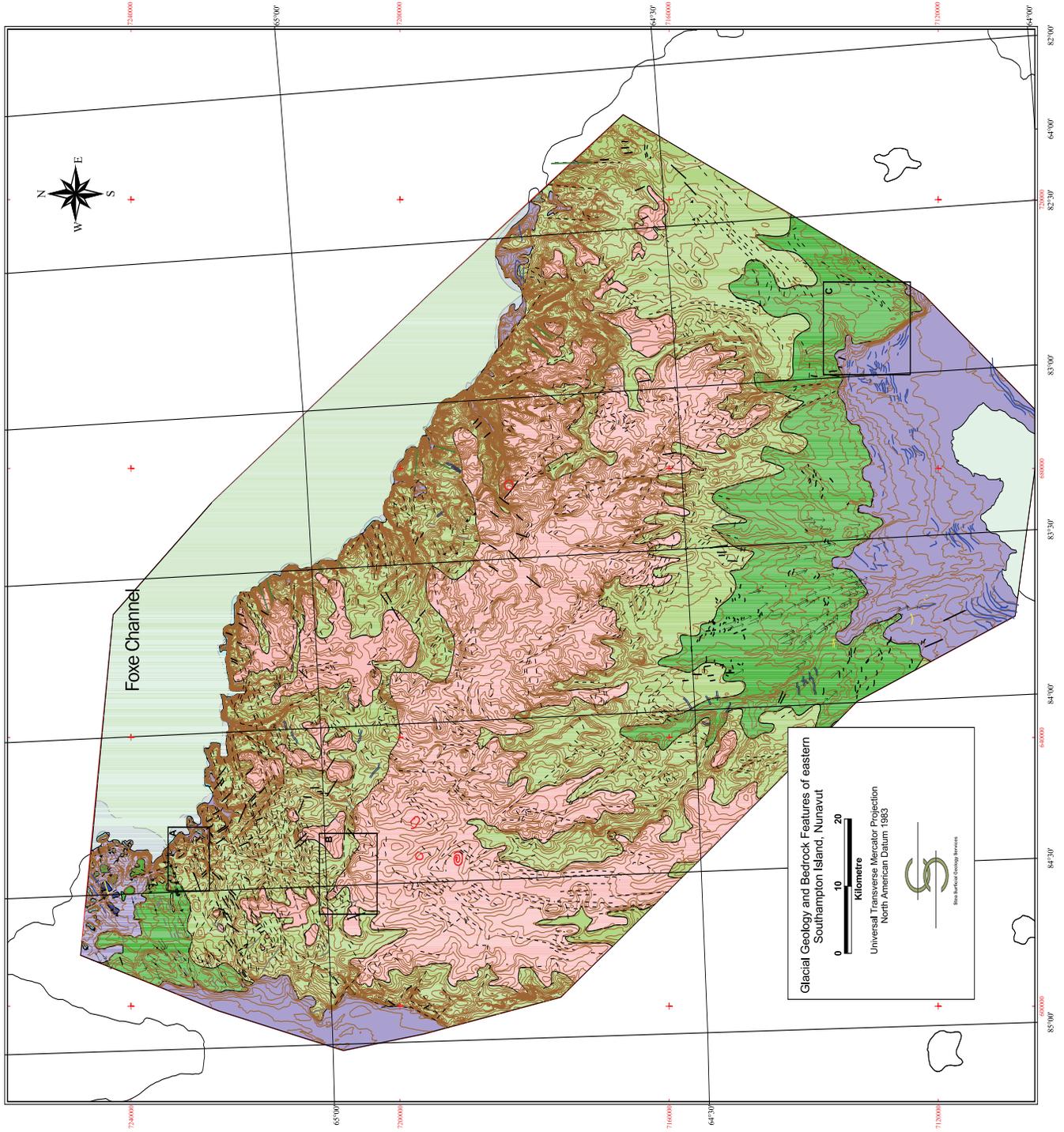
A regional-scale 'follow up' to indicator minerals of interest from the reconnaissance-scale survey is carried out at a sample spacing of 1 sample per 25 km<sup>2</sup> with a sample size of 15 kg. Results from this scale of survey should define better the reconnaissance-scale signature and will provide 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 the 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, 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 Canada where, although very effective, a higher than necessary density was used at the regional scale.



## 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.

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

## 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 may occur in the near-surface environment due to 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 1 m 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 portable data capture device (PDA). 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 geographic information system (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 samples and better interpretation of results easily justifies the extra cost of a field capture device.

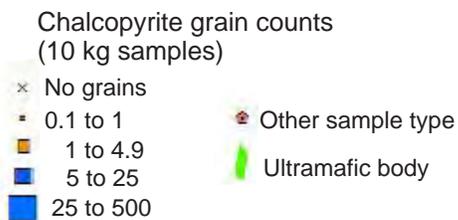
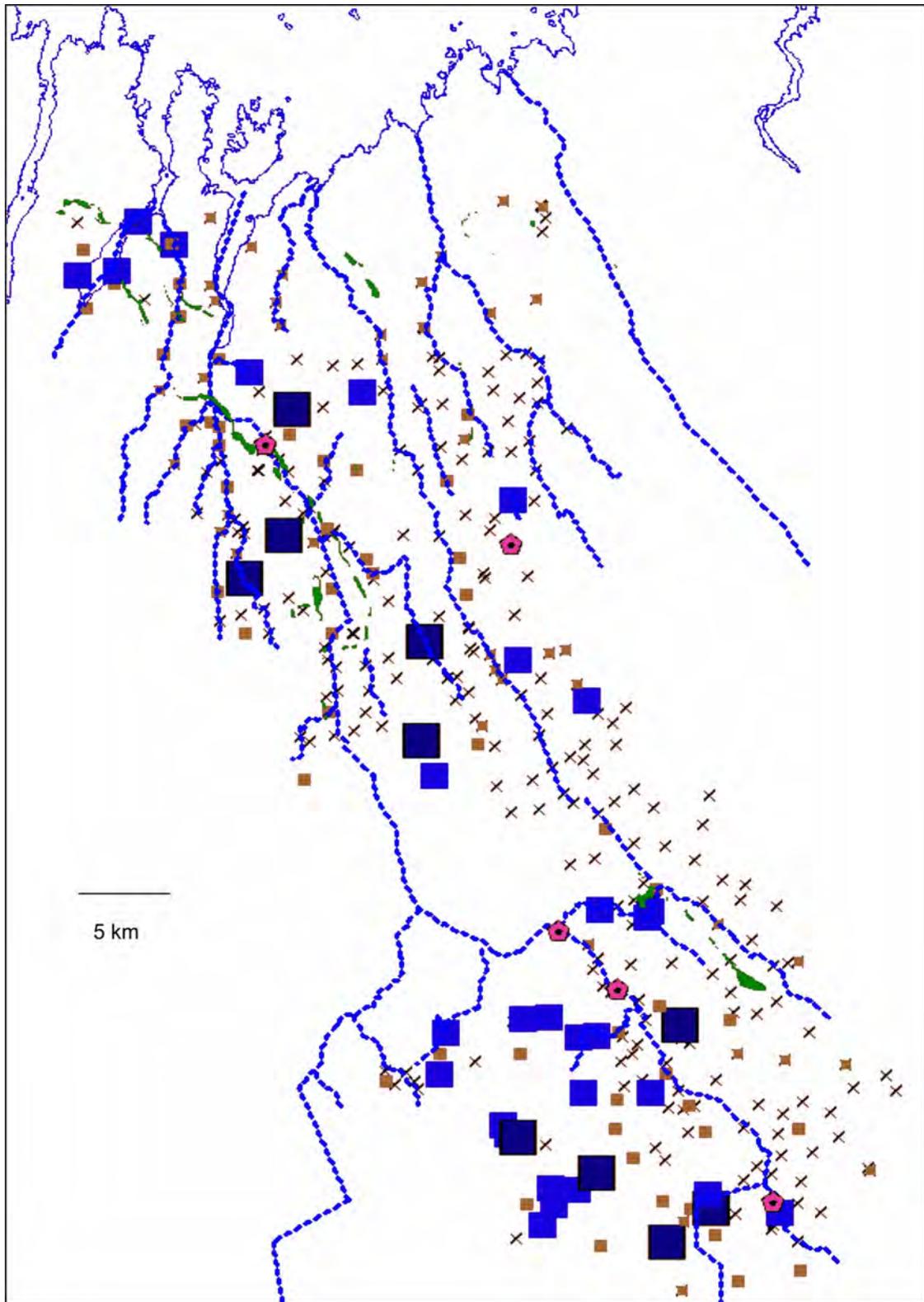


Fig. 2. Indicator mineral survey from Canada showing an example of greater than necessary sample density.

**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.

### COST OF SURVEYS

The design of a survey is strongly influenced by the cost. 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

M. Beth McClenaghan

Geological Survey of Canada, 601 Booth Street, Ottawa, Ontario, K1A 0E8  
(e-mail: [bmcclena@nrcan.gc.ca](mailto:bmcclena@nrcan.gc.ca))

## INTRODUCTION

The application of indicator mineral methods to mineral exploration has grown and developed significantly over the past two decades and they are now used around the world to explore for a broad spectrum of commodities. Heavy mineral suites have been identified for detecting a variety of ore deposit types including diamond, gold, Ni-Cu, PGE, metamorphosed VMS, porphyry Cu, uranium, tin, and tungsten. Indicator minerals, including ore, accessory, and alteration minerals, are usually sparsely distributed in their host rocks. They may be sparser in surficial sediments, therefore sediment samples must be concentrated in order to recover and examine them. Many indicator minerals have a moderate to high ( $>2.9 \text{ g/cm}^3$ ) specific gravity (SG), 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 10 kg 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) without contaminating the sample and do so 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 can also be recovered from both weathered and fresh bedrock, as well as from mineralized float. The combinations of processing techniques used by exploration companies or government agencies for recovering indicator minerals are quite varied (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. As most oxide and silicate indicator minerals (e.g. kimberlite, Ni-Cu-PGE, and metamorphosed massive sulphide indicator minerals) are medium to coarse sand sized (0.25-2.0 mm; Averill 2001), concentration techniques that recover the sand-sized heavy minerals are required. In contrast, approximately 90% of gold grains, platinum group minerals (PGMs), and sulphide minerals are silt sized ( $<0.063 \text{ mm}$ ), thus concentration of these indicators requires a preconcentration technique that 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

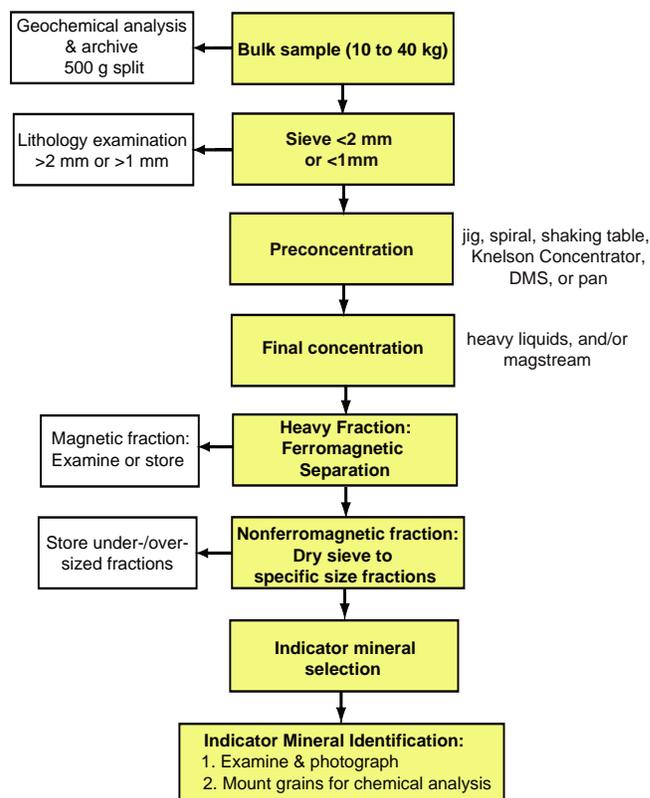


Fig. 1. Generalized flow sheet showing steps in sample processing used to reduce sample weight, concentrate heavy minerals, and recover indicator minerals.

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 20 to 50 kg (or more) in order to recover a sufficient weight of sand-sized heavy minerals (Table 2, sample 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, samples 1 to 4). Sediment samples collected for recovery of porphyry Cu indicator minerals (PCIM) need only be  $\sim 0.5 \text{ kg}$  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.

## BEDROCK PREPARATION

Bedrock and float samples often need to be crushed prior to processing to recover heavy minerals. Crushing or disaggregating reduces rock fragment size to about 2 mm, or the average size of mineral grains in the sample. Since rock crushers can be difficult to clean between samples, barren quartz can be crushed as a blank between each bedrock sample to reduce cross contamination. To monitor heavy mineral carryover, the

**Table 1.** Examples of the 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

blanks can then be processed and examined for heavy minerals along with the actual sample concentrates.

### PRECONCENTRATION METHODS

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 % 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 as well as time consuming, and the available methods may not provide optimal recovery of the indicator minerals of interest.

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 (or <1) mm fraction is preconcentrated most commonly using sieving and/or density methods (e.g. jig, shaking table, spiral, dense media separator, pan, or 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.

#### Panning

Panning is the oldest method used to recover indicator minerals, primarily for gold and PGMs. Sediment is placed in a pan

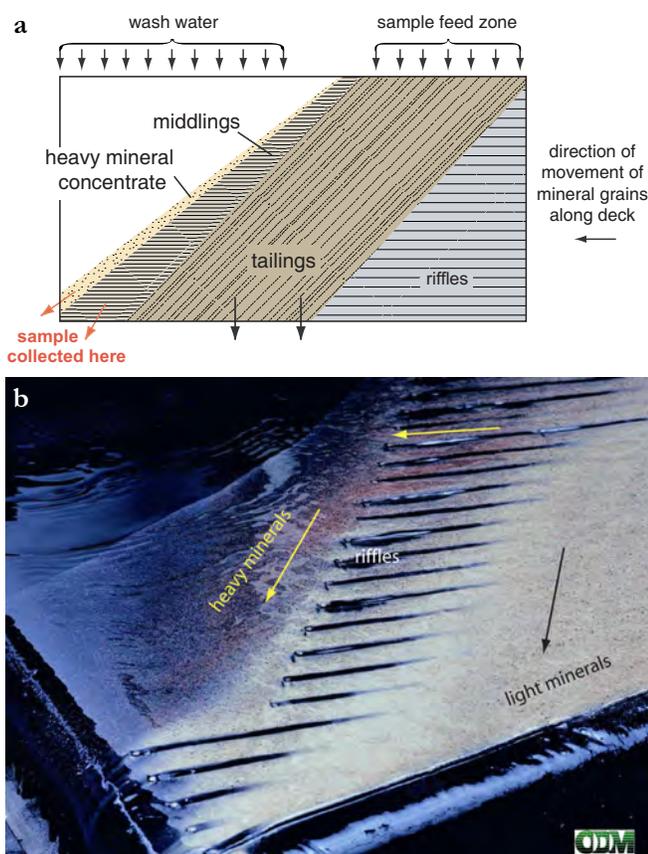


**Fig. 2.** Panning is one of the oldest methods used to reduce sample volume and prepare a heavy mineral preconcentrate (photo from W. Spirito, Geological Survey of Canada).

and shaken sideways in a circular motion while being held just under water, heavy minerals sink to the pan bottom and light minerals rise and spill out over the top (e.g. Silva 1986; English *et al.* 1987; Ballantyne & Harris 1997). Pans have varying shapes (flat bottomed or conical) and sizes (Fig. 2), and can be made out of plastic, metal, or wood. The advantages of this technique are that it can be a field or lab-based operation, is inexpensive in terms of equipment costs, and, if used in the field, it reduces sample shipping weight and thus cost. Panning is

**Table 2.** Weight of fractions generated by a combination of tabling and heavy liquid separation (MI) to reduce till sample weight, concentrate heavy minerals, and recover indicator minerals. **A)** initial sample weight; **B)** sieving of <2 mm; **C) & D)** tabling; **E)** heavy liquid separation; **F)** ferro-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 occurrence, Sudbury, Ontario; 3) Pamour Au 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 of >2 mm clasts (kg)	C: Weight of sample put across the shaking table (kg)	D: Weight of concentrate produced from shaking table (g)	E: Weight of heavy liquid light fraction (g)	F: Weight of the magnetic fraction (g)	G: Weight of 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



**Fig. 3. a)** Schematic plan view of a shaking table (Silva 1986); **b)** close up of heavy minerals separating from light minerals on a shaking table (photo from Overburden Drilling Management Ltd.).

often used in combination with other preconcentration methods to recover the silt-sized precious metal grains (e.g. Grant *et al.* 1991; Leake *et al.* 1991, 1998; Ballantyne & Harris 1997; Wierchowicz 2002). The disadvantages of this method are that it is slow, is highly dependent on the experience and skill of the operator, and therefore requires consistent personnel to perform the panning.

### Tabling

Preconcentration using a shaking (Wilfley) table is a commonly used method for separating minerals on the basis of density (Silva 1986). It recovers silt to coarse sand-sized heavy minerals for a broad spectrum of commodities, including diamonds, precious and base metals, and uranium (Averill & Huneault 2006). Silva (1986) describes this method consisting of a table with the deck covered with up to 1 cm high riffles along half of the surface (Fig. 3a). A motor mounted to the side drives a small arm that shakes the table along its length. A slurry of <math><2.0\text{ mm}</math> sample material and water is fed along the top of the table, perpendicular to the direction of the table motion. The table is shaken sideways using a slow forward stroke and a fast return strike that causes the grains to crawl along the deck parallel to the direction of motion. The shaking motion, combined with the water wash, moves grains diagonally across the deck from the feed end and separates them on the table according to size and density (Fig. 3b). If kimberlite indicators are targeted, the sample may be tabled twice to ensure higher recovery of the key lower density minerals, such as Cr-diopside and forsteritic olivine, as well as the coarsest grains. The advan-

tages 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 and metallic metal mineral grains, as well as kimberlite indicator minerals (e.g. English *et al.* 1987; McClenaghan *et al.* 1998; 2002). 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 commonly used to preconcentrate kimberlite indicator minerals is the micro-scale dense media separator (DMS) (Fig. 4). As described by Baumgartner (2006), heavy mineral concentration is carried out using a gravity-fed high-pressure cyclone. The <math><1\text{ mm}</math> fraction of a sample is mixed with ferrosilicon (FeSi) to produce a slurry that has a controlled density. The slurry is fed into a cyclone where the grains travel radially and helically, forcing the heavier particles toward the wall of the cyclone and the lighter particles toward the centre. The lighter and heavier particles exit the cyclone through different holes, with the light fraction discarded and the heavy fraction collected on a 0.25 or 0.3 mm screen. The >math>>0.25/0.3\text{ mm}</math> heavy fraction 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 approximately 3.1 g/cm<sup>3</sup> and is calibrated to recover the common kimberlite



**Fig. 4.** Micro dense media separator used at Mineral Services Canada to separate kimberlite indicator minerals (photo from Mineral Services Canada).



**Fig. 5.** Lab-based Knelson concentrator used to prepare a preconcentrate for recovery of kimberlite indicator minerals from till by the Geological Survey of Finland (from Marmo *et al.* 2008).

indicator minerals that have a specific gravity of  $>3.1 \text{ g/cm}^3$ : 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 to maintain accurate specific-gravity thresholds. 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 recover silt-sized precious and base metal indicator minerals.

#### Knelson concentrator

The Knelson concentrator is a fluidized centrifugal separator (Fig. 5) that was originally designed for concentrating gold and platinum from placer and bedrock samples. However, in recent years, it has also been used to recover kimberlite indicator minerals from sediment samples (e.g. Chernet *et al.* 1999; Lehtonen *et al.* 2005). The concentrator can handle particle sizes from  $>10$  microns up to a maximum of 6 mm. The general processing procedure, from the Knelson Concentrator website: <http://www.knelsongravitysolutions.com/>, states that water 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. Particles with high specific gravity 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 takes 5 to 11 minutes per sample. The advantages of the Knelson concentrator are that it is fast, inexpensive, and can be used in a lab or mobilized to the field to reduce the weight of material to be shipped to the lab. 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).

#### Rotary spiral concentrator

Heavy minerals can be recovered using a rotary spiral concentrator, which consists of a flat circular stainless steel bowl with rubber ribs that spiral inward (Fig. 6), a detailed description of which is reported by Silva (1986). A spiral concentrator is mounted on a frame so it can be tilted and has a water wash bar extending laterally from one side of the bowl to the centre. As the bowl spins, water is sprayed from the bar and heavy mineral grains move up and inward along the spirals to the central opening, where they are collected in a container behind the bowl. Water washes light minerals down to the bottom bowl. The heaviest minerals are recovered first. The advantages of the spiral concentrator are that it can be field based and thus reduce sample weight to be shipped, it is inexpensive to acquire and operate, it is fast if the material is sandy, and it recovers indicator minerals across a broad size range, from silt- to sand-size grains. The method, however, is dependent on the experience and skill of the operator, the lower density threshold is variable, there is some loss of heavy minerals, and the method is slow if the sample is clay-rich. It is used mainly for gold recovery (e.g. Maurice & Mercier 1986; Silva 1986) but in the past 10 years it also has been used for the recovery of kimberlite indicator minerals (e.g. Sarala & Peuraniemi 2007).

#### Magnetic separation and sieving

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 specific methods include wet screening, typically at 0.86 mm and 0.25 mm. The 0.25 to 0.86 mm fraction 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 / diamagnetic, 2) weakly paramagnetic, and 3) strongly paramagnetic. The weak (2) and strong (3) paramagnetic fractions are combined in one "magnetic concentrate" that is then processed through heavy liquids. The advantages of this procedure are that it is fast and inexpensive. Their methods, however, do not allow for the recovery of silt-sized precious and base metal grains, and do not recover coarse ( $>1$  mm) indicator minerals.



**Fig. 6.** Lab-based rotary spiral concentrator used by the Geological Survey of Finland (photo from P. Sarala, Geological Survey of Finland).



**Fig. 7.** Separation of heavy and light minerals is carried out using a specific heavy liquid of known density in separatory funnels. Heavy minerals sink and light minerals float to the top (photo from Overburden Drilling Management Ltd.).

### FINAL CONCENTRATION METHODS

A preconcentrate is usually further refined using heavy liquids of a precise density to further reduce the size of the concentrate prior to heavy mineral selection (Table 2, column E). Heavy liquid separation (Fig. 7) provides a sharp separation between heavy (sink) and light minerals (float) at an exact known 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 SG of 3.3 g/cm<sup>3</sup> and tetrabromoethane (TBE) with a SG 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 SG 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 g/cm<sup>3</sup> in order to recover the mid-density indicators tourmaline (dravite), alunite, and turquoise (Averill 2007).

Ferromagnetic minerals may 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 from sampling or processing are removed using a magnetic separator and commonly set aside unless magnetite, pyrrhotite, or magnetic ilmenite are important indicator minerals.

The non-ferromagnetic fraction is commonly sieved into two or three size fractions (e.g. 0.25-0.5 mm, 0.5-1.0 mm, and 1.0-2.0 mm) 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 0.25-0.5 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 non-ferromagnetic 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). Paramagnetic minerals are not magnetic, but are weakly attracted into a magnetic field. Paramagnetic minerals with different degrees of paramagnetism can be separated from one another by varying the strength of the magnetic field produced by an electromagnet. 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 remove 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 non-ferromagnetic heavy mineral concentrates during a visual scan, in most cases, of the finer size fractions (e.g. 0.25-0.5 mm, or 0.3-0.5 mm, 0.25-0.86 mm) 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/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. 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 such 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 identified and counted under short-wave ultraviolet light because they fluoresce. Gold and PGM grains may be panned from preconcentrates that were prepared in such a way that the silt-sized fraction has been retained (e.g. tabling). The grains may be counted and classified with the aid of an optical or scanning electron microscope. Commonly, gold grains are classified according to their shape/degree of wear (e.g. DiLabio 1990; Wierchowicz 2002), as this 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 (Jackson 2009) 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; Averill 2007; Scott & Radford 2007; Spry *et al.* 2009). 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; Wyatt *et al.* 2004; Grütter 2007). Gold, PGM, and sulphide grains may be analyzed to determine their trace element chemistry (e.g. Grant *et al.* 1991; Podlipsky *et al.* 2007) or isotopic compositions (e.g. Hattori & Cabri 1992).

### QUALITY CONTROL

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

### SUMMARY

This paper has described some of more common procedures available in commercial labs for processing surficial media and rocks to recover indicator minerals for mineral exploration. 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, and should 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

Simon Jackson

*Geological Survey of Canada, 601 Booth Street, Ottawa, Ontario K1A 0E8*

*(e-mail: simon.jackson@NRCan.gc.ca)*

## INTRODUCTION

Indicator mineral studies rely heavily on rapid, in situ analytical techniques because of their speed and the avoidance of laborious chemical dissolution and, for isotopic analysis, chemical separation. Historically, indicator mineral studies have utilized largely major and minor element chemical data. However, with the advent of a range of rapid, in situ techniques that can measure trace element concentrations and, now, highly precise isotope ratios, indicator mineral studies are starting to benefit from multi-dimensional analytical data.

This contribution will not only describe the analytical techniques currently available but emphasize the multiple types of information that can be generated from common indicator minerals as a result of recent technological breakthroughs. For example, on a single mineral, zircon, modern analytical techniques can rapidly extract physical information on crystal structure (e.g. core and overgrowth history), major and trace element chemistry (source magma type), rare earth element (REE) patterns (oxidation state of magma), U-Pb ratios (age(s)) and Hf isotope ratios (magma source). This wealth of diagnostic information, which was not attainable even a few years ago, represents a powerful arsenal in the exploration for certain magmato-hydrothermal mineral deposits that are genetically linked to specific magma types (in terms of magma source, degree of fractionation and oxidation).

This contribution will discuss the major in situ analytical methodologies with an emphasis on laser-based techniques because their relatively low capital cost and speed has made them affordable and widely accessible relative to conventional techniques.

## IN SITU ANALYTICAL METHODOLOGIES

### Major (and minor) element analysis by electron probe micro-analysis

Major element chemistry of minerals is generally determined using an electron probe micro-analyzer (EPMA), more commonly known as the electron microprobe. In this technique, low-energy electrons from a tungsten filament (cathode) are accelerated, collimated, and focused into an approximately 1  $\mu\text{m}$  diameter spot on the sample surface. The focussed electron beam excites various effects in the upper few microns of the sample, including cathodo-luminescence (visible light fluorescence), characteristic X-ray radiation, and backscattered electron (BSE) production.

For elemental analysis, the characteristic X-rays are measured either by wavelength-dispersive spectrometry (WDS) or energy-dispersive spectrometry (EDS). In WDS, selected X-ray wavelengths specific to each element of interest are separated by a monochromator. The monochromator is a crystal or synthetic multi-layered material that separates X-rays by Bragg diffraction. As WDS spectrometers measure the intensity of one wavelength at a time, electron microprobes are generally equipped with several monochromators, which will include

crystals of differing d-spacing to allow measurement of a wide range of X-ray wavelengths, and allow simultaneous measurement of several elements. The intensities of the separated X-rays are measured using gas-flow or sealed proportional detectors. In EDS, a solid state semiconductor detector (Si(Li) – lithium drifted silicon) simultaneously accumulates all X-ray photon energies produced from the sample and measures the intensities of characteristic X-ray energies. Simultaneous detection is advantageous for many applications, such as rapid identification of target grains and compositional mapping, which can require a prohibitively long time using WDS. However, the EDS has poorer spectral resolution and is, therefore, less well suited to quantitative analysis, especially for trace elements.

Chemical composition is determined by comparing the intensities of characteristic X-rays from the sample material with intensities from minerals of known composition (standards). To yield quantitative chemical compositions, the signal intensities from the sample must be corrected for matrix effects - ZAF correction, in reference to the three components of matrix effects; atomic number (Z), absorption (A), and secondary fluorescence (F).

Another important capability of the electron probe is imaging of the internal structure of minerals (e.g. zircons) using back-scattered electron (BSE) imaging. Since the BSE detector is actually a light-sensitive diode, the image obtained of the internal structure of a mineral is a combination of the variation in mean atomic number (composition) as well as the cathodo-luminescence (CL). Knowing the internal structure of, say, a zircon, especially where two or more generations of growth are apparent, is critical for guiding further study of the zircon by other methods.

### *Figures of merit*

The high continuum background in EPMA results in relatively high detection limits. While use of very long count times can produce detection limits to ppm levels, the realistic detection limit of the technique is approximately 0.01% (typical analysis time is approximately 10 minutes per grain). With its excellent spatial resolution, variations in chemical composition within a material (e.g. zoning) can be determined by traversing or by rastering the electron beam over the sample to produce a distribution map of the major elements in the crystal. Because of its unequalled spatial resolution and maturity, EPMA remains the preeminent technique for in situ major (and minor) element analysis in minerals.

### *Applications*

The EMPA has been utilized in a number of indicator mineral applications. The classical application is using it to search for specific kimberlite indicator mineral compositions (e.g. 'G10' eclogitic garnet, Mg-ilmenite, Mg-chromite), determined to be significant trace components of some diamondiferous kimberlites (e.g. Gurney & Switzer 1973; Grütter et al. 2004; Schulze 1997; Armstrong 2009; Clements et al. 2009). It has also been

used heavily in the mineral characterization of gold and PGE placers and lode deposits (e.g. Cabri et al. 1996) and as a guide in the search for metamorphosed massive sulphide deposits (Spry & Teale 2009).

Another recent application is determining the major and trace element contents of iron oxide minerals (magnetite and hematite), which occur widely in a number of mineral deposits and have highly desirable physical and chemical properties (e.g. high density, resistance to physical and chemical weathering, and easy separation from heavy mineral concentrates). Studies of iron oxides from a variety of ore deposit settings show that they possess major and minor elemental variations that can be used to discriminate grains from different source deposit type, and thus they can be applied in mineral exploration (Dupuis & Beaudoin 2008).

The EMPA also plays a critical role in providing major element data that are required for calibrating trace element analyses by LA-ICP-MS and providing BSE images of minerals (e.g. zircons) for age dating studies and trace element analysis (discussed below).

### Trace element analysis

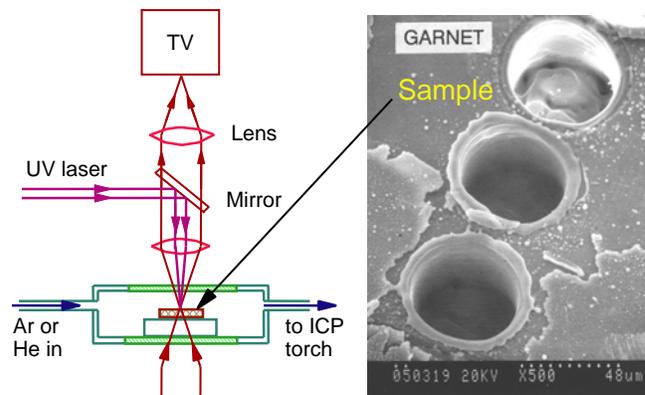
Trace elements generally show orders of magnitude more variability in rocks and minerals than major elements. Thus they are much more sensitive indicators of geological processes and environments than major elements. Additionally, many important ore elements typically occur at trace element levels in common indicator minerals.

Technology developments in the past two decades now allow rapid in situ trace element analysis of minerals to be performed routinely by a variety of techniques, including Secondary Ion Mass Spectrometry (SIMS), Proton Induced X-ray Emission (PIXE), and laser ablation-inductively coupled plasma-mass spectrometry (LA-ICP-MS). Thus the application of trace elements in indicator minerals studies is growing. In terms of capital cost and throughput, and thus accessibility, the most widely used technique today by far is LA-ICP-MS. Thus this technique is highlighted in this article.

#### *Laser ablation-inductively coupled plasma-mass spectrometry*

Unlike the older technique of ICP-atomic emission spectroscopy (ICP-AES), which uses an inductively coupled Ar plasma (ICP) to excite atomic *emission* of electromagnetic radiation from elements, the ICP-MS utilizes the powerful *ionizing* capability of the ICP. When this is combined with the unparalleled detection power of a mass spectrometer, it provides an incredibly sensitive instrument that allows elemental concentrations to be determined in solutions at concentrations down to ppq levels (parts per  $10^{15}$ ). The ICP is a very flexible ion source that can be coupled to a variety of sample introduction devices, most significantly from an indicator mineral standpoint, a laser ablation sampler. Combining the ICP-MS with a LA sampler provides a simple but incredibly flexible technique for in situ trace element and isotopic analysis.

In laser ablation analysis, a pulsed UV laser is steered and focused onto the sample, which is mounted in a cell through which a carrier gas (usually He) is flowing (Fig. 1). The interaction of the focused laser beam with the sample ablates material, which is transported as an aerosol to the ICP by the carrier gas. In the ICP, the aerosol particles are volatilized and converted to ions, which are extracted through an interface into the mass spectrometer for isotopic quantification. A vari-

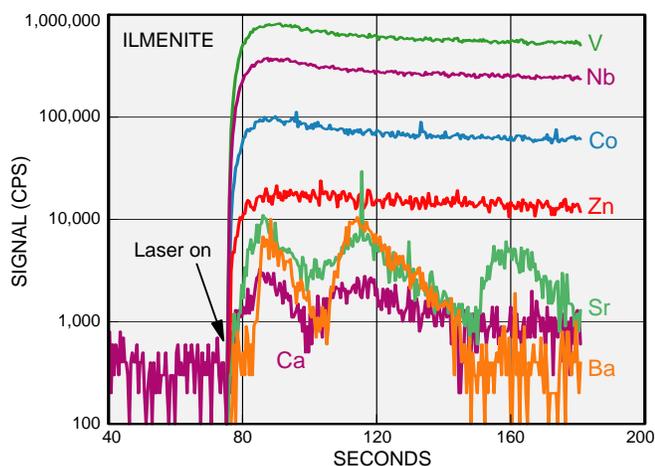


**Fig. 1.** Principles of a laser ablation sampling system. The sample may be a thin section, polished block or grain mount, or rock slab. Ablation pits shown are approximately 50  $\mu\text{m}$  in diameter.

ety of laser sources are currently used, with the most common being the UV harmonics of the Nd:YAG laser (4<sup>th</sup> harmonic at  $\lambda = 266$  nm or 5<sup>th</sup> harmonic at 213 nm) and the ArF excimer ( $\lambda = 193$  nm). Although more expensive to produce, shorter wavelengths are more strongly absorbed by transparent matrices and thus are preferred for diverse geological applications. Laser ablation systems feature controllable incident laser power and repetition rate, a number of spot size selections (typically about 10–150  $\mu\text{m}$ ), and a system for visualizing the sample. Sample preparation requirements are minimal; no coating or polishing is required. Although commercial LA systems have historically had rather small sample cells (approximately 50 mm diameter) large format cells are now being developed that can hold multiple samples and allow automated analysis of very large sample runs.

A laser sampler can be coupled to any ICP-MS system, including instruments that measure each mass sequentially (quadrupole and single-collector magnetic sector instruments) and those that measure each mass simultaneously (multi-collector magnetic sector and the rarely used time-of-flight instruments). Sequential instruments are more flexible but the simultaneous instruments offer better precision for isotopic ratio measurements.

The most common ICP-MS system, because it is the cheapest and offers the greatest flexibility in terms of mass scanning speed, employs a quadrupole mass filter. This consists of four parallel rods between which the ions are focused. The quadrupole mass filter separates the ions based on the stability of their trajectories in oscillating electric fields that are generated by superimposing RF voltages on to DC voltages applied between adjacent rods. Once inside the quadrupole, the ions start to oscillate in response to the rapidly alternating electric fields. For any combination of DC and RF frequency, the oscillations are only stable for ions of a single mass-to-charge ratio ( $m/z$ ); these ions will reach the detector while those with unstable oscillations will strike the rods and dissipate. The stable  $m/z$  ratio can be scanned very rapidly by changing the DC voltage and the RF frequency in tandem. Modern ICP-MS systems have dual pulse-analogue detectors or other technologies that provide up to nine orders of dynamic range, allowing measurement of trace (ppb), minor (ppm), and major elements (percent) simultaneously. In addition, many instruments come equipped with a gas collision/reaction cell that can attenuate some molecular ion interferences (e.g.  $^{40}\text{Ar}^{16}\text{O}^+$  on  $^{56}\text{Fe}^+$ ).



**Fig. 2.** Time-resolved laser ablation signals for a kimberlitic ilmenite. Note the smooth signals for V, Nb, Co, and Zn, which parallel the signals for major elements, Ti and Fe (not shown), indicating that they are substituted for these major elements. The highly variable signals for Sr, Ca, and Ba suggest the presence of inclusions in this grain.

Magnetic sector single collector instruments employ both a magnetic sector and an electrostatic sector to separate and focus the ions. Such an arrangement is called a double-focusing high-resolution mass spectrometer. In these instruments, the reverse Nier-Johnson geometry - where the magnetic sector is before the electrostatic sector - is used. The magnetic sector disperses ions based on the degree of deflection in a magnetic field, which is dependent on their mass and ion energy, and the electrostatic sector focuses the selected dispersed ions into the detector. This geometry has the advantage that the electrostatic sector can be used to scan rapidly between masses, although several slower magnet jumps are required to scan across the entire working mass range.

Magnetic sector instruments offer the highest sensitivity and high mass-resolution capability (at substantially reduced sensitivity), which allows resolution of a number of molecular ion interferences, although this has few significant applications in LA-ICP-MS, particularly when the increase in detection limits is factored in. They are also more complex and thus are significantly more costly than a quadrupole ICP-MS instrument to purchase and to maintain. Finally, they have slower scanning speeds than a quadrupole system due to the longer settling times required by the magnet when the voltages are adjusted to achieve a mass jump. This makes them less well suited for transient signal analysis typical of laser ablation sampling.

Time-of-flight instruments use an electrostatic field to accelerate packets of ions towards the detector. Since the velocity of the ions depends on the mass-to-charge ratio, ions of different mass can be separated based on their arrival time at the detector. These instruments offer 'quasi-simultaneous' analysis, and are thus very fast and suffer less from signal noise created by 'plasma flicker'. However, they are beset by space charge effects (mutual repulsion of ions), which cause relatively large matrix effects and substantially limits sensitivity. They have not, therefore, had widespread uptake. The other simultaneous ICP-MS instrument is the multi-collector-(MC)-ICP-MS. These instruments are designed specifically for high-precision isotopic analysis and are discussed below.

In laser ablation analysis, data are acquired using a fast 'time resolved analysis' protocol (signal intensity with time), which

allows the evolution of the signals to be viewed as a function of analysis time (and thus ablation depth). This allows heterogeneity within the ablation volume to be evaluated and recognition of zoning, inclusions, etc. (Fig. 2). This is of critical importance since it provides useful information on how elements are bound in the mineral (cleavage planes, fractures, metamict zones, etc.) and generally allows the presence of inclusions to be recognized. The contribution of the inclusion to the signal can then be rejected by selective integration.

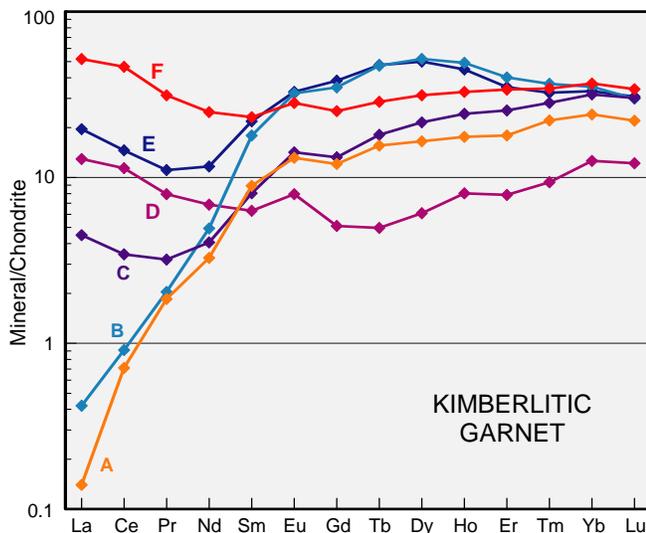
LA-ICP-MS does not suffer severe element-specific matrix effects and thus calibration is generally remarkably straightforward. For most applications, calibration is accomplished by referencing against a synthetic glass standard and correcting for 'ablation yield' via internal standardization using a naturally occurring internal standard (i.e. a major element in the sample, the concentration of which is either known from the mineral's stoichiometry or from measurement by EPMA). Procedures have been described recently that allow composition to be determined without knowledge of a major element concentration (Halicz & Günther 2004; Guillong et al. 2005).

#### *Figures of merit*

LA-ICP-MS throughput is rapid. A typical analysis determines 30-50 elements in 2-3 minutes. For elements well above detection limit, external precision (1 standard deviation) of 2 to 5% is normal. This is often much less than the natural variation of trace elements in minerals. Detection limits are a function of the analytical hardware and the number of elements determined, and are highly spot size dependent. However, for determination of 30 to 40 elements at typical sampling resolution (40  $\mu\text{m}$ ), detection limits down to low ppb can be achieved for heavy mass elements ( $z > 80$ ). Detection limits are higher for most low-mass elements but still quite sufficient to measure minor and major elements in most matrices. Good accuracy is generally attained, although there are some limitations related to fractionation of elements during the laser ablation process (e.g. determination of chalcophile elements in silicate minerals - see Jackson 2008). Use of shorter wavelengths and new ultrashort pulse lasers (femtosecond) have recently been shown to reduce fractionation (Horn & Von Blanckenburg 2007), and a variety of algorithms have been developed to correct for this effect (Jackson 2008). A host of commercial and freeware data-reduction packages are now available (see Appendix in Sylvester 2008).

#### *Applications*

With its inherent speed, detection capability, and flexibility, LA-ICP-MS is ideally suited to indicator mineral analysis. The most widespread application of LA-ICP-MS in indicator minerals surveys to date is in diamond exploration programs where trace element analysis of garnet has provided critical information on prospectivity via Ni geothermometry (Griffin et al. 1989) and the use of trace element/REE analysis to identify metasomatic events that may have resulted in the formation, or destruction, of diamonds (Fig. 3). LA-ICP-MS is also being applied to exploration for granite-related deposits where trace element chemistry of indicator minerals, such as zircon and apatite, provides important genetic information, such as source rock chemistry and origin, degree of fractionation and oxidation state and, potentially, therefore, the mineral prospectivity of the indicator mineral source rocks (Belousova et al. 2002a,b; Griffin et al. 2007). Other applications include the use of



**Fig. 3.** Range of rare earth element patterns, determined by LA-ICP-MS, for garnets from a single sample of kimberlite (data courtesy of M. Escayola). Analysis A is typical of a normal garnet. The higher LREE concentrations in analyses B-F and development of sinusoidal patterns (especially, analysis E) are indicative of variable degrees of metasomatism. Note that analyses B and E are from the same grain, which emphasizes the critical importance of careful in situ analysis.

chromite chemistry as an indicator for magmatic Ni deposits (Yao 2000) and the chemistry of native gold for typing deposits (Fig. 4).

Another potential application of LA-ICP-MS in indicator mineral exploration that has not yet been exploited is its capability of determining the trace element composition of fluid inclusions, which could potentially indicate whether quartz, for example, might have been deposited from a metal-rich fluid and is therefore potentially derived from an ore system

#### Low-precision isotopic analysis (U-Pb):

An important feature of a variety of resistate minerals (e.g. zircon, monazite, titanite, apatite, perovskite) is that they can be dated using radiogenic isotopic decay systems, especially the U-Pb scheme. Particularly when coupled with trace element and other isotopic data, age can be an important exploration criterion. Two main techniques are used for in situ U-Pb age dating. Historically, the main technique has been Secondary Ion Mass Spectrometry (SIMS). More recently, however, with its extremely rapid growth and advances in analytical methodologies in the past decade, LA-ICP-MS U-Pb dating has become extremely widespread and has overtaken SIMS in terms of output.

#### Secondary Ion Mass Spectrometry

Although Secondary Ion Mass Spectrometry (SIMS) can measure elemental and various isotopic ratios in minerals, its most widespread application is in U-Th-Pb geochronology. The ion microprobe uses a focused primary beam of O or Cs ions to sputter small areas of the sample (typically 10-30  $\mu\text{m}$  in diameter and a few microns deep). This releases 'secondary ions' from the target, which are accelerated and separated in a double-focussing mass spectrometer and measured by one, or more, detectors.

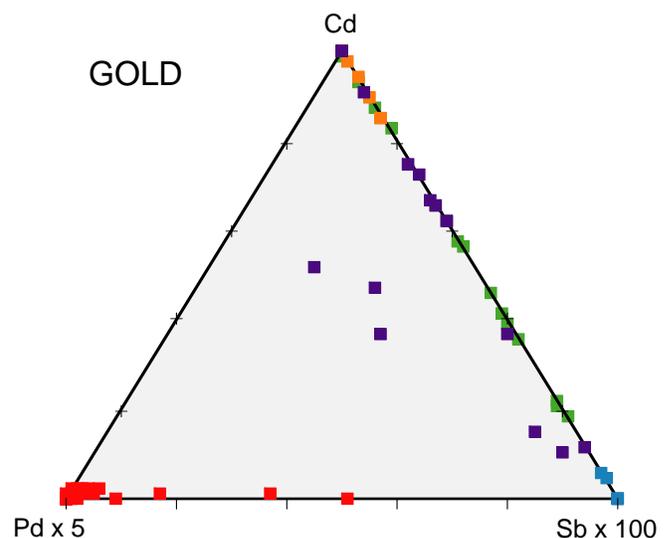
For U-Th-Pb geochronology, a beam of primary ions ( $\text{O}^{2-}$ ) is used to sputter secondary ions of U, Pb, and Th that are

measured successively, or simultaneously on MC instruments, along with certain reference peaks (e.g.  $\text{UO}^+$ ). Since the important U-Pb ratios (e.g.  $^{206}\text{Pb}/^{238}\text{U}$ ) are inter-element ratios and the sputtering yields differ between elements, between minerals, and with time during an analysis (due to increasing crater depth, charging effects, etc.), the measured relative isotopic abundances are not true isotopic abundances in the target. Thus, they must be calibrated against a standard material (matrix-matched material of known isotopic composition) by determining an analytical-session specific calibration factor. Since Pb/U ratios co-vary with the relative abundances of  $\text{U}^+$ ,  $\text{UO}^+$ , and  $\text{UO}_2^+$  ions, the  $\text{U}^+/\text{UO}^+$  ratio is used to monitor and correct the U/Pb fractionation effect.

#### Laser Ablation-Inductively Coupled Plasma-Mass Spectrometry

Since Laser Ablation Inductively Coupled Plasma-Mass Spectrometry (LA-ICP-MS) is an ion detecting technique, it is capable of isotopic measurements. However, single collector (sequential) instruments are limited by the signal noise (plasma flicker) to a best precision of approximately 0.1%. This restricts their application to isotopic systems that do not require very high precision. By far the most important of these is U-Pb dating, which is now a major application of the technique. With the inherent speed of LA-ICP-MS, individual labs are producing many thousands of zircon U-Pb age dates per year. Perovskite, titanite, monazite, and other minerals have also been dated. Note that, while MC-ICP-MS (discussed below) is also used for this application, no major analytical advantage has been reported as the primary source of analytical noise is variable fractionation of U and Pb during the ablation process.

Although a number of data acquisition and calibration approaches have been proposed, most rely on referencing against a matrix matched standard to correct for the inherent mass bias of the ICP-MS and the matrix-dependent fractionation of Pb and U during the ablation process (e.g. Jackson *et al.* 2004; Simonetti *et al.* 2008).



**Fig. 4.** LA-ICP-MS analyses of gold grains from five different locations (distinguished by symbol colour). Note that gold from each area is characterized by different elemental enrichments: red - Pd,(Sb); purple - Cd,Sb,(Pd); blue - Sb; orange - Cd; green - Cd,Sb.

*Figures of merit*

The external precision and accuracy of SIMS and LA-ICP-MS are both of the order of 1 to 3% (2 s.d.) and thus there is often debate as to which is the superior technique. SIMS has the advantage of better spatial resolution, particularly depth, although small spot size (20-40  $\mu\text{m}$  diameter, 5-10  $\mu\text{m}$  deep) LA-ICP-MS U-Pb dating has been reported (Simonetti et al. 2008). SIMS is therefore better suited for analysis of small zircons or zircons that have very small cores and/or overgrowths. SIMS also allows a  $^{204}\text{Pb}$ -based common Pb correction, which is compromised in LA-ICP-MS by an overlap from  $^{204}\text{Hg}$ , a ubiquitous contaminant in Ar supplies. The main advantages of LA-ICP-MS are much lower capital cost and speed - typically 60-80 age determinations per 8 hour day, although up to 40 age determinations per hour are reported! (<http://www.geo.arizona.edu/alc/Background.htm>). Thus many geological research institutions and companies now own one or more LA-ICP-MS systems, allowing widespread, cost-competitive accessibility to U-Pb geochronology (the so-called 'geochronology for the masses'). LA-ICP-MS generally handles the common Pb problem by selective integration of signals (common Pb often resides in discrete domains that can be identified and rejected during data reduction), regression of data (Jackson et al. 2004) and alternative common Pb correction procedures (e.g. Anderson 2002). LA-ICP-MS is also more flexible in terms of samples; for example, analysis of grains in thin section is straightforward.

**High-precision isotopic analysis - the multi-collector-inductively coupled plasma-mass spectrometer**

A largely unexplored approach in indicator mineral exploration is high-precision stable isotopic and radiogenic analysis. Yet, modern technology allows high-precision in situ isotopic analysis using LA-multi-collector (MC)-ICP-MS with a speed comparable to LA-ICP-MS trace element analysis (a few minutes per analysis) (Jackson et al. 2001). The MC-ICP-MS, which couples an ICP source to a double-focussing mass spectrometer with multiple collectors, has provided a new-found capability to measure the isotopic ratio with precision comparable to the benchmark technique of thermal ionization mass spectrometry (TIMS) but with all the advantages of an ICP ion source (high ionization efficiency, flexibility, steady-state ionization process). This development has led to a surge in research in the stable isotopic systems of elements such as Cu, Fe, Zn, Mo, W, etc., that had previously received scant attention owing to analytical limitations of TIMS.

The MC-ICP-MS uses a double-focussing mass spectrometer to separate the ions, which are then directed to a collector array consisting of numerous (~15) analogue and pulse counting detectors. The double-focusing mass spectrometer provides high transmission and flat-topped peaks, while multiple collections allow simultaneous measurement of the isotopes of interest, which circumvents the problem of plasma flicker-related signal noise that substantially limits the precision of sequential ICP-MS systems. The technology has become widely accepted and there are now a large number of MC-ICP-MS systems in geological establishments. In addition to its very high ionization efficiency, the ICP-source accepts a wide range of sample introduction systems. Coupling a laser ablation system to a MC-ICP-MS allows rapid, high-precision in situ isotopic analysis of a large array of elements.

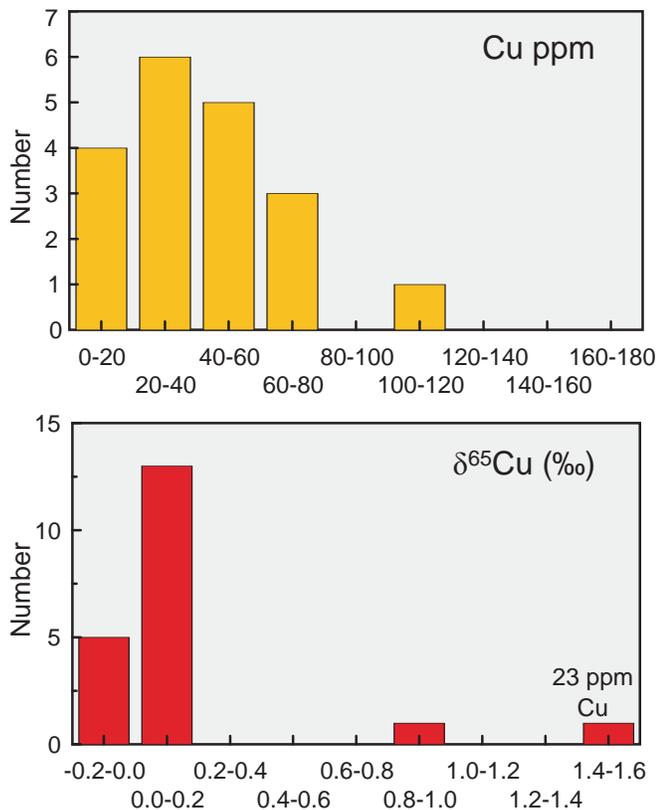
*Figures of merit*

LA-MC-ICP-MS allows extremely rapid (typically 3-5 minutes), in situ determination of a wide variety of isotope ratios with internal precisions as low as 10 ppm. Best precision is achieved mostly in minerals with analyte contents ranging from 1000 ppm upwards and with laser spot sizes ranging from approximately 50 to 150  $\mu\text{m}$  in diameter. Similar precision has been achieved using smaller spot sizes in minerals with percent levels of analyte and, no doubt, with larger spot sizes, rastering, or extended ablations similar precision could be achieved on minerals with analyte concentrations as low as a few hundred ppm. At low masses ( $z < 80$ ), laser-induced isotopic fractionation is significant and must be corrected using external standards. This limits typical precision to approximately 0.1‰, which is easily sufficient for measuring the typical variations in metal isotopes (e.g. >1‰ for Cu in Cu-rich sulphides in hydrothermal deposits). As in trace element analysis using LA-ICP-MS, time-resolved data acquisition is an essential tool for recognition of laser-induced isotopic fractionation and real isotopic heterogeneities in minerals.

*Applications*

In situ LA-MC-ICP-MS mineral analysis has already been applied to a large number of isotopic systems, including Mg, Si, Cu, Fe, Sr, Nd, Os, and Pb. However, so far, the applications of isotopic analysis in indicator mineral studies have been limited. By far the greatest has been the use of Hf isotopes in detrital zircons as part of the 'Terranechron' approach in which Hf isotope analyses of zircon substantially increase the value of the trace element and U-Pb age data, thus allowing terrane-scale interpretation of the crustal evolution of the drainage basin (Griffin et al. 2007).

Recent studies have shown that the Cu isotopic composition of granites (Li et al. 2009) is a more sensitive indicator of cryptic hydrothermal activity than trace element chemistry (Fig. 5), and that the isotopic composition of Cu in Cu-rich sulphides in porphyry systems can be used as a vector to ore (Graham et al. 2004). Thus, there exists the potential for stable isotopes of, for example, Fe and Si to be applied in resistate indicator mineral analysis. For example, Graham et al. (2004) showed that some skarn sulphides show extremely light ( $^{57}\text{Fe}$ -poor) Fe isotopic compositions relative to typical igneous rocks and porphyry deposits in the same district. If the Fe isotopes in common Fe-rich resistate hydrothermal minerals (e.g. magnetite, epidote) show similar isotopic trends, Fe isotope compositions might be useful for distinguishing hydrothermal versus magmatic versus metamorphic, and porphyry versus skarn, magnetite, or epidote. Iron isotope data would provide another dimension to the elemental data, potentially resulting in a very powerful tool for application in discrimination/exploration exercises using resistate minerals. Similarly, a further unexplored potential application of LA-ICP-MS is using Si isotopes of common resistate hydrothermal minerals (e.g. quartz) to interpret the genesis (e.g. magmatic or hydrothermal) and, potentially, crystallization temperature of the quartz. Another potential application of LA-MC-ICP-MS is analysis of resistate indicator minerals for their Pb isotopic composition, a system that has historically seen wide usage in mineral deposit research and exploration.



**Fig. 5.** Cu concentrations and  $\delta^{65}\text{Cu}$  values ( $^{65}\text{Cu}/^{63}\text{Cu}$  of the sample relative to a standard in parts per thousand) for I-type granite suites of southeastern Australia (data from Li *et al.* 2008). The two samples with anomalous Cu isotope ratios ( $>0.8\%$ ) are from closely spaced outcrops with trace hydrothermal chalcopyrite, the presence of which is not evident in the elemental data. Thus isotopes have the potential to be extremely sensitive indicators of cryptic hydrothermal activity, such as might occur in distal parts of hydrothermal Cu deposits.

## SUMMARY

The speed, level of maturity and automation, and thus cost effectiveness, of modern in situ trace element analytical techniques will see their increased application in resistate indicator mineral analysis. Similarly, with analytical advances (particularly development of isotopic mineral standards), there is huge potential for discovery of new isotopic applications in indicator mineral investigations. In particular, the ability of modern analytical techniques to provide multiple data sets (major, minor, and trace elements and isotopic data) on the same grains increases the variables with which to discriminate grains and with which to interpret their source rock settings.

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# Gahnite composition as a guide in the search for metamorphosed massive sulphide deposits

Paul G. Spry<sup>1</sup> & Graham S. Teale<sup>2</sup>

1. Department of Geological and Atmospheric Sciences, 253 Science I, Iowa State University, Ames, Iowa 50011-3212  
(e-mail: pgspry@iastate.edu)

2. Teale & Associates, P.O. Box 740, North Adelaide, South Australia 5006  
(e-mail: teales@ozemail.com.au)

## ABSTRACT

Gahnite occurs in metamorphosed massive sulphide deposits and sulphur-poor rocks in Mg-Ca-Al alteration zones, metamorphosed massive sulphide deposits in Fe-Al metasedimentary and metavolcanic rocks, marble, skarn, quartz veins, metabauxite, granitic pegmatite, contact metamorphic aureoles, granite, unaltered and hydrothermally altered Fe-Al-rich metasedimentary rocks, Al-rich granulite, and iron formation. Gahnite found in situ or as resistate grains in unconsolidated sediments (e.g. glacial till, alluvium) will have a composition of gahnite<sub>45-90</sub>hercynite<sub>0-45</sub>spinel<sub>0-25</sub> and gahnite<sub>0-65</sub>hercynite<sub>0-50</sub>spinel<sub>25-90</sub> in Mg-poor and Mg-rich massive sulphide deposits/alteration zones, respectively. Pressure, temperature, and  $f_{S_2}$ - $f_{O_2}$ - $f_{H_2O}$  conditions will affect the composition of gahnite within these fields and must be taken into account when exploring for Broken Hill-type Pb-Zn-Ag deposits, and metamorphosed SEDEX Pb-Zn and volcanogenic massive sulphide Cu-Zn-Pb deposits.

## INTRODUCTION

Resistate indicator minerals signify the presence of a rock, alteration type, or specific type of sulphide deposit (McClenaghan 2005). The physicochemical characteristics of these minerals mean that they can occur in soils, alluvium, and glacial and aeolian sediments. Indicator minerals are increasingly being used in the exploration for base metal sulphides, porphyry copper, magmatic (Ni-Cu-PGE), gold, tungsten, tin, and diamond deposits, with much of the emphasis being placed on the search for diamond, gold, and base metal deposits (e.g. Johansson *et al.* 1986; Stendal & Theobald 1994; Averill 2001, 2007a,b; McClenaghan 2005). There are almost twenty indicator minerals that have been used for magmatic deposits, including chromite, olivine, pyroxene, andradite, corundum, chalcopyrite, and native gold, whereas gahnite, spessartine, sphalerite, apatite, tourmaline, staurolite, rutile, anthophyllite, and chalcopyrite have been used in the search for metamorphosed massive sulphide deposits (e.g. Morris *et al.* 1997; Walters 2001; Averill 2007b). Gahnite and spessartine are particularly useful indicator minerals in the search for metamorphosed massive sulphide deposits because of their resistate nature; sphalerite and chalcopyrite are, for example, much more susceptible to physical and chemical attack, especially in non-glacial terranes.

In view of the close spatial relationship between gahnite and metamorphosed massive sulphide deposits [Broken Hill-type (BHT), sedimentary exhalative (SEDEX), and volcanogenic massive sulphide (VMS)], gahnite and gahnite-bearing rocks have long been used as exploration guide for ores of these types (e.g. Stillwell 1922; Ririe & Foster 1984; Sheridan & Raymond 1984, Spry & Scott 1986a,b; Spry 2000; Spry *et al.* 2000, 2003; Walters 2001, Walters *et al.* 2002; Taylor *et al.* 2005). However, it should be stressed that gahnite is also found in marble, pegmatite, aluminous metasedimentary rocks, aluminous granulite, iron formation, metabauxite, granite, and unconsolidated rocks (stream sediments, glacial till, and soils), in addition to being found in and adjacent to Sn-W skarn,

replacement-type Sn deposits associated with A-type granite (e.g. Prospect Hill, Australia), gold deposits, and non-sulphide zinc deposits (Table 1). As noted by Griffin *et al.* (2007), empirical fingerprints using resistate indicator minerals can only work if the target and host sequence are understood. Therefore, if gahnite is to be sought in resistate indicator surveys in stream sediments, glacial till, and soils, it is paramount that gahnite from the various geological settings can be distinguished. The aim of the present contribution is to summarize the compositional aspects of gahnite and to see how it can be used in the exploration for metamorphosed massive sulphide deposits. A key aspect of this work is to note how physicochemical variables and geological setting affect the composition of gahnite.

## COMPOSITION OF GAHNITE

### General considerations

Gahnite (ZnAl<sub>2</sub>O<sub>4</sub>) is a Zn end-member of the spinel group of minerals that also contains hercynite (FeAl<sub>2</sub>O<sub>4</sub>), spinel (MgAl<sub>2</sub>O<sub>4</sub>), galaxite (MnAl<sub>2</sub>O<sub>4</sub>), magnetite (Fe<sub>3</sub>O<sub>4</sub>), franklinite (ZnFe<sub>2</sub>O<sub>4</sub>), magnesioferrite (MgFe<sub>2</sub>O<sub>4</sub>), maghemite ( $\gamma$ -Fe<sub>2</sub>O<sub>3</sub>), ulvospinel ((FeTi<sub>2</sub>O<sub>4</sub>), jacobsonite (MnFe<sub>2</sub>O<sub>4</sub>), trevorite (NiFe<sub>2</sub>O<sub>4</sub>), magnesiochromite (MgCr<sub>2</sub>O<sub>4</sub>), and chromite (FeCr<sub>2</sub>O<sub>4</sub>). Of these spinel end-members, gahnite, hercynite, and spinel *sensu stricto* are the most important in the search for metamorphosed massive sulphide deposits. Minor amounts of the galaxite molecule are also present in natural gahnites but they generally contain less than 1-2 mole %. The other spinel group end-members rarely form solid solutions with the gahnite molecule, except for magnetite and franklinite, which form solid solutions with gahnite in the Franklin and Sterling Hill non-sulphide zinc deposits (Carvalho & Sclar 1988). Gahnite with >1 weight % of various metals also occurs in rare examples: Sn (Cotelo Neiva *et al.* 1955), Co (Shannon 1923; Spry & Scott 1986b; Feenstra *et al.* 2003), Cr (Uhlir *et al.* 1998), and Sb (Jancev 2001). It should also be noted that gahnite with a high V content also occurs in gneiss in the Mt. Painter area, South Australia (G.S. Teale unpubl. data).

**Table 1.** Selected examples of the geological settings of gahnite.

Setting/ Deposit Type	Location/Deposit	Reference
Broken Hill-type deposit	Broken Hill, South Africa	Spry (1987)
	Broken Hill, Australia	Spry <i>et al.</i> (2003)
	Cannington, Australia	Kim & Bell (2005)
	Pinnacles, Australia	Parr (1992)
Volcanogenic massive sulphide deposit	Palmeiropoulos, Brazil	Araujo <i>et al.</i> (1995)
	Linda, Canada	Zaleski <i>et al.</i> (1991)
Sedimentary exhalative deposit	Wheal Ellen, Australia	Spry <i>et al.</i> (1988)
	Rampura-Agucha, India	Hoeller & Gandhi (1997)
	Bleikvassli, Norway	Rosenberg <i>et al.</i> (2000)
Non-sulphide zinc deposits	Franklin, New Jersey	Johnson & Skinner (2003)
Gold deposit	Mount Gibson, Australia	Yeats & Groves (1998)
Tin-tungsten skarn	Dusky Sound, New Zealand	Challis (1986)
Contact metamorphic aureole	Ardara pluton, Ireland	Atkin (1978)
Diamonds	Ivancauti, Romania	Gardu (2004)
Marble	Bohemian Massif, Czech Rep.	Novak <i>et al.</i> (1997)
	Ganesh Himal, Nepal	Uhlir <i>et al.</i> (1998)
Iron formation	West Greenland	Appel (1986)
	Aggeneys, South Africa	Spry (1987)
Quartz veins	Broken Hill area, Australia	Wall (1977)
Al-rich metasedimentary rocks	New Hampshire, U.S.A.	Schumacher & Robinson (1987)
	Wind River Mts, U.S.A.	Frost (1973)
Aluminous granulite	Araku, India	Sengupta <i>et al.</i> (1991)
Metabauxite	Menderes massif, Turkey	Yalçin <i>et al.</i> (1993)
	Samos Island, Greece	Henry & Dutrow (2001)
Pegmatite	California	Kleck & Foord (1999)
	Siedlimowice, Poland	Szuskiewiz & Lobos (2004)
Granite	Buryat, Russia	Lisitsyn & Yurkina (1974)
Glacial sediments	Kallala Lake, Ontario	Morris <i>et al.</i> (2000)
Beach sands	Martha's Vineyard, U.S.A.	Kaye & Mrose (1965)
Soil	Unspecified location	Nachtegaal <i>et al.</i> (2005)
Stream sediments	Georgia, U.S.A.	Siegel <i>et al.</i> (1991)
	Kallala Lake, Ontario	Morris <i>et al.</i> (2000)
	James Bay, Ontario	Crabtree (2003)
	Sault Ste. Marie, Ontario	Reid (2003)

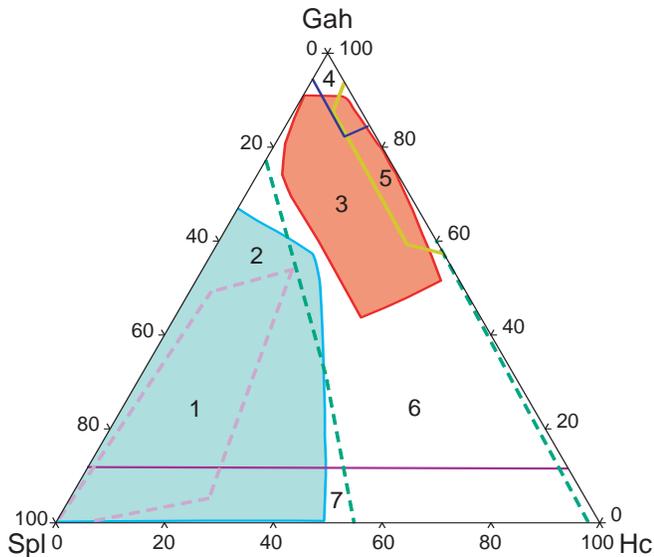
### Methods of analyses and trace element compositions of gahnite

In situ analytical methods that involve the electron microprobe (EMP), ion microprobe (Secondary Ion Mass Spectrometer, or SIMS), proton microprobe (Proton Induced X-ray Emission, or PIXE; Proton Induced Gamma Emission, or PIGE), and laser-ablation inductively coupled mass spectrometry (LA-ICP-MS) have been used to analyze major and trace element or just trace element composition of indicator minerals, particularly for diamond exploration (Griffin *et al.* 2007). Other than EMP analyses, the application of other in situ analytical techniques to determine the trace element compositions of indicator minerals, such as garnet, gahnite, apatite, and tourmaline, which have been used to specifically explore for metamorphosed base metal deposits, are relatively few in number (e.g. Griffin *et al.* 1996; Walters 2001; Stalder & Rozendaal 2005; Spry *et al.* 2007). Unpublished, preliminary LA-ICP-MS analyses of gahnite from the Broken Hill deposit shows that they contain REE that are close to or just above detection limits, which is in contrast to well defined patterns for garnet in garnet-rich rocks from the deposit (Spry *et al.* 2007). Analyses of the trace element content of gahnite in unconsolidated stream sediments or glacial till and in their source rocks by SIMS, PIXE, and LA-

ICP-MS are essentially lacking, but these data should be obtained and evaluated given the great success in the application of trace element data to resistate minerals associated with diamond exploration (e.g. Griffin *et al.* 2007).

### Major element compositions of gahnite

The close spatial relationship between gahnite and massive sulphides, either as grains of gahnite within high-grade sulphides or as gahnite-bearing rocks along strike from metamorphosed massive sulphide deposits, has meant that gahnite has long been used as an empirical exploration guide to ore (e.g. Stillwell 1922; Forwood 1968; Sheridan & Raymond 1984; Sandhaus & Craig 1986; Spry and Scott 1986a,b). The experimental and/or thermodynamic studies of Froese (1969), Wall & England (1979), and Spry & Scott (1986a) verified the proposal of, for example, Juve (1969), that gahnite may have formed as a result of chemical reactions involving sphalerite. However, it became apparent to Spry & Scott (1982, 1986a) and BHP Limited (Walters 2001; Walters *et al.* 2002) in the early 1980s that gahnite formed in various geological environments, and that there needed to be a way to attribute gahnite to a specific geological setting before it could be used as a tool to explore for metamorphosed massive sulphide deposits. While field mapping of



**Fig. 1.** Triangular plot of zincian spinel compositions in terms of gahnite (Gah), hercynite (Hc), and spinel (Spl) components from worldwide localities in (1) marble, (2) metamorphosed massive sulphide deposits and S-poor rocks in Mg-Ca-Al alteration zones, (3) metamorphosed massive sulphide deposits in Fe-Al metasedimentary and metavolcanic rocks, (4) metabauxite, (5) pegmatite, (6) unaltered and hydrothermally altered Fe-Al-rich metasedimentary and metavolcanic rocks, and (7) Al-rich granulite (area below the red line where the gahnite component <12%). Data in this plot are from Spry & Scott (1986a) and Heimann *et al.* (2005).

gahnite-bearing rocks can be used to follow gahnite-bearing rocks back to known or unknown sulphide occurrences, it is impossible to use gahnite found in glacial tills, solid horizons, and stream sediments as an exploration guide unless the composition of the gahnite is known (e.g. Morris *et al.* 1997).

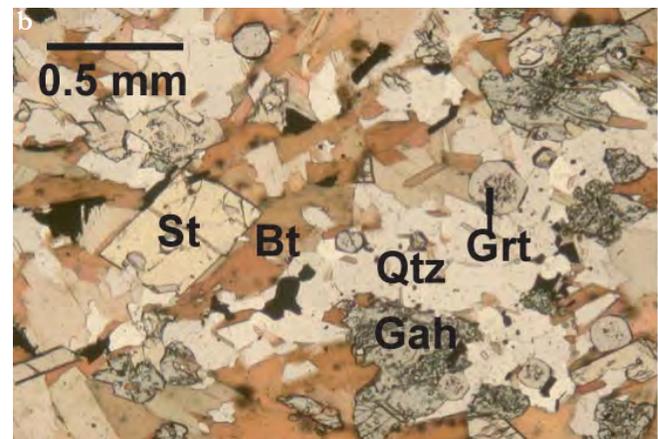
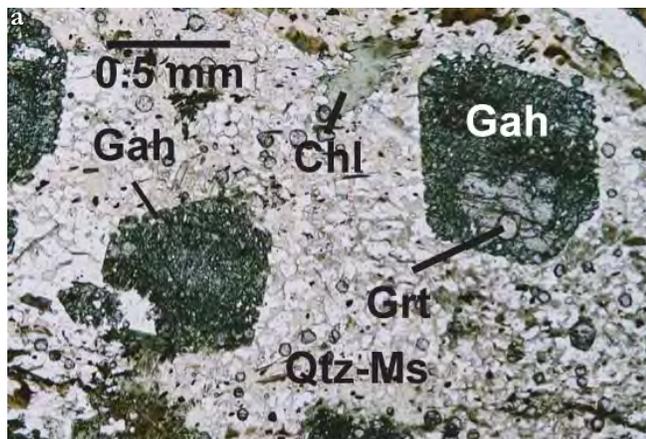
Various studies have used discrimination diagrams to distinguish gahnite from different source rocks. Walters (2001) used proprietary bivariate plots involving Zn-Fe-Mg-Mn, while most other workers have used ternary plots of Zn-Fe-Mg±Mn, or their wt.% oxide or spinel end-member equivalents, because the Mn content of gahnite is generally <1 wt.%. (e.g. Vokes 1962; Sandhaus & Craig 1986). Spry & Scott (1982, 1986a) distinguished zincian spinel compositions from 106 locations for metamorphosed massive sulphide deposits, aluminous

metasediments, marble, and pegmatite using a ternary plot of Zn-Fe-Mg, while Batchelor & Kinnaird (1984) distinguished fields for “igneous” and “metamorphic associations” using both bivariate [(Zn+Mn/Al) versus (Fe+Mg/Al)] and ternary plots (Zn-Fe-Mg). More recently, Heimann *et al.* (2005) utilized available zincian spinel compositions reported in the literature at that time to expand the work of Spry & Scott (1986a) in order to distinguish gahnite compositions for (1) metamorphosed massive sulphide deposits and sulphur-poor rocks in Mg-Ca-Al alteration zones; (2) metamorphosed massive sulphide deposits in Fe-Al metasedimentary and metavolcanic rocks; (3) marble; (4) metabauxite; (5) granitic pegmatite; (6) unaltered and hydrothermally altered Fe-Al-rich metasedimentary rocks; (7) Al-rich granulite; and (8) iron formation (Fig. 1). Gahnite is also found in skarn (Meca 1973), contact metamorphic aureoles of plutons (e.g. Atwin 1978), granite (Tulloch 1981), and sulphide-free quartz veins (Wall 1977), but the amount of compositional data available in the literature from these settings is limited.

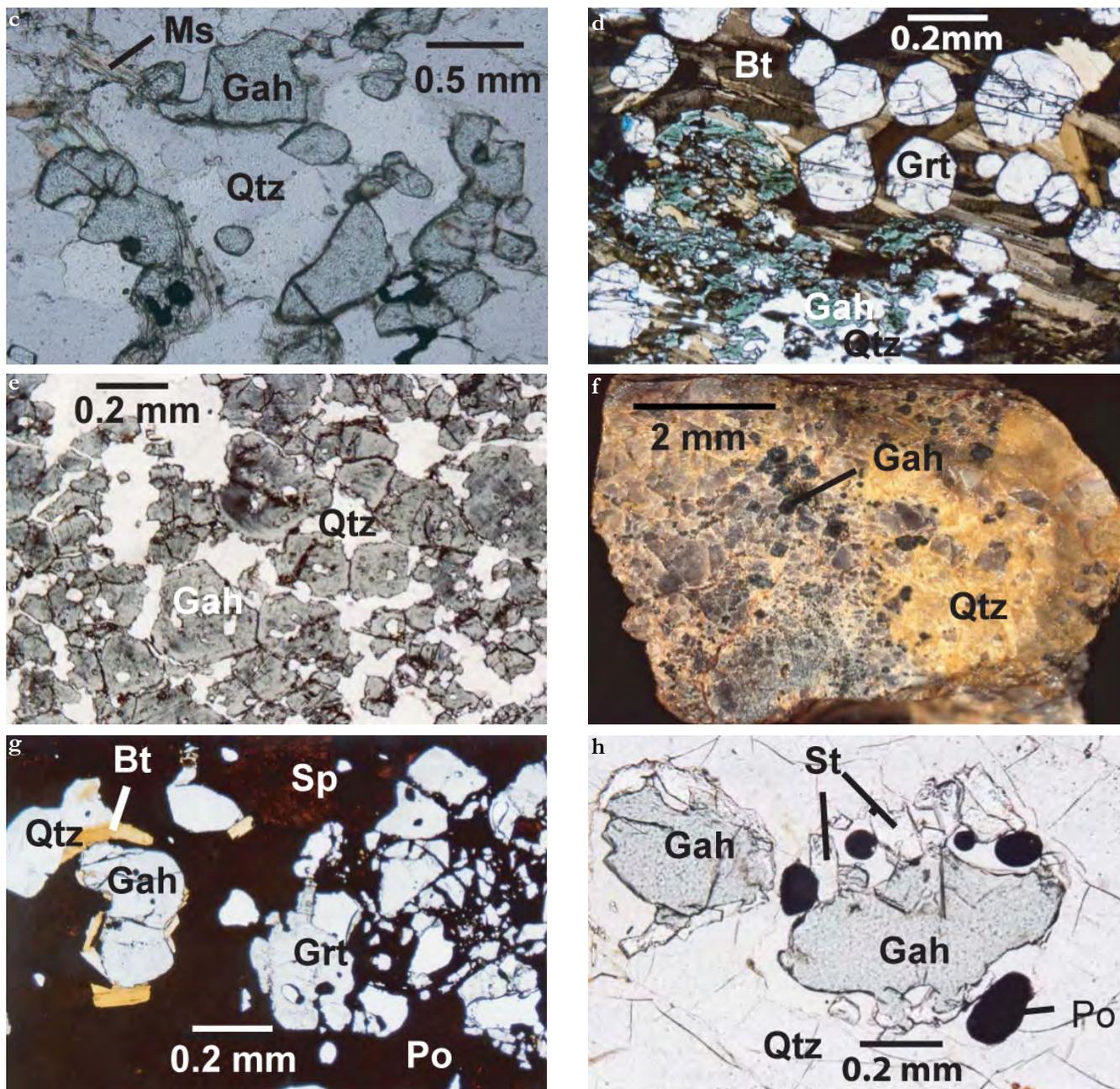
Morris *et al.* (1997) analyzed gahnite from C horizon till in the Separation Lake greenstone belt and from alluvium in the Kinniwabi Lake area, Ontario. Using ternary plots of ZnO-MgO-FeO<sub>t</sub>, Morris *et al.* (1997) recognized three groups of gahnite compositions from the Separation Lake greenstone belt and four from the Kinniwabi Lake area, and suggested that the composition of gahnite in groups 1 and 2 from both areas were derived from pegmatite and massive sulphide deposits, respectively. They also proposed that group 3 gahnite from the Separation Lake greenstone belt falls in the field of aluminous metasedimentary rocks of Spry & Scott (1986a). Groups 3 and 4 gahnites from Kinniwabi Lake were not ascribed to any source rock but group 3 overlaps the field of aluminous metasedimentary rocks, whereas group 4 overlaps the fields of marble as well as that of metamorphosed massive sulphide deposits and S-poor rocks in Mg-Ca-Al alteration zones of Heimann *et al.* (2005).

#### CONDITIONS OF GAHNITE FORMATION

Gahnite is stable from upper greenschist grade [e.g. Venn Stavelot Massif, Belgium (Kramm 1997) and the Mulyungarie Antiform area, South Australia, Fig. 2a], through middle amphibolite grade (e.g. Angas Pb-Zn-Ag deposit, South Australia, Fig. 2b), upper amphibolite grade (e.g. Foster River



**Fig. 2.** Plane-polarized photomicrographs, except for 2f, which is a hand-specimen photograph. **a)** Poikilitic grains of gahnite (Gah) with garnet (Grt) inclusions in a quartz (Qtz)-muscovite (Ms)-chlorite (Chl) matrix from the Mulyungarie Antiform, South Australia (upper greenschist facies). **b)** Gahnite-staurolite (St)-biotite (Bt)-quartz rock from the Angas Pb-Zn-Ag deposit, South Australia (middle amphibolite facies).



**Fig. 2 continued.** Plane-polarized photomicrographs, except for 2f, which is a hand-specimen photograph). **c)** Gahnite-quartz-muscovite rock from the George Pb-Zn-Ag prospect, Foster River area, northern Saskatchewan (upper amphibolite facies). **d)** Corroded gahnite in a biotite-garnet-quartz rock, Thunderdome (New South Wales, upper amphibolite facies). **e)** Gahnite-quartz rock in the Emu Pb-Zn-Ag prospect, Mutooroo, South Australia (granulite facies). **f)** Quartz-gahnite lode from the Centennial Pb-Zn-Ag prospect, New South Wales (granulite facies). **g)** Grains of gahnite, garnet, quartz, and biotite in massive sulphide [sphalerite (Sp) and pyrrhotite (Po)] in C-lode, Broken Hill Pb-Zn-Ag deposit, New South Wales, Australia (granulite facies). **h)** Euhedral zincian staurolite crystals that have formed as a breakdown product of corroded gahnite from the Berta Tank Pb-Zn-Ag deposit, Mutooroo, South Australia. Note that staurolite grew during a retrograde amphibolites-facies event during the Delamarian Orogeny, whereas gahnite formed during the Olarian Orogeny at the granulite facies.

Zn-Pb prospect, Saskatchewan, Fig. 2c; Thunderdome Pb-Zn-Ag prospect, New South Wales, Fig. 2d), and to the granulite facies (Centennial Pb-Zn-Ag deposit, New South Wales, Fig. 2e; Emu Ridge Pb-Zn-Ag prospect, South Australia, Fig. 2f; Broken Hill Pb-Zn-Ag, New South Wales, Australia, Fig. 2g), but it can breakdown to staurolite (Fig. 2h), chlorite, and muscovite during retrograde metamorphism. Clearly, it is also stable under magmatic conditions since it occurs as unaltered grains in pegmatite and granite (e.g. Tulloch 1981; Kleck &

Foord 1999). Heimann *et al.* (2005) pointed out that the composition of gahnite was dependent on (1) bulk composition of the host rock; (2) pressure and temperature; and (3) sulphur, oxygen, and water fugacity conditions.

Thermodynamic and experimental studies of Wall & England (1979) and Spry & Scott (1986a) suggest that gahnite in metamorphosed massive sulphides forms from various reactions in the system Zn-Fe-Al-Si-O-H-S that involve gahnite, sphalerite, pyrite, pyrrhotite, garnet, quartz, and either, silli-

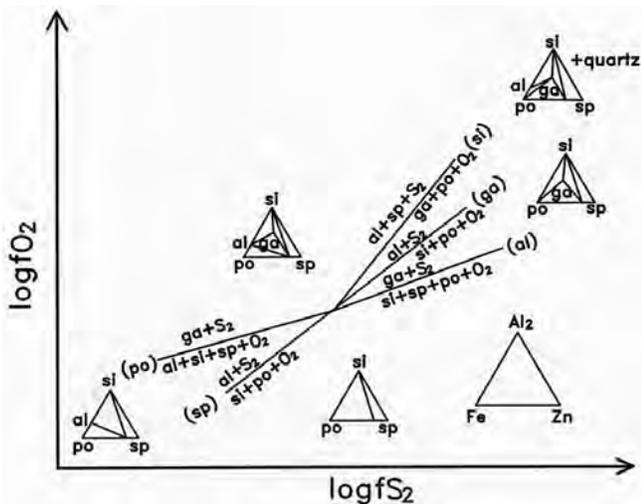


Fig. 3. Schreinemaker's analysis of the system Zn-Fe-Al-Si-S-O in  $\log fO_2$ - $\log fS_2$  space (after Spry & Scott 1986a).

manite, kyanite, or andalusite (Fig. 3). The system is particularly relevant to gahnite that forms in and adjacent to sulphides in BHT and SEDEX deposits (Fig. 2a-e, 2g). A Schreinemaker's analysis of this system shows gahnite can form by the desulphidation of sphalerite and that gahnite can also coexist with sphalerite without it having undergone a desulphidation reaction (Fig. 3). Gahnite will form under low  $fS_2$  and high  $fO_2$  conditions in ore zones. Gahnite that forms in the Mg-rich alteration zones of metamorphosed VMS deposits will form by a series of more complicated reactions in the systems Zn-Fe-Mg-Al-Si-K-S-O-H (Froese *et al.* 1989) and Zn-Fe-Mg-Al-K-F-O-H-S (Zaleski *et al.* 1991), which can involve the same minerals noted above for the system Zn-Fe-Al-Si-O-H-S, as well as Mg-rich such as anthophyllite, staurolite, clinocllore, and cordierite (Fig. 4).

Although massive sulphides are spatially associated with Mg-Ca-Al alteration zones like those found in Colorado, for example, such deposits serve to show the influence that Mg-rich assemblages have on the composition of gahnite, which are also Mg-rich (Fig. 1). Gahnite in most massive sulphide deposits (excluding those in Mg-rich rocks) have the following compositional range: gahnite<sub>45-90</sub>hercynite<sub>0-45</sub>spinel<sub>0-25</sub>. The reason for the restricted gahnite-to-hercynite ratio is because the activity of Fe in sphalerite, pyrite, and pyrrhotite and coexisting Fe-silicates will dictate the sulphur and oxygen fugacities of the ore system and adjacent wall rocks. As shown by the experiments and thermodynamic calculations of Spry and Scott (1986a), the buffering capacity of  $fS_2$  will fix the Zn:Fe ratio of gahnite to the range shown for most massive sulphide deposits. However, the buffering capacity of the gahnite-bearing rock will not be reached if the amount of sphalerite, pyrite, and pyrrhotite is less than 1 volume %. For these rocks, which are usually aluminous metasediments, the Zn:Fe ratio markedly decreases. Since the Mg component of silicates does not undergo sulphidation reactions, a magnesium-rich bulk composition will simply cause the gahnite to have a high spinel sensu stricto component. An increase in  $fS_2$  and  $fO_2$  with proximity to sulphides likely accounts for the increase in the Zn/Fe ratio of gahnite (in otherwise homogeneous country rocks) with proximity to the metamorphosed Zn-rich deposits at Cotopaxi, Colorado and Montauban, Quebec (Ririe & Foster 1984; Bernier *et al.* 1987). In the Broken Hill area,

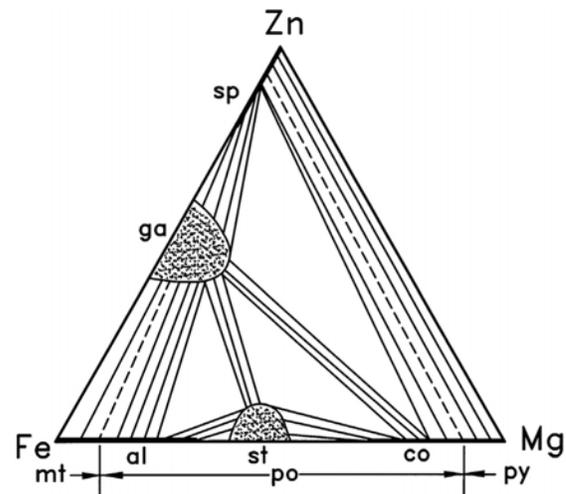


Fig. 4. Phase relations among gahnite, staurolite, and aluminous silicates shown in an Fe-Zn-Mg ternary diagram for the system Zn-Fe-Mg-Al-Si-S-O-H in the alteration zone of a volcanogenic massive sulphide deposit metamorphosed to the upper amphibolite facies (after Spry 2000).

Australia, the composition of gahnite in the Broken Hill deposit can be distinguished from gahnite in small BHT deposits (Fig. 5), because, in the latter occurrences, either the volume of sphalerite and pyrrhotite are insufficient to buffer the composition of gahnite or gahnite formed under low  $fS_2$  conditions. It should be noted that gahnite from the Mutooroo area in the southwestern part of the Proterozoic Willyama Domain, which hosts the Broken Hill deposit, contains gahnite that is essentially indistinguishable in composition to those from Broken Hill. This observation highlights the potential for significant BHT mineralization in the Mutooroo area (Fig. 6). The composition of gahnite may also be affected by an increase in  $fH_2O$ , especially during retrograde metamorphism,

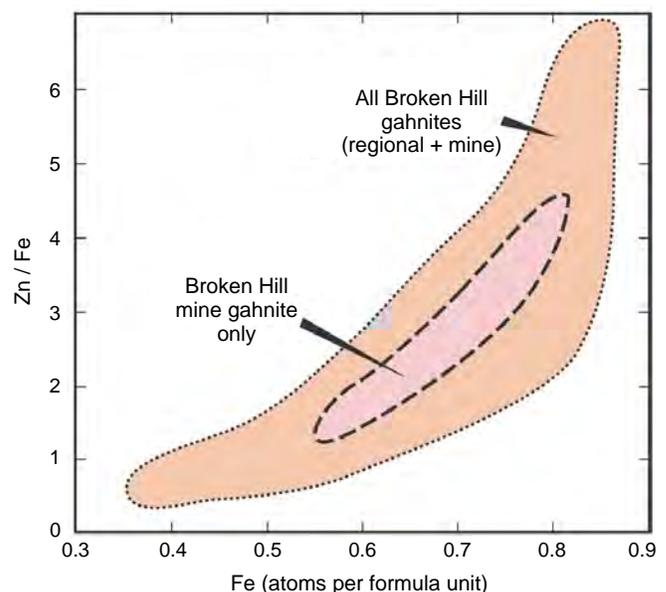
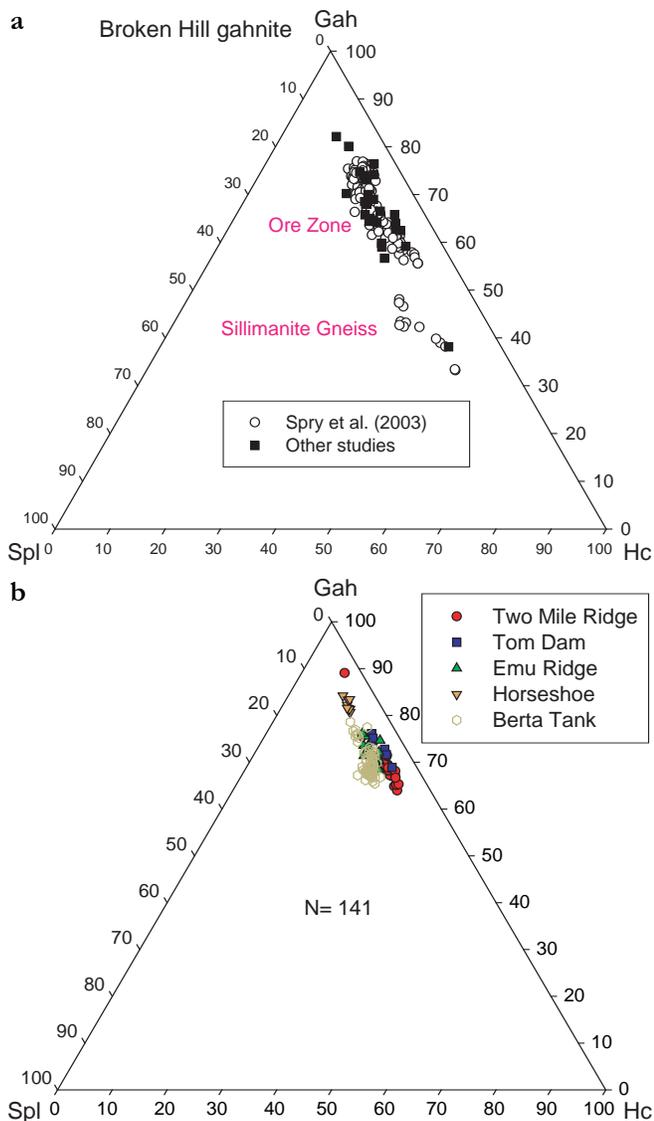


Fig. 5. Composition of gahnite (Fe vs. Zn/Fe) from the Broken Hill deposit along with gahnite from the Broken Hill area. Note the more restricted composition of gahnite from ore samples, which coexists with and is buffered by sphalerite and pyrrhotite.



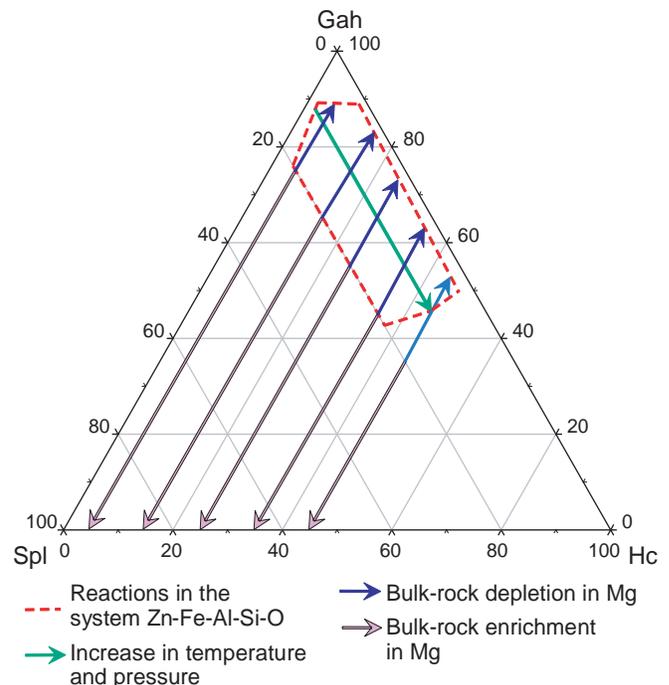
**Fig. 6.** Ternary plots of gahnite compositions from (a) the Broken Hill lode (ore zone and sillimanite gneiss adjacent to the deposit) and (b) minor Broken Hill-type deposits in the Mutooroo area, South Australia (Two Mile Ridge, Tom Dam, Emu Ridge, Horseshoe, and Berta Tank prospects) (after Spry *et al.* 2003).

as indicated also from the Mutooroo area, since gahnite can easily convert to zincian staurolite or muscovite (Fig. 2f).

Temperature, and to a lesser extent, pressure, also have a major effect on the composition of gahnite. Zincian spinels in greenschist-facies rocks (e.g. Venn Stavelot massif) have among the highest proportion of the gahnite molecule yet reported in the literature. Whereas gahnite from the Broken Hill deposit, Australia, which has been metamorphosed to granulite facies, have among the lowest Zn:Fe ratios of gahnite yet reported for any metamorphosed massive sulphide deposits (Fig. 1). The effects of bulk rock composition,  $fS_2$ - $fO_2$ , temperature, and pressure are shown schematically in Figure 7.

## DISCUSSION AND CONCLUSIONS

Gahnite occurs in metamorphosed massive sulphide deposits and sulphur-poor rocks in Mg-Ca-Al alteration zones, metamorphosed massive sulphide deposits in Fe-Al metasedimentary and metavolcanic rocks, marble, skarn, quartz veins,



**Fig. 7.** Schematic ternary plot showing the gahnite, hercynite, and spinel content of zincian spinels in metamorphosed massive sulphide deposits. Zincian spinels in metamorphosed massive sulphide deposits in Fe-Al-rich rocks typically form an area containing  $10 \pm 5$  mole %  $MgAl_2O_4$  and between 50 and 90 mole %  $ZnAl_2O_4$  (dashed line). The Mg content of the spinel is low due to the low Mg to Fe ratio of the ore and host rocks, whereas the Zn to Fe content of the zincian spinel is buffered by  $fS_2$ , which is dictated by the composition of sulphides in the Zn-Fe-S system. The Zn:Fe ratio of the zincian spinel will decrease with increasing temperature and to a lesser extent pressure. In Mg-rich rocks, zincian spinel composition shifts towards the Mg apex. These competing physical and chemical parameters allow for a complete range of compositions of spinels in metamorphosed massive sulphide deposits from  $MgAl_2O_4$ -rich to  $MgAl_2O_4$ -poor and  $ZnAl_2O_4$  contents  $>50$  mole% (after Heimann *et al.* 2005).

metabauxite, granitic pegmatite, contact metamorphic aureoles, granite, unaltered and hydrothermally altered Fe-Al-rich metasedimentary rocks, Al-rich granulite, and iron formation. The composition of gahnite in such settings virtually covers the entire range of compositions when plotted in a ternary diagram of Zn-Fe-Mg, but is restricted in range for individual geological settings.

Morris *et al.* (1997) measured the composition of gahnite in surficial sediments and known consolidated rocks in northern Ontario. The approach they took for using gahnite as an exploration guide in unconsolidated sediments is recommended, when used in conjunction with the ternary plot shown in Figure 1. Using the compositions of gahnite of Morris *et al.* (1997) along with the compositional fields for gahnite derived by Heimann *et al.* (2005), suggests that their group 2 spinels are spatially associated with metamorphosed massive sulphide deposits.

Figure 1 relates the composition of gahnite to a known geological setting, but it should be emphasized that the variation of gahnite within a given field is affected by pressure-temperature and  $fS_2$ - $fO_2$  conditions, and the bulk rock chemistries of the source rocks. If these parameters are known, Figure 1 should then be used in conjunction with Figure 7.

Regardless of whether or not the composition of gahnite is derived from rocks in situ or from unconsolidated sediments, the potential presence of metamorphosed massive sulphides is maximized if the gahnite analyzed has a composition that falls in the range of gahnite<sub>45-90</sub>hercynite<sub>0-45</sub>spinel<sub>0-25</sub> (excluding those in Mg-rich rocks). However, for terranes that are known to contain metamorphosed massive sulphide deposits with Mg-Ca-Al alteration zones, gahnite within the compositional range gahnite<sub>0-65</sub>hercynite<sub>0-50</sub>spinel<sub>25-90</sub> will be excellent guides to ore. During the collection of gahnite from unconsolidated sediments, other potential Zn-bearing resistate minerals, such as zincian staurolite, ecandrewsite-zincian ilmenite, and zincian h ogbomite (e.g. Spry & Scott 1986b; Spry & Petersen 1989; Plimer 1990) should also be considered as exploration guides in the search for metamorphosed massive sulphide deposits.

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# Quality control in indicator mineral methods

Mary Doherty

*ALS Chemex, 994 Glendale Unit #3, Sparks, Nevada 89431*

*(e-mail: Mary.Doherty@alschemex.com)*

## ABSTRACT

A discussion of quality control in indicator mineral analyses requires consideration of the value of quality, the sources of error, the methods for the measurement of error, and systems to minimize and control error. In addition, practitioners require awareness of reporting requirements and obligations related to Canada's National Instrument 43-101 (NI 43-101), as well as International Organization for Standardization (ISO) standards, including ISO 9001 and ISO 17025. At the outset, it is recognized that corporate wealth is directly tied to quality. As outlined by Pitard (2005), a sound management strategy that recognizes the value of quality will realize financial benefits, add stakeholder value, and improve market perception. This strategy needs to be founded on three pillars. Firstly, the causes of problems need to be linked to proactive management. Secondly, a strong commitment to good sampling and good laboratory practices is essential. Thirdly, practitioners require the capability to understand variability and must be able to perform reliable statistical studies.

Definition of the sources of error requires must take into account field variability, field collection sampling error, sample preparation error, mineral identification error, and analytical error. Indicator mineral sampling is best guided by broader principles that were developed to guide sampling in general, particularly those outlined by Gy (Gy 1992, 1995, 1998; Pitard 1993; Minnitt *et al.* 2007). Fundamental to an assessment of error is the initial recognition of the character of field variability, and thus the scale of sample heterogeneity. Consideration of indicator mineral sample processing error requires consideration of the various steps, including sieving, density preconcentration and concentration, magnetic separation, and mineral picking. Sample processing is complicated by mineral chemistry variations, as these variations may cause differences in the magnetic susceptibility and density. In addition, loss of sample, sample mix ups, grain losses, contamination, carry-over, and splitting errors must all be taken into account.

Taking a representative split from a large sample is a challenging task. It is important to split the entire sample, to split it evenly, to avoid density separation, to avoid the loss of fines, and to avoid size segregation. According to Allen & Kahn (1970), error in the cone-and-quarter method is 13.6%, for scoop sampling the error is 10.3%, in riffle splitting it is 2%, and with a rotary splitter, the error is 0.25%. Error related to riffle splitting maybe as good if the operator takes great care, but error increases for larger samples requiring multiple splits.

To measure and monitor quality control, field duplicates may be required, usually insertion rate of approximately 5%. Blank samples may be added to detect contamination, carry-over, and switching, using material such as ceramic-grade coarse sand. Blanks should be inserted as the first sample in a batch, to detect carry-over from previous sample batches.

Standards may also be developed, using a set of samples taken from a known field site. In addition, spiked samples may

be prepared with a known number of distinct grains, such as uniquely coloured diamonds or laser-etched grains. Appropriate reference materials are also required to support elemental analysis in mineral chemistry, for example, to recognize baseline shifts and instrumental drift in electron microprobe and laser ablation analyses. Repeat analyses will support assessment of precision, i.e., the ability to repeat analyses with consistent results, while trusted reference materials are required to support assessment of accuracy, which is the ability to produce what is regarded as the true value. In mineral grain elemental analysis by electron microprobe and laser ablation, one can insert known grains, randomize the order of grain analysis, or insert duplicate grains. One can also conduct batch repeats or send duplicate samples to an alternate lab.

National Instrument 43-101 (NI 43-101) is a Canadian regulation governing disclosure of technical information by exploration/mining companies listed on Canadian stock exchanges and has been widely adopted as a general guideline of best practices by international companies. The regulation stipulates that all technical information must be verified by a "Qualified Person", and requires regular reporting of all relevant technical information according to industry-standard guidelines. The qualified person is required to validate all technical information reported and to set up and maintain a quality control program on projects, including aspects of indicator mineral surveys related to sampling, sample security, sample preparation, analysis, and testing. The regulation recommends the use of accredited laboratories and industry standard practices. NI 43-101 was created to help re-instill confidence in companies overseen by the Canadian Securities Authority after the Bre-X scandal of 1997.

ISO 9001:2000 is a set of international quality standards developed by the International Organization of Standards in Geneva, Switzerland. This organization has a membership of standards groups representing 110 countries, with the American National Standards Institute (ANSI) being the United States representative. Over 140 countries recognize the standards and more than 8,500 companies are certified as meeting these standards in the United States. Certification under ISO 9001:2000 requires evidence of a quality management system covering all aspects of an organization. It reviews what the organization does to fulfill the customer's quality requirements and applicable regulatory requirements, and to achieve continual improvement of its performance in pursuit of these objectives. Registration requires an audit by an external agency accredited by ISO and requires annual renewal. ISO 9001:2000 is a model/standard that lists requirements for a system to manage quality assurance, although it is not a strict set of rules, it is a series of common sense guidelines consisting of a Level I Quality Manual, Level II Procedures, Level III Instructions, and Level IV Records and Documents. The Quality manual contains a series of policy statements for each of the elements of the ISO 9000 quality standard. Procedures

define whom, what, and where, as well as outlining responsibilities and actions. Work instructions define specific steps to accomplish tasks and show how to use of the system. The key to ISO 9001:2000 is to “say what you do, and do what you say”.

To effectively monitor quality control, it is recommended that project geologists visit the lab(s) that will perform the heavy mineral concentrating, picking, and mineral chemistry analysis, in order to review the project needs with lab personnel, define potential sources of error, and determine what the lab is doing to monitor and control the quality of the sample processing and/or analyses. It is recommended that 10% of all samples submitted should be quality control samples in both the processing and grain analytical batches. Field duplicates, used to measure site variability, are recommended to be inserted in every 20 to 30 samples. Blanks, which are used to monitor for carry-over and contamination, analytical control samples, which are designed to assess accuracy, and lab replicates, used to measure precision, should each be inserted at a frequency of 1 in 20 samples. Results can then be confirmed with follow-up sampling, consisting of up to an additional 5% to the sample count, while the number of replicate samples

sent to a second lab should be approximately 10% of the total number of samples.

The goal in any indicator mineral survey or study is a well designed quality control system that will recognize error, and measure and minimize error, while recognizing that the wealth of a Corporation is dependent upon the quality of the exploration and reserve data.

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# Gold and platinum group element indicator mineral methods in mineral exploration

M. Beth McClenaghan

*Geological Survey of Canada, 601 Booth Street, Ottawa, Ontario, K1A 0E8*

*(e-mail: bmcclena@nrcan.gc.ca)*

## INTRODUCTION

Indicator minerals of Au and platinum group element (PGE) deposits include gold and platinum-group minerals (PGM), as well as associated metallic, oxide, and silicate minerals. The greatest advantages of precious metal indicator mineral methods over traditional geochemical analysis of sediment samples are that ore grains are visible and can be examined, and that the method is sufficiently sensitive to detect only a few grains, equivalent to ppb-level indicator mineral abundances, even in regions where regional rocks dilute concentrates with non-indicator heavy minerals (Brundin & Bergstrom 1977; Averill 2001). This paper reviews indicator mineral methods applied to surface sediment sampling in Au and PGE exploration.

## METHODS

Indicator mineral abundance in the sediments will depend on the primary indicator mineral content of the source rock, degree of post-emplacment/-formation weathering of the indicator mineral source, and dispersion/dispersal transport mechanisms (i.e. fluvial, glacial, aeolian). All of these factors must be considered when planning sampling strategies (i.e. sample medium, size, and spacing) (Benn 2009). The choice of sample media will depend on the climate, topography, and size of area to be sampled. Typically, at least 10 to 20 kg (~5 to 10 litres) of sediment are collected for heavy mineral surveys.

Recovery of precious metal indicator minerals is carried out using a combination of sizing and gravity techniques, and some of the more common methods available are described by McClenaghan (2009). Processing methods for precious metals are two-phased to allow for the recovery of indicator minerals from two size fractions. Most gold, PGM, and associated sulphide minerals are silt-sized and are commonly recovered by panning. Coarser sand-sized gold, PGM, silicate, and oxide indicator minerals can be recovered using a table, jig, dense media separator (DMS), or spiral concentrator. Once recovered, gold, PGM, or other related indicator minerals may then be examined and analyzed using microanalytical techniques, including scanning electron microscope (SEM), electron microprobe (EMP) (e.g. Leake *et al.* 1991; Knight *et al.* 1994; Loen 1994; Wierchowiec 2002; Shcheka *et al.* 2004b; Barkov *et al.* 2005), proton-induced X-ray emission (PIXE) (e.g. Shcheka *et al.* 2004b), secondary ion mass spectrometer (SIMS) (e.g. Hattori & Cabri 1992), or laser ablation ICP-MS (LA ICP-MS) (e.g. Watling *et al.* 1994; McCandless *et al.* 1997; Mortensen *et al.* 2004).

## GOLD INDICATOR MINERALS

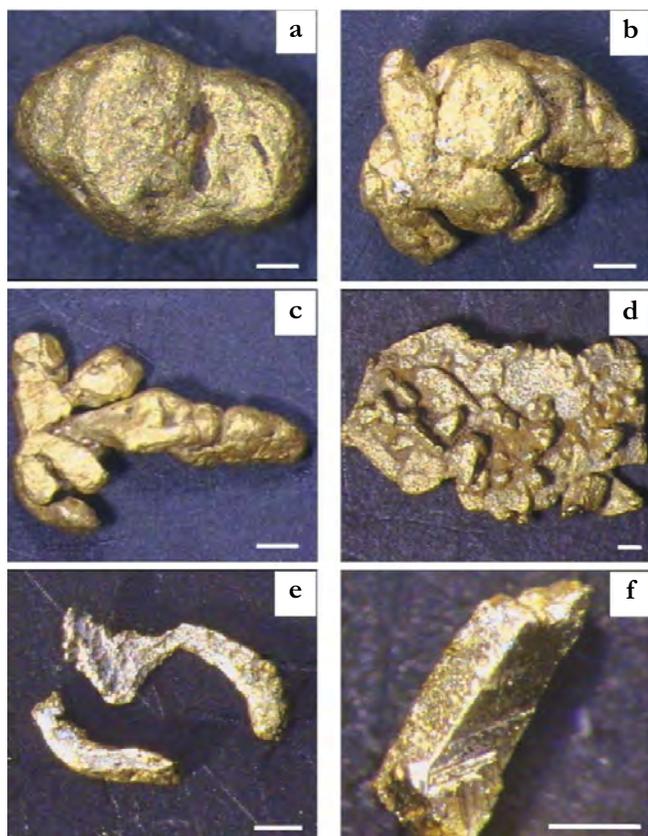
Gold grains are the best indicator mineral for detecting the presence of gold deposits, although sulphides (pyrite, pyrrhotite, arsenopyrite, chalcopyrite, sphalerite, galena, pyrrhotite, proussite), platinumoids, tellurides, scheelite, and rutile, as well as secondary minerals (jarosite, limonite, goethite, and pyrolusite) may also be useful in specific regions (e.g. Boyle & Gleeson 1972; Gleeson & Boyle 1980; Barakso & Tegart 1982;

Knight *et al.* 1994; Scott & Radford 2007). Till and stream sediments are the most effective media for using gold grain methods, which include documenting gold grain abundance, size, shape, and fineness (e.g. Giusti 1986; Grant *et al.* 1991). The optimal size range of grains that are most easily recovered from these media is 0.010 to 2.0 mm. In glaciated terrain, till is a much more effective sampling medium than stream sediments because most gold grains are silt sized (0.002-0.063 mm) and such fine grains are expelled rather than concentrated during stream sedimentation (Averill 2001). Although gold grains have been panned from stream sediments since pre-Roman times, the systematic use of gold grain abundance data and grain characteristics to find their source has only been applied in the past 35 years.

Much of what has been published on gold grain characteristics relates to size, shape, and chemical composition. Many studies have been published about exploration for lode and placer deposits using different sample media in a variety of terrains, including 1) till sampling in glaciated terrain (e.g. MacEachern & Stea 1985; Sopuck *et al.* 1986; Pronk & Burton 1988; Sibbick & Fletcher 1993; Plouffe 2001); 2) stream sediment sampling in various terrains (e.g. Giusti 1986; Youngson & Craw 1999; Chapman & Mortenson 2006); 3) exploration for, and sampling of, Au placer deposits (e.g. Knight *et al.* 1994, 1999a,b; Márquez-Zavalía *et al.* 2004); and, 4) lateritic terrain (e.g. Freyssinet *et al.* 1989; Grant *et al.* 1991; Porto & Hale 1996; Freyssinet & Butt 1998; Gedeon & Butt 1998; Larizzatti *et al.* 2008).

## Gold grain condition

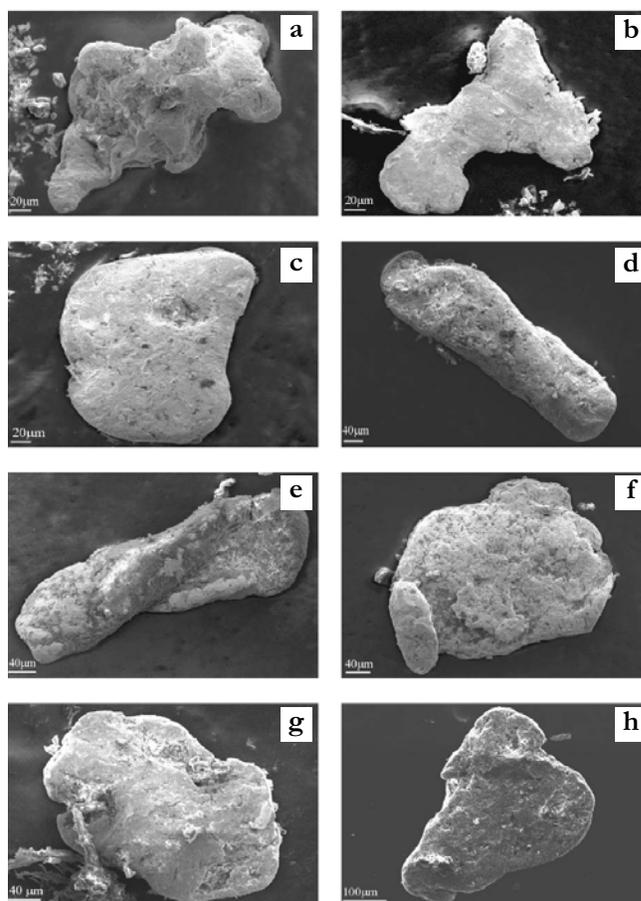
Because gold is malleable, gold grains will change shape as they are transported or as they are subjected to chemical weathering in situ during laterite formation. In general, the degree of rounding, polishing, and bending of the gold grains provides information about transport distance in streams (e.g. Halbauer & Utter 1977; Knight *et al.* 1999b; Youngson 1998; Townley *et al.* 2003; Márquez-Zavalía *et al.* 2004) and glaciers (e.g. Averill 1988; Hérail *et al.* 1989; DiLabio 1990; Nikkarinen 1991; Kinnunen 1996). The shapes of gold grains from streams are usually described in terms of their flatness, roundness, folding, and surface texture (Figs. 1 and 2) and these characteristics can be used to estimate the distance of transport (e.g. Knight & McTaggart 1986; Loen 1994, 1995; Youngson & Craw 1999; Wierchowiec 2002). Hammering and abrasion processes control grain shape and increase the roundness. Particle rounding results mainly from abrasion of particle edges and in folding of delicate protrusions. The Flatness Index,  $FI = a+b/2c$  ( $a$  = long axis,  $b$  = intermediate axis,  $c$  = short axis dimensions of a grain) is one shape factor used to characterize and compare gold grains (e.g. Callieux 1945; Wierchowiec 2002). Grain flattening, and hence Flatness Index, gradually increase with increasing transport distance downstream. For example,  $FI = 2$  is typical of bedrock deposits or gold grains very close to source, whereas  $FI = 45$  reflects grains that have been transported sev-



**Fig. 1.** Typical morphologies of placer gold grains recovered from the San Luis Range, Argentina. **a)** Rounded gold grain. **b)** Folded leaf of gold, giving the appearance of a rounded gold grain. **c)** Slightly rounded arborescent masses of branching distorted octahedra of gold. **d)** Leaf of gold with overgrowth of small flattened, distorted octahedral of gold. **e)** Wire gold. **f)** Gold crystal. From Márquez-Zavalía *et al.* (2004). Scale bar 300  $\mu\text{m}$ .

eral 10s km (e.g. Hérial *et al.* 1989; Loen 1995; Wierchowicz 2002). Knight *et al.* (1999a,b) noted that both gold grain roundness and flatness increase rapidly within the first 3 km from the source. After approximately 5 km, the flatness continues to increase slowly whereas roundness remains essentially unchanged. Roundness is a more sensitive estimator for distances less than 5 km and less reliable than flatness for distances greater than 5 to 10 km. Gold grains in bedrock lateritic terrain change shape due to progressive chemical weathering over time, from primary grains that have irregular, dendritic, and prismatic forms with sharp edges to grains with rounded edges and corrosion pits (e.g. Larizzatti *et al.* 2008).

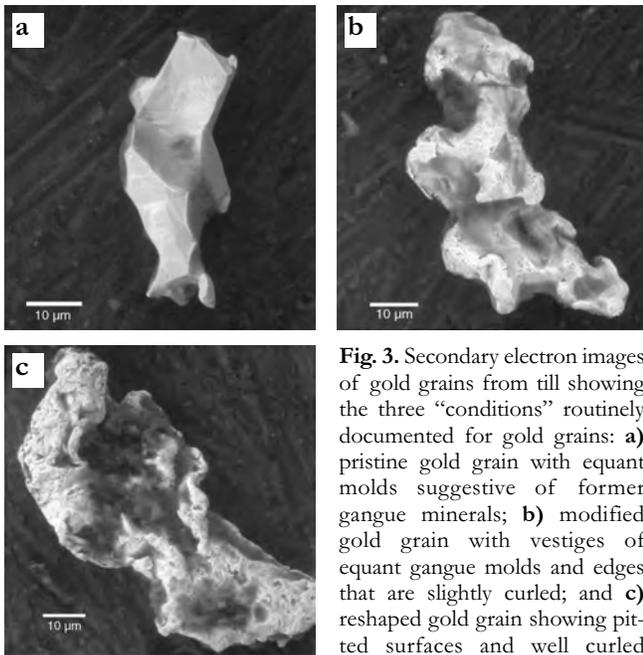
The graphically descriptive classification scheme (pristine-modified-reshaped) of DiLabio (1990) for describing conditions and surface textures of gold grains builds on Averill's (1988) descriptions of gold grain shape related to glacial transport distance. Although Averill's and DiLabio's schemes were initially designed for gold grains recovered from till, they can also be applied to gold from stream sediments. Their scheme is described in detail here as it is widely used for till in the glaciated terrain of Canada. The progression from pristine to reshaped grains represents increasing distance of glacial transport. However, caution should be used when utilizing gold grain condition as an indication of transport distance because gold grain morphology can be quite variable in the bedrock



**Fig. 2.** Secondary electron images of typical fluvial gold particles from placers in the East Sudetic Foreland, Poland. **a)** Irregular grain with angular appendages and locally rounded rims. **b)** Branched particle showing evidence of abrasion and rounding. **c)** Elliptical particle with straight edges (fold hinges). **d)** Rod-shaped particle produced by folding of discoid. **e)** Rod-like particle that has been twisted about its a axis. Note freshly deposited ferric oxides and clay minerals coating the surface of gold grain. **f)** Typical platy particle, it has been folded during fluvial transport. Note fold hinges and pitting on the grain surface. **g)** 'Sandwich-like' particle, showing remnants of curved sutures where the pre-fold particle edge has been hammered into the face of the a-b plane. **h)** 'Envelope-like' particle with marks of intense deformation. From Wierchowicz (2002).

source, and gold grains can be released from mineralized bedrock fragments at any distance during transport or during subsequent post-depositional weathering.

Pristine gold grains (Fig. 3a) retain primary shapes and surface textures and appear not to have been damaged in transport. They occur as angular wires, rods, and delicate leaves that once infilled fractures, as crystals with grain molds, and as inclusions in sulphides. The transport history of pristine grains may be interpreted in two ways: 1) gold grains were eroded from a bedrock source nearby and transported to the site with little or no surface modification, thus the transport distance is generally short; and 2) gold grains were liberated from rock fragments during in situ weathering of transported grains containing gold. Modified gold grains (Fig. 3b) retain some primary surface textures but all edges and protrusions have been damaged during transport and they are commonly striated. Irregular edges and protrusions are crumpled, folded, and curled. Grain molds and primary surface textures are preserved



**Fig. 3.** Secondary electron images of gold grains from till showing the three “conditions” routinely documented for gold grains: **a)** pristine gold grain with equant molds suggestive of former gangue minerals; **b)** modified gold grain with vestiges of equant gangue molds and edges that are slightly curled; and **c)** reshaped gold grain showing pitted surfaces and well curled edges (from McClenaghan 2001).

only on protected faces of grains. Samples that contain elevated concentrations of modified grains are generally proximal to the bedrock source. Reshaped gold grains (Fig. 3c) have undergone sufficient glacial transport that all primary surface textures have been destroyed and the original grain shape is no longer discernible. Reshaped grains are flattened to rounded, resulting from repeated folding of leaves, wires, and rods. Grain surfaces may be pitted from impact marks from other grains but, in most cases, surfaces are not leached of silver in glaciated terrain. Although these grains can have a complex transport history, the presence of large numbers of reshaped grains in discrete areas may be significant. Most background gold grains have a reshaped morphology (Averill 1988).

#### Gold grain composition

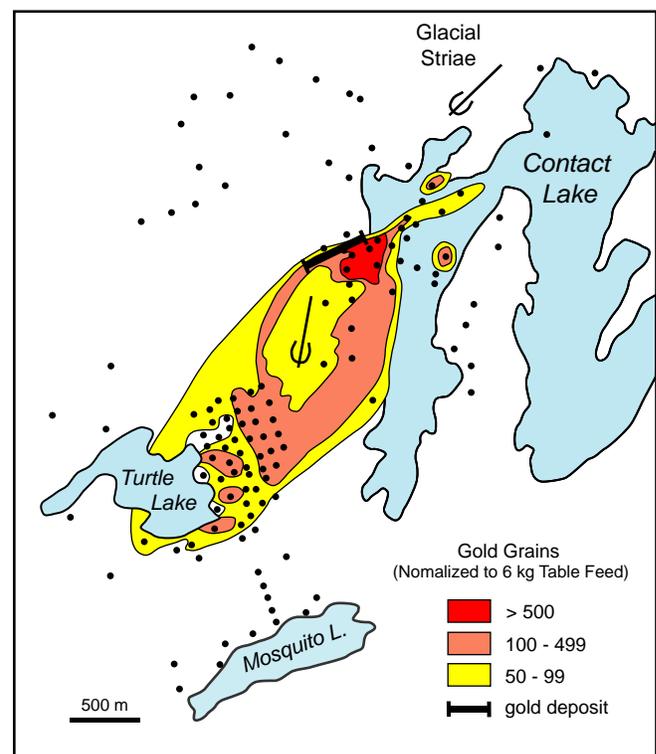
Gold grain core compositional studies are carried out to identify different populations and compare their compositions to potential bedrock sources (Knight & McTaggart 1986). Grain compositions are most commonly characterized using EMP analysis for Au, Ag, Cu, Hg, Bi, and Te. Limitations on applying these methods include the facts that large numbers of grains should be analyzed to characterize the compositional range and that gold from different deposit types does not necessarily have unique trace element signatures (Mortensen *et al.* 2004). Large volumes of gold grain compositional data have been published for various types of lode and placer deposits worldwide (see references listed in this paper). Trace element analysis by gold grains by LA ICP-MS for elements such as Mo, Bi, Te, Sb, and Sn (e.g. Watling *et al.* 1994; McCandless *et al.* 1997; Mortensen *et al.* 2004) allows for more specific fingerprinting of groups of gold grains or deposit types. Placer gold grains frequently exhibit Au-enriched, Ag-depleted rims that are typically 1 to 20 µm thick and surround the grain core. These rims may form by the leaching of Ag or by overgrowth of higher fineness gold subsequent to deposition (e.g. Groen *et al.* 1990; Wierchowicz 2002).

The study of micro-inclusions in gold grains can provide information on the ore and gangue minerals present in the

bedrock source and thus provide a signature of the deposit type or even a specific gold deposit (e.g. Leake *et al.* 1998; Chapman *et al.* 2000, 2002; Mortensen *et al.* 2004). For example, enargite inclusions may be indicative of a high-sulphidation epithermal source, while argentite may be indicative of mesothermal gold (Mortensen *et al.* 2004). In addition to inclusion composition, the relative proportion of specific mineral classes (e.g. tellurides, sulphides, sulpharsenides, or sulphosalts) in gold grains can be useful for identifying different grain populations and/or sources. Inclusion studies, however, are limited by the fact that their abundance may decrease due to post-depositional alteration/weathering in the surficial environment and due to physical grain degradation with increasing transport downstream (Loen 1995; Mortensen *et al.* 2004).

#### Examples

In glaciated terrain, the ability to recover silt- to sand-sized gold grains in till has led to much exploration in the past 30 years (e.g. Huhta 1988, 1993; Bernier & Weber 1989; Héral *et al.* 1989; Nikkarinen 1991; Saarnisto *et al.* 1991; McClenaghan *et al.* 1998; McMartin 2007) and the discovery of gold deposits (e.g. Sopuck *et al.* 1986; Sauerbrei *et al.* 1987). One of the earliest published examples of utilizing gold grains in till to explore for a lode source is from 100 years ago (Prest 1911; Stea & Finck 2001). In northern Saskatchewan, Canada, till sampling to recover gold grains was used to explore the area around a lake sediment gold anomaly, and led to discovery of the Bakos gold deposit. In this area, gold grains in till defined a well developed 2 km long by 0.5 km wide dispersal train southwest (down-ice) of the deposit (Fig. 4). Gold grain content in 6 kg till samples varied from background concentrations of zero



**Fig. 4.** Abundance of gold grains in 6 kg till samples around the Bakos gold deposit in northern Saskatchewan, Canada (modified from Chapman *et al.* 1990) showing a well developed dispersal train extending 2.8 km southwest of the deposit.

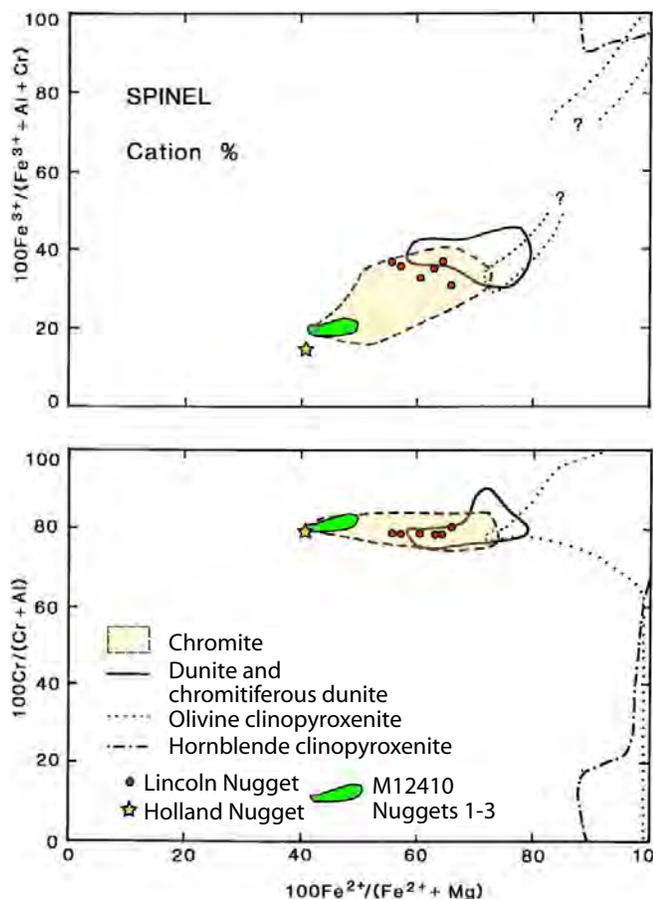


Fig. 5. Plots comparing the compositional range of chromiferous spinel in placer nuggets ( $n=32$ ) to spinel compositions ( $n=264$  analyses) in the major rock types of the Tulameen complex, British Columbia, Canada (from Nixon *et al.* 1990).

grains to the highest value of 2751 grains (Chapman *et al.* 1990). At Waddy Lake, also in Saskatchewan, gold grain abundance in combination with native copper, galena, chalcocite, and pyromorphite grains in unweathered till were used to detect glacial dispersal and ultimately discover the E-P Zone (Averill & Zimmerman 1986). Gold deposits in the world-class Timmins gold camp of the Abitibi Greenstone Belt, Canada display well developed gold grain signatures in till characterized by 100s to 1000s of pristine silt-sized gold grains per 10 kg till sample (McClenaghan 2001).

Smith *et al.* (1993) examined gold grains in surface soils in the semi-arid to arid Hatu mining district of China, where the shape, trend, and distribution of soil gold anomalies, with respect to known lode deposits, suggested they may have formed by eolian dispersion. The most prominent soil anomaly was 30 km long and 5 km wide. Soil samples weighing 6 and 18 kg were sieved and panned to recover gold grains, which varied in abundance from zero to 378 grains, depending on distance from source. Using gold grain abundance, morphology, size, and compositional features, combined with the grains' areal distribution, they concluded that the major component of the gold grains in the soil anomalies were likely of fluvial origin, not eolian.

In Wayamaga and Cokioco areas of French Guiana, gold grain size and degree of physical wear in stream sediments reflects proximity to bedrock mineralization (Kelley *et al.* 2003).

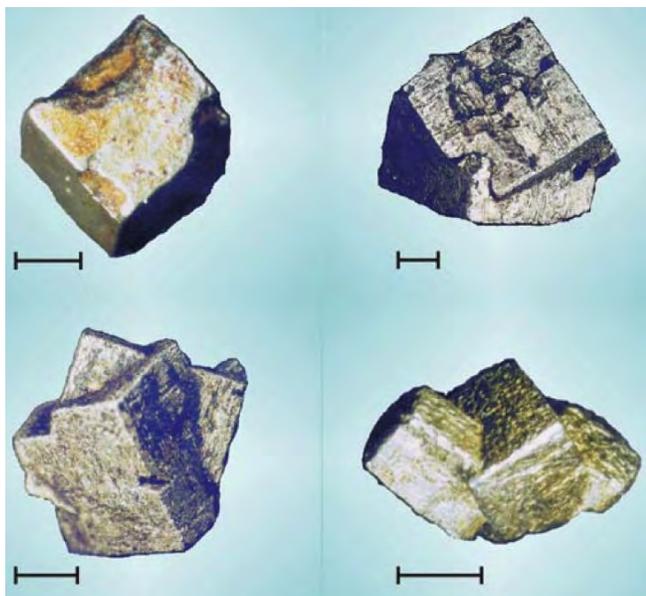
Gold grains at Cokioco display complete reshaping of all grains, coarser average grain size (125-200  $\mu\text{m}$ ), total leaching of any alloyed silver in cores and rims of grains, absence of unstable mineral inclusions, and possible presence of supergene gold in aluminosilicate inclusions. These characteristics suggest placer gold concentration but lack input from a proximal or preserved lode gold source and none is known in the area. In contrast, at Wayamaga the incomplete reshaping of some grains, finer average grain (50-125  $\mu\text{m}$ ), incomplete leaching of silver with average inner fineness of 953, and the presence of unstable mineral inclusions suggest gold was shed from a proximal lode source. The Wayamaga area has a significant upstream bedrock lode source defined (Kelley *et al.* 2003).

#### PLATINUM GROUP ELEMENT INDICATOR MINERALS

Similar to gold, platinum group minerals (PGMs) can be the best indicators of their own deposits. Initially, PGMs were indicators of Au and PGE placer deposits (e.g. Nixon *et al.* 1990; Podlipsky *et al.* 2007) and until the early 20<sup>th</sup> century, placer deposits were the principal source of PGEs (Cabri *et al.* 1996). Today, they are indicators of both placer and lode PGE deposits. Other minerals that indicate the presence of lode PGE mineralization include oxide and silicate minerals that reflect the mafic to ultramafic host rocks, such as enstatite, forsterite, low-Cr diopside, chromite, and minerals that reflect mineralization such as PGM, gold, and Fe and Ni sulphides, arsenides, and antimonides (Averill 2007a,b, 2009). Fe and Ni sulphides and Pt and Pd sulphides and tellurides are highly unstable in oxidized surficial sediments and thus are not often present in surficial sediments in large enough numbers to be useful as PGE indicator minerals. Chalcopyrite and Pt, Pd, and FeNi arsenides and antimonides are sufficiently stable to be used as Ni-Cu-PGE indicator minerals (Averill 2007a,b, 2009).

#### Platinum group mineral grain composition

Despite their more than 300 year history of recovery, only in past 40 years have PGM grain compositions been characterized and documented. Platinum group minerals, especially Pt-Fe alloys, usually contain inclusions of other PGMs, as well as silicates, spinels, and base metal sulphides, and may be intergrown with other minerals. Platinum group mineral chemistry, as well as that of the inclusions and intergrowths, can be used to characterize the genetic history of the grains and their source rocks, and provide a fingerprint by which grain populations can be identified and compared (e.g. Slansky *et al.* 1991; Auge & Legendre 1992; Cabri *et al.* 1996; Shcheka *et al.* 2004a,b; Podlipsky *et al.* 2007). For example, Pd-rich ferroan platinum nuggets may indicate derivation from a clinopyroxenite source, whereas Ir-rich Pt-Fe alloys may suggest a chromitite source (Malitch & Thalhammer 2002). In the Tulameen placer PGM district in southern British Columbia, Canada, Nixon *et al.* (1990) compared PGM chemistry as well as the compositions of chromite (Fig. 5) and olivine inclusions in placer PGM nuggets to those in various rocks in the Tulameen ultramafic complex. They demonstrated that the placer minerals were derived from chromitite in the dunitic core of the nearby complex. Os and S isotope ratios also may be useful for understanding the origin of PGM grains (e.g. Hattori *et al.* 1991; Hattori & Cabri 1992; Malitch & Thalhammer 2002; Hattori *et al.* 2004). PGM grain compositions are most com-



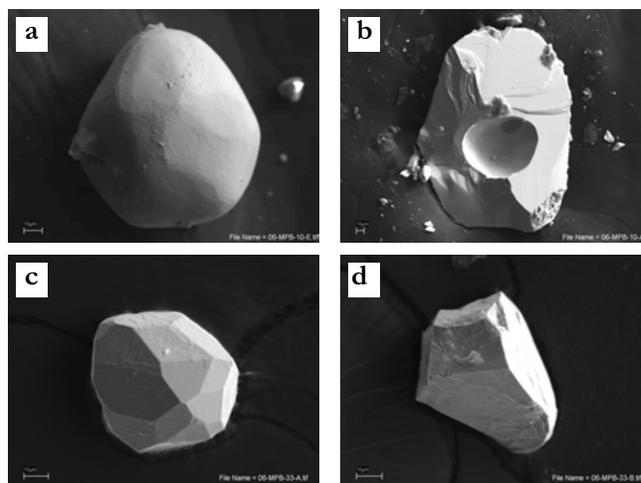
**Fig. 6.** Examples of coarse hexahedral-shaped Pt-Fe alloy crystals from the Kondyor PGE placer, Khabarovskiy Kray, eastern Siberia, Russia. Penetration twins and intergrowth aggregates of two or more crystals are common, such as shown here. Scale bar is 1 mm (from Shcheka *et al.* 2004b).

monly characterized using EMP analysis for Pt, Pd, Os, Ir, Ru, Rh, Fe, Cu, Ni, and S.

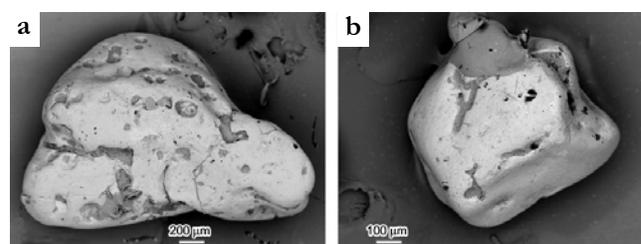
#### Platinum group mineral grain morphology

Platinum group mineral grain morphology and size in surficial sediments depends on the distance traveled from source, with the largest grains and best preserved crystal faces usually found closer to the source rocks (Cabri *et al.* 1996). In contrast to gold, no systematic classification scheme for characterizing PGM morphology has been developed. However, a wide variety of grain morphologies have been reported (e.g. Cabri *et al.* 1996; Podlipsky *et al.* 2007), including preserved crystal faces on PGMs from stream placers (Fig. 6) and till (Fig. 7), to rounded PGMs from placers in Russia (Fig. 8) and Colombia (Fig. 9).

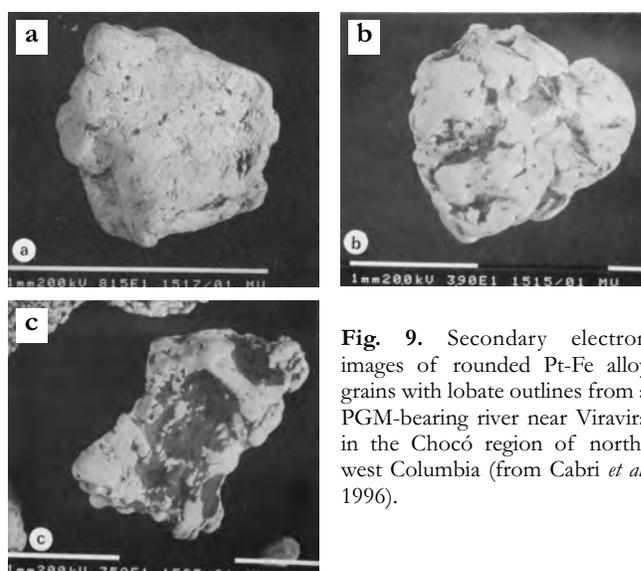
Numerous accounts of PGMs in placers around the world include deposits in Columbia, Brazil, Russia, Canada, Indonesia, and Madagascar (e.g. Cabri *et al.* 1996; Cassedanne & Alves 1992; Podlipsky *et al.* 2007). Much of what is known about PGM transport, morphology, and inclusions has been learned from studies of placer deposits and alluvial occurrences. Historically, reports of PGMs in glacial sediments (till) have been rare and, where found, usually included only a few silt-sized grains obtained from samples collected within 500 m of PGE mineralization (Cook & Fletcher 1992; Bajc & Hall 2000; Searcy 2001; Barnett & Dyer 2005; Kojonen *et al.* 2005). The presence of as few as two PGM grains in a till sample is significant as it likely indicates nearby (<500 m) proximity to PGE mineralization (Averill 2007a,b). More recent examples of PGM anomalies in till demonstrate the improving methods for recovery and recognition of PGMs in till. One example is the Broken Hammer Cu-(Ni)-PGE resource on the North Range of the Sudbury Structure, central Canada. In this deposit, a large chalcopyrite vein contains abundant PGMs (Fig. 10) and gold. Till immediately down-ice of the subcropping mineral-



**Fig. 7.** Secondary electron images of sperrylite grains from the Broken Hammer Cu-(Ni)-PGE resource. **a)** Pristine crystal with resorbed faces recovered from gossan sample 06-MPB-10. **b)** Broken angular grain with mold left by spherical inclusion; recovered from gossan sample 06-MPB-10. **c)** Pristine pyritohedron crystal from till sample 06-MPB-33, 150 m down-ice of the occurrence. **d)** Angular, broken grain in till sample 06-MPB-33.

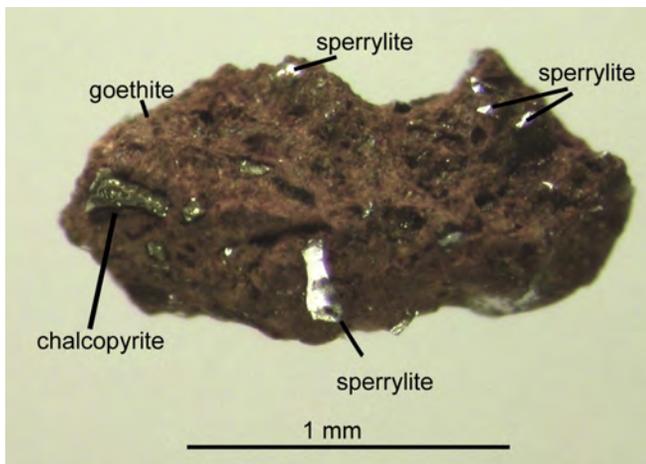


**Fig. 8.** Secondary electron images of rounded PGE mineral grains from the Simonovsky Brook gold placer, Salair Range, Russia (from Podlipsky *et al.* 2007).



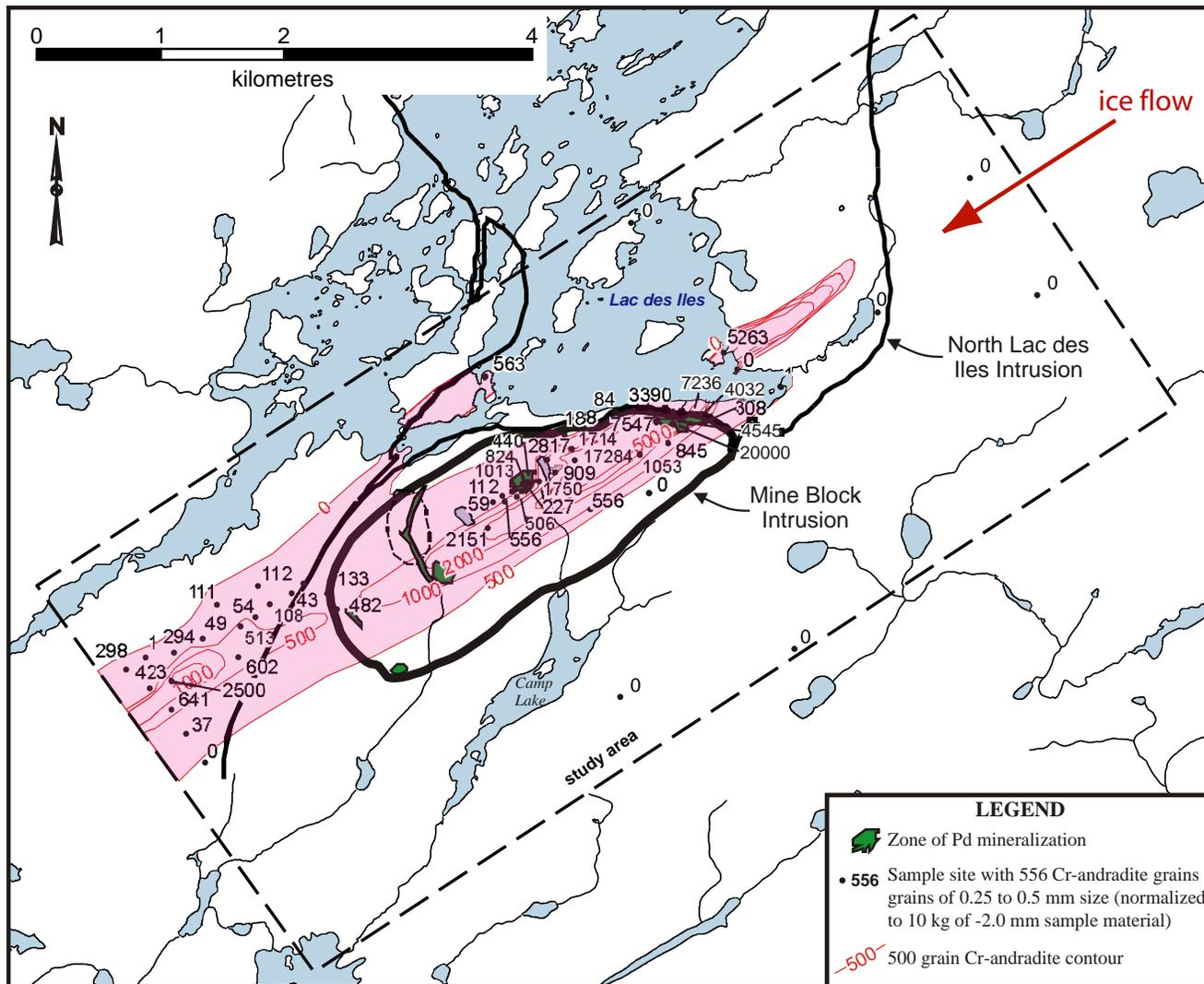
**Fig. 9.** Secondary electron images of rounded Pt-Fe alloy grains with lobate outlines from a PGM-bearing river near Viravira in the Chocó region of north-west Columbia (from Cabri *et al.* 1996).

ization contains up to 700 sperrylite grains/10 kg, as well as 10s to 100s of gold grains and 100s to 1000s of chalcopyrite grains (Ames *et al.* 2007). Sperrylite grains recovered from postglacial gossan developed on the subcropping surface of



**Fig. 10.** Small gossan fragment from the Broken Hammer Cu-(Ni)-PGE resource on the North Range of the Sudbury Structure, Ontario, Canada, showing sperrylite hosted in chalcopyrite-goethite (modified from Averill 2007a).

the deposit display both pristine intact crystals and angular fragments of crystals (Fig. 7a,b). Sperrylite grains recovered from till collected 150 m down-ice of mineralization display similar pristine crystals and angular fragments of crystals (Fig. 7c,d). The Ni-Cu deposits in the Thompson Nickel Belt (TNB), in central Canada, also have noteworthy PGM signatures in till. Up to 14 sperrylite grains/10 kg were recovered from till 250 m down-ice of the Pipe Ni-Cu deposit (McClenaghan *et al.* 2009). Peregrine Diamonds' 2007 discovery of sperrylite grains in till on Baffin Island in the Canadian Arctic is another example. Here a sperrylite anomaly in till occurs over a 10 km by 10 km area. Several samples outside of the anomalous area also contain sperrylite, including one sample containing 60 sperrylite grains (Peregrine Diamonds <http://www.pdiam.com/s/NorthAmerica.asp#baffin>). In northwestern Ontario, Canada, a single phase of ice flow to the southwest has eroded and transported debris from the Lac des Iles PGE deposits more than 5 km to the southwest. Only a few PGM grains (stillwaterite, sperrylite) were recovered from till down-ice of the deposit (Searcy 2001; Averill 2007a,b). Instead of using PGM grains, glacial dispersal is best



**Fig. 11.** Distribution of Cr-andradite in the 0.25 to 0.5 mm fraction of till normalized to a 10 kg sample weight down-ice of the Lac des Iles Pd mine area, northwestern Ontario, Canada (modified from Averill 2007a,b).

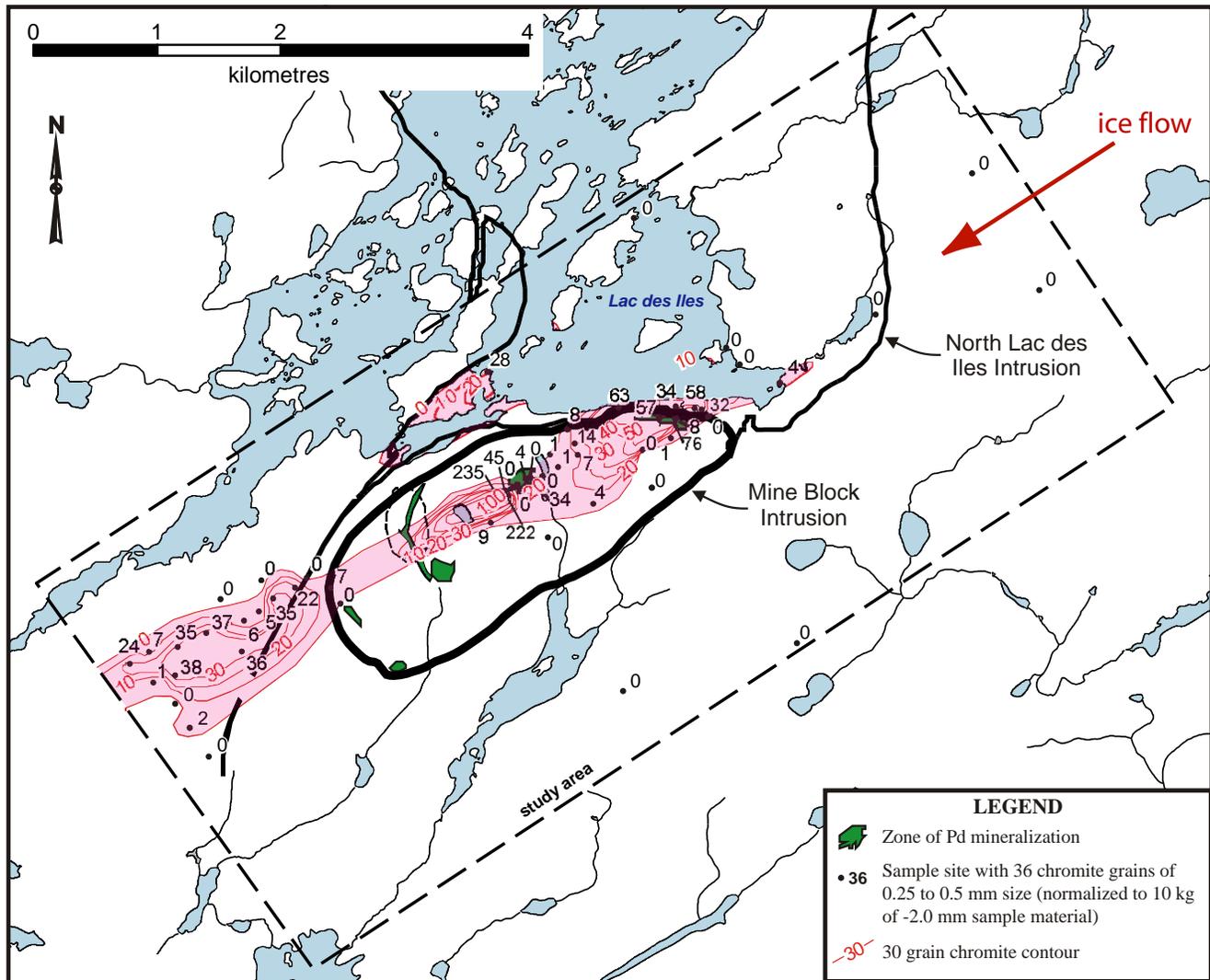


Fig. 12. Distribution of chromite in the 0.25 to 0.5 mm fraction of till normalized to a 10 kg sample weight down-ice of the Lac des Iles Pd mine area, northwestern Ontario, Canada (modified from Averill 2007a,b).

defined in till by 100s to 1000s per 10 kg of Cr-andradite (green garnet) and 10s to 100s per 10 kg of chromite grains (Figs. 11, 12).

### SUMMARY

Both gold and PGMs are the best detrital indicators of their own deposits and often each other's deposits. For gold deposits, gold grains, sulphides, platinoids, tellurides, scheelite, and rutile, as well as some secondary minerals may also be useful in specific regions. For PGM deposits, oxide and silicate minerals that reflect the mafic to ultramafic host rocks and minerals that reflect mineralization such as PGMs, gold, sulphides, arsenides, and antimonides are all useful indicators. Because gold grains are their own best deposit indicator, their size, shape, and chemical composition have been studied and reported in detail and classification schemes for gold grain shapes in placers and glacial sediments have been developed. Composition and morphology of PGMs have also been studied and described in detail, although no systematic shape classification has been developed. Both gold and PGMs have been recovered from stream sediments for more than 100 years. Gold grains in glacial sediments have been recoverable and

used as indicators for at least 100 years. Recovery of PGM grains in glacial sediments have not been widely reported. Recently published examples in glaciated terrain of Canada (e.g. Sudbury, Baffin Island, Thompson Ni Belt) demonstrate the increased sensitivity of indicator mineral methods for PGE exploration in glaciated terrain.

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# Kimberlite indicator mineral anomalies in a regional, local, and bedrock context: Examples from Canada

John P. Armstrong

*Stornoway Diamond Corporation, 116-980 West 1st Street, North Vancouver, British Columbia V7P 3N4  
(e-mail: jarmstrong@stornowaydiamonds.com)*

## INTRODUCTION

Large-scale regional surficial sediment sampling programs are a fundamental underpinning of diamond exploration programs within Canada. Sampling campaigns change in scope and style based upon raw kimberlite indicator mineral grain counts and subsequent mineral chemistry. Additional sampling and exploration techniques may be required when bedrock sources return abundances and chemistry that differ from the surficial signature. Patience and forethought are required as lag times for receipt of picking counts is further exacerbated by additional waiting periods for mineral chemistry data.

Recent advances in determining the origin of an indicator mineral suite based upon major oxide mineral chemistry and crystal structure allows for relatively inexpensive, timely, and informed decisions regarding the nature and diamond potential of the subcontinental lithospheric mantle underlying regional exploration targets. Pressure and temperature constraints for peridotitic garnet assemblages (Grütter *et al.* 2006; Creighton 2008); graphical presentation of the diamond-graphite transition (Grütter & Sweeney 2000); clinopyroxene single-grain pressure-temperature estimations (Nimis & Taylor 2001); ilmenite/picroilmenite kimberlitic chemical classification (Wyatt *et al.* 2004), kimberlitic spinel variances (Grütter & Apter 1998); and the hydration state of olivine (Matveev & Stachel 2007) all become important factors when evaluating an indicator mineral suite.

During the last 20 years, diamond exploration and associated surficial sediment sampling campaigns have been conducted by industry and government over broad regions within Canada. Government-sponsored studies complement industry derived data sets and, in certain areas, provide a regional context in the absence of industry derived data. Regional compilations also provide an opportunity to compare and contrast results from sampling conducted in greenfield regions or areas lacking a comprehensive public data set. Internal industry-generated data sets may be used for comparative purposes against data generated by government, competitors, and new discoveries.

Several examples are presented for discoveries in regions of mature exploration, greenfield discoveries, multiple source types, and new innovative techniques.

## MATURE EXPLORATION PLAY - TIMISKAMING

Property-scale till sampling was completed in proximity to the BA-19 target on the Quebec portion of the Timiskaming kimberlite field. Encouraging picking counts and chemistry, in conjunction with geophysical surveys, lead to a drill test of the BA-19 target. BA-19 is associated with a ground magnetic and aeromagnetic geophysical anomaly extending for over 400 m along strike, with another possible 200 m strike extent across an offsetting magnetic lineament (Fig. 1). Tuffisitic kimberlite breccia was intersected between 93 and 124 metres on a 45 degree inclined hole, implying a true thickness of 22 m for a vertical

body. Based on drilling, overburden is estimated to have a thickness of approximately 28 m. Representative portions of drill core were submitted for indicator mineral extraction.

Garnet recovered from the till samples have Cr<sub>2</sub>O<sub>3</sub> contents that range from 1.8 to 6 wt.% and a restricted range for CaO of 4 to 6.5 wt.%; the population parallels the lherzolitic trend and is offset to the right of the G9-G10 line. One subcalcic G10 garnet was recovered. The garnet population from the kimberlite body conforms to the upper portion of the lherzolite trend, the bulk of recovered garnet contains 5 to 12 wt.% Cr<sub>2</sub>O<sub>3</sub> and a range in CaO of 4 to 9 wt.%. Four garnets are classified as harzburgitic (G10) with three grains sitting just below the graphite-diamond constraint (Grütter & Sweeney 2000; Grütter *et al.* 2006). Comparison of the kimberlite-derived indicators with the till samples results indicate that the geochemical source for the till anomaly is substantively different than that of the intersected kimberlite (Fig. 2). The till anomaly may be derived from another phase of the BA-19 kimberlite or from an as yet to be discovered kimberlite. The suite of garnet data from the BA-19 target conforms well to the regional garnet signature and falls within the upper lherzolitic trend for the region (Fig. 3).

## AREA SELECTION - AVIAT

The Aviat Project is located on the northern portion of the Melville Peninsula, 69.27°N 83.32°W, approximately 60 km west-southwest of the hamlet of Igloodik, Nunavut and 2700 km north-northeast of Ottawa, Ontario. Reconnaissance-scale heavy mineral sampling of till, eskers, and beaches over the northern portion of the Melville Peninsula commenced in 2001. Recovery of diamond indicator minerals was achieved in the first tranche of samples. One sample in particular returned a suite of grains with promising mineral chemistry. Follow-up sampling, including a check/duplicate sample at the beach location of the first sample, resulted in the recovery of diamond indicators with adhering kimberlite. Soon after, the AV1 kimberlite was discovered approximately 650 metres east of the original sample site.

The Aviat kimberlite bodies intruded into the Archean basement rocks of the northern Rae craton between 500 and 560 Ma. Since the initial discovery, in excess of 80 000 line kilometres of airborne geophysics have been flown and approximately 11 000 surface samples have been collected across the property. Complex magnetic bedrock responses limit the usefulness of airborne surveys in identifying kimberlite. Combined sample campaigns revealed a kimberlite indicator mineral-rich zone approximately 65 km long and 10 km wide, trending 286°, which is parallel to the dominant glacial-flow direction. This regional-scale indicator mineral train is referred to as the Tremblay Corridor and all presently known kimberlites occur within a 10 km long by 3 km portion at the eastern terminus of the Tremblay Corridor (Armstrong *et al.* 2008).

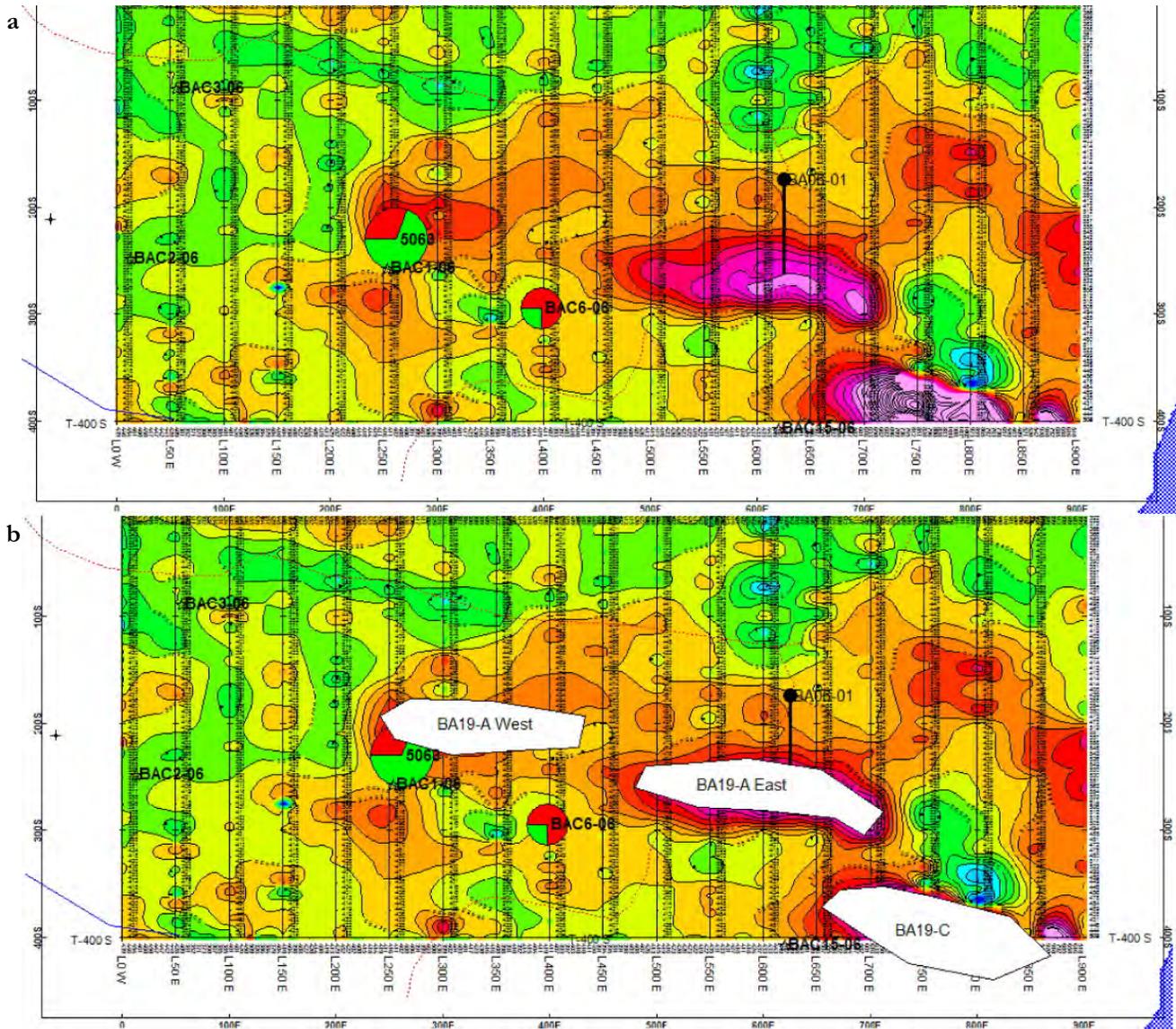


Fig. 1. a) BA-19 Ground total magnetic intensity result with diamond drillhole location and till sample sites. b) BA-19 ground total magnetic intensity with interpretation.

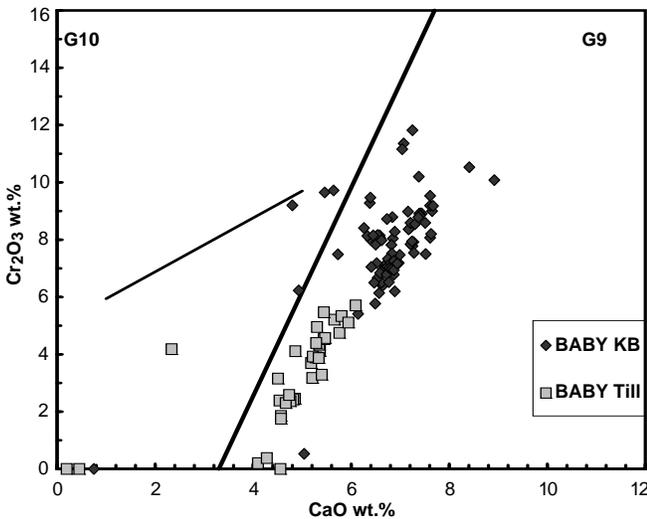


Fig. 2. Cr<sub>2</sub>O<sub>3</sub> versus CaO (wt.%) in garnets from the BA-19 kimberlite and down-ice till samples.

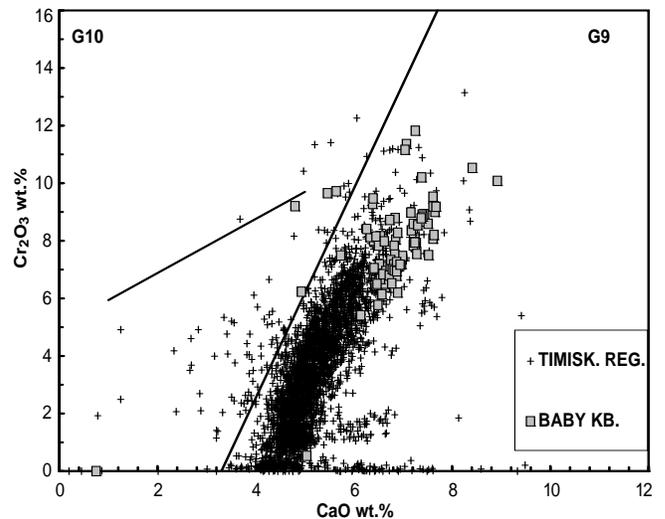


Fig. 3. Cr<sub>2</sub>O<sub>3</sub> versus CaO (wt.%) in garnets from the BA-19 kimberlite versus Timiskaming regional garnet data.

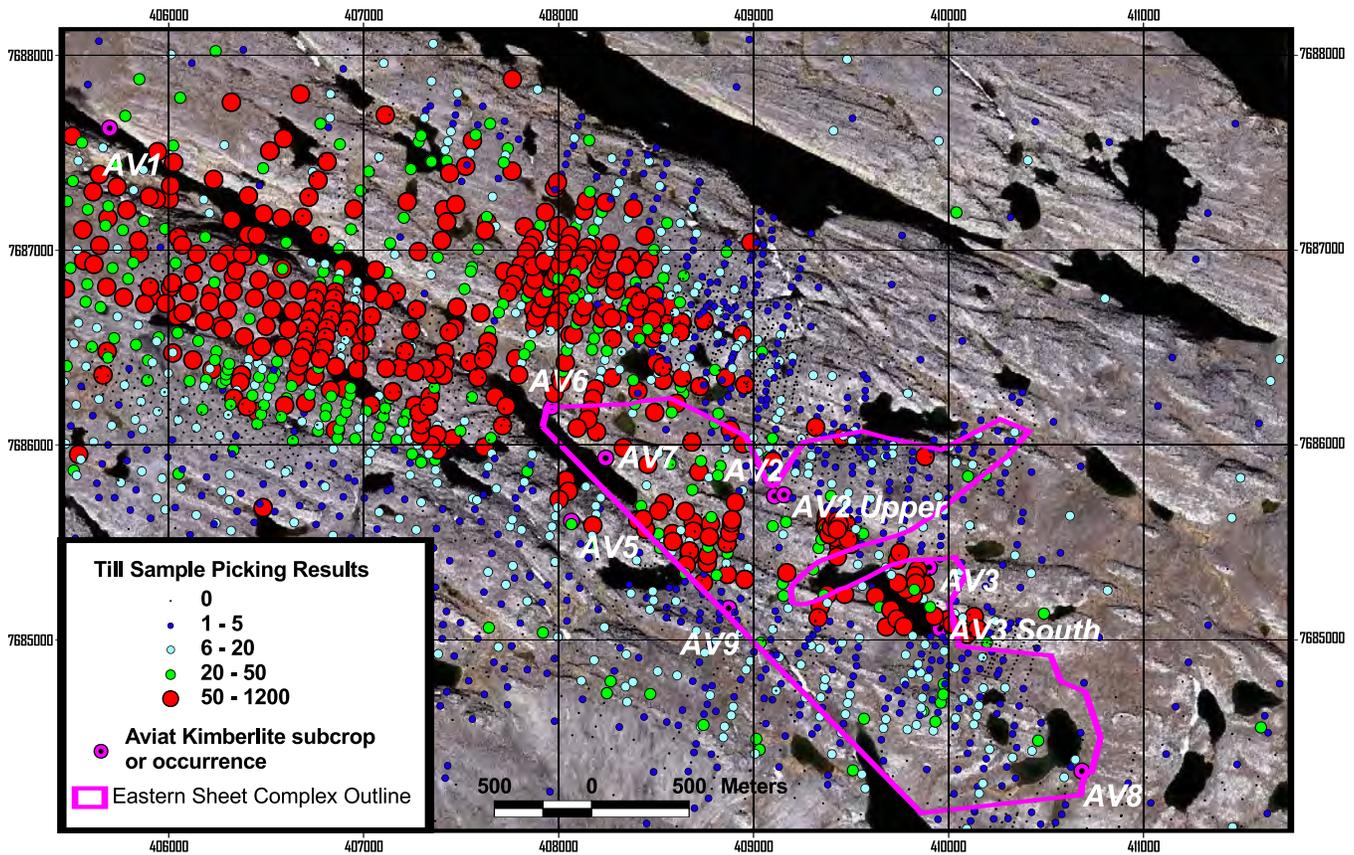


Fig. 4. Projected surface trace of the Aviat ES1 kimberlite sheet and the picked indicator mineral anomaly associated with the eroded leading edge of the kimberlite sheet complex.

Preferred sample media for the sampling campaigns was glacial till collected from frost boils. Sample size for coarse sample spacing was 20 kilograms and for detailed sample grids, 10 kg samples were taken. The density of sample spacing decreases from 1000 m, 500 m, and 250 m, to a final 30 m spaced sampling along lines near up-ice geochemical cut-offs.

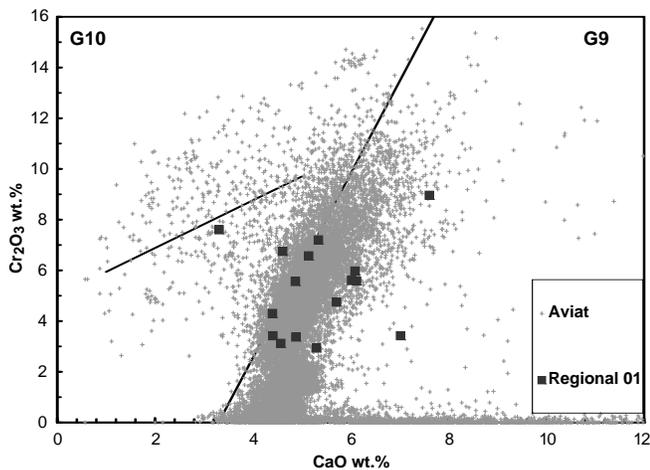
Two distinct geometries of kimberlite intrusions are observed within the 10 x 3 km eastern portion of the Tremblay Corridor: pipe-like intrusions and subhorizontal sill-like intrusions. Three kimberlite pipes have been identified along a 4 km strike length, within and south of a northwest-trending sinistral strike-slip regional fault zone. North of the fault zone, kimberlite sheet complexes comprise sets of stacked, subhorizontal to shallow-dipping, hypabyssal kimberlite intrusions that have been delineated over a 10 km<sup>2</sup> area. The Eastern Sheet Complex (ESC) forms a broadly conical to radial pattern, dipping inward toward the northwest-trending fault system. The ES1 sheet is the dominant kimberlite within the ESC and has been traced through surface exposures and drill intersections over an area of 280 hectares and to depths of 100 metres below surface. This sheet is manifested by several lobes: an eastern segment trending 065°/10°, and a western segment trending 110°/8°. The lobes join along a north-northeast/south-southwest hinge/rollover zone. The eastern portion of the ES1 sheet outcrops at a location known as AV8 and displays a 210°/20° attitude (Armstrong *et al.* 2008).

Dominant ice-flow indicators are orientated at approximately 286° degrees, perpendicular or at a high angle to the surface trace of the ESC kimberlites. Kimberlite indicator mineral dispersions in the eastern portion of the Tremblay

Corridor are therefore manifested by broad swaths of grains, and within 250 m of the ESC surface trace, overall abundances are uniformly high (Fig. 4). Linear trains are uncommon; one such train terminates in a small bay adjacent to the strike extent of AV8, and remains untested. Abundant anomalous KIM samples that did not resolve as linear trains, in conjunction with complicated bedrock geophysical responses required intensive ground follow-up and prospecting. As a result, the greatest success in identifying kimberlite occurrences has come through intensive prospecting and detailed infill sampling in the immediate vicinity of anomalous till samples.

The AV1 kimberlite was discovered during follow-up sampling and prospecting of one of the original regional till samples. ESC-AV2 was discovered by following a train of kimberlite boulders. ESC-AV3 was discovered approximately 250 metres up-ice of a highly anomalous till sample [garnet (n=56), spinel (n=7), microilmenite (n=9), olivine (n=5)]. AV4 boulders were discovered during the course of a 250 m spaced grid sample program. ESC-AV6 and ESC-AV7 were discovered during prospecting in the vicinity of highly anomalous till samples [garnet (n=46), spinel (n=10), microilmenite (n=11), olivine (n=5)]. ESC-AV8 was discovered while prospecting a subtle indicator anomaly at the up-ice terminus of the regional till anomaly [garnet (n=6), spinel (n=1), microilmenite (n=8)].

The full suite of mantle-derived garnet, clinopyroxene, chromite, orthopyroxene, olivine, and kimberlite-derived ilmenite are present within the surficial and kimberlite samples. The recovered garnets include significant proportions of high-Cr and low-Cr types. The high-Cr (>2 wt.% Cr<sub>2</sub>O<sub>3</sub>) population is dominated by ilherzolitic garnets with CaO contents greater



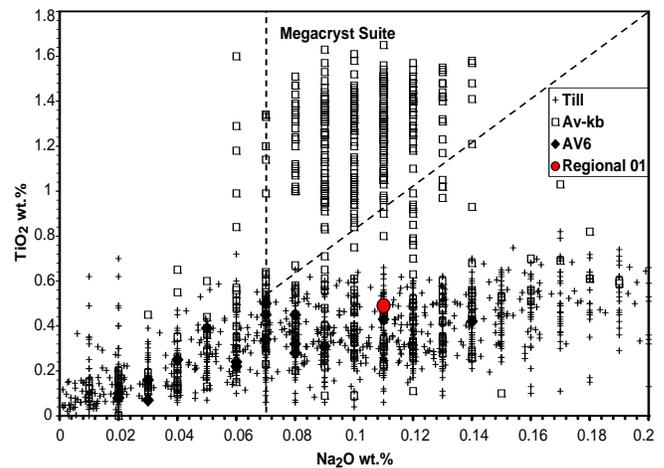
**Fig. 5.**  $\text{Cr}_2\text{O}_3$  versus  $\text{CaO}$  (wt.%) in garnets from Aviat till samples and kimberlites versus garnet results from the anomalous regional sediment sample that attracted interest to the area.

than the G9-G10 line. The harzburgitic or G10 population contains a significant proportion that lie on the high  $\text{Cr}_2\text{O}_3$  side of the diamond graphite line, as defined by Grütter and Sweeney (2000) and Grütter *et al.* (2006). There is also a significant population of Ti-enriched megacrystic garnets. A suite of eclogitic garnets are present in kimberlite and till samples. The eclogitic suite includes high  $\text{MgO}$ , low  $\text{MgO}$ , and high  $\text{CaO}$  varieties, including diamond-inclusion chemistries with  $\text{Na}_2\text{O}$  concentrations of  $>0.07$  wt.%. The importance of the eclogitic component was reinforced by the recovery of a small ( $<1.5$  cm in diameter) diamond-bearing eclogite nodule from the ES1 kimberlite. High Cr chromites are recovered in tills and kimberlite samples, a significant proportion (with  $>0.7$  wt.%  $\text{TiO}_2$ ) display increasing  $\text{TiO}_2$  concentrations with decreasing  $\text{Cr}_2\text{O}_3$ , indicative of the phenocryst suite of chromites. A proportion of chromites with  $<0.7$  wt.%  $\text{TiO}_2$  have  $\text{Cr}_2\text{O}_3$  concentrations of between 59 and 69 wt.%, and may be considered as sourced from a diamondiferous spinel-peridotite paragenesis (Armstrong *et al.* 2008).

A data set of approximately 20,000 garnet analyses now provides a regional context for this portion of the Rae Craton. A retrospective evaluation of the regional geochemical survey indicates that the first-received indicator mineral chemistry, although significant, is certainly not amongst the best recovered, nor entirely reflective of the AV1 kimberlite (Figs. 5, 6). This should be considered as a cautionary note when reviewing initial mineral chemistry results.

### CORONATION GULF

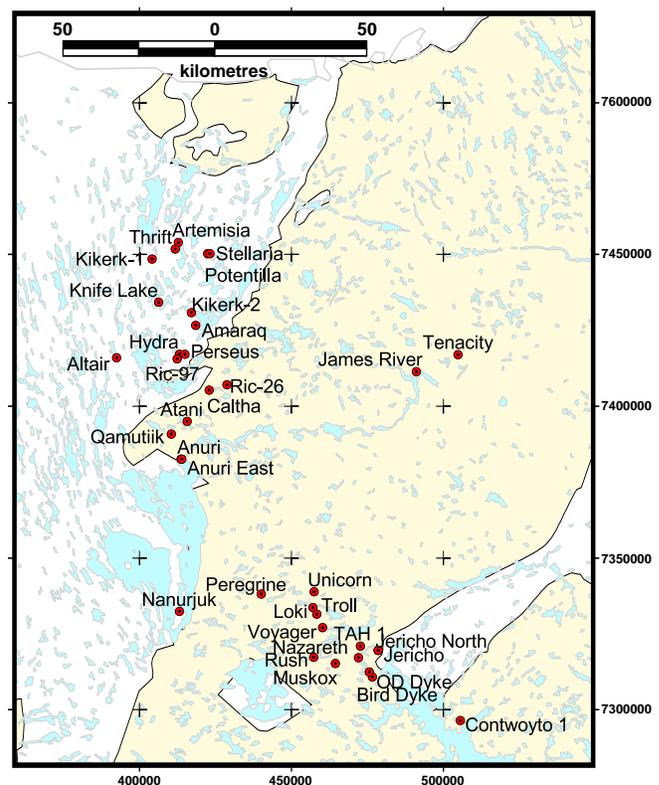
Exploration for kimberlites in the Coronation Gulf region of the Northern Slave Craton has taken place since the mid-1990s, with the discovery of numerous kimberlite bodies (Fig. 7). Exploration underwent a resurgence with the discovery of the Artemisia kimberlite in 2000. The government-generated KIDD and KIMC compilations of industry data (Armstrong 2001; Armstrong & Chatman 2001) provided a regional indicator mineral and mineral chemistry base for comparative work. The region is host to approximately 24 kimberlites, several of which have been shown to be diamondiferous (10 to 25 cph), and a suite of barren to poorly diamondiferous bodies.



**Fig. 6.**  $\text{Na}_2\text{O}$  versus  $\text{TiO}_2$  (wt.%) in eclogitic (G3,G4) garnets from Aviat till samples and kimberlites versus eclogite garnet results from the anomalous regional sediment sample that attracted interest to the area.

Kimberlite magmatism has been episodic, with emplacement in the late Proterozoic, Cambrian, and Jurassic.

Dispersion trains within the region are linear in nature (Armstrong & Kjarsgaard 2003; McClenaghan & Kjarsgaard 2007) and overall mineral abundances suggest dramatically different source rocks. Ilmenite-dominant trains are sourced from a variety of barren to weakly diamondiferous bodies (Kikerk 1, Kikerk 2, Perseus); trains with lherzolitic and weak harzburgitic signatures are sourced from the Artemisia, Potentilla, Stellaria, and Knife kimberlites (Fig. 8). The availability of public domain data sets allows for comparison of both the silicate and oxide mineral signatures from kimberlite sources



**Fig. 7.** Coronation Gulf kimberlite occurrences.

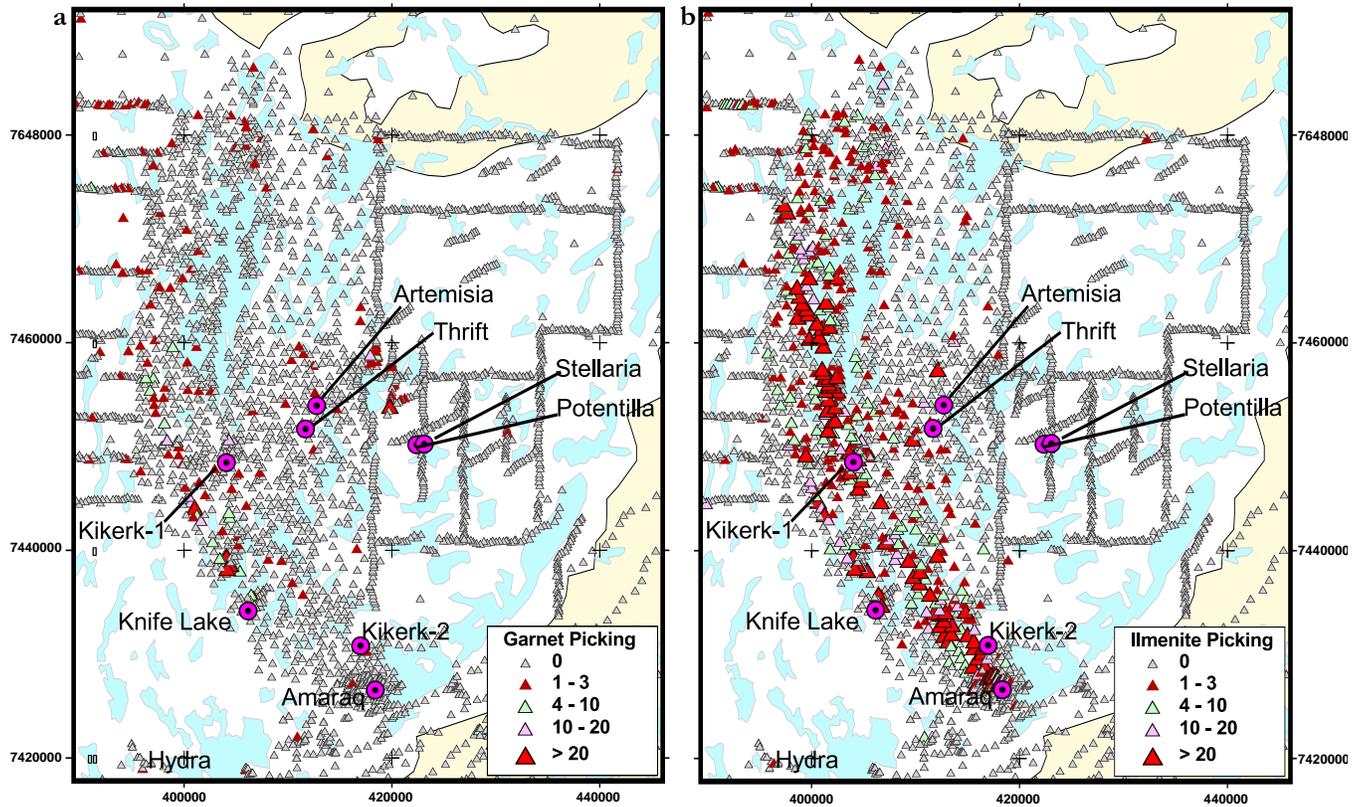


Fig. 8. a) Ilmenite picking results for the northern Coronation Gulf region. b) Total garnet picking results for the northern Coronation Gulf region. Data from Armstrong & Chatman (2001).

(Armstrong 2001; Armstrong & Chatman 2001; Hetman *et al.* 2004). Ilmenite chemistry appears similar, although ilmenite is much more abundant in trains sourced from diamond-poor kimberlites. The garnet signatures display subtle differences between till data from the Knife and Potentilla kimberlites. The Potentilla data are offset to the right of the G9-G10 line and span the lherzolitic trend from 2 to 12 wt.%  $\text{Cr}_2\text{O}_3$ , several grains plot to the left of the line and are classified as G10s. There is a cluster of data with CaO concentrations of 4 to 6 wt.% and  $\text{Cr}_2\text{O}_3$  concentrations of 2 to 4 wt.%, indicative of the megacryst garnet suite. The Knife Lake data also plot parallel to the G9-G10 line but lie closer to, and overlap the line

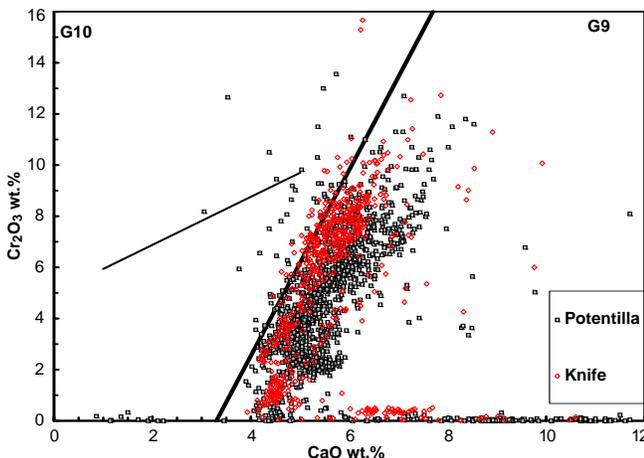


Fig. 9.  $\text{Cr}_2\text{O}_3$  versus CaO (wt.%) in garnet from till samples in the vicinity of the Potentilla and Knife Lake kimberlites.

(Fig. 9). The Knife Lake data also apparently lack the megacryst component. The Potentilla and Knife Lake garnet signatures also contain a suite of eclogitic garnets (Fig. 10a). Potentilla eclogite is similar to that of Jericho and the Knife population contains a subset with  $\text{Cr}_2\text{O}_3$  concentrations between 0.1 and 2.0 wt.%, and CaO concentrations of 6 to 8 wt.% (Fig. 10b), which is similar to compositions reported for central Slave eclogite (Aulbach *et al.* 2007).

#### NEW TECHNIQUES IN EMERGING REGIONS: FOXTROT/RENARD

The Foxtrot property is located within the northern Otish Mountains of Quebec. The property is host to at least 9 kimberlite pipes (Renard pipes) and two extensive flat-lying kimberlite dyke intrusions (Lynx and Hibou) (Fig. 11). Two distinct emplacement ages have been returned for the Foxtrot kimberlites: a Neoproterozoic age of approximately 640 Ma for the Renard bodies (Birkett *et al.* 2004; Fitzgerald *et al.* 2008) and a Cambrian age of  $522 \pm 30$  Ma for the Lynx dyke (McCandless *et al.* 2008).

One sample from a regional program of 48 samples, which was conducted in 2000, returned anomalous grain counts (O'Connor & Lepine 2006) and a suite of indicator minerals [garnet (n=33), spinel (n=1), picroilmenite (n=800), chromium diopside (n=11)]. Mineral chemistry was strongly suggestive of a source region with high diamond potential (Fig. 12). In 2001, a series of 248 till samples were collected on the Foxtrot property and in the early fall of 2001, after results were returned from the additional sampling and geophysical surveys, the discovery phase of drilling commenced (O'Connor & Lepine 2006).

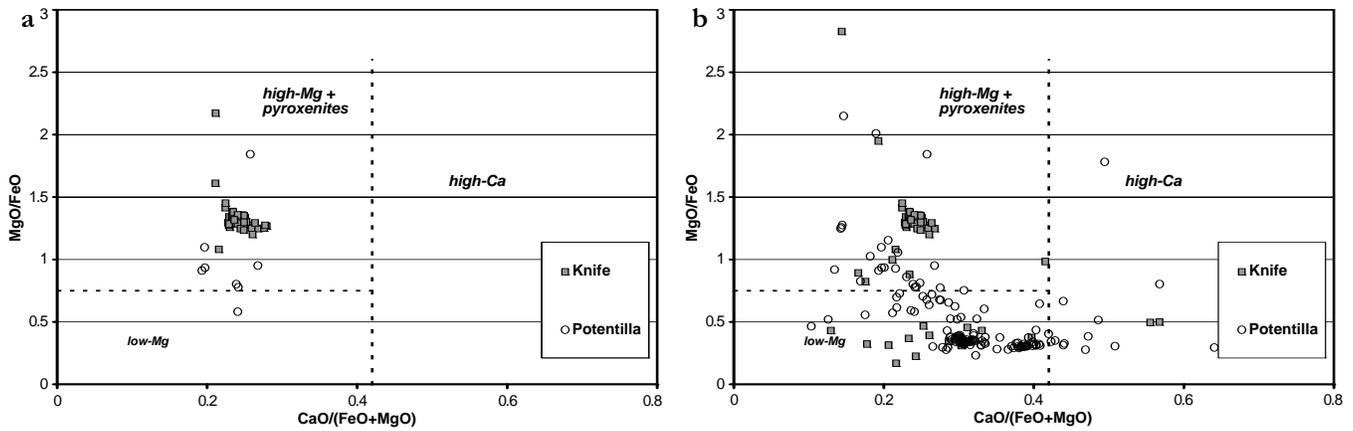


Fig. 10. a)  $\text{Na}_2\text{O}$  versus  $\text{TiO}_2$  (wt.%) and b)  $\text{CaO}/(\text{MgO}+\text{FeO})$  versus  $\text{MgO}/\text{FeO}$  (wt.%) in eclogitic (G3,G4) garnet from till samples in the vicinity of the Potentilla and Knife Lake kimberlites.

The dominant ice direction is approximately 215° degrees and numerous indicator mineral trains with the traditional suite of indicator minerals are present on the property. An olivine-dominant train, referred to as the Ruler train, lies 12 km to the

northeast of the Renard cluster, has a width of 200 to 600 metres, and a defined length of approximately 6 kilometres (Fig. 11). Within the Ruler train, olivine grain counts range up to 500 grains per sample. FTIR spectroscopy, employing the method-

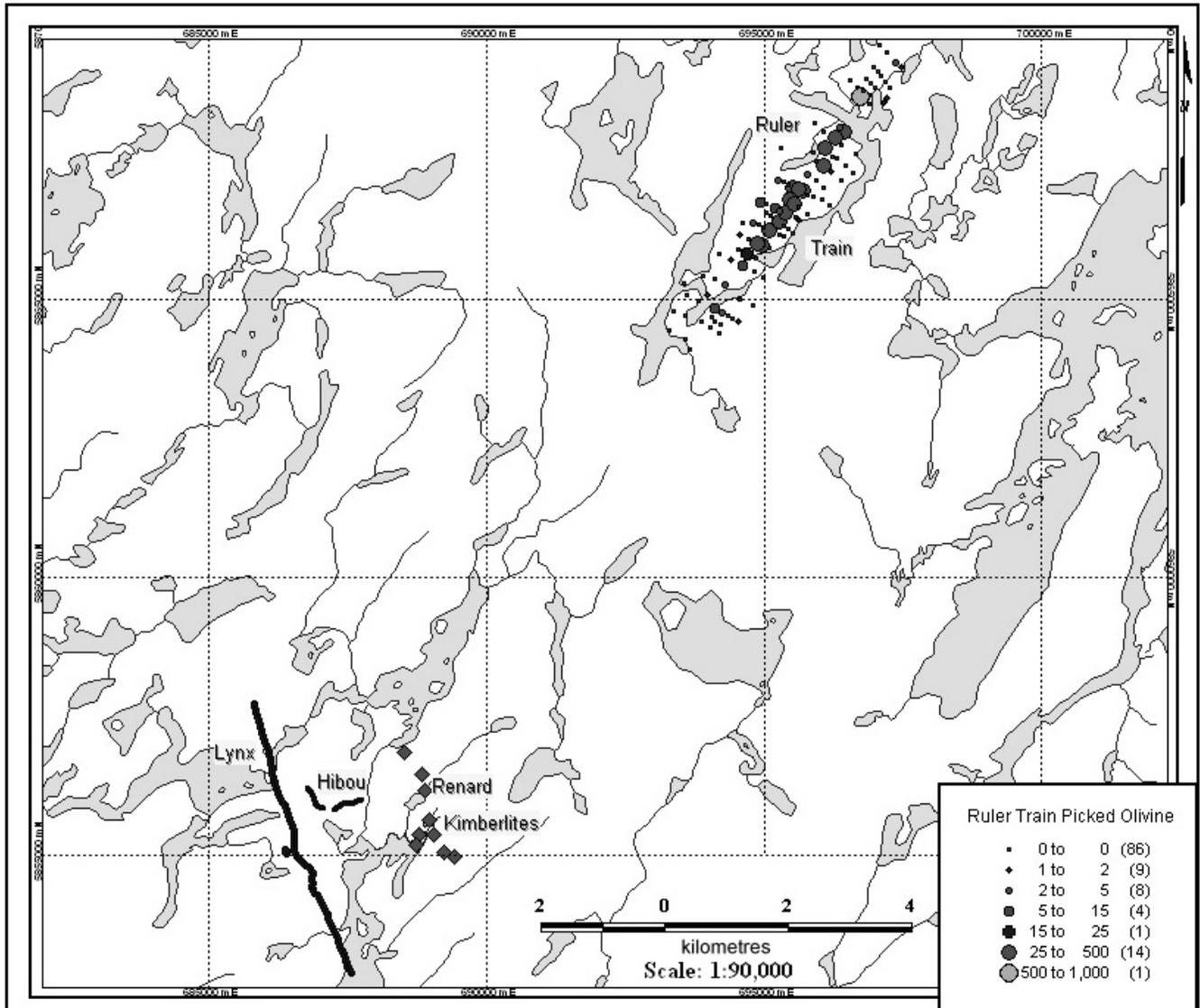


Fig. 11. Location of the Renard kimberlite pipes, Lynx and Hibou dykes, and the olivine-dominant Ruler train, Foxtrot property.

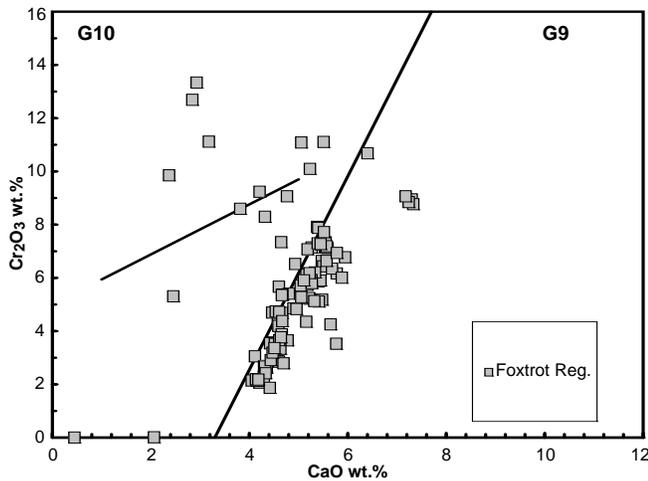


Fig. 12.  $\text{Cr}_2\text{O}_3$  versus  $\text{CaO}$  (wt.%) in garnets from the anomalous Foxtrot regional till sample collected in 2000.

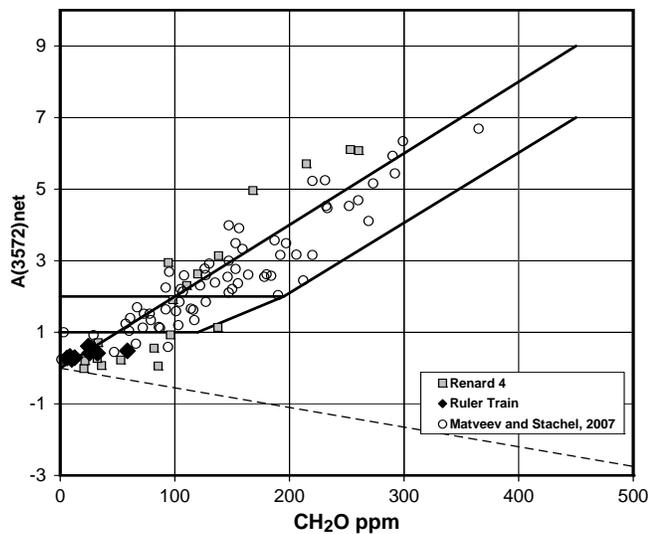


Fig. 13.  $\text{H}_2\text{O}$  (ppm) versus OH absorption at  $3572\text{ cm}^{-1}$  after Matveev & Stachel (2007). Data shown for the Ruler train, Renard 4 kimberlite, and published data for various Canadian kimberlite occurrences (Matveev *et al.* 2007).

ology of Matveev and Stachel (2007), was undertaken on a suite of 20 olivine grains recovered from the Ruler Train in order to assist in determining olivine paragenesis. In addition, olivine from kimberlite concentrate was submitted for FTIR spectroscopy. Results of the small study indicate that the Ruler train olivine is anhydrous and therefore not of kimberlitic provenance (Fig. 13). The suite of kimberlite-concentrate olivine overlaps with hydrous olivine from other Canadian kimberlite occurrences (Fig. 13; Matveev & Stachel 2007). Coincident with the FTIR work, additional Ruler train olivine grains were recovered that are intergrown with non-kimberlitic mineral assemblages. The FTIR method was approximately one third the price on a per-grain basis compared to traditional mineral chemistry, and quick turnaround times were achieved. Grains remain intact if mounting and mineral chemistry are required.

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

S. A. Averill

Overburden Drilling Management Limited, 07-15 Capella Court, Nepean, Ontario K2E 7X1  
(e-mail: odm@storm.ca)

## 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, and 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 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

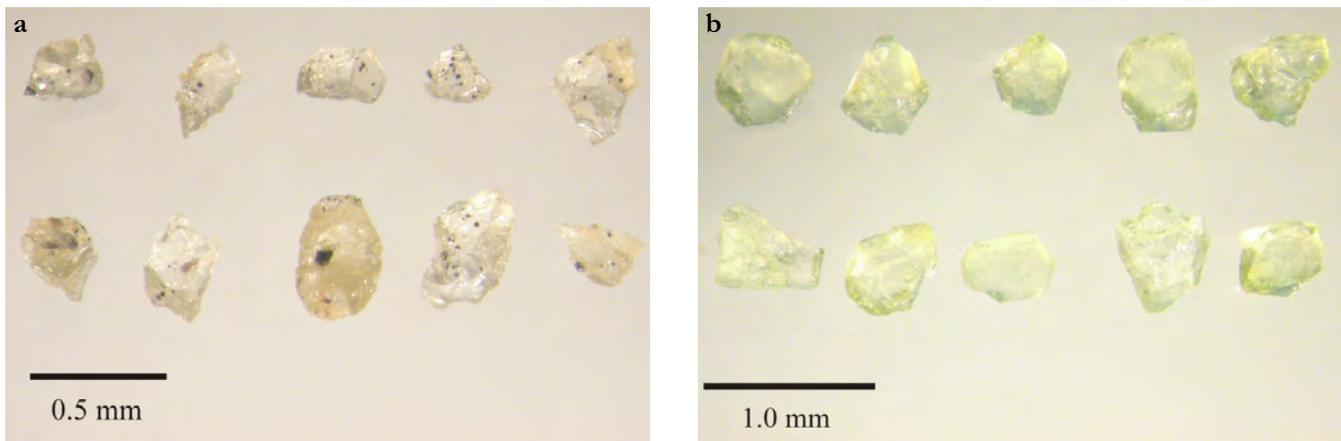
Four types of indicator minerals are potentially present in the vicinity of Ni-Cu-PGE deposits (Table 1; Averill 2007b). Each type is inexorably linked to one or more of the conditions of formation of such deposits because these conditions involve chemical factors that influence mineral compositions.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



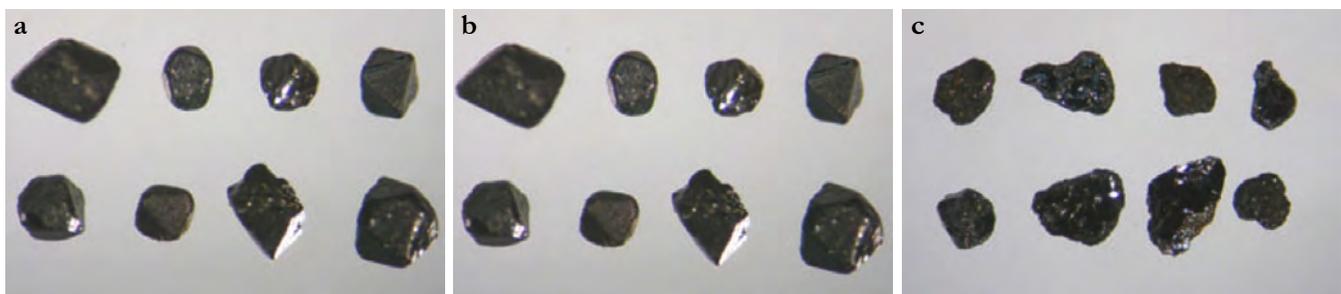
**Fig. 1.** Large (~40 cm) upper mantle xenolith of garnet peridotite from the Premier kimberlite pipe, South Africa. Specimen 116686, National Museum of Natural History, Smithsonian Institution.

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 nonparamagnetic. 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 >20:1 compared to <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



**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.



**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.

**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>

**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	Ratio of 0.25-0.5 to 0.5-1.0 mm grains	Ratio of 0.25-0.5 to 0.5-1.0 mm grains		Number of forsterite grains 0.25-0.5 mm	Ratio of 0.25-0.5 to 0.5-1.0 mm grains	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

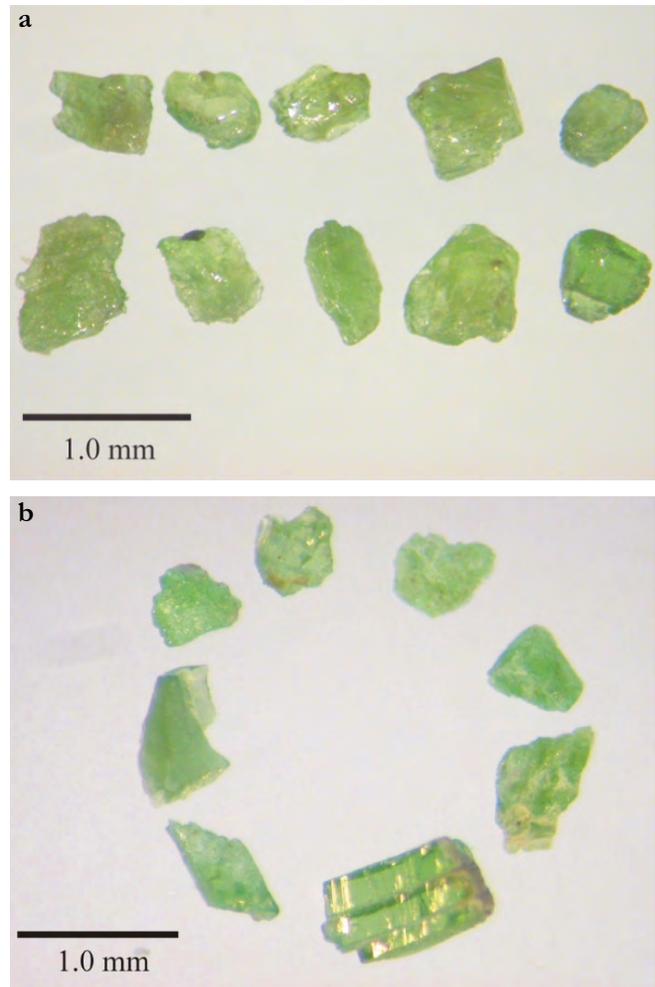
than the kimberlitic variety, reducing the amount of emerald-green pigment (Fig. 4). A 1.25% Cr<sub>2</sub>O<sub>3</sub> boundary is helpful for distinguishing this “low-Cr” diopside from xenocrystal kimberlitic Cr-diopside grains but visual identification is generally more reliable due to significant 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). A similarly long chromite anomaly was identified by the Ontario Geological Survey in 2003 in the bed of the Attawapiskat River west of James Bay (Fig. 6; Crabtree 2003). This anomaly begins in the river headwaters near the edge of the Precambrian Shield. Downstream to the east, the river traverses the Paleozoic carbonate platform of the Hudson Bay Lowland where it crosses a cluster of chromite-bearing kimberlite pipes. However, the chromite content of the riverbed does not increase significantly in this area. Furthermore, most of the chromite grains are smaller than typical kimberlitic chromite and some occur in cemented aggregates similar to the banded cumulus chromitite found in certain layered mafic intrusions such as the Bushveld Complex. Recent exploration along the Shield/Lowland boundary by KWG Resources Inc., Spider Resources Inc., Freewest Resources Canada Inc., and Noront Resources Ltd. has resulted in the discovery of three chromite deposits and two Ni-Cu-PGE-bearing massive sulphide lenses in an area that also contains significant volcanogenic massive sulphide (VMS) deposits and diamondiferous kimberlites; this area has been dubbed the “Ring of Fire” (Fig. 7; Stott 2008). The chromite is concentrated in banded to massive chromitite zones up to 70 m thick (Fig. 8). However, the three deposits discovered to date, Big Daddy, Blackbird, and Black Thor are partly covered by carbonate rocks and are significantly removed from the Attawapiskat River, suggesting that the principal source of the large chromite anomaly in the riverbed remains to be discovered.

While a fertile melt is essential to the formation of a Ni-Cu-PGE deposit, such a 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 concen-



**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 Cr<sub>2</sub>O<sub>3</sub> content. Source: Overburden Drilling Management client files.

trated 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. 9).

The indicator minerals produced by assimilation of felsic country rocks by a komatiitic melt are hybrid alteration minerals containing both felsic elements, primarily 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. 9). The hydrated grains are cryptocrystalline and drusy (Fig. 10a), 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. 10b) 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).

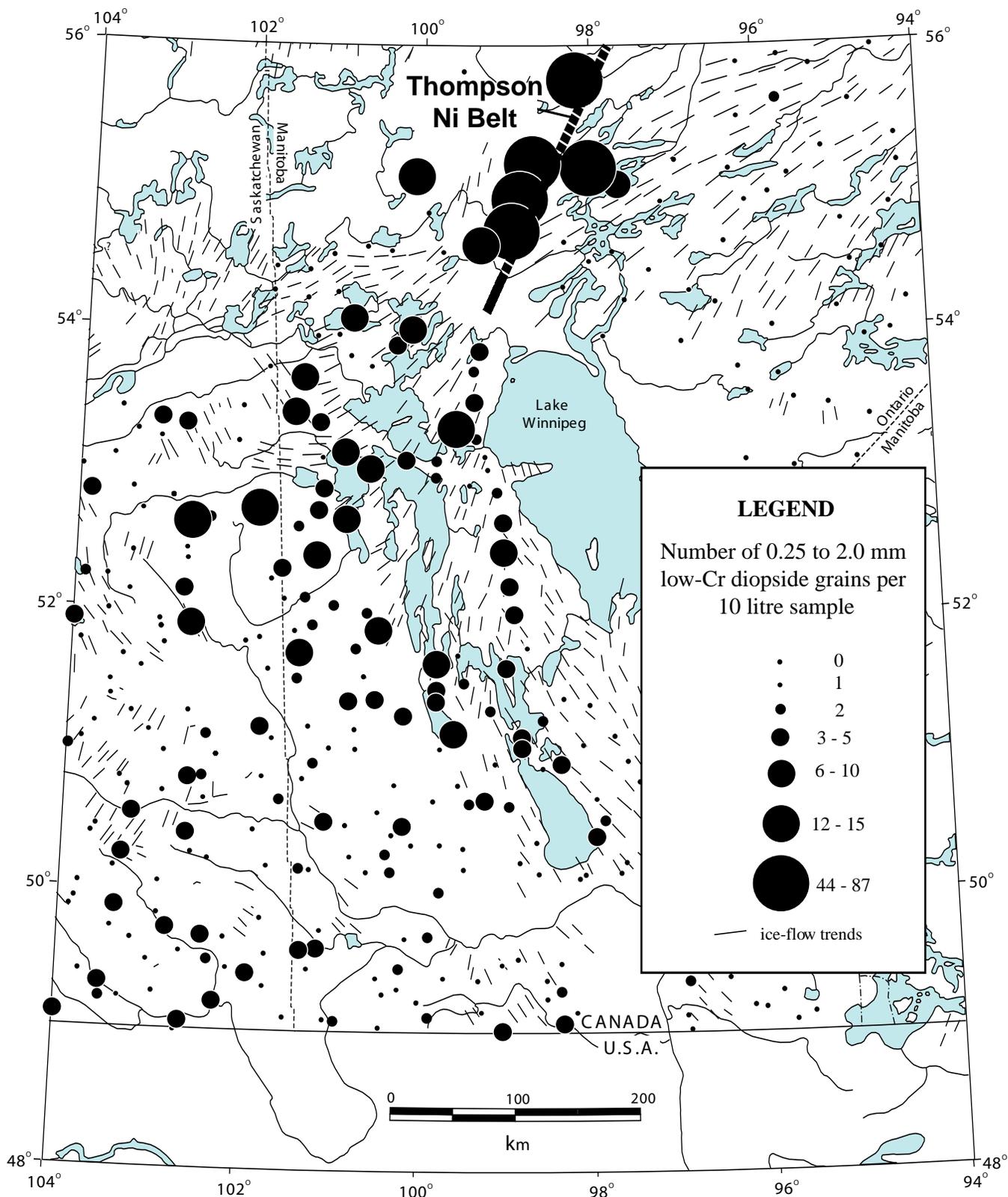
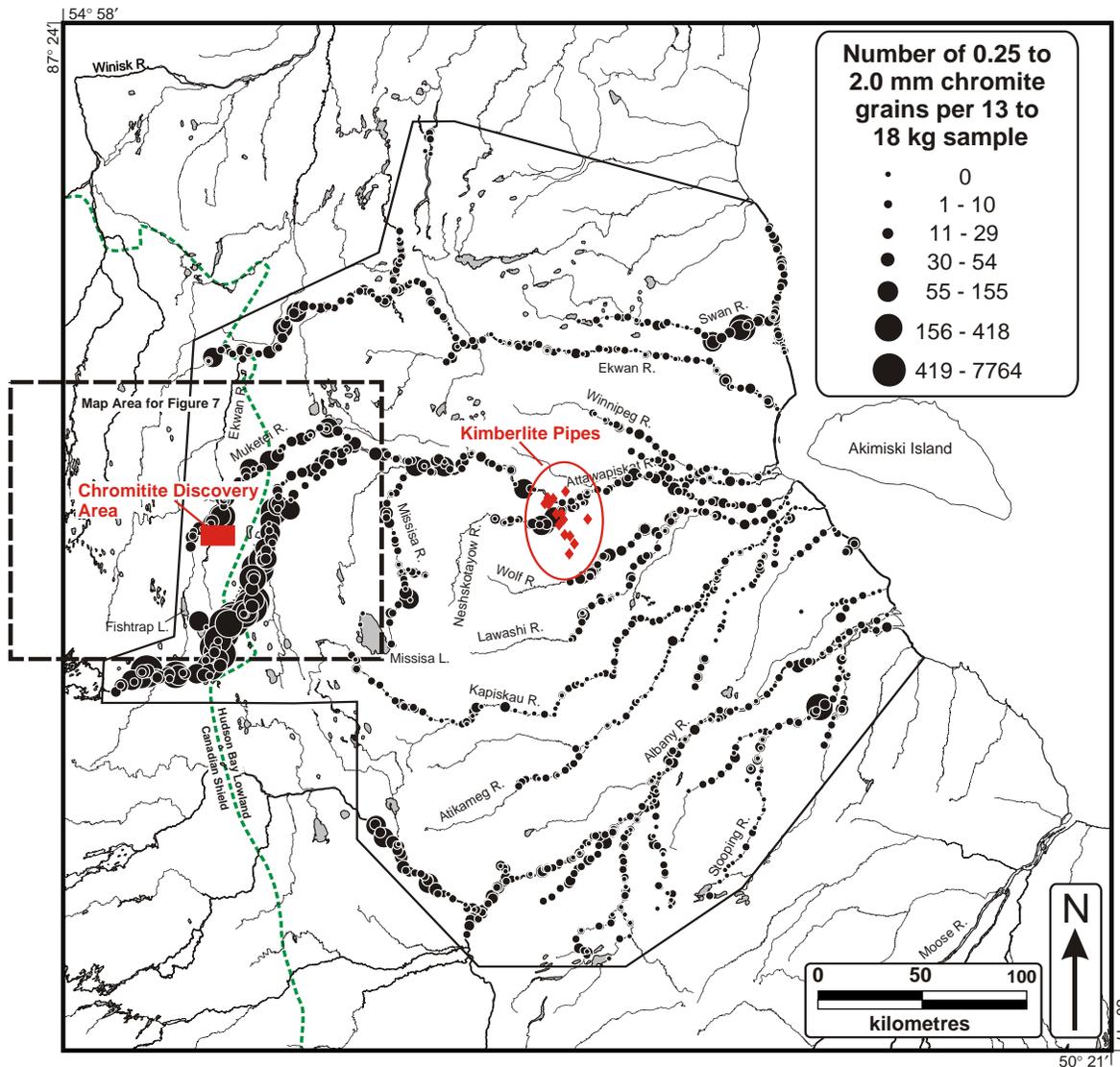


Fig. 5. Regional distribution of low-Cr diopside in till in western Manitoba and eastern Saskatchewan. Note the broad dispersal train extending 400 km southwest from the Thompson Nickel Belt. Modified from Thorleifson & Garrett (1993).

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 and pyrite grains present in the



**Fig. 6.** Regional distribution of chromite in alluvial gravel in the James Bay Lowland, Ontario. Note the major buildup in chromite grains toward the western headwaters of the Attawapiskat River. Source: Crabtree (2003).

**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				
			Ontario		Nunavut		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			

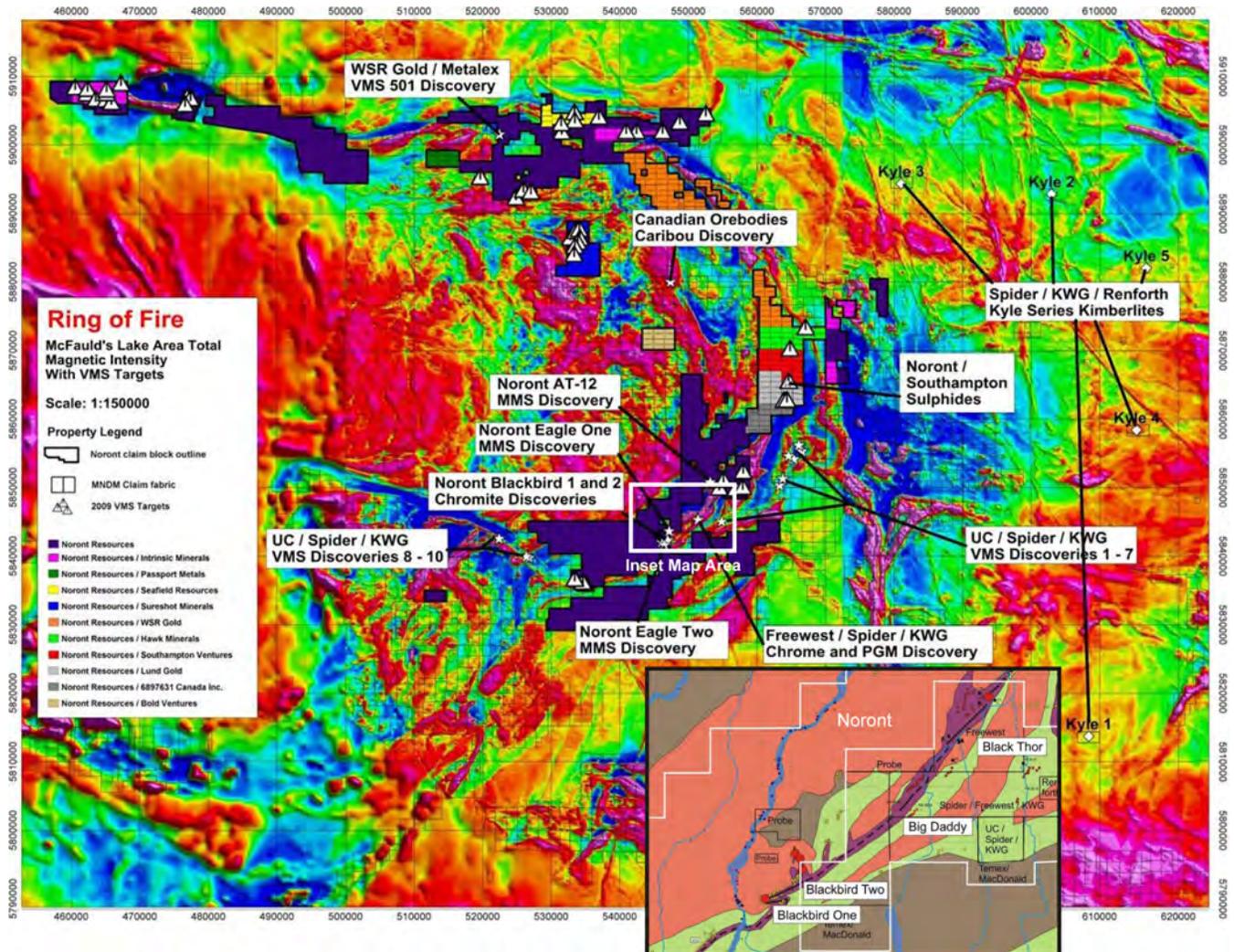


Fig. 7. Total field magnetic map of the Ring of Fire area, Ontario, showing the locations of known mineral occurrences. The map outline is also shown on Figure 6. The inset shows the locations of the Blackbird, Big Daddy, and Black Thor chromite discoveries. Courtesy of Greg Stott, Ontario Geological Survey, and Noront Resources Ltd.

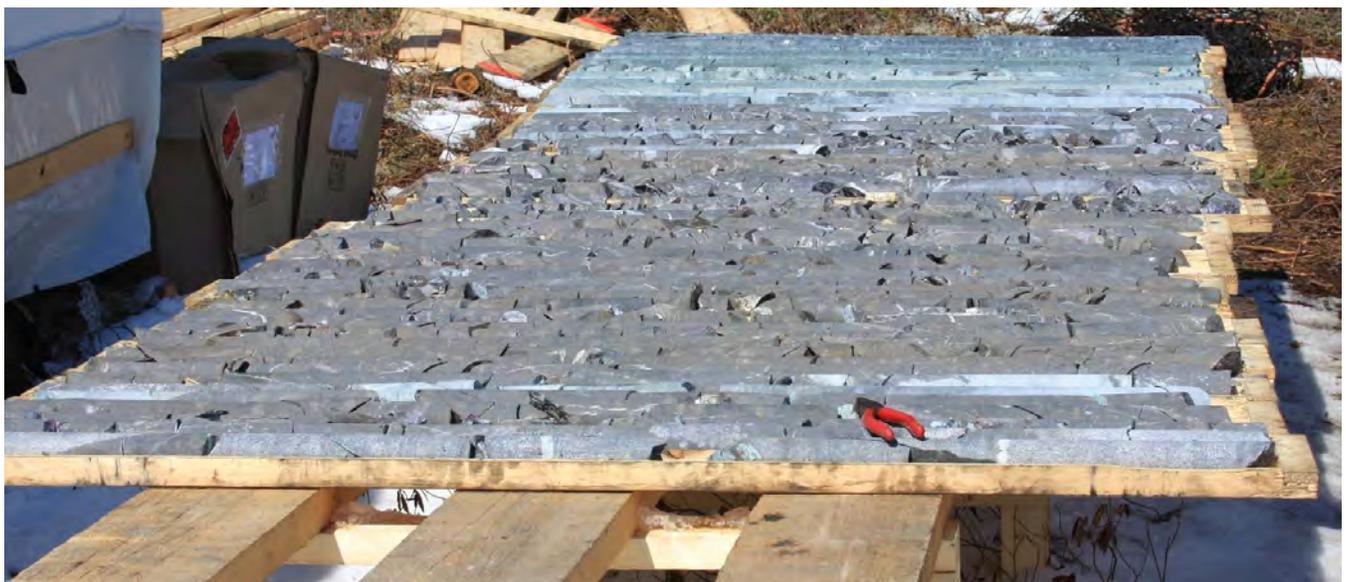


Fig. 8. A 70 m section of massive chromitite from the Blackbird deposit. Courtesy of Greg Stott, Ontario Geological Survey, and Noront Resources Ltd.

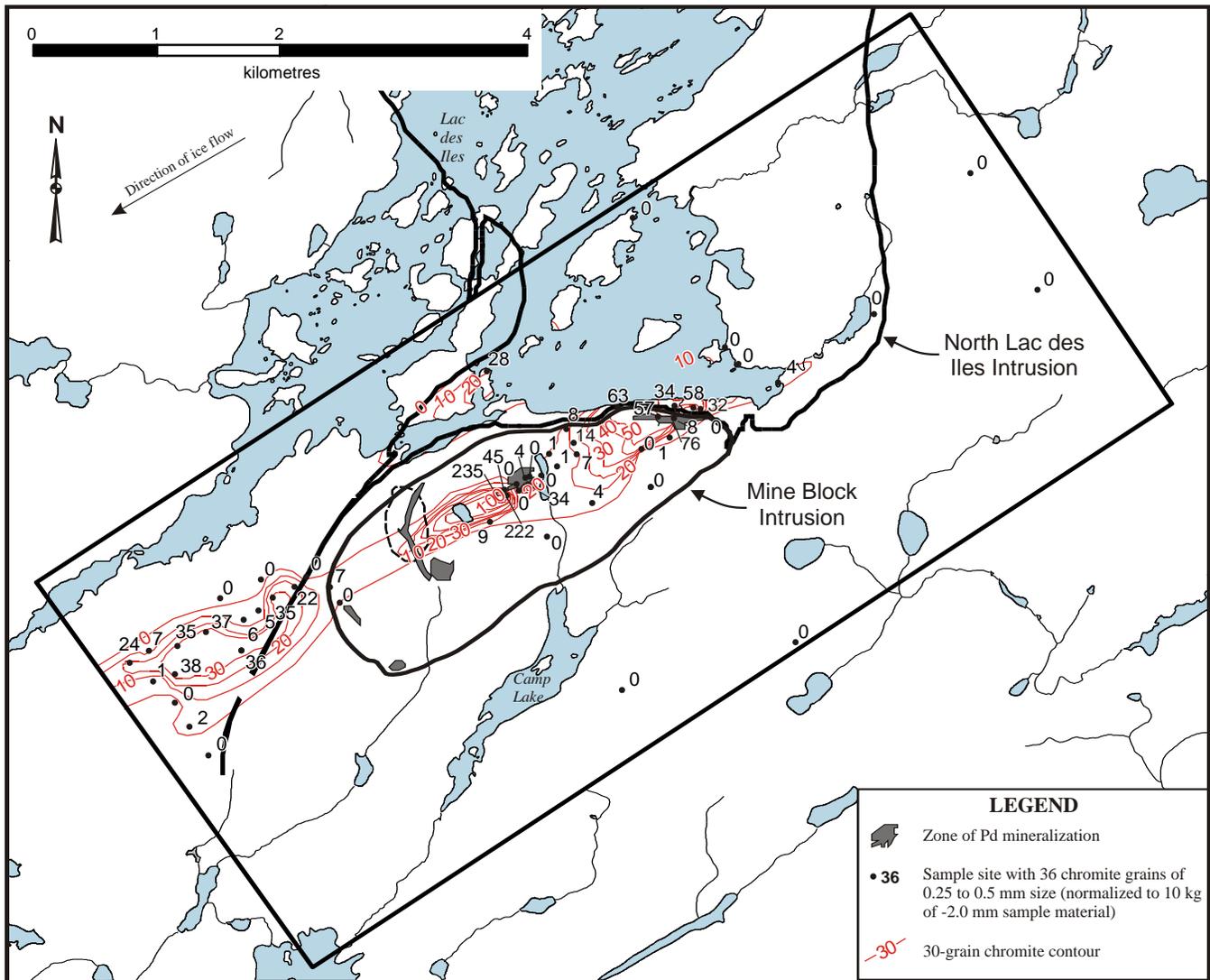


Fig. 9. Distribution of chromite in till near the Lac des Iles Pd mine, Ontario. From Barnett & Averill (in press).

sample, as shown for the four sample batches in Table 3. In most rocks, pyrite is at least 500 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, despite weathering conditions that are more severe than in Canada, the degree of chalcopyrite survival is sufficient to detect sulphide mineralization having a significant Cu component.

Till sampling near eight widely scattered PGE occurrences in Ontario, including Lac des Iles in the northwest and Broken Hammer near Sudbury, 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 anti-

monides, 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. 11) 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.

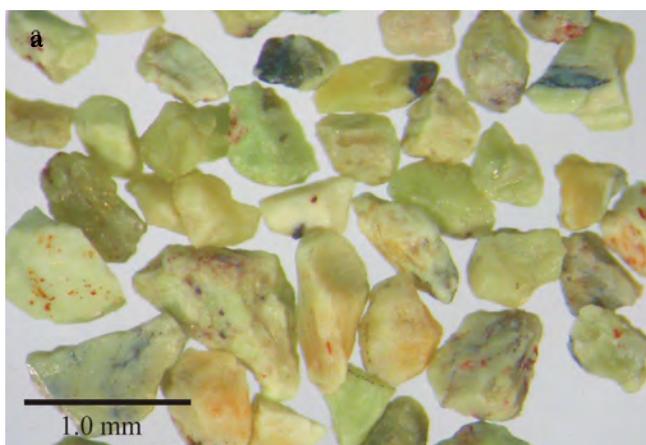
#### PORPHYRY Cu INDICATOR MINERALS

Porphyry Cu indicator mineralogy (PCIM®)<sup>1</sup> has emerged as a significant exploration tool only in the last seven years (Averill 2007a). The initial focus was on porphyry deposits in arid regions, such as the Atacama Desert in Chile where the climate has stabilized primary sulphide mineralization by converting it 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

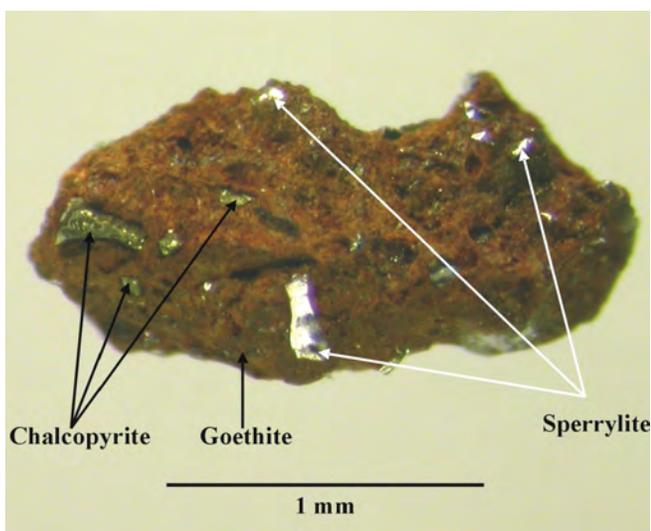
1. PCIM is a registered trademark of Overburden Drilling Management Limited.

**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 (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 Cr<sub>2</sub>O<sub>3</sub> content and with decreasing distance from the chromitite bands. From Barnett & Averill (in press).



**Fig. 11.** 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.

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 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 (0.25-2.0 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 collection and shipping; just one sample/km<sup>2</sup> has proven sufficient to identify and outline significant porphyry Cu and epithermal Au systems (Fig. 12). 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 (S.G.) of 2.8 to 3.2] and heavy (S.G. >3.2) mineral concentrate.

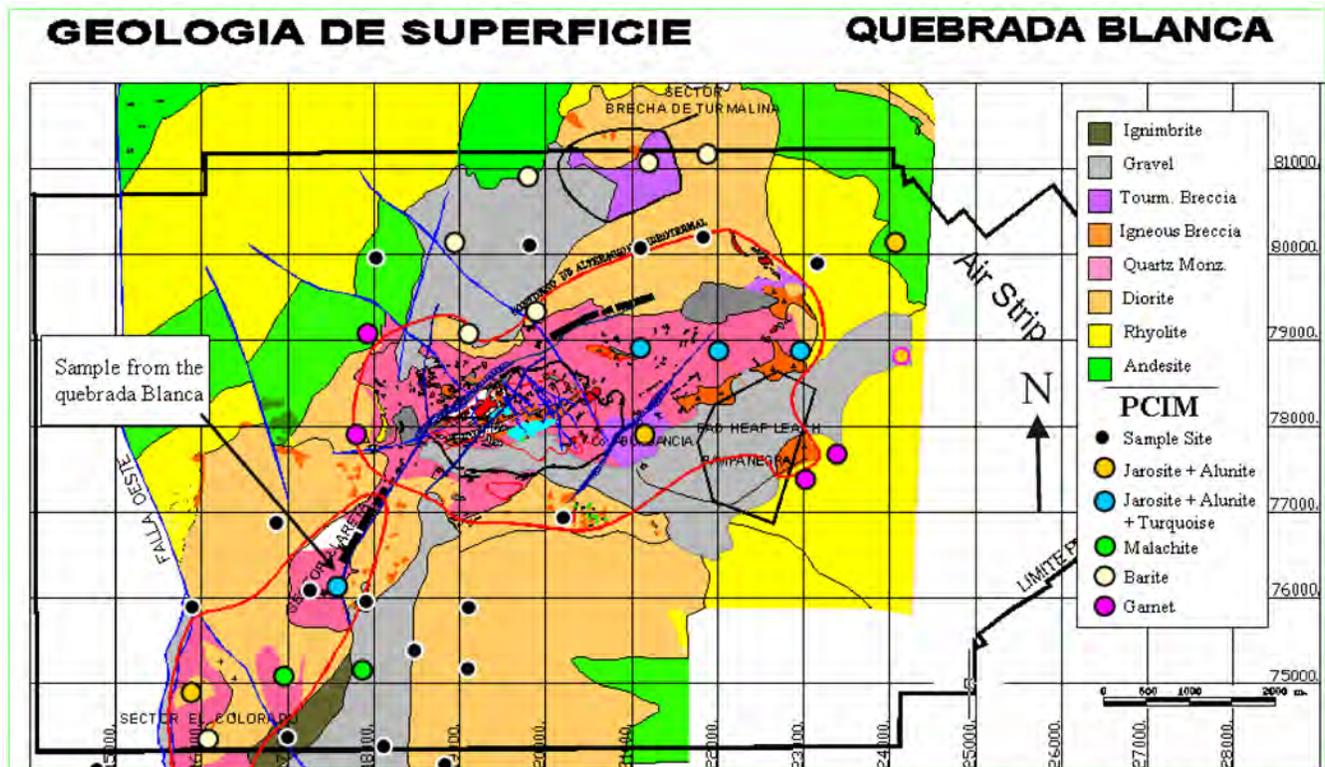


Fig. 12. Distribution of porphyry Cu indicator minerals (PCIMs) in weathered alluvium near the Quebrada Blanca deposit, Chile. Courtesy of Aur Resources Inc.

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), primary 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 less than 1 to approximately 20 m. Aur collected 38 samples at approximately 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. 12) clearly outline the outer propylitic (andradite garnet) and more central advanced argillic/potassic (jarosite, alunite, and turquoise) alteration zones and, at higher elevations, suggest a change to epithermal (barite) alteration.

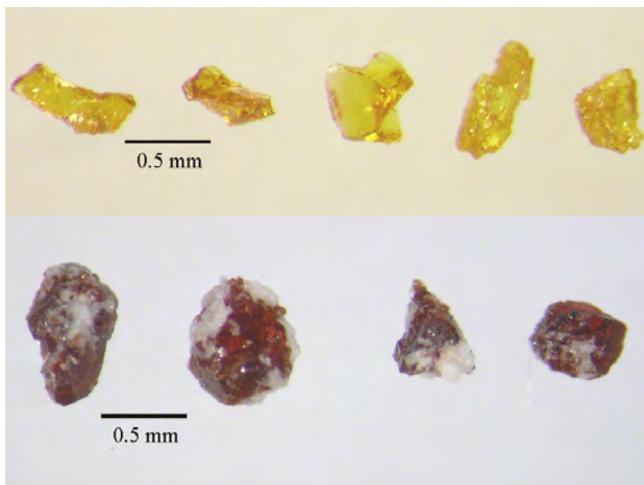
Andradite garnet, together with its grossular and spessartine variants, appears to be the “holy grail” of PCIMs. Anomalous concentrations of this mineral have been encountered in the

surficial sediments at every porphyry Cu deposit tested to date, even though garnet alteration has only been reported in bedrock at a few deposits, such as Escondida (Padilla Garza et al. 2001). Andradite forms the largest dispersal anomaly at most porphyry deposits, including Quebrada Blanca (Fig. 12), 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. 13) 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 kilo-



**Fig. 13.** 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.

metres 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, which is 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 is 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 tighter sample spacing is required.

Porphyry Cu indicators have been used for only seven 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 km<sup>2</sup> appears to

provide very effective exploration coverage. The ten indicator mineral species presently proven for arid regions can 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 and 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, and 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? The long-term need for large quantities of copper to further grow the emerging economies of China, India, and other populous countries suggests that the answers to such questions are of more than academic interest.

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# Introduced particles in indicator mineral processing: Considerations in preparing test samples for QA/QC and examples of anthropogenic contamination

Michael D. J. Michaud & Stuart A. Averill

Overburden Drilling Management Limited, 107-15 Capella Court, Nepean, Ontario K2E 7X1  
(e-mail: odm@storm.ca)

## INTRODUCTION

Heavy mineral laboratories that process till and other sediment samples for indicator minerals can expect to encounter particles that are not native to the samples. These introduced particles are of two main types: 1) spiked grains that have been added to the samples to check laboratory recovery rates; and 2) mining, industrial, and other anthropogenic contaminants, primarily in samples collected in settled areas. This paper discusses the considerations involved in designing an effective spiking program and provides examples of anthropogenic contamination that can negatively impact mineral exploration programs and thus must be recognized and reported by the sample processing laboratory.

### PART 1. CONSIDERATIONS IN PREPARING TEST SAMPLES FOR QUALITY ASSURANCE/QUALITY CONTROL

Quality Assurance/Quality Control (QA/QC) testing of an indicator mineral laboratory is typically performed using blank, duplicate, and/or spiked samples. Although blank samples can identify carry-over problems, they provide no recovery data. Due to the natural heterogeneity of till and other sediments, duplicate samples can only measure recovery rates on a qualitative basis. The processing of samples carefully spiked with indicator minerals is the only reliable method of quantitatively measuring indicator mineral recovery rates. Since 1999, Overburden Drilling Management Ltd. (ODM) has conducted seven extensive, blind, internal spike tests to measure kimberlite indicator mineral (KIM) recovery in its laboratory (Averill & Huneault 2003). Each test was consisted of exactly 300 spiked grains inserted throughout 10 base samples, which were processed intermittently within projects with more than 200 samples over a period of three to six months. One of the most enlightening lessons of the tests was the degree of care needed to prepare the spiked samples to permit a high degree of accuracy in determining recovery rates.

The first three internal tests used six of the seven, best known KIM species: 1) purple to red Cr-pyrope garnet (GP); 2) orange Cr-poor pyrope garnet (GO); 3) Cr-diopside (DC); 4) Mg-ilmenite (IM); 5) chromite (CR), and 6) forsteritic olivine (FO). The seventh mineral, orange eclogitic pyrope-almandine garnet, was not used because it is too similar visually to Cr-poor pyrope, requiring differentiation by scanning electron microscope (SEM) or electron microprobe analysis. It is slightly heavier than Cr-poor pyrope (specific gravity of 3.9 versus 3.7; Table 1) and is assumed to be at least as recoverable because gravity processing is the principal means of KIM extraction. In the subsequent tests, only three KIM species — Cr-pyrope, Cr-diopside, chromite — were used. These three

Table 1. Relative densities of kimberlite indicator minerals.

Mineral	Symbol	Specific Gravity
Forsterite	FO	3.25
Cr-diopside	DC	3.3
Cr-poor pyrope	GO	3.7
Cr-pyrope	GP	3.8
Pyrope-almandine	GO	3.9
Mg-ilmenite	IM	4.7
Chromite	CR	5.1

species are easier to recognize than the other KIMs and span the specific gravity range of all KIM species (Table 1).

In any gravity concentrating process, recovery is governed by particle size (Stokes= Law) and shape, in addition to mineral density (Fig. 1). Similarly, electromagnetic separations, which are sometimes employed in conjunction with gravity separations, are influenced by particle size and density. As a result, samples are sized at various stages to suit the specific equipment and techniques used by the laboratory. Since the size of most KIMs is naturally between 0.25 and 2.0 mm (Averill 2001), populations of spiked grains from at least the 0.25-0.5 and 0.5-1.0 mm fractions are required to adequately test all of the processing circuits of a laboratory. A common error made by clients is to prepare test samples with only one size range of KIMs, for example 0.5-1.0 mm grains may be chosen because large grains are easier to see and handle.

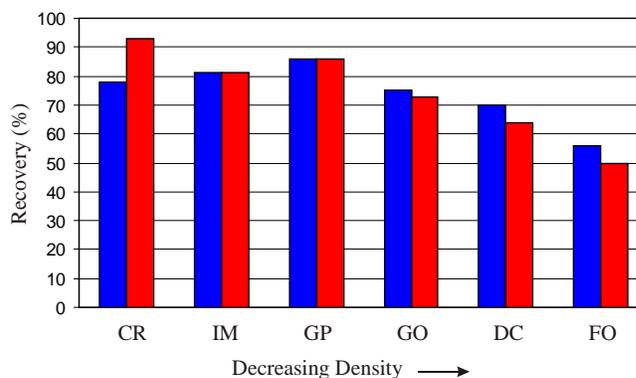


Fig. 1. Relationship between KIM density, particle size, and recovery for 0.25-0.5 (blue) and 0.5-1.0 mm (red) grains. Ten spiked till samples were tested and the concentrates were obtained by tabling, followed by heavy liquid separation. Tabling is more efficient for 0.25-0.5 mm grains and heavy liquid separation for 0.5-1.0 mm grains, the net result being slightly better recovery of 0.25-0.5 mm grains. At both sizes, recovery improves with increasing density. Modified from Averill & Huneault (2003). CR = chromite, DC = Cr-diopside, FO = forsteritic olivine, GO = Cr-poor pyrope garnet, GP = Cr-pyrope garnet, IM = Mg-ilmenite.



**Fig. 2.** Typical density tracers used in dense media separation circuits. The different colours represent different densities. Courtesy of Bateman Engineering N.V.

For the ODM tests, the KIMs were subjected to a detailed examination prior to selection. The spiked KIMs were natural, transported grains extracted from anomalous sediments, not fresh grains milled from kimberlite. Grains with dimensions too close to the upper and lower boundaries of their respective size fractions were avoided to ensure that they reported to the correct size fraction. Grains included both typical and atypical specimens; however, structurally weak grains that could break during processing were avoided. Slightly magnetic ilmenites were not chosen, to ensure they would not be removed during the ferromagnetic separation. Only unequivocal KIMs were used, with the composition of potentially ambiguous grains confirmed by scanning electron microscope (SEM) analysis. Alternatively, laser-etched KIMs could have been used but at a considerably higher cost.

Some clients prepare their own spiked samples for blind tests. Common shortcomings encountered include 1) using KIM grains freshly milled from kimberlite rather than natural grains from anomalous sediments; 2) not verifying the composition of KIMs before adding them to the sample (e.g. kimberlite may contain orange almandine, and grossular and spessartine garnet derived from metamorphic rocks, in addition to the mantle-derived orange pyrope and pyrope-almandine garnet that are used as KIMs); and 3) adding an unnatural number, size, or variety of KIM grains. In a few instances, the client even forgot to add the intended KIMs to the sample or added them to a different sample. Synthetic density tracers of various densities and sizes (Fig. 2) are commonly used to monitor the efficiency of primary, large-scale dense media separations (DMSs). They are not an effective substitute for KIMs because 1) they are of a cubic to tabular shape whereas most KIMs have a rounded form; 2) if magnetic, they are removed during the ferromagnetic separation and if nonmagnetic, they contain Pb but are variably paramagnetic; and 3) solvents such as acetone resorb their surfaces changing their shape to spherical and therefore affect their size.

To obtain meaningful test results, just as much care is needed in selecting the base material for the test samples. It is essential that the base be a natural sample consisting of minerals with a broad range of specific gravities, i.e., not low-density quartz sand, because the efficiency of the separation of heavy from light minerals in all gravity-based devices is directly proportional to the density contrast between the minerals (Pryor 1965). A natural base of glacial till or alluvial or glaciofluvial sand and gravel should be used. In ODM's early tests, as well

**Table 2.** Relative densities of some common base metal indicator minerals. The natural size range of these minerals is the same as kimberlite indicator minerals (0.25-2.0 mm; Averill 2001) and three minerals (forsterite olivine, Cr-diopside, and chromite) double as KIMs.

Mineral	Specific Gravity
Alunite	2.8
Apatite	3.2
Jarosite	3.2
Forsterite	3.25
Sillimanite	3.3
Cr-diopside	3.3
Kyanite	3.6
Staurolite	3.7
Chalcopyrite	4.2
Gahnite	4.6
Pyrite	5.0
Chromite	5.1
Arsenopyrite	6.0

as those from client-prepared spikes, two problems were encountered when natural samples were used. The first problem was the presence of an excessive amount of heavy minerals in some samples, making identification of spiked KIMs very time consuming and difficult due to the large volume of heavy mineral concentrate (HMC) obtained. The second problem was the presence of natural populations of low-Cr diopside, chromite, and forsterite from non-kimberlitic mafic, ultramafic, or metamorphic rocks, which often resulted in misleading recovery rates of more than 100 percent. These "pseudoKIMs" are difficult to distinguish visually or chemically from their kimberlitic counterparts, especially from well travelled KIMs that have completely lost their distinctive alteration mantles and resorption textures. To eliminate these problems, ODM switched to using only previously processed samples as spike test bases. A sample is acceptable for use as a spike base if 1) the weight and mineralogy of the heavy mineral fraction matches that of the project samples; 2) the HMC is essentially KIM- and pseudoKIM-free; and 3) *all* sample fractions (with the exception of the original indicator minerals) are available to be recombined. Prior to recombining the sample fractions, the HMC should be logged again to determine if any KIMs or pseudoKIMs were previously missed.

Most labs, including ODM, remove a representative archival split or subsample from every sample at the beginning of the processing circuit. Clients preparing their own spikes should carefully blend the KIMs into the entire sample to ensure that the laboratory's archival split does not contain a disproportionate number of grains. Simply adding the grains to the top of the bag will bias the results. It may also compound any losses that occur due to accidental spillage or damage to the sample bag or container. Upon completion of the processing, the client should ask that the archival split be processed to determine the number of contained grains.

Indicator mineralogy has evolved into an effective tool for base metal exploration (Averill 2001, 2007; Coker 2003). The above guidelines for KIM spiking can easily be adapted to develop spike tests for base metal indicator programs since 1) the size (0.25-2.0 mm) and specific gravity range (Table 2) of base metal indicators are the same as those of KIMs (Averill 2001); and 2) three KIMs (forsteritic olivine, Cr-diopside, and chromite) are also important base metal indicators.

## PART 2. EXAMPLES OF ANTHROPOGENIC CONTAMINATION

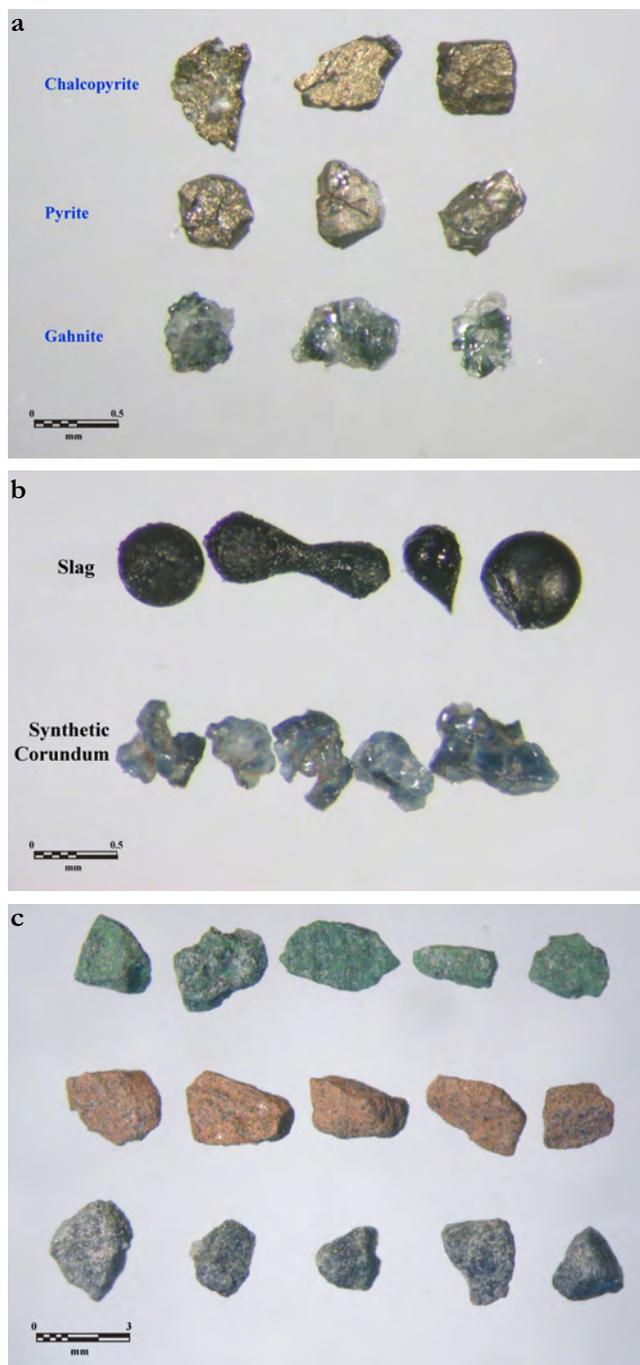
Present-day mineral exploration takes place not only in remote areas but also in areas of current and historical mining and industrial activity. Cultural features (roads, railways, bridges, smelters, tailings ponds, etc.) may introduce anthropogenic contamination which, dependent on the contaminant type, 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) document a heavy mineral sampling survey in the area of the closed Winston Lake and Zenmac zinc mines near Schreiber, Ontario. Sediment samples taken in stream drainages were highly anomalous in metamorphosed massive sulphide indicator minerals (MMSIM®) sphalerite, chalcopyrite, pyrite, galena, and gahnite (Fig. 3a), which is a Zn-bearing spinel that occurs in the alteration haloes of metamorphosed volcanogenic massive sulphide deposits. The entire populations of chalcopyrite and pyrite are angular, have milling stress cracks and are very fresh, which is unusual because sulphides are extremely susceptible to weathering in the surface environment. Some of these grains also have chemically leached surfaces. The gahnite is very fresh and angular, and is always attached to quartz, which is again characteristic of liberation by milling rather than glaciation. The results of this survey were properly interpreted as contamination because the authors were aware of the presence of the mine dumps, 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. 3b), a byproduct of metal smelting commonly used as railbed ballast, and lesser amounts of aluminum oxide (synthetic corundum; Fig. 3b), an abrasive commonly used to sandblast rusted steel. The source of the contamination was determined to be railroad beds and steel 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 historical 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 epidote (roofing granules used in the production of asphalt shingles; Fig. 3c), 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 zaraitite, 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 tailings pond. Similarly, a large Pd-in-humus anomaly, identified by Hattori & Cameron (2004), near the Lac des Iles Pd



**Fig. 3.** 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.

mine in northwestern Ontario was subsequently shown by Barnett & Dyer (2005) to be due to wind-blown dust from the open-pit mine operation. Although humus is not sampled in indicator mineral surveys, contamination of underlying sediments may result if the sediments are exposed or if humus is accidentally incorporated in the sample.

## CONCLUSION

Indicator mineral spike tests can be useful for measuring laboratory recovery rates but designing an effective test

requires knowledge of the physical laws governing mineral separations. In addition, due care must be exercised in choosing the indicator mineral grains and base samples used in the tests, and in placing the grains in the base samples. However, high laboratory recovery rates are only of limited value if the project samples are of poor quality or significantly contaminated. When sampling in settled areas, adjustments must be made to minimize the potential for introducing anthropogenic contamination. The indicator mineral laboratory must also identify and report any contaminants present in the samples.

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# Heavy mineral signature of the NICO Co-Au-Bi deposit, Great Bear magmatic zone, Northwest Territories, Canada

I. McMartin<sup>1</sup>, L. Corriveau<sup>2</sup>, G. Beaudoin<sup>3</sup>, S. Averill<sup>4</sup> & I. Kjarsgaard<sup>5</sup>

1. Geological Survey of Canada, 601 Booth Street, Ottawa, Ontario K1A 0E8

2. Geological Survey of Canada, 490 de la Couronne, Québec, Québec G1K 9A9

3. Department of Geology and Geological Engineering, Laval University, Québec, Québec G1K 7P4

4. Overburden Drilling Management Limited, 07-15 Capella Court, Nepean, Ontario K2E 7X1

5. Mineralogical Consultant, 15 Scotia Place, Ottawa, Ontario K1S 0W2

(e-mail: imcmarti@nrcan.gc.ca)

## INTRODUCTION

Iron oxide copper-gold (IOCG) deposits can host significant resources in base, precious, and strategic metals, as well as energy, but due to the extraordinary range of deposits and intrinsic complexity, they present one of the most challenging targets for explorers and researchers in economic geology (Corriveau 2007; Corriveau & Mumin 2009). The Great Bear magmatic zone (GBMZ) in the Northwest Territories is now considered the most prospective setting for IOCG deposits in Canada. It includes two economic IOCG deposits: the magnetite-group IOCG NICO Co-Au-Bi deposit, scheduled to commence production in 2011, and the nearby hematite-group IOCG Sue-Dianne Cu-Ag-Au deposit (Fig. 1). Moreover, many past-producing vein-type U, Ag, and Cu mines and Kiruna-type showings are now recognized to be parts of large polymetallic IOCG systems (Mumin *et al.* 2009). Despite past and current exploration success along the belt, the GBMZ remains largely unexplored and unmapped because local geological exploration models are immature and key regional and local indicators of ore have not been identified. As part of a joint government-industry-academia research project taking place under the government Targeted Geoscience Initiative 3 (TGI-3), Geo-mapping for Energy and Minerals (GEM), and Strategic Investments in Northern Economic Development (SINED) program umbrellas (Corriveau *et al.* 2007), the Co-Au-Bi NICO deposit in the GBMZ was selected as a test site to characterize the heavy mineral and geochemical signature of IOCG±U deposits and derived glacial sediments and assess if some heavy minerals have a potential as indicator minerals for IOCG deposits in glaciated terrain (McMartin *et al.* 2008). At NICO, mineralized zones and alkali-altered host rocks are exposed, ore mineralogy is well known, and active resource evaluation by Fortune Minerals Ltd. provides logistical support in an otherwise remote, poorly accessible area (Corriveau *et al.* 2007). The mineralogy and chemistry of heavy minerals from bedrock and C-horizon till samples collected in 2007, over and down-ice from mineralization and host rocks (barren), and up-ice in background terrain are presented here.

## REGIONAL SETTING

The NICO deposit is located at the south end of the GBMZ in the Proterozoic Bear Structural Province of the Canadian Shield, about 160 km northwest of Yellowknife, Northwest Territories (Fig. 1). It represents an economically significant source of Co-Au-Bi-Cu-Fe with calculated reserves of 21.8 Mt with 1.08 g/t Au, 0.13% Co, and 0.16% Bi (Fortune Minerals,

2008). Mineralization at NICO consists of a number of showings predominantly hosted in brecciated and altered siltstone and wacke of the ca. 1.88 Ga Treasure Lake Group, overlain by felsic ignimbrite sheets of the Faber Group (Goad *et al.* 2000a,b; Ghandi & van Breeman 2005; Fig. 2). Minor mineralization extends into the overlying volcanic rocks and subvolcanic porphyry dykes. Ore mineralogy mainly includes Fe-, As-, Co-, and Cu-sulphides, native Au, and Bi. The NICO deposit is now considered a member of the IOCG family because of features such as abundant iron oxides, hydrothermal mineralization, Cu and/or Au as the primary economic metals, and regional- to deposit-scale intense K and Fe alteration (Ghandi *et al.* 1996; Goad *et al.* 2000a; Corriveau *et al.* 2009; Williams 2009). Intense, pervasive, polyphase iron-oxide (magnetite-dominant)-hornblende-biotite-tourmaline-K-feldspar-carbonate replacive alteration occurs in the upper metasedimentary sequence below the volcanic-sedimentary unconformity (Mumin *et al.* 2009). Namely polyphase and stratabound magnetite-amphibole alteration is pervasive and can be fairly cryptic (Corriveau *et al.* 2009).

Sedimentary rocks of the Treasure Lake Group and overlying volcanic sequence generally form prominent bedrock ridges striking southeast-northwest with relief exceeding 100 m and are dominated by exposed bedrock and thin, discontinuous till (<2 m). Great Bear intrusive rocks form more poorly drained lowlands covered by generally thin till. Striations, roches moutonnées, and crescentic fractures indicate the area was influenced primarily by the Laurentide Ice Sheet flowing to the west-southwest during the last Wisconsinan glaciation, from an ice mass centred in Keewatin (McMartin *et al.* 2008). Pebble lithology data show that surface till in the area is locally derived and its composition reflects local provenance of the underlying bedrock (McMartin *et al.* 2008). Late during deglaciation (ca. 8.5-10.5 ka BP, Dyke 2004), Glacial Lake McConnell occupied the area as a result of glacioisostatic depression reversing the regional drainage in the Great Bear, Great Slave, and Athabasca lake basins (e.g. Lemmen *et al.* 1994; Smith 1994). Evidence for reworking of glacial sediments by glaciolacustrine processes, to varying degrees, is present at all elevations as veneers of silt and clay in topographic depressions, namely around Lou Lake and other lake basins, or as veneers of winnowed till and littoral sands overlying the glacial deposits. The area is underlain by extensive discontinuous permafrost with mixed deciduous and conifer open forest vegetation. Brunosolic static cryosols are prevalent in glacial sediments.

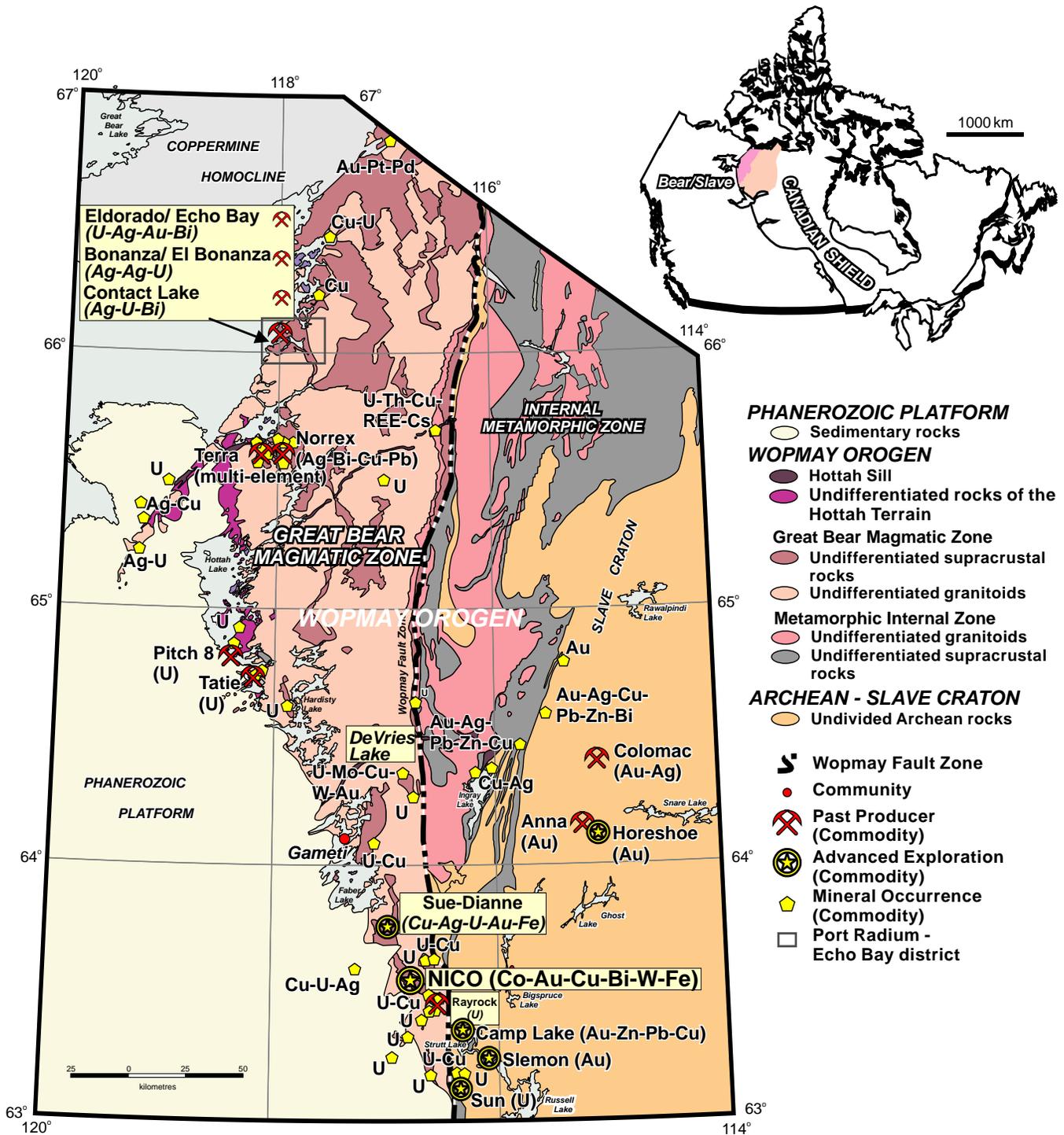


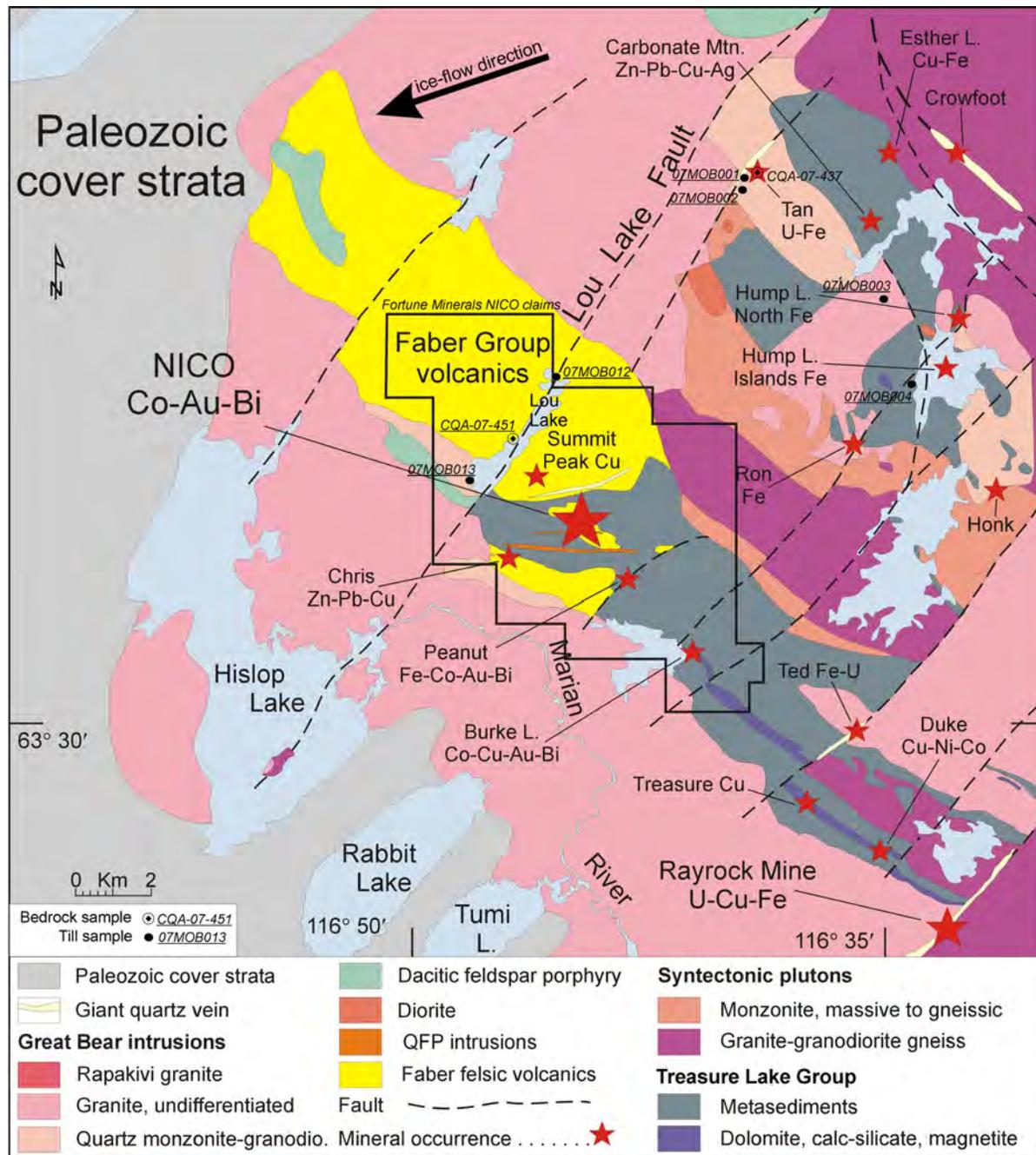
Fig. 1. The Great Bear magmatic zone (GBMZ) geology and mineral showings, prospects, and past-producing mines (modified from Corriveau *et al.* 2007). The NICO deposit is located in the southern part of the GBMZ. Inset map locates the Wopmay Orogen (Bear Province) and adjacent Slave Craton at the western end of the Canadian Shield.

**METHODS**

**Field procedures**

A total of thirteen 5-12 kg till samples were collected from twelve sites in the vicinity of the NICO deposit at the end of May 2007 (Figs. 2, 3). The samples were collected from hand dug pits in the upper C horizon, at an average depth of 50 cm, to obtain relatively unaltered parent material. Permafrost, or sometimes bedrock, was encountered at the bottom of most

holes. Three samples were collected over or immediately down-ice (<100 m) from known mineralized zones (07MOB006: #3 Zone; 07MOB005: #2 and #25 Zones; 07MOB007: Discovery outcrop). On the northwest side of the Bowl Zone, one section exposing a glacial diamicton in a gravel pit was sampled above (07MOB009: 180 cm depth) and below (07MOB010: 350 cm depth) the zone of oxidation to evaluate the effects of surface weathering on till composition. Four till samples were collected at different sites offset from mineral-



**Fig. 2.** Mineral occurrences and regional geology of the NICO deposit area (modified from Goad *et al.* 2000b). Sample site locations are shown (see Table 1 for bedrock sample numbers collected at till sampling sites). Detailed sampling closer to the NICO deposit is shown on Figure 3.

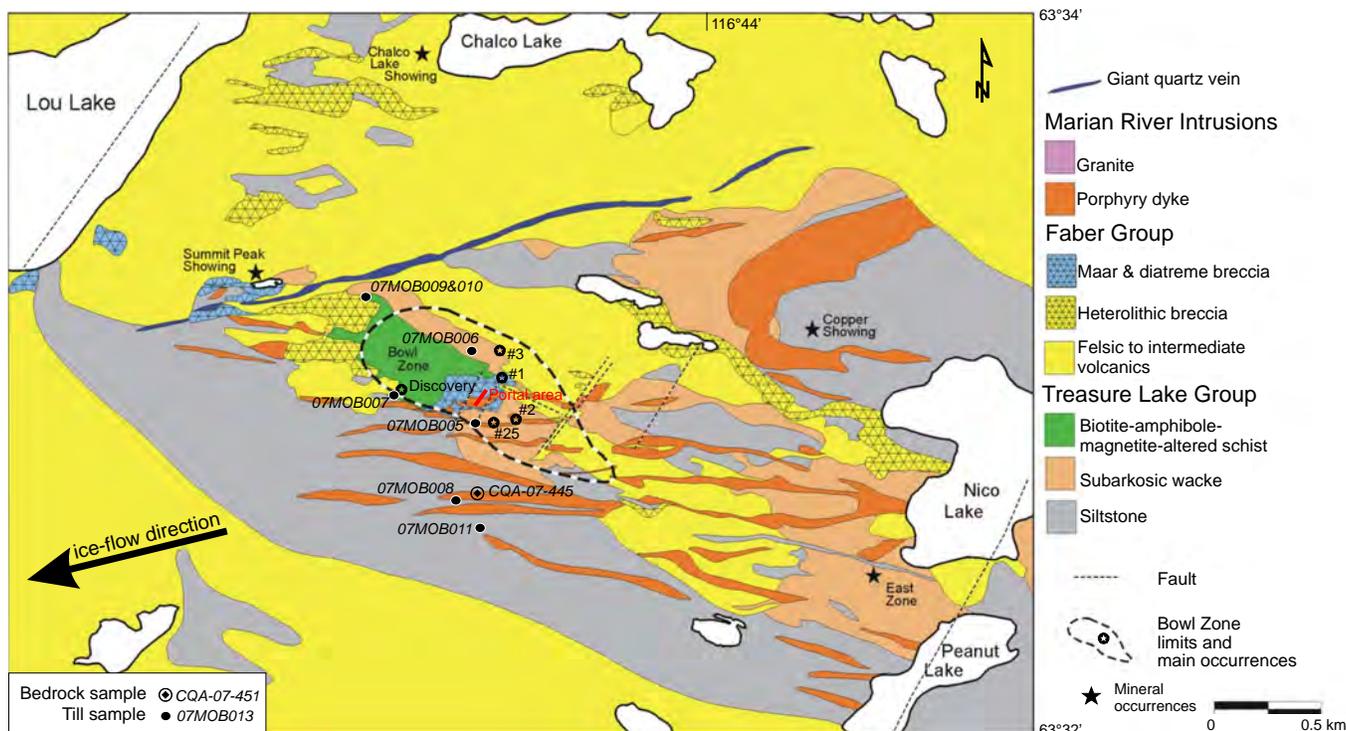
ization (barren): two samples along Lou Lake over weakly altered porphyritic rocks of the Faber Group (07MOB012) and monzonite of the Great Bear intrusions (07MOB013); and two samples closer to mineralization over magnetite-altered metasediments and porphyry (07MOB008 and 011). Four till samples were collected approximately 10 km up-ice (NE) from the NICO deposit area (background), over weakly altered metasedimentary and porphyritic volcanic rocks of the Treasure Lake and Faber groups (07MOB001 to 004). One of these sites is located immediately down-ice from the Tan U-Fe showing (07MOB001).

Twenty-seven representative 0.5 to 2 kg bedrock samples were collected for indicator mineral recovery at most drift sam-

pling sites ( $n=14$ ), in the vicinity of and at nearby surface occurrences ( $n=9$ ), and from the subsurface “Portal” area at various bulk sample stock piles ( $n=4$ ) (see Figs. 2, 3). Bedrock samples included both mineralized and unmineralized host rocks and some regional rocks to determine what heavy minerals the regional and local rocks are shedding and to establish the mineralogical background (Table 1).

#### Analytical procedures

Bedrock samples were examined at Overburden Drilling Management (ODM) and described carefully prior to crushing and processing. The unmineralized or weakly mineralized samples were milled to  $<2$  mm at Als Chemex in Val d’Or and the



**Fig. 3.** Mineral occurrences and detailed geology of the NICO deposit area (modified from Goad *et al.* 2000b). Sample site locations are shown. Bedrock samples collected at till sampling sites and at mineral occurrences are listed in Table 1.

mineralized rocks were milled at ODM. All crushed bedrock samples were processed at ODM for heavy mineral separation, indicator mineral picking, and gold grain counts. Samples were crushed and processed in order of increasing visible mineralization to minimize the potential for indicator mineral carry-over between samples. Crushed quartz vein samples (i.e. mineral blanks) were inserted between samples to evaluate cross-sample contamination and/or contamination from laboratory equipment. The crushed bedrock fraction was pre-concentrated with respect to density using a shaker table (two runs). Visible gold grains recovered from this table concentrate and by subsequent panning were counted and their morphology noted before being returned to the table concentrate. Methylene iodide (S.G. 3.2 g/cm<sup>3</sup>) was used to produce a heavy mineral concentrate (HMC) from the table concentrate. After ferromagnetic separation, the non-ferromagnetic HMCs (NFM-HMCs) were sieved to obtain the sand fraction (0.18–2 mm) for picking. The ferromagnetic fraction was sieved (0.25–2 mm) and was stored for further analysis.

Till samples were processed at ODM for heavy mineral analysis, using a method similar to that used for the bedrock samples. However, instead of milling, till samples were disaggregated and sieved to obtain the <2 mm (matrix) fraction prior to two shaking table runs; blank samples were not inserted. After heavy liquid and ferromagnetic separation, the NFM-HMCs in till were sieved to obtain the 0.25–2 mm fraction. Only half of this fraction was picked for indicator minerals (the other half was used for HMC geochemistry). The light-weight (S.G.<3.2) and heavy finer sand fraction (0.063–0.25 mm) will be examined and minerals described and counted to evaluate the presence of finer grained and/or mid-weight indicator minerals in till. Till samples were also analyzed for near-total and partial geochemistry, total/organic carbon content, textural analysis, and pebble lithology (McMartin *et al.* 2008).

Prior to indicator mineral picking, the NFM-HMCs recovered from till and bedrock samples were sieved to 0.25–0.50 mm, 0.5–1 mm, and 1–2 mm. In the bedrock samples, only the 0.25–0.5 mm fraction was examined as it consisted of individual mineral grains; the 0.5–1 and 1–2 mm fractions consisted of impure lithic fragments containing the same heavy minerals. A finer grained, 0.18–0.25 mm concentrate was prepared as a backup for the bedrock concentrates but did not need to be used. The 0.25–0.5 mm fraction was further sorted with an electromagnetic separator into fractions with different paramagnetic characteristics to reduce the volume to be examined (Averill & Huneault 2006). All fractions were examined under a stereoscopic microscope at ODM for metamorphosed or magmatic massive sulphide indicator minerals, kimberlite indicator minerals (KIMs), and potential IOCG indicator minerals; scanning electron microscope (SEM) checks were performed on selected grains by energy dispersive x-ray spectrometer (EDS). Grains considered to have possible IOCG affinities were hand picked. These grains included mainly sulphides (arsenopyrite, bismuthinite, chalcopyrite, molybdenite) and silicates (tourmaline, actinolite). Because of their abundance in some bedrock samples, no more than 20 to 40 representative grains of the same species were picked per sample. In the till samples, any other unusual mineral plus KIMs were also hand-picked.

A total of 94 grains from the till samples (1 lost) and 532 grains from the bedrock samples (1 lost) were mounted on 25 mm epoxy-impregnated stubs at SGS Lakefield, sorted by grain size, mineral species, and sample number, and polished. The grains were then probed to confirm their identity and quantify their chemical composition. The electron microprobe analyses were conducted at the Geological Survey of Canada Microbeam laboratory using a CAMECA SX50 electron microprobe equipped with four wavelength-dispersive spec-

**Table 1.** Summary of bedrock samples.

Sample ID	Location	Nearby till sample	Lithology
CQA-07 228A	Discovery outcrop	07MOB007	Biotite-amphibole-magnetite alteration of Treasure Lake sediment
CQA-07 230D	#1 Zone	-	Hydrothermal breccia with amphibole-biotite matrix and K-feldspar-altered fragment
CQA-07 436C	near Tan showing	07MOB001	Weakly magnetite-altered metasediment
CQA-07 437A	Tan showing	-	Weakly albite-magnetite-altered metasediment
CQA-07 438C	near Tan showing	07MOB002	K-feldspar-magnetite-altered porphyritic andesite
CQA-07 438D	near Tan showing	07MOB002	coarse-grained granitoid
CQA-07 439A	Crow Foot Lake area	07MOB003	Weakly altered porphyritic rhyodacite
CQA-07 440A	Hump Lake	07MOB004	Least altered porphyritic dacite
CQA-07 441A	#2 Zone	-	Mineralized metasediment with disseminated arsenopyrite
CQA-07 441B	#2 Zone	-	K-feldspar-altered, very fine-grained, massive, chilled margin of porphyry
CQA-07 441D	#2 Zone	-	Quartz-paragonite porphyry
CQA-07 442A	near #25 Zone	07MOB005	Weakly mineralized quartz porphyry with disseminated arsenopyrite and chalcopyrite
CQA-07 442D	near #25 Zone	07MOB005	Mineralized amphibole-altered metasediment with arsenopyrite
CQA-07 443A	#25 Zone	-	Mineralized amphibole-altered metasediment with cobalt-arsenopyrite
CQA-07 443B	#25 Zone	-	Altered metasediment intersected by cobaltian magnetite and chalcopyrite veins
CQA-07 445A	end of "Portal A" road	07MOB008	Magnetite-altered metasediment
CQA-07 445B	end of "Portal A" road	07MOB008	Porphyry
CQA-07 445C	end of "Portal A" road	07MOB008	Tourmaline-filled crackle breccia in weakly magnetite-altered quartz-paragonite porphyry
CQA-07 447A	East Zone	-	Weakly mineralized amphibole-biotite-magnetite-altered metasediment with arsenopyrite
CQA-07 448A	northeast end of Lou Lake	07MOB012	Weakly magnetite-altered porphyry
CQA-07 451A	Lou Lake centre	-	Specular hematite stonework in K-feldspar-hemitite-altered porphyry
CQA-07 452A	south end of Lou Lake	07MOB013	Hornblende-magnetite-bearing monzonite
CQA-07 458F	NICO Bulk Sample Stock Pile, 13-10-06 day shift	-	Amphibole-biotite-altered sediments with veins of quartz-amphibole-magnetite-magnetite-chalcopyrite(?)-bornite(?)
CQA-07 465A	NICO Bulk Sample Stock Pile, 08-03-07 night shift	-	Amphibole-biotite-magnetite-arsenopyrite-altered sediments
CQA-07 480Q	NICO Bulk Sample Stock Pile, 05-10-06 day shift	-	Arsenopyrite-tourmaline vein crosscut by magnetite-rich amphibole-biotite-altered sediments
CQA-07 482F	NICO Bulk Sample Stock Pile, 06-10-06 day shift	-	Titanite-epidote-chalcocite-amphibole-biotite-arsenopyrite vein in mineralized amphibole-biotite-magnetite-altered metasediment
CQA-07 487A2	#3 Zone	07MOB006	Weakly mineralized, phyllic altered porphyry

trometers. Operating conditions were 20 kV accelerating voltage, 10 nA beam current using a focused spot. Count times on peak were 10 seconds, with 5 seconds off-peak. The raw data were processed with the ZAF matrix correction. A mixture of natural and synthetic pure metals, simple oxides, and simple compounds were used as standards. The analysed grains were classified (or re-classified when necessary) on the basis of their chemical composition. Theoretical chemical compositions of mineral end-members (LeMaitre 1982) were used to calculate cut-off values (at approximately 50:50 mol %) for members of binary solid solution series. For minerals that contain substantial amounts of more than two end-members, the threshold values were lowered accordingly.

Grains from the 0.25-2 mm ferromagnetic fraction were picked randomly by SGS Lakefield for each sample. Five grains per till sample, for a total of 65, and ten grains per rock sample, for a total of 265, were mounted on 25 mm epoxy-impregnated stubs at SGS Lakefield. The grains were analyzed using Université Laval's CAMECA SX-100 5-WDS electron microprobe under a beam of 15 kV at 100 nA, using a range of natural and synthetic standards. After counting over the peak for 20 to 30s, background was measured on both sides for 10s at offset from the peak empirically determined to improve sensitivity. Matrix corrections were computed using CAMECA's implementation of the PAP method. These settings yield minimum detection limits as low as 20 ppm for elements such as K, Ca, Al, Si, Ti, and Mg, 50 ppm for Mn, Cr, and V, 200 ppm for Cu and Ni, and 400 ppm for Zn. The optimized analytical routine allows analysis of one spot in approximately 20 min. Of

the 65 till and 265 rock sample grains, only 46 and 210, respectively, were analyzed for minor and trace elements because some of the ferromagnetic fraction grains consisted of a ferromagnetic mineral inclusion too small for accurate analysis.

#### Quality Assurance / Quality Control

At ODM, most of the bedrock concentrates contained 5 to 15 grains of common heavy minerals, principally hornblende, almandine, and green epidote. These grains represent carryover from the much larger sediment samples processed in this lab that typically yield hundreds of thousands of these common species; therefore the carryover is minuscule. One arsenopyrite and two pyrite grains were found in the pre-batch quartz blank. Seven of the thirteen quartz blank samples processed at ODM yielded no heavy mineral grains >0.25 mm. Traces (1 to 11 grains) of 0.25-0.5 mm arsenopyrite and/or pyrite were found in 4 of the other 6 blank concentrates, indicating that crusher carryover at this particle size occurred but was negligible. Stronger and more consistent carryover (up to 1000 grains) of finer, silt-sized arsenopyrite was evident in the pan concentrates of the blank samples; however carryover of elemental arsenic is negligible due to the extremely small size of the grains.

Thirty hematite grains were found in the pre-batch quartz blank at Chemex and are presumed to represent crusher carryover from a previous client's samples. Otherwise, crusher carryover at Chemex was negligible with the exception that five blanks did contain one to four pyrite or arsenopyrite grains, suggesting that carryover at Chemex was negligible only

**Table 2.** Summary of heavy minerals in bedrock and till (S.G.>3.2; <2 mm).

Mineral specie	Bedrock	Till (from 50% of concentrate)
Arsenopyrite	16 out of 27 samples trace amounts to >60 000 grains 0.25-1 mm	None
Cobaltite	2 out of 27 samples 1 to 2 grains 0.25-0.5 mm	None
Bismuthinite	2 out of 27 samples inclosed in arsenopyrite <0.1mm	1 out of 13 samples 1 grain 0.25-0.5 mm
Chalcopyrite	8 out of 27 samples trace amounts to ~1000 grains 0.25-0.5 mm	1 out of 13 samples 8 grains 0.25-1 mm
Molybdenite	1 out of 27 samples up to 50 grains 0.25-0.5 mm	None
Pyrite	17 out of 27 samples trace amounts to ~5000 grains 0.25-0.5 mm	6 out of 13 samples trace amounts 0.25-0.5 mm
Tourmaline	3 out of 27 samples 150 to 650 grains <0.05mm (intergrown with quartz) or 0.25-0.5 mm	9 out of 13 samples trace amounts 0.25-0.5 mm
Ferroactinolite/ actinolite	7 out of 27 samples trace to minor amounts 0.25-0.5 mm	1 out of 13 samples >21 grains 0.25-0.5 mm
Gold	4 out of 27 samples 1 to 2 grains <125 µm Pristine shape	12 out of 13 samples 1 to 28 grains <50 µm Pristine to reshaped
Magnetite	All 0.01-35.20 g /table concentrate 0.25-2 mm individual grains, bedrock fragments, or inclusions in mineral grains	All 0.5-128.1 g /table concentrate 0.25-2 mm individual grains, bedrock fragments, or inclusions in mineral grains

Note: Numbers of grains/sample are not normalized.

because the NICO samples processed in this laboratory (unmineralized or weakly mineralized) contained insignificant levels of sulphides or other heavy minerals.

## HEAVY MINERAL SIGNATURE

### Sulphides

Six sulphide species were observed in the NFM-HMCs of bedrock samples: arsenopyrite >> pyrite > chalcopyrite > bismuthinite = molybdenite = cobaltite (Table 2). Arsenopyrite is the foremost As-bearing mineral observed in this fraction. Although arsenide minerals such as loellingite and cobaltite were observed in mineralized bedrock samples at NICO (e.g. Goad *et al.* 2000a), only one grain of loellingite (FeAs<sub>2</sub>) and three grains of cobaltite (CoAsS) were recovered in the NFM-HMCs, all from mineralized bedrock samples. Arsenopyrite occurs in 16 out of 27 bedrock samples in trace amounts in background samples up to about 60 000 grains/sample in mineralized metasediments collected at #25 Zone and from the subsurface Portal area. The grains are fresh and nonparamagnetic. A few grains are euhedral in shape and some grains contain Co, as confirmed by the SEM. The arsenopyrite sometimes contains inclusions of amphibole and bismuthinite (Fig. 4a). The grains also occur in large amounts in the pan concentrates but in the much finer fraction (25-150 µm). Arsenopyrite grains visibly do not survive post-glacial weathering; they are entirely absent in the till concentrates, even in those samples located directly down-ice from mineralized zones exceptionally enriched in arsenopyrite (e.g. #2 and #25 zones).

Bismuthinite grains occur in two bedrock samples, both collected from the subsurface Portal area. They are so fine grained (mostly silt-sized) that no individual grains are present in the 0.25-0.5 mm fraction. The grains mainly occur as small inclusions in larger arsenopyrite grains (Fig. 4a). They are also recovered in abundance in the same two samples from the pan concentrates. Bismuthinite is basically absent from the till samples except in one till sample collected at the south end of Lou Lake (07MOB013), where a single bismuthinite grain was recovered in the 0.25-0.5 mm fraction.

Chalcopyrite occurs in 8 out of the 27 bedrock samples at varied mineralization zones from the NICO deposit. It occurs generally in trace amounts (2- 4 grains) but samples were found with up to 400 grains at #3 Zone and 1000 grains in one of the subsurface Portal samples. The grains are fresh and nonparamagnetic in bedrock. About 20 grains of chalcopyrite were also observed in the pan concentrate (100 µm) of one bedrock sample collected at #3 Zone. In till, chalcopyrite occurs in only one sample (07MOB010) but in minor amounts (8 grains), mainly in the 0.25-0.5 mm fraction (Fig. 4b).

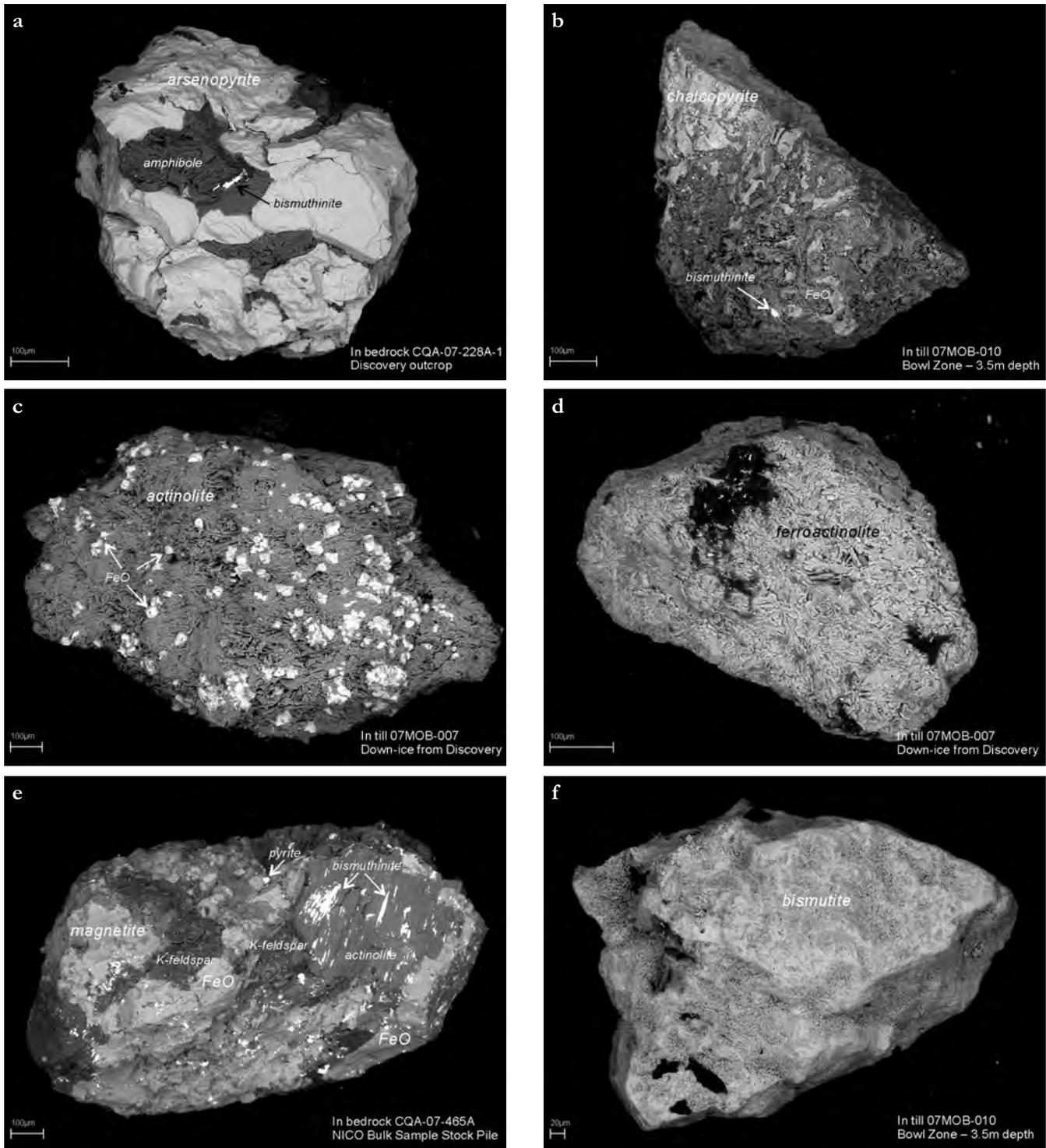
Fifty grains of molybdenite were counted in one bedrock sample collected from a bulk sample stock pile in the Portal subsurface area. Twenty-one of these grains were mounted for microprobe analysis. Molybdenite is completely absent from the till sample concentrates.

Pyrite is commonly absent or occurs mainly in trace to minor amounts in the bedrock samples. However it does occur in abundance (up to 5000 grains) in three bedrock samples collected at #3 and #25 zones and from the Portal area. Nevertheless, pyrite occurs only in trace amounts in a few till samples. Seven representative pyrite grains with minor Co from two mineralized bedrock samples were handpicked. Goethite, an alteration product of pyrite, was found in minor amounts (25 grains) in till sample #07MOB013. Being non-distinctive and non-diagnosis of IOCG deposits, goethite grains and the rest of the pyrite grains were not hand picked for microprobe analysis.

### Silicates

Tourmaline occurs in three bedrock samples in considerable amounts, varying from 150 to 650 grains/sample. In two of these samples, both from barren brecciated porphyry (end of Portal A road: CQA-07-445), tourmaline occurs as very small grains (<0.05 mm) intercalated with quartz so that few grains are heavier than S.G. 3.2 in the 0.25-0.5 mm sand fraction. The other sample is from the Tan U-Fe showing and contains a fair number (150 grains) of sand-sized tourmaline grains with Ti-magnetite inclusions. In till, tourmaline, which occurs in trace amounts and is not visually distinctive, is probably related to background concentrations found in Great Bear intrusive rocks or in Slave Craton rocks that lie 15 km east of NICO. One till sample collected near the Tan showing contains one distinctive elbaite tourmaline (picked for microprobe analysis).

Ferroactinolite grains are present in the HMCs of seven mineralized bedrock samples, mainly from the subsurface Portal area, but only in minor amounts. Based on the pre-processing binocular examination however, ferroactinolite was actually a major constituent of these same bedrock samples. Therefore the paucity of this mineral in the bedrock concentrates suggests that the grains are not sufficiently heavy to be concentrated in the HMCs except where they contain inclusions of heavier minerals such as magnetite (Fig. 4c). In such



**Fig. 4.** SEM photographs of mineral grains from bedrock and till samples collected in the NICO deposit area. **a**) Arsenopyrite grain with amphibole and bismuthinite inclusions, Discovery outcrop. **b**) Chalcopyrite grain from a till sample collected near the Bowl Zone below the oxidation zone. **c**) Magnetite grains occurring as small inclusions in an actinolite grain from a till sample collected near the Discovery outcrop. **d**) Subrounded ferroactinolite grain from a till sample collected near the Discovery outcrop. **e**) Magnetite grain from a subsurface bedrock sample occurring in association with a mixture of other minerals common to the mineralization zones. **f**) Bismutite grain in the unoxidized till sample collected at depth near the Bowl Zone. Scale bar is 100 µm in length except for (f) where it is 20 µm.

cases, ferroactinolite grains are recovered with the ferromagnetic fraction. In addition, ferroactinolite commonly occurs as small inclusions in arsenopyrite grains of several bedrock samples (cf. Fig. 4a). Ferroactinolite/actinolite are virtually absent

from till samples, with the exception of one sample collected near the Discovery outcrop that contains a relatively large number (>21) of grains in the 0.25-0.5 mm fraction (Fig. 4d).

### Other nonferromagnetic minerals

Other distinctive minerals were observed in minor amounts or only in a few samples. Allanite was found in minor amounts in one bedrock sample from #2 Zone. Scheelite was found in minor amounts in the two bismuthinite-bearing bedrock samples. The unoxidized till sample collected at depth near the Bowl Zone (07MOB010) yielded major (25% of concentrate) gedrite, an aluminous anthophyllite alteration mineral. Approximately 100 sand grains of bismutite, an oxidation product of bismuthinite or native bismuth, were also found in the same till sample (Fig. 4f). All of these mineral species were picked for microprobe analysis. Apatite was observed in trace amounts in various till samples but the grains are not distinctive and are probably related to background in Great Bear or Slave Craton intrusive rocks; hence they were not hand picked for microprobe analysis.

### Gold

Only five gold grains (<125  $\mu\text{m}$ ) were recovered from the entire suite of bedrock samples, suggesting that most of the gold is encapsulated in sulphide minerals and therefore is not recovered during the panning. Gold is known to occur as microscopic grains ranging from <1 to >100  $\mu\text{m}$  in size, typically as inclusions within sulphides, particularly cobaltian arsenopyrite, or attached to sulphide and telluride grain boundaries (Goad *et al.* 2000a,b). In contrast to bedrock, gold grains are relatively abundant in till, averaging 11 grains/10 kg, and up to 39 grains/10 kg in a till sample collected near #25 Zone. Pristine gold grains are abundant down-ice of #25 and #3 zones whereas none of the gold grains from background terrain (up-ice of NICO) are pristine in shape. High gold values were also found in the pulverized <2 mm HMC fraction of till analyzed geochemically by FA-MS (unpublished data), implying that some of the gold in till also occurs as inclusions in sulphides.

### Magnetite

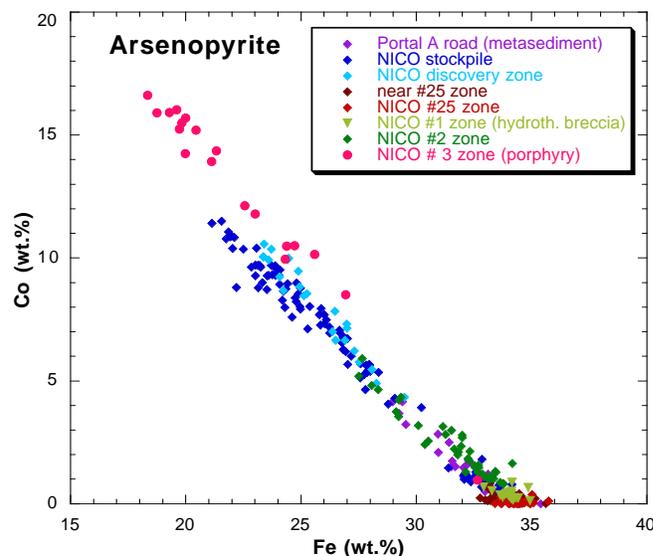
Magnetite occurs in all bedrock and till samples in varying concentrations. In bedrock, the ferromagnetic fraction (0.25–2 mm) is particularly abundant in a few samples collected from the Discovery outcrop and the East Zone and from two subsurface samples from the Portal area (up to 32% by weight of table concentrate). In till, magnetite is highly abundant in the sample collected down-ice from the Discovery outcrop, forming close to 9% by weight of the <2 mm table concentrate. In both bedrock and till, magnetite occurs as individual grains but also disseminated in bedrock fragments (Fig. 4e) or as inclusions in mineral grains (Fig. 4c). A few probed grains from the magnetite fraction were also reclassified as hematite.

## HEAVY MINERAL CHEMISTRY

### Nonferromagnetic heavy mineral concentrates

#### Sulphides

One of the most abundant minerals in this study is arsenopyrite (FeAsS) with 318 grains analyzed. Compositions range from pure FeAsS to  $\leq 17$  wt.% Co, trending towards cobaltite (CoAsS) end-member composition, with the highest Co values in NICO stockpile and Discovery zone metasediments and NICO #3 Zone porphyry (Fig. 5). The arsenopyrite data provided by Sidor (2000) for the NICO area show the same compositional variations as the data presented here: there is a high degree of substitution of Co for Fe, particularly in Au-rich



**Fig. 5.** Fe versus Co in 318 arsenopyrite grains. The Co  $\pm$  Ni substitution for Fe trends towards the cobaltite end-member composition (at approximately 35% Co and 0% Fe, not shown). Slight offsets in the trend towards higher Co at a given Fe content in the porphyry samples are due to lesser amounts of Ni substituting for Fe in these samples.

ironstone (Co concentrations range from 1.2 to 17.5 wt.%), which also contains end-member cobaltite and slightly arsenian pyrite, whereas unmineralized ironstone contains arsenopyrite with less than 4.9 wt.% Co and As-poor pyrite but no cobaltite.

One löllingite and three cobaltite grains were also analyzed. Two cobaltite grains came from the NICO bulk sample stockpile and one from the Discovery zone. The löllingite came from metasediment near the #25 Zone at NICO. The cobaltite grains are near stoichiometric in composition with approximately 2 wt.% Fe and 0.5–1.15 wt.% Ni.

Seventy-nine chalcopyrite grains were analyzed, mainly from metasediments, but 22 grains were from porphyry samples. Chalcopyrite is generally near stoichiometric in composition in most ore assemblages and this is also the case here. There is no discernible difference in the composition of chalcopyrite from metasediments versus those from porphyry samples.

Twenty-one molybdenite grains were analyzed from mineralized metasediments in a NICO bulk sample stockpile. Molybdenite - like chalcopyrite - is near stoichiometric in composition with only trace amounts of Se (up to 0.6 wt.%) substituting for S. Seven pyrite grains were analyzed: five from mineralized metasediments and two from mineralized porphyry near the NICO # 25 Zone. The pyrite grains are very stoichiometric in composition with the grains from porphyry containing only slightly higher As and Bi than those from the metasediments.

#### Silicates

Sixty tourmaline grains were analyzed: one grain from background till (07MOB002), 20 grains from tourmaline-rich breccia in porphyry (CQA-07 445C-1), 19 grains from porphyry bedrock (CQA-07 445B-1), and 20 from metasediments at the Tan showing (CQA-07 437A-1). Meta-sedimentary tourmaline from the Tan showing is considerably higher in CaO and Mg# (closer to the dravite end-member) compared to tourmaline from porphyry, tourmaline breccia, and background till, which overlap in their compositional range and correspond to schörl.

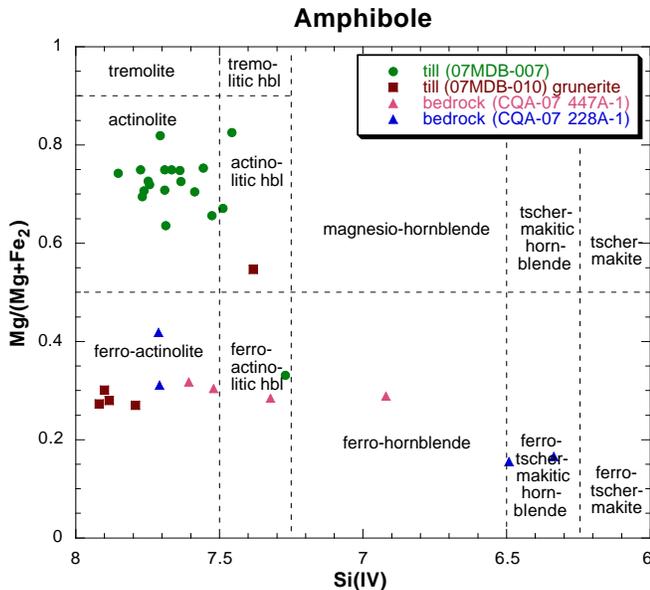


Fig. 6. Si(IV) vs. Mg-# for amphibole compositions. Note that the amphiboles from sample 07MOB010 (brown squares) are not ferro-actinolite but grunerite (Mg-Fe-amphibole).

Thirty-two amphibole grains were analyzed from two till samples (07MOB007 and 07MOB010) and from mineralized bedrock samples CQA-07-447-1A (road junction Portal A) and CQA-07-228A-1 (NICO Bowl zone). The amphibole grains from till sample 07MOB007 are all fairly Fe-poor actinolite grading to actinolitic hornblende. Amphibole from till sample 07MOB010 plots in the ferro-actinolite field in Figure 6 but is in fact grunerite. The mineralized bedrock samples contained much more Fe-rich amphibole than the till sample, spanning the range from ferro-actinolite to ferro tschermakitic hornblende (Fig. 6). The amphibole data from mineralized bedrock correspond to data from Sidor (2000) from mineralized and unmineralized ironstones and hornfels, which have variable but generally low Mg-# (<50) and variable Si/Al ratios with Si(IV) ranging from approximately 6.0 to 8.0. The amphiboles from Au-rich ironstone analyzed by Sidor (2000) have the lowest Mg-#s and a large variation in Si/Al.

#### Other minerals

Twenty allanite grains were analyzed from porphyry of the NICO #2 Zone. They are quite homogeneous in their compositions with only small variations in  $\text{FeO}_{\text{tot}}$  and rare earth content. Twenty-two scheelite grains were analyzed from mineralized metasediments from the NICO bulk sample stockpile. Scheelite - like chalcopyrite and molybdenite - is perfectly stoichiometric in composition with no significant trace element substitutions. Forty-one bismutite grains were analyzed from till from the NICO Bowl Zone. The totals of the analyses vary widely from 83 to 106 wt.%, indicating either analytical problems or intergrowth with other minerals with elements not analyzed here (e.g. Si from quartz). The most common trace in bismutite is  $\text{SO}_3$ , which occurs with up to 0.8 wt.%.

#### Ferromagnetic fraction

Magnetite and hematite are minerals that have a range of divalent and/or trivalent cation solid-solution substitutions. The chemical composition of representative samples from a num-

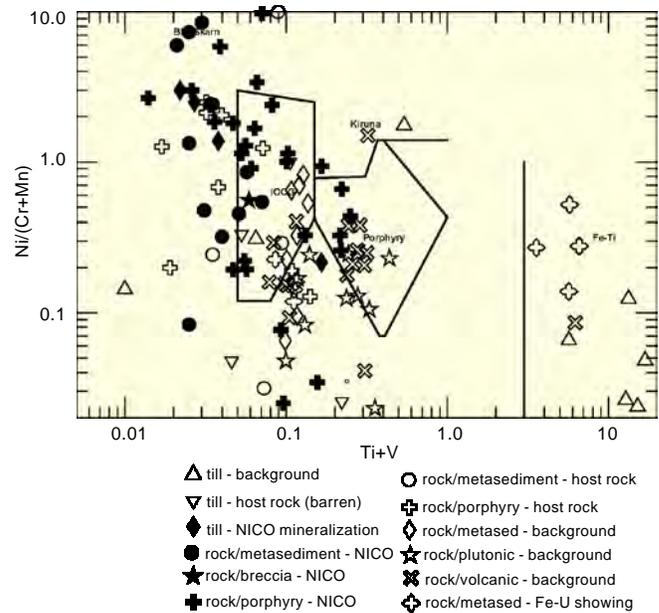


Fig. 7. Ni/(Cr+Mn) versus Ti+V diagram to discriminate IOCG, porphyry Cu-Mo-Au, banded iron formation, skarn, and Fe-Ti-V deposits.

ber of representative deposits world-wide are used to define discriminant diagrams that are useful to fingerprint a range of mineral deposit types (Beaudoin & Dupuis 2009). In the Ni/(Mn+Cr) versus Ti+V diagram (Fig. 7), most grains from background till have higher Ti+V compositions compared to grains from till collected over NICO and have a similar composition to grains collected in metasediments at the Tan Fe-U showing. There is a good correspondence between the composition of grains from NICO metasediments and that of grains from till collected over NICO, and a reasonable differentiation between NICO and background metasediments. These preliminary results indicate the potential use of discriminant diagrams for iron oxides to fingerprint IOCG deposits. Further tests will be conducted to determine the optimum grain size, number of grains, and other sample preparation methods for using iron oxide mineral chemistry in mineral exploration over the GBMZ.

#### DISCUSSION AND CONCLUSIONS

Study of the heavy mineral signature of the NICO deposit area and overlying glacial sediments demonstrates that, apart from gold and magnetite, few mineral species present in the mineralized rocks at NICO have clear potential as indicator minerals in surficial sediments. The non-ferromagnetic heavy minerals at NICO are either not chemically stable in surface glacial sediments, not sufficiently coarse grained or visually distinctive to be readily identified, not abundant enough in the mineralized bedrock, or not sufficiently heavy (concentratable).

Many of the sulphides that are highly to moderately abundant in the bedrock (i.e. arsenopyrite, chalcopyrite, pyrite) are relatively unstable in soils; hence they are absent or poorly preserved in surface till samples. Shallow till sampling (<1 m) is an effective method in discontinuous and continuous permafrost terrain of the Canadian Shield and has been widely used for geochemical and indicator mineral sampling by government agencies and by exploration companies (e.g. McMartin & McClenaghan 2001 and references herein; McMartin &

Campbell 2009). However, in weakly oxidized C-horizon material, easily weathered metal-rich ore minerals, such as many sulphides and tellurides, are oxidized in the shallow glacial sediments. Chalcopyrite, which usually survives near-surface weathering better than the other sulphides (e.g. Peuraniemi 1984; Averill 2001), has been used as an indicator mineral in exploring for metamorphosed or magmatic massive sulphide deposits (Averill 2001). At NICO, chalcopyrite is not recovered readily from the bedrock concentrates and only occurs in one till sample collected below the zone of oxidation at 3.5 m depth near the Bowl Zone. This suggests that chalcopyrite is not a practical indicator of mineralization at NICO. Although the Co-rich composition of arsenopyrite is possibly the strongest vector to Au-rich polymetallic mineralization in the study area, arsenopyrite is absent in C-horizon tills, thus arsenopyrite more readily oxidizes than chalcopyrite and pyrite in till. Perhaps in the northern part of the GBMZ, where soils developed on till are generally thin and immature because of relatively less chemical weathering, chalcopyrite (and arsenopyrite?) have better potential as indicator minerals for IOCG mineralization.

Some of the heavy minerals are present in bedrock and till concentrates as small inclusions intergrown in larger minerals; it is difficult to recover them in the sand-fraction HMCs. For example, bismuthinite is relatively abundant in two mineralized bedrock samples but occurs as silt-sized inclusions of brittle, acicular crystals in larger arsenopyrite grains. When the arsenopyrite is oxidized in the soils, the bismuthinite grains are released but can not be recovered in the >0.25 mm fraction of till concentrates. Also, no bismuthinite grains were recovered from the pan concentrates of any till sample, indicating that, even if present in sufficiently large concentration and coarse-grained fraction, bismuthinite is probably poorly resistant to glacial transport and/or post-glacial weathering.

Tourmaline, an accessory alteration mineral at NICO, occurs abundantly as very small grains (<50  $\mu\text{m}$ ) intercalated with quartz in the NFM-HMCs of two barren and heavily brecciated porphyry samples closed to NICO. In this case, the grains are too small to be recovered in the sand fraction and/or are not sufficiently heavy enough to be recovered in the S.G. >3.2 fraction. Brecciation is not only common on the NICO property, particularly in the Treasure Lake Group immediately below the volcanic unconformity, but also into the overlying volcanic rocks (Goad *et al.* 2000b). Fractures and breccia zones, created in part by the hydrothermal activity, acted as channels for the mineralization solutions. The fragmental nature of the tourmaline grains in crackle breccia hampers the use of tourmaline as an indicator mineral for the NICO deposit. Sand-sized tourmaline grains with Ti-magnetite inclusions and distinct composition do occur in abundance in bedrock from the nearby Tan showing, and tourmaline has been observed in other IOCG settings in the GBMZ, namely in the Echo Bay District (Mumin *et al.* 2009). Tourmaline has shown potential as a resistate indicator mineral that is able to survive weathering and mechanical dispersal in both deeply weathered and glaciated terrains (Ramsden *et al.* 1993; Slack *et al.* 1999; Averill 2001, 2007). If sufficiently abundant and coarse grained in the bedrock, tourmaline could have some potential as an indicator mineral in the GBMZ.

Ferroactinolite, although forming a pervasive alteration mineral in mineralized bedrock at NICO (cf. Corriveau *et al.* 2009), is present only in minor amounts in several bedrock

concentrates. The grains are not sufficiently heavy to be concentrated in the NFM-HMCs, except where they contain inclusions of magnetite. In any case, amphibole compositions in mineralized and unmineralized metasediments overlap to a certain degree and could therefore only be used with caution. Additionally, ferroactinolite typically occurs as loose, silt-size aggregates of prismatic crystals in mineral grains of several bedrock samples. These mineral inclusions are probably destroyed during glacial transport and comminution; hence they have poor preservation potential in till samples. One till sample collected directly down-ice from the Discovery outcrop does contain a relatively large amount of sand-sized actinolite grains, suggesting that the mere presence of these grains in till indicates a close proximity to intensively altered (mineralized?) bedrock.

Some of the heavy minerals at NICO are not particularly abundant enough in bedrock to show up in till, or are too soft to survive glacial transport. Molybdenite, a very soft sulphide, is scarce in the bedrock concentrates and absent in the tills. Allanite was recovered in one mineralized bedrock sample but is absent in the till samples. Scheelite, a useful and distinctive resistate indicator mineral for skarn, volcanogenic massive sulphide, lode gold, and tungsten deposits in both glaciated and deeply weathered terrains (e.g. Lindmark 1977; Averill 2001), occurs as an accessory mineral in mineralized metasediments at NICO (Goad *et al.* 2000a). It was recovered in the two bismuthinite-bearing subsurface bedrock samples but in none of the till samples. Major gedrite and bismutite occur in the unoxidized till sample collected near the Bowl Zone. In effect, this till sample is the only one collected at NICO that yielded a distinct suite of heavy minerals (with chalcopyrite) that could be used as a guide to mineralization.

Relatively high numbers of Au grains are found in surface tills collected over and near mineral showings of the NICO deposit area (up to 39 grains/10 kg sample) in comparison with those collected over barren host rocks (up to 10) and background terrain (up to 4). Pristine Au grains, which indicate a local source and a short distance of glacial transport, are mostly abundant close to mineralized zones indicating that documenting gold grain abundance, size, shape, and fineness (e.g. Grant *et al.* 1991) remains a valuable surface exploration method for Au-bearing IOCG deposits in the GBMZ.

All of the mineralization discovered to date at NICO occurs within an intense zone of potassium and iron metasomatism around Lou Lake (Goad *et al.* 2000b). Iron oxides, dominated by magnetite, comprise approximately 20% of mineralized zones and produce a variety of coincident geophysical anomalies, namely a regional positive Bouguer gravity anomaly and a positive total-field magnetic-gradient anomaly (Goad *et al.* 2000a). Magnetite occurs in all bedrock and till samples collected around NICO and the abundance of ferromagnetic minerals alone in till produces a distinct anomaly directly down-ice from the Discovery outcrop. As magnetite occurs commonly in mineralized zones and alteration haloes of many hydrothermal and metamorphosed ore deposits, is resistant to weathering and mechanical transport, and separates easily from heavy mineral concentrates, using trace elements in oxides to fingerprint IOCG deposits glacially eroded and deposited into glacial sediments offers considerable yet untapped potential. At NICO, iron oxide composition, using preliminary discriminant diagrams, shows some potential, namely using the Ni/(Cr+Mn) versus Ti+V plot. To this effect, the study of the mineral

chemistry of iron oxides (magnetite and hematite) from a range of mineral deposit types at worldwide locations, including the NICO deposit, is under way (Beaudoin & Dupuis 2009).

The potential utility of magnetite and other unusual, distinctive, and propitious heavy minerals as indicators of IOCG, together with till geochemistry, will be further tested with detailed bedrock and drift sampling around the Sue Dianne deposit in 2009. Further sampling around additional deposits and re-picking from existing archived samples in the GBMZ (in collaboration with the mineral exploration industry) is also planned for the remainder of the project.

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# Following kimberlite indicator minerals to Chidliak, Baffin Island: Canada's newest diamond district

Brooke Clements, Jennifer Pell, Peter Holmes & Hugo Grenon

*Peregrine Diamonds Limited, 201-1250 Homer Street, Vancouver, British Columbia V6B 1C6  
(e-mail: brooke@pdiam.com)*

## INTRODUCTION

The most widely used technique in the early stages of a diamond exploration program is sediment sampling, where distinctive heavy minerals that can be associated with diamond are recovered. This methodology has been in practice for over one hundred years, since it was discovered that in South Africa, kimberlites were the primary host of diamonds. Samples of surface sediments are collected and tested for the presence of the easily recognizable kimberlite indicator minerals (KIMs) pyrope and eclogitic garnet, chromite, picroilmenite, and chrome diopside. Sediment-sampling techniques vary to suit local conditions (Atkinson 1989; Fipke *et al.* 1995). In glaciated areas like Canada, glacial tills have proven to be an effective medium to sample for KIMs and the application of this technique has led to major discoveries, such as the Ekati™ and Diavik™ diamond mines in the Northwest Territories and the Victor diamond mine in Ontario. Detecting the presence of KIMs can lead to the discovery of kimberlites and studying the chemistry of the grains helps to distinguish diamond-bearing kimberlites from barren kimberlites (Gurney 1984; Gurney *et al.* 1993; Fipke *et al.* 1995).

The discovery of diamond-bearing kimberlites on the 980,000 hectare Chidliak property ("Chidliak"), approximately 120 kilometres northeast of Iqaluit, on the Hall Peninsula of Baffin Island, Nunavut (Fig. 1), is a classic example of applying traditional diamond exploration techniques in an exploration program. Reconnaissance-scale sampling of glacial sediments led to the identification of an area containing KIMs and follow-up sampling better defined the KIM anomalies. Abrasion and electron-microprobe studies of the KIMs further prioritized the area. Studies of the glacial history and indicator dis-

person trains allowed for the design of focused airborne geophysical surveys. Kimberlite-type anomalies were identified from the survey and prospecting resulted in the discovery of three kimberlites in 2008. Caustic fusion analyses of the kimberlites have proven them to be significantly diamond-bearing. This work sets the stage for the next phase of work, the evaluation of the economic potential of this new diamond district.

## REGIONAL GEOLOGY

The geology of the Chidliak area and the Hall Peninsula is poorly understood. Chidliak lies along the eastern edge of the Paleoproterozoic Cumberland Batholith, which is in contact, to the east, with Paleoproterozoic supracrustal rocks believed by some to be correlative with the Lake Harbour Group strata on southern Baffin Island. To the east of this is Archean gneiss that locally, has been subjected to Paleoproterozoic tectonothermal overprinting. Some workers (St-Onge *et al.* 2009) correlate the Archean gneiss on Hall Peninsula with Archean units of western Greenland, northern Quebec, and Labrador that may have been reworked during the Trans-Hudson Orogen. Other workers consider the Archean gneiss on Hall Peninsula to be part of the Archean North Atlantic Craton (Scott 1996; Scott *et al.* 2002).

Hall Peninsula was covered by ice during much of the Quaternary period by the Foxe Dome of the Laurentide Ice Sheet, which was centred on the Foxe Basin approximately 500 kilometres northwest of Chidliak. The ice direction at Chidliak during this time is interpreted to be northwest-southeast. During the waning of Laurentide glaciation, the ice flow at Chidliak was modified or overprinted by ice radiating from the smaller Hall Dome centred over the Hall peninsula.

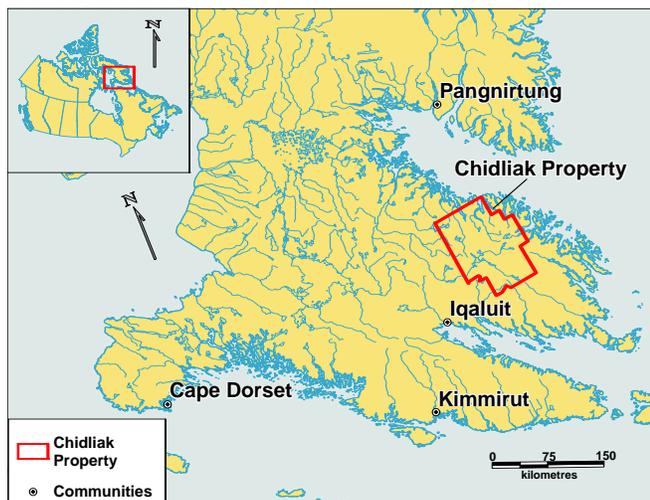


Fig. 1. Map showing the location of the Chidliak property on Baffin Island, Nunavut.



Fig. 2. Peregrine geologist, Sonya Neilson, collecting a till sample on the eastern part of the Chidliak property. An active glacier, a remnant of the Laurentide Ice Sheet, is evident on the hills in the background.

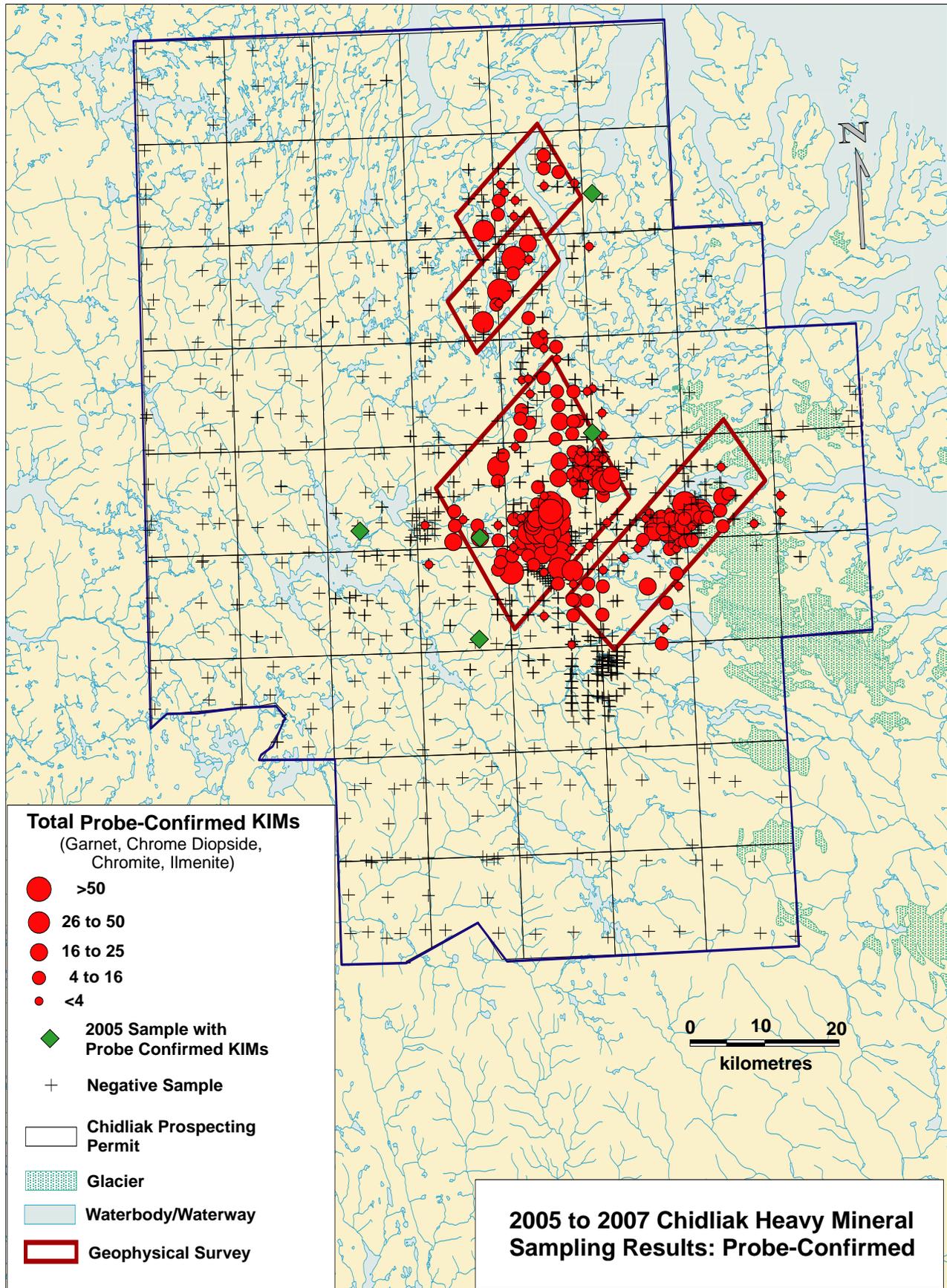
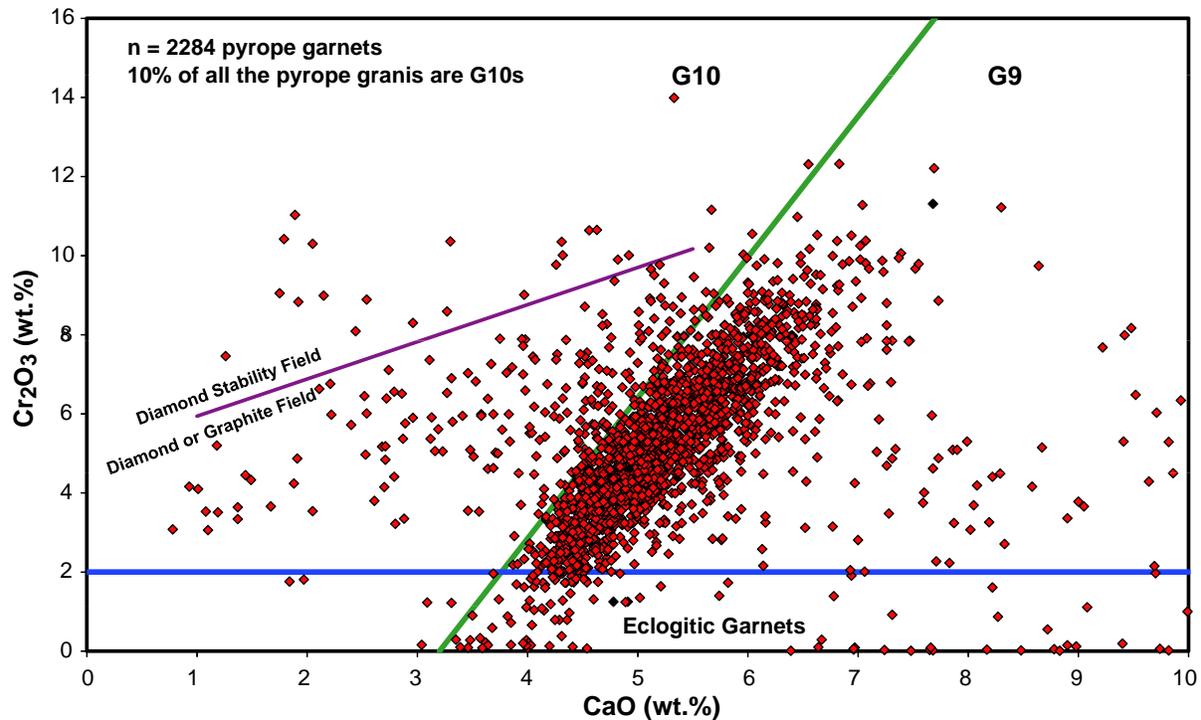


Fig. 3. Map showing the 2005 to 2007 kimberlite indicator mineral results (red circles) and outlines of the 2008 geophysical survey blocks (red rectangles).



**Fig. 4.** Chromium versus calcium plot for peridotitic garnets from Chidliak exploration till samples. Plot modified from Gurney (1984). 85% of garnets that occur as inclusions in diamonds plot to the left of the steep green diagonal line and are referred to as G10 garnets. The blue horizontal line at 2 wt.%  $\text{Cr}_2\text{O}_3$  is used as an arbitrary division between eclogitic garnets, below the line, and peridotitic garnets, above the line. The shallow purple diagonal line is the diamond-graphite constraint line of Grütter *et al.* (2004), where garnets that plot above the line are in equilibrium with diamond, and garnets below the line could co-exist with either diamond or graphite.

Remnants of this ice dome are still in existence today as manifested by small, active glaciers in the highlands of Hall Peninsula (Fig. 2).

#### REGIONAL INDICATOR MINERAL SURVEY (2005)

Prior to 2005, there is no record of diamond exploration on what is now Chidliak. In 2005, Peregrine and BHP Billiton jointly funded a reconnaissance till sampling program over a portion of southern Baffin Island that was operated by BHP Billiton. Samples of glacial till weighing approximately 15 kilograms were collected throughout the area. On what is now Chidliak, five samples contained probe-confirmed KIMs; two contained multiple mineral species (garnet, chrome diopside, and picroilmenite). One of the 2005 samples contained 21 KIM grains.

#### FOLLOW-UP INDICATOR MINERAL SAMPLING (2006 and 2007)

In 2006 and 2007, Peregrine funded and operated programs that increased the sample density and collected follow-up till samples in the anomalous area discovered in 2005 (Fig. 3). The first prospecting permits at Chidliak were acquired in February, 2007. The abrasion characteristics of many of the indicator minerals recovered from the 2006 and 2007 programs suggested minimal glacial transport and proximal kimberlite sources. The mineral chemistry of the same grains showed evidence of being within the diamond stability field. Microprobe analysis of over 2,000 peridotitic pyrope garnets showed that approximately ten percent of the grains had a high-chrome/low-calcium harzburgitic, or “G10” geochemical signature (Fig. 4). This indicated significant diamond potential for the as-yet undiscovered sources. Diamond inclusion field

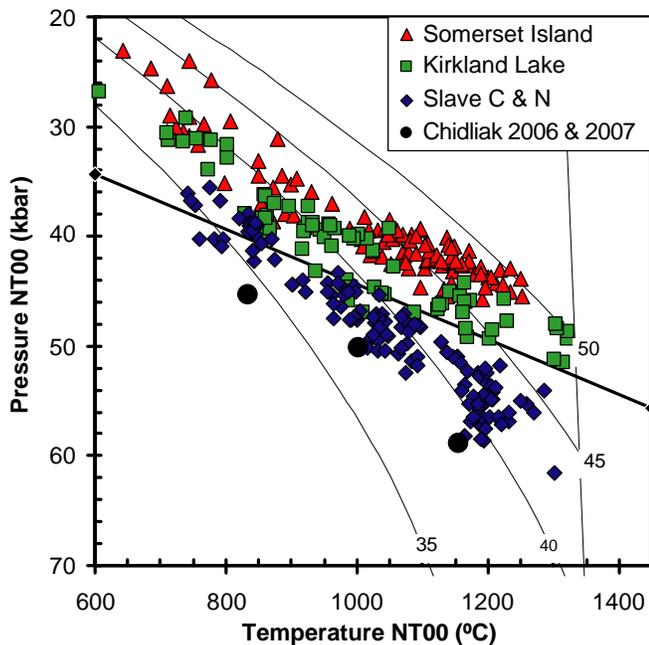
eclogitic garnets and chromites were also recovered. Clinopyroxene thermobarometry indicated that the area was characterized by a cool geotherm, similar to that of the Slave Province (Fig. 5). A cool geotherm indicates diamond preservation within the deep lithospheric mantle and is typical of most world-class diamond deposits. Studies of the mineral chemistry, particularly the picroilmenites, suggested that multiple sources, with distinct chemical signatures, were present.

#### KIMBERLITE AND DIAMOND DISCOVERY (2008)

Interpretation of the results of the 2006 and 2007 sampling programs allowed Peregrine to define probable source areas for the KIMs. Glacial striae on outcrops are locally abundant. When combined with the observed KIM dispersion trains, it was interpreted that till transport was most influenced by the waning stages of glaciation and that glacial transport distances in the area were likely to be short.

In the summer of 2008, an 11,700 line kilometre helicopterborne geophysical survey was completed over the areas with the greatest concentrations of KIMs (Fig. 3). Approximately 175 kimberlite-type anomalies were selected from the geophysical data. Field checking of three of these anomalies resulted in the discovery of three kimberlites at surface, CH-1, CH-2, and CH-3. Kimberlite outcrops were discovered at CH-1 and CH-2, and a collection of kimberlite boulders, interpreted to represent a kimberlite pipe, were discovered at CH-3. The CH-1 and CH-2 kimberlites comprise both coherent (magmatic) and pyroclastic kimberlite. Coherent kimberlite was found at CH-3.

The diamond results from the CH-1, CH-2, and CH-3 kimberlites confirm that all three are significantly diamond-bearing



**Fig. 5.** Pressure-temperature (P-T) results using the Nimis & Taylor (2000) thermobarometer for clinopyroxenes from Chidliak exploration samples, plotted over P-T results for peridotite xenoliths from other Canadian kimberlite provinces (after Grütter & Moore 2003). Note that Chidliak P-T results plot close to P-T data of high-grade central Slave kimberlites, and do not overlap P-T data from low-interest kimberlites at Kirkland Lake and Somerset Island. Model conductive geotherms are labeled at 35, 40, 45, and 50 mW/m<sup>2</sup>. The diagonal line represents the graphite/diamond equilibrium.

(Table 1). A 2.28 tonne mini-bulk sample, collected by hand from the surface of the CH-1 kimberlite, yielded 168 diamonds larger than 0.425 mm, including 34 commercial-size diamonds larger than 0.85 mm, weighing a total of 3.55 carats, for a diamond content of 1.56 carats per tonne. The largest stone recovered was a 2.01 carat gem. With geophysical surface expressions estimated at six, three, and two hectares, respectively, CH-1, CH-2, and CH-3 need to be evaluated by drilling and additional sampling to accurately determine the geology, volume, and diamond content of the different kimberlite phases.

In November 2008, BHP Billiton elected to exercise its earn-in rights at Chidliak and under the terms of the earn-in

agreement must incur a total of \$22.3 million dollars Canadian in exploration expenditures in order to earn a 51 percent interest. Upon successful completion of the earn-in, BHP Billiton will have a one-time election to earn an additional seven percent interest by delivering a fully engineered, bankable feasibility study on Chidliak.

#### FUTURE WORK (2009)

As a result of the encouraging results, a 2009 exploration program with a budget of \$9.2 million has been approved. The approved program includes the following principal elements.

- Additional indicator mineral sampling to prioritize geophysical anomalies and increase sample density over the entire property.
- Ground geophysical surveys to prioritize anomalies selected from the airborne survey.
- The collection and processing of a 50 tonne sample from the CH-1 kimberlite.
- Definition drilling of the known kimberlites and the drilling of new targets.
- Diamond testing of the different phases in the known kimberlites and new kimberlite discoveries.

#### CONCLUSIONS

Chidliak represents a brand new Canadian diamond district; the nearest known kimberlite district is located approximately 700 kilometres to the east in Greenland. This discovery is another classic case of the successful application of sediment sampling for kimberlite indicator minerals by Canadian diamond explorers. Since the dramatic discoveries in the Lac de Gras district in the Northwest Territories in the early 1990s, the Canadian diamond exploration community has discovered hundreds of kimberlites, most of them in remote locations, and Canada is now the world's third leading diamond producer. The discovery at Chidliak in 2008 illustrates that significant new diamond districts can still be discovered in Canada, and by extension the rest of the world, by using conventional exploration techniques.

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**Table 1.** Caustic fusion diamond results from the CH-1, CH-2, and CH-3 kimberlites.

INITIAL CAUSTIC FUSION RESULTS													
Sample	Sample Weight (kg)	Numbers of Diamonds According to Sieve Size Fraction (mm)										Total Diamonds	Total Carats (>0.850 mm)
		0.075 to 0.106	0.106 to 0.15	0.15 to 0.212	0.212 to 0.3	0.3 to 0.425	0.425 to 0.6	0.6 to 0.85	0.85 to 1.18	1.18 to 1.7	1.7 to 2.221		
CH-1A	100	9	11	9	9	2	2	2	0	0	0	44	
CH-1B	94.9	34	37	28	19	13	5	4	2	2	2	146	0.157
CH-1C	94	51	32	33	27	11	15	8	5	2	0	184	0.101
CH-2	356.6	249	101	15	3	1	0	2	0	1	0	372	0.029
CH-3	253.7	79	60	27	11	11	1	0	0	0	0	189	

MINI-BULK SAMPLE CAUSTIC FUSION RESULTS													
Sample	Sample Weight (kg)	Numbers of Diamonds According to Sieve Size Fraction (mm)							Total Diamonds	Total Carats >0.850 mm	Total Carats >1.18 mm	Diamond Content (>0.850 mm, cpt)	
		0.425 to 0.6	0.6 to 0.85	0.85 to 1.18	1.18 to 1.7	1.7 to 2.36	2.36 to 3.35	3.35 to 4.75					
CH-1A	1174	38	31	9	5	0	1	0	1	85	2.55	2.45	2.17
CH-1B	1106	37	28	9	3	4	2	0	0	83	1	0.8	0.9
TOTAL	2280	75	59	18	8	4	3	0	1	168	3.55	3.25	1.56

The diamond analyses were completed by the Saskatchewan Research Council Geoanalytical Laboratories, which is accredited to the ISO/IEC 17025 standard for microdiamond recovery via caustic fusion.

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# Public sector case study: Indicator mineral survey of Minnesota

L. H. Thorleifson

*Minnesota Geological Survey, 2642 University Avenue West, St Paul, Minnesota 55114  
(e-mail: thorleif@umn.edu)*

## 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 nonferromagnetic 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 likeli-

hood 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, anticipated 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



Fig. 1. Typical field sampling conditions.

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 possible, 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, wherever 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. The field data included site identification, WGS84 latitude, WGS84 longitude, date, collector, topographic map name, site name based

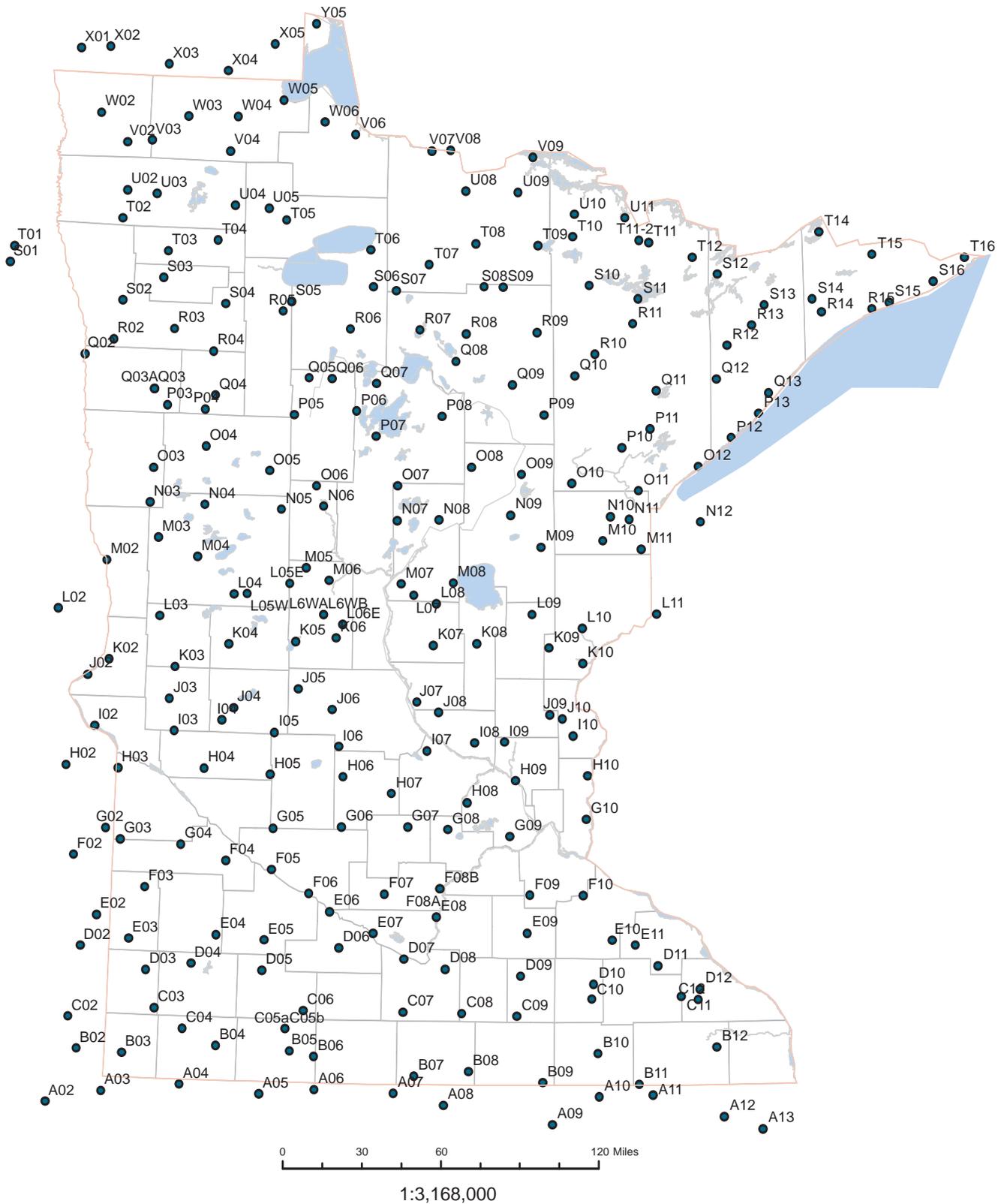


Fig. 2. Location of Minnesota survey sample sites (from Thorleifson *et al.* 2007).

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 lithol-

ogy (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,



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

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



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

abundance, the bulk samples were split in 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®), 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.

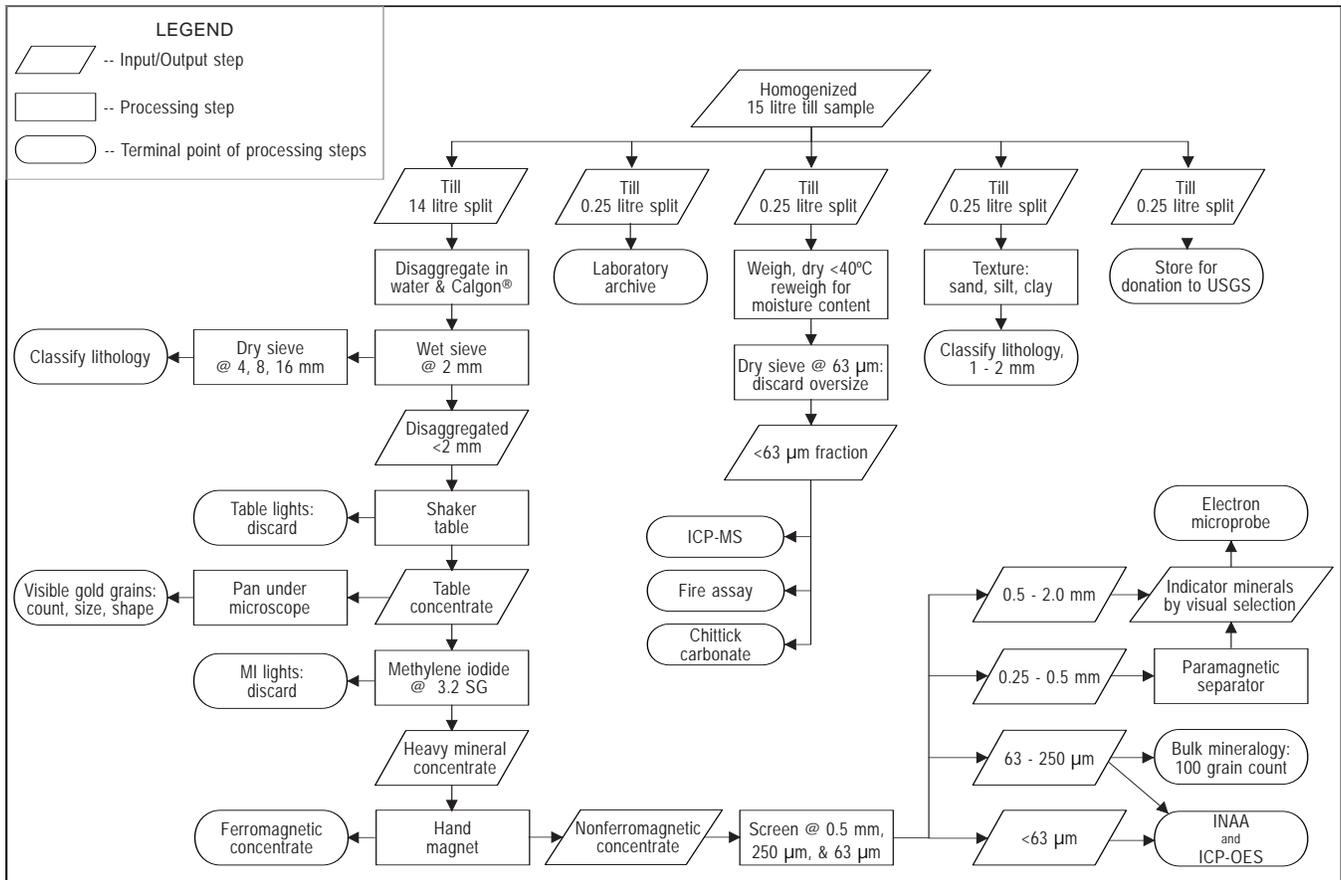


Fig. 5. Sample processing flow sheet (from Thorleifson *et al.* 2007).

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  $\text{Al}_2\text{O}_3$ ,  $\text{CaO}$ ,  $\text{Cr}_2\text{O}_3$ ,  $\text{FeO}$ ,  $\text{K}_2\text{O}$ ,  $\text{MgO}$ ,  $\text{MnO}$ ,  $\text{Na}_2\text{O}$ ,  $\text{SiO}_2$ ,  $\text{TiO}_2$ , and  $\text{ZnO}$  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 non-ferromagnetic 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 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 non-ferromagnetic, <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 clinopyroxene 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 tentative 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

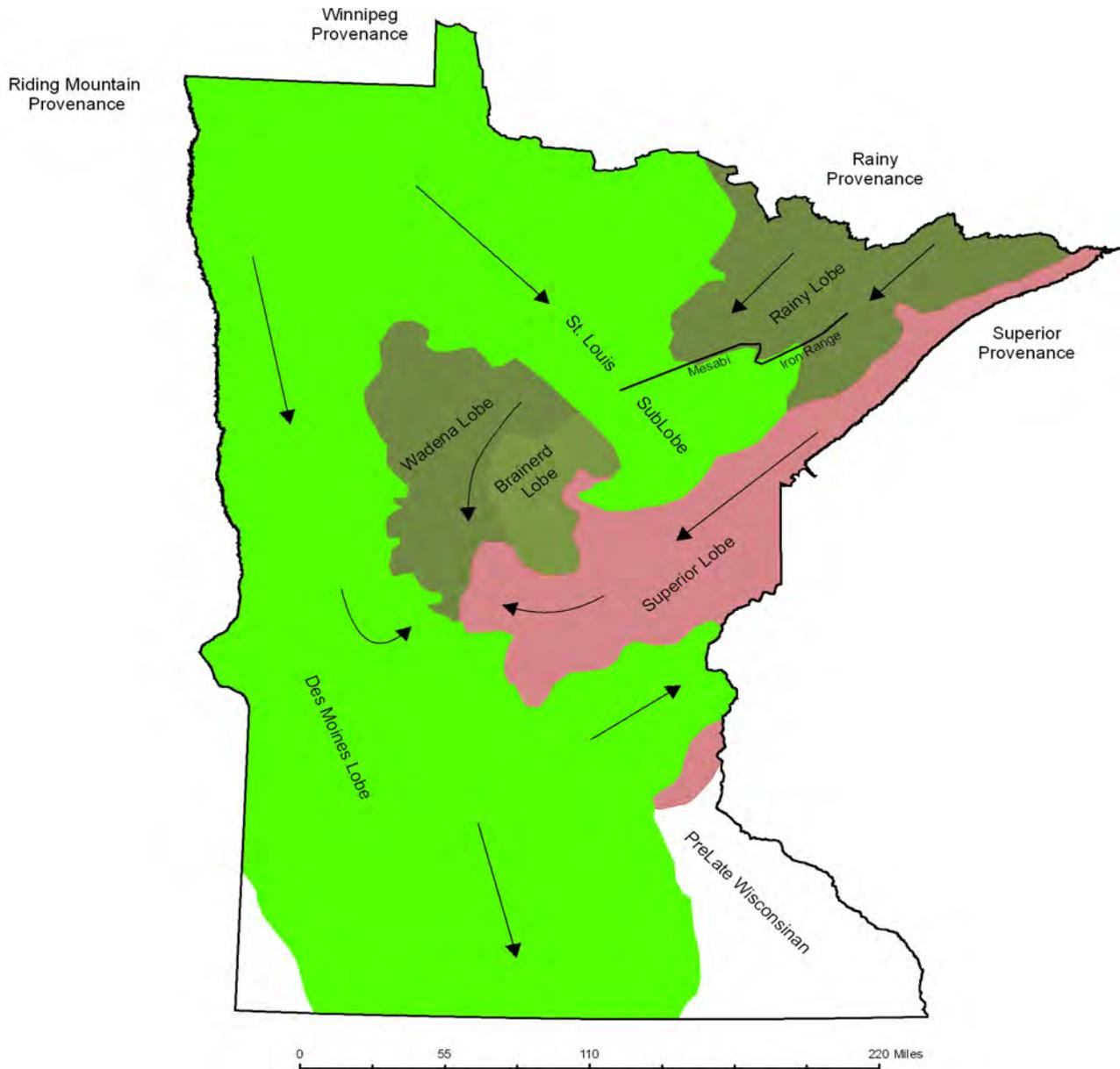


Fig. 6. Overview of till provenance trends (from Thorleifson *et al.* 2007).

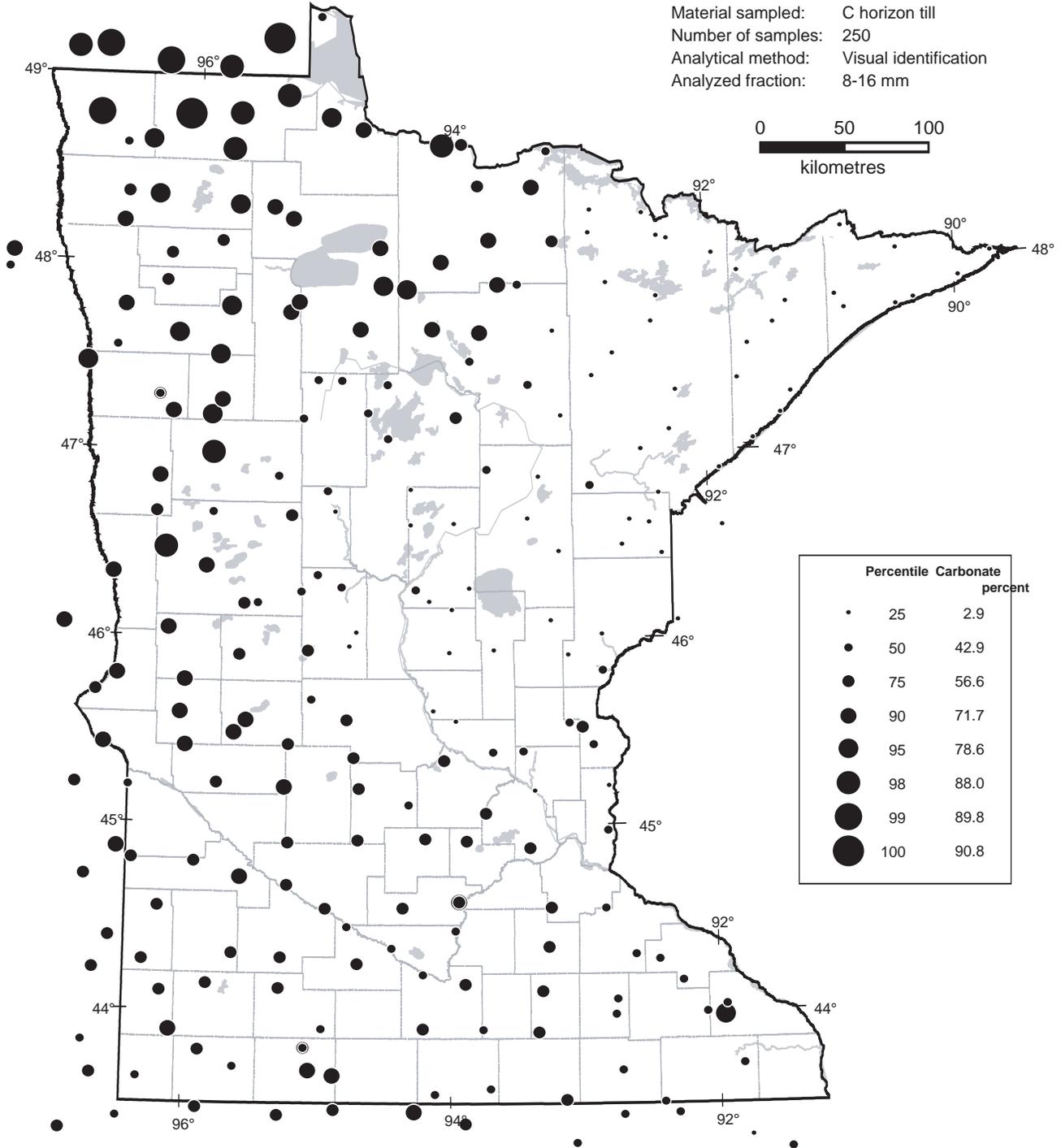
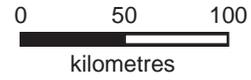
region and derivation from the Superior Basin, while goethite (Fig. 12) is elevated in the old tills of the southeast, ilmenite (Fig. 13), at least in the size fraction analyzed, is elevated in a zone of the Superior sediments, while siderite (Fig. 14) is an example of a zone along the Minnesota River 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.

# 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



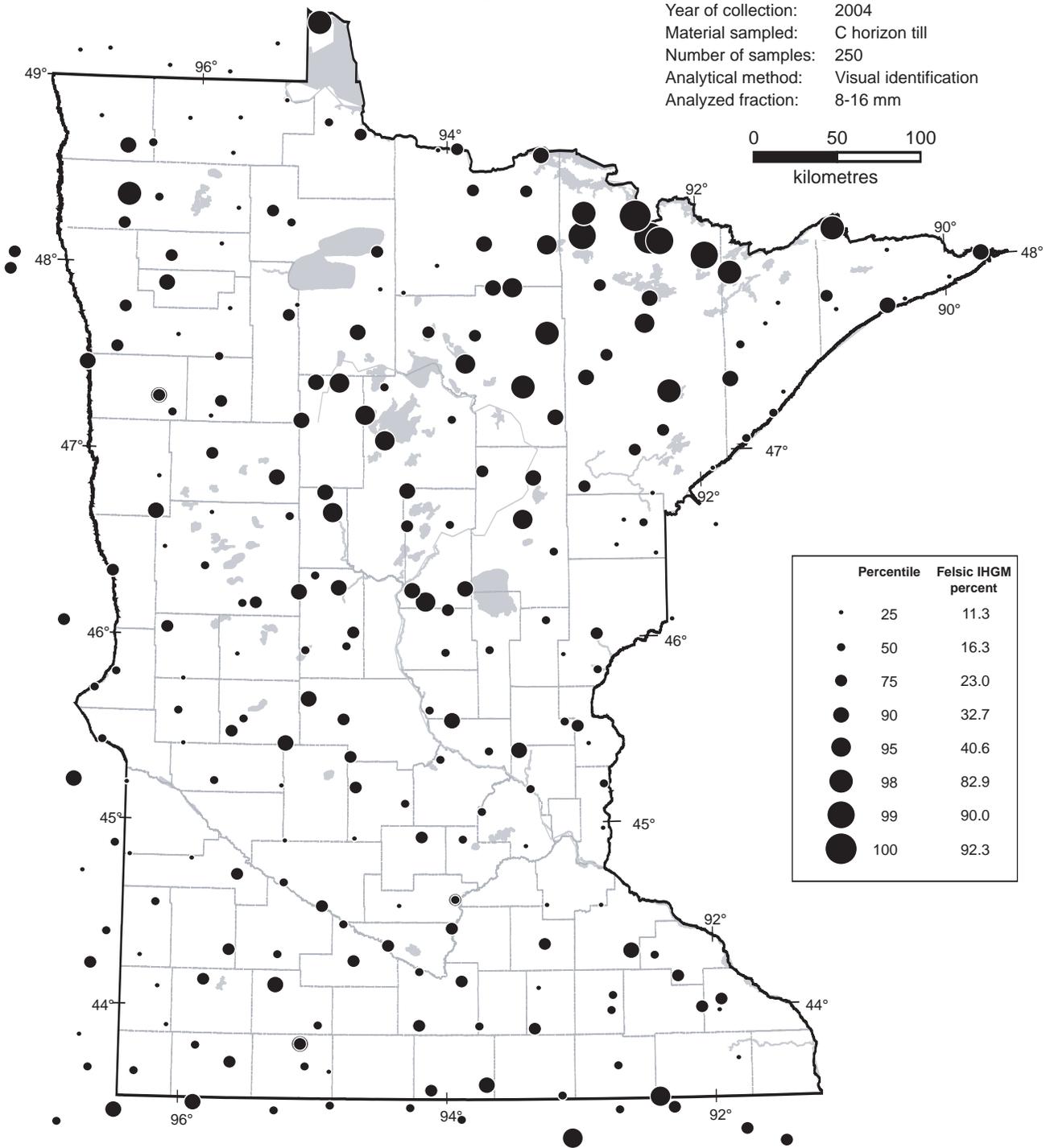
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Fig. 7. Carbonate in the 8 to 16 mm gravel fraction (from Thorleifson *et al.* 2007).

Soil Geochemical and Indicator Mineral Reconnaissance  
Survey of Till in Minnesota

Felsic Intrusive and  
High-Grade Metamorphic



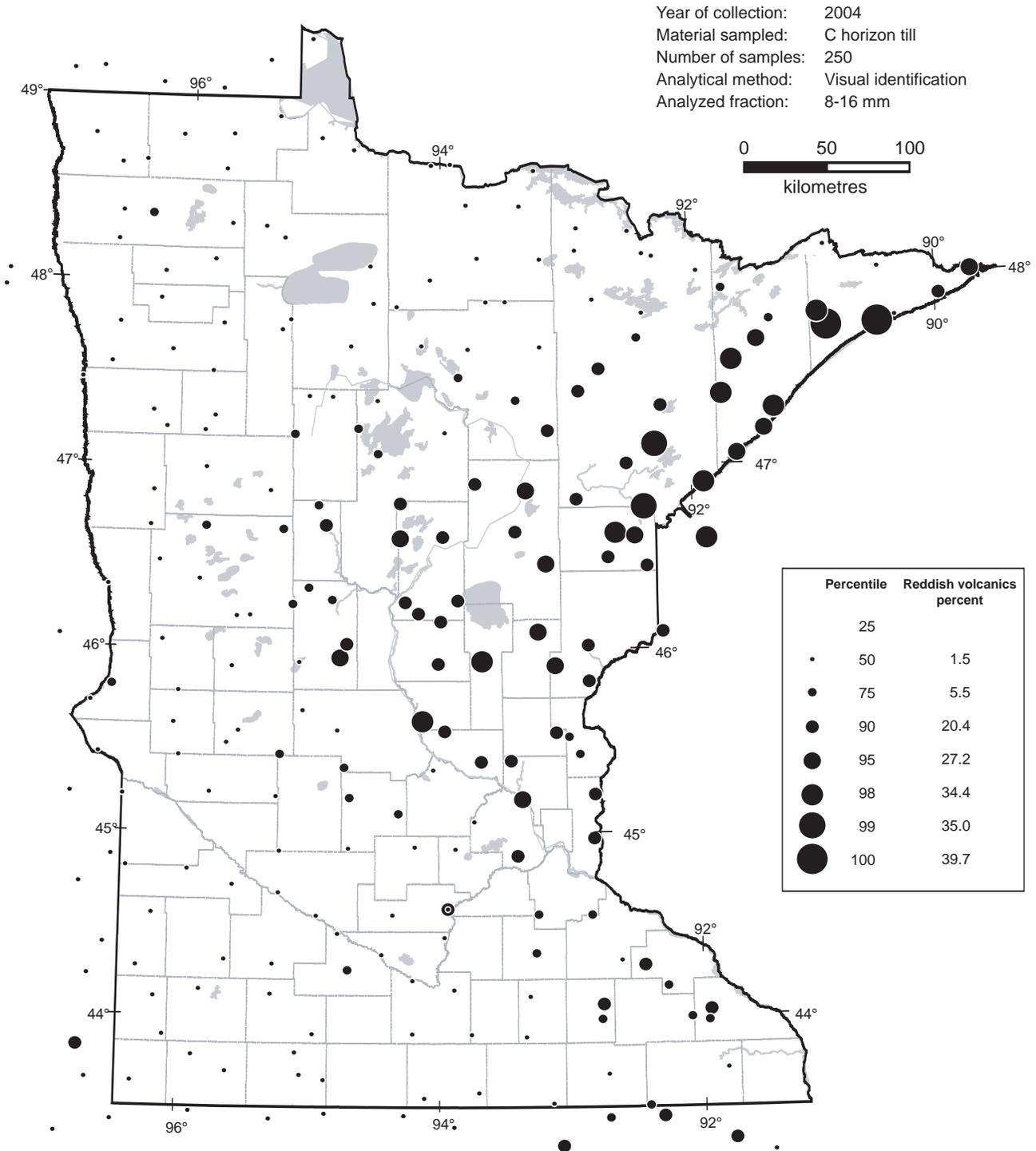
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Fig. 8. Granite and similar rocks in the 8 to 16 mm gravel fraction (from Thorleifson *et al.* 2007).

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

## Reddish Volcanics



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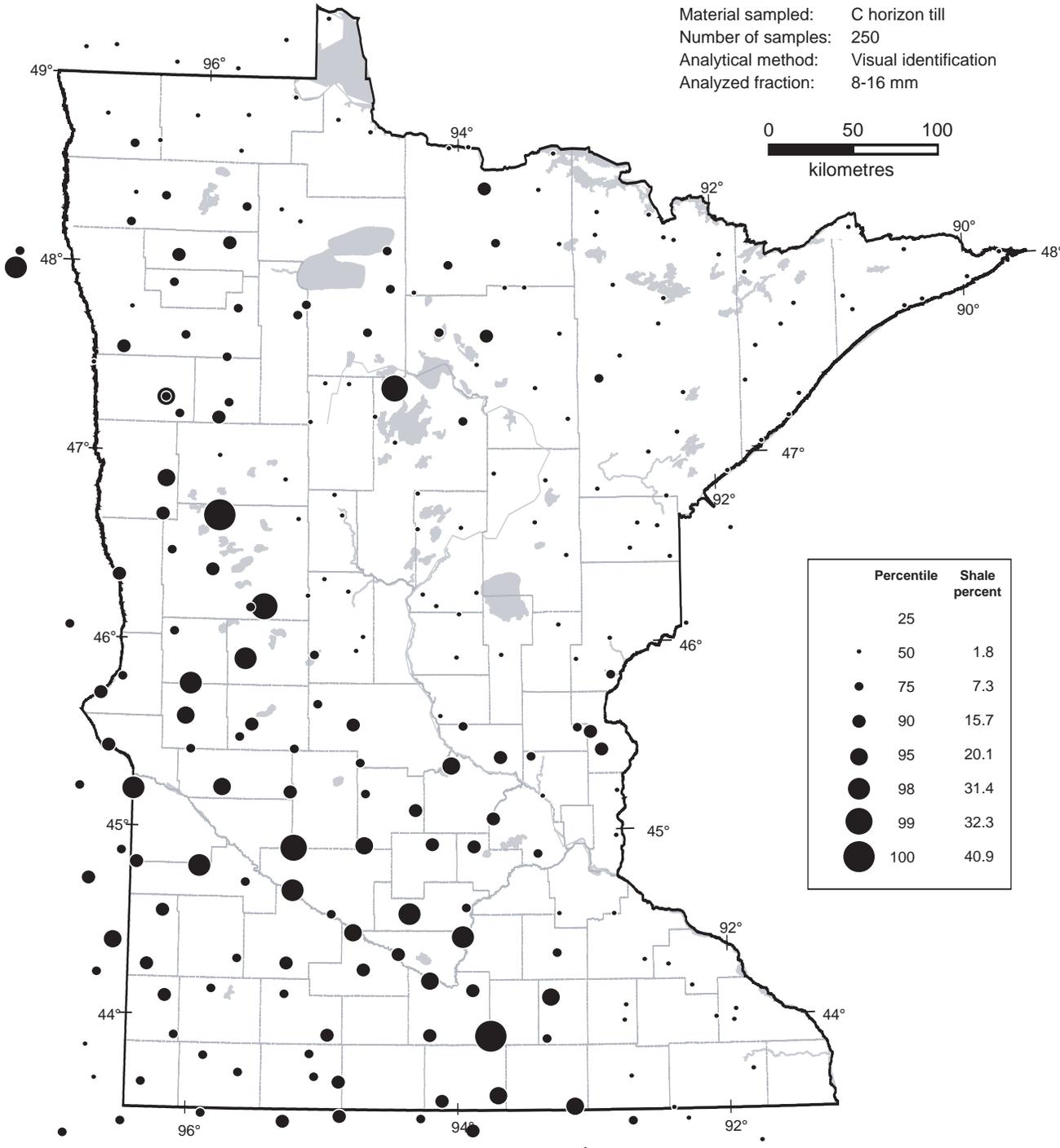
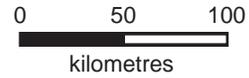


Fig. 9. Reddish volcanics in the 8 to 16 mm gravel fraction (from Thorleifson *et al.* 2007).

# 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	
•	1.8
•	7.3
•	15.7
•	20.1
•	31.4
•	32.3
•	40.9

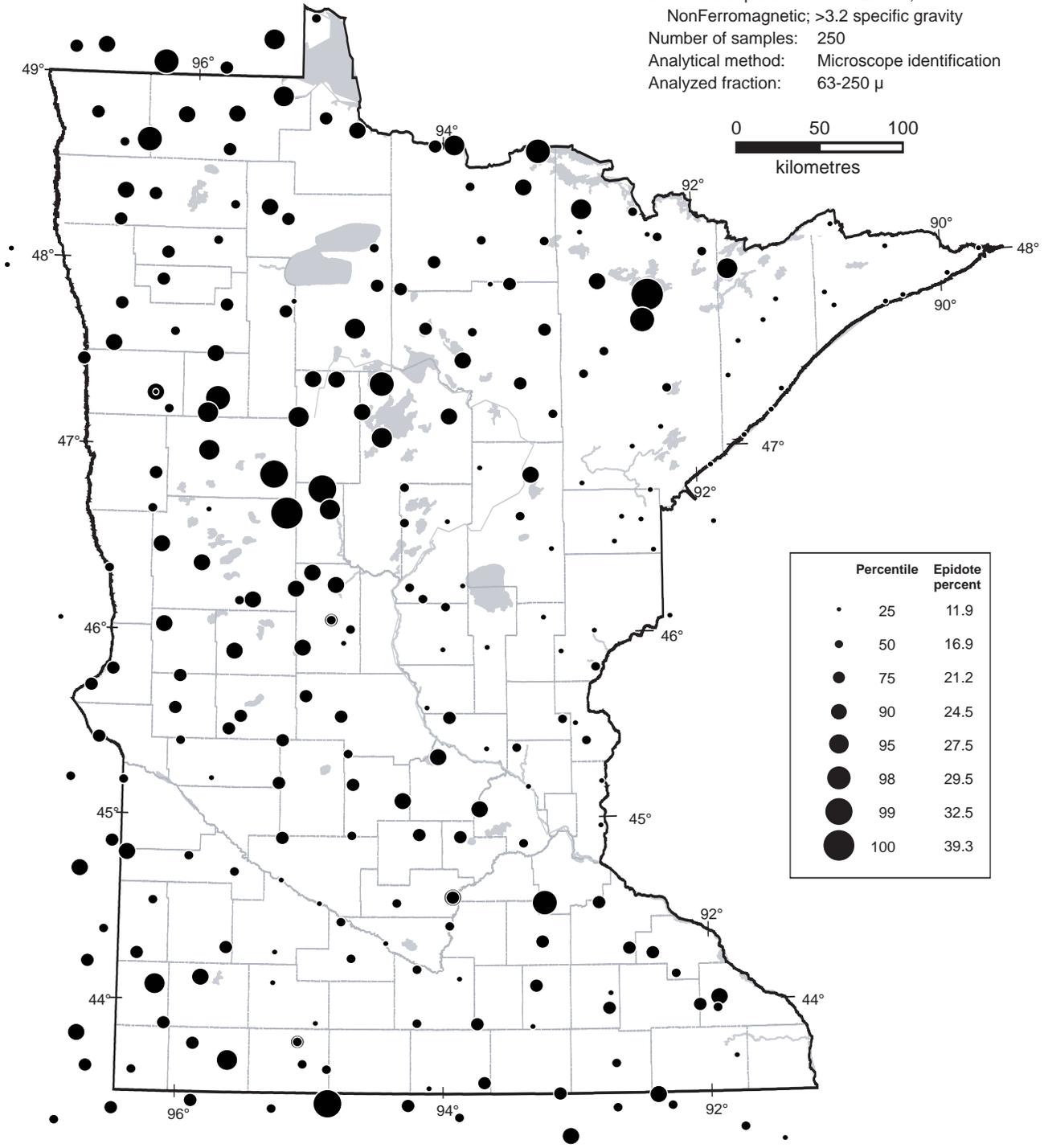


Fig. 10. Shale in the 8 to 16 mm gravel fraction (from Thorleifson *et al.* 2007).

# 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 μ



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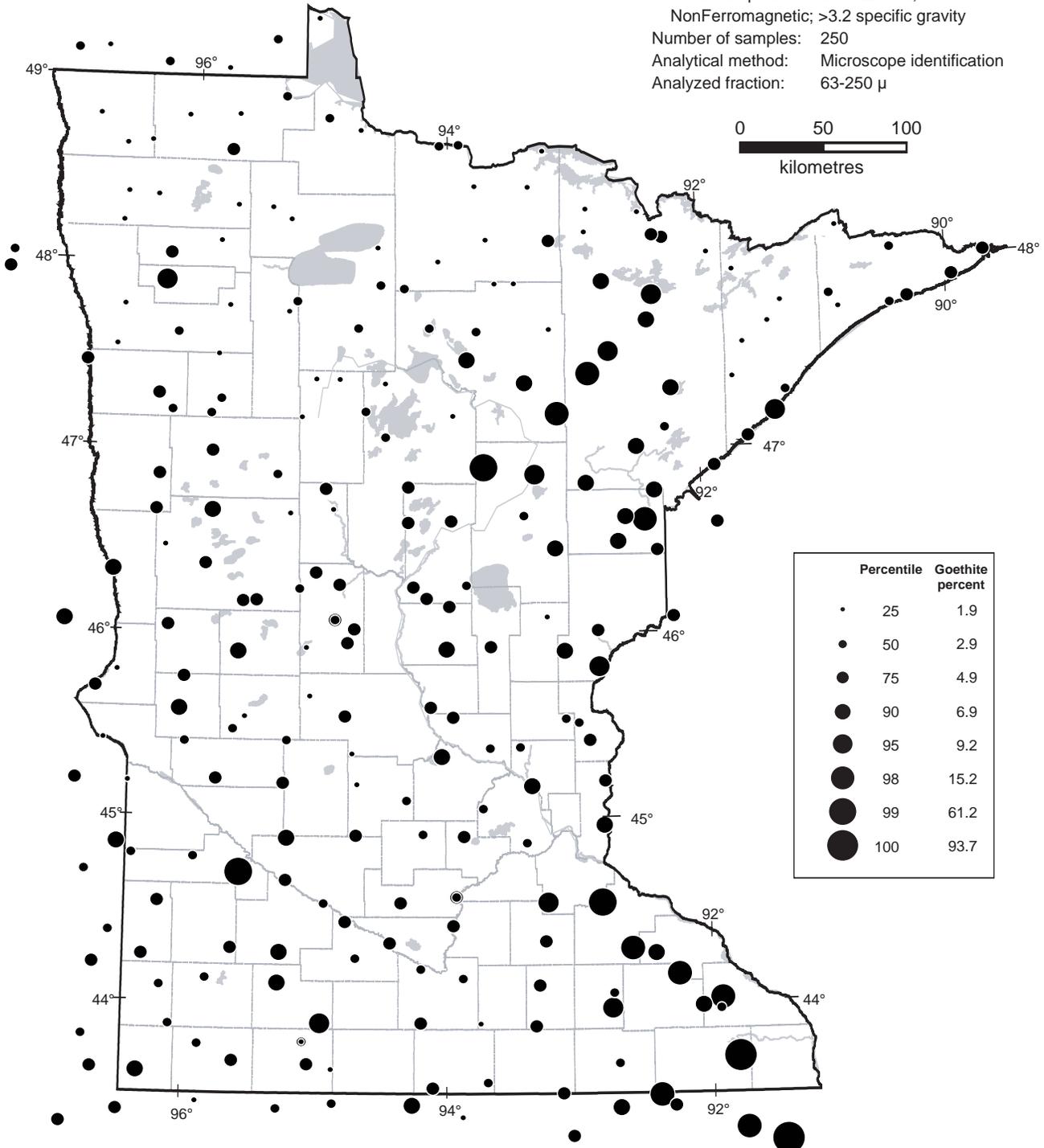


Fig. 11. Epidote in the 63 to 250 micron nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# 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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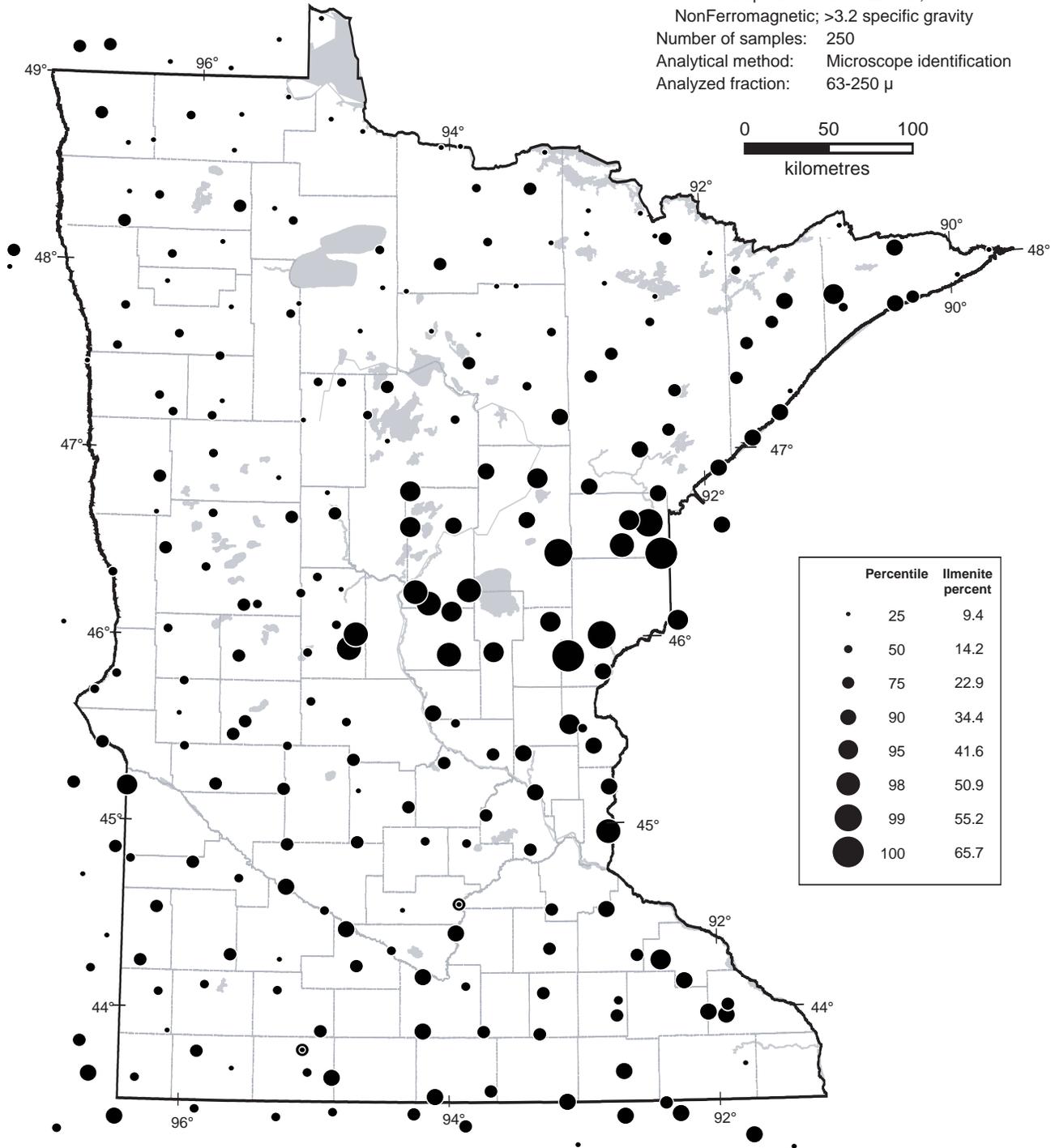


Fig. 12. Goethite in the 63 to 250 micron nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# 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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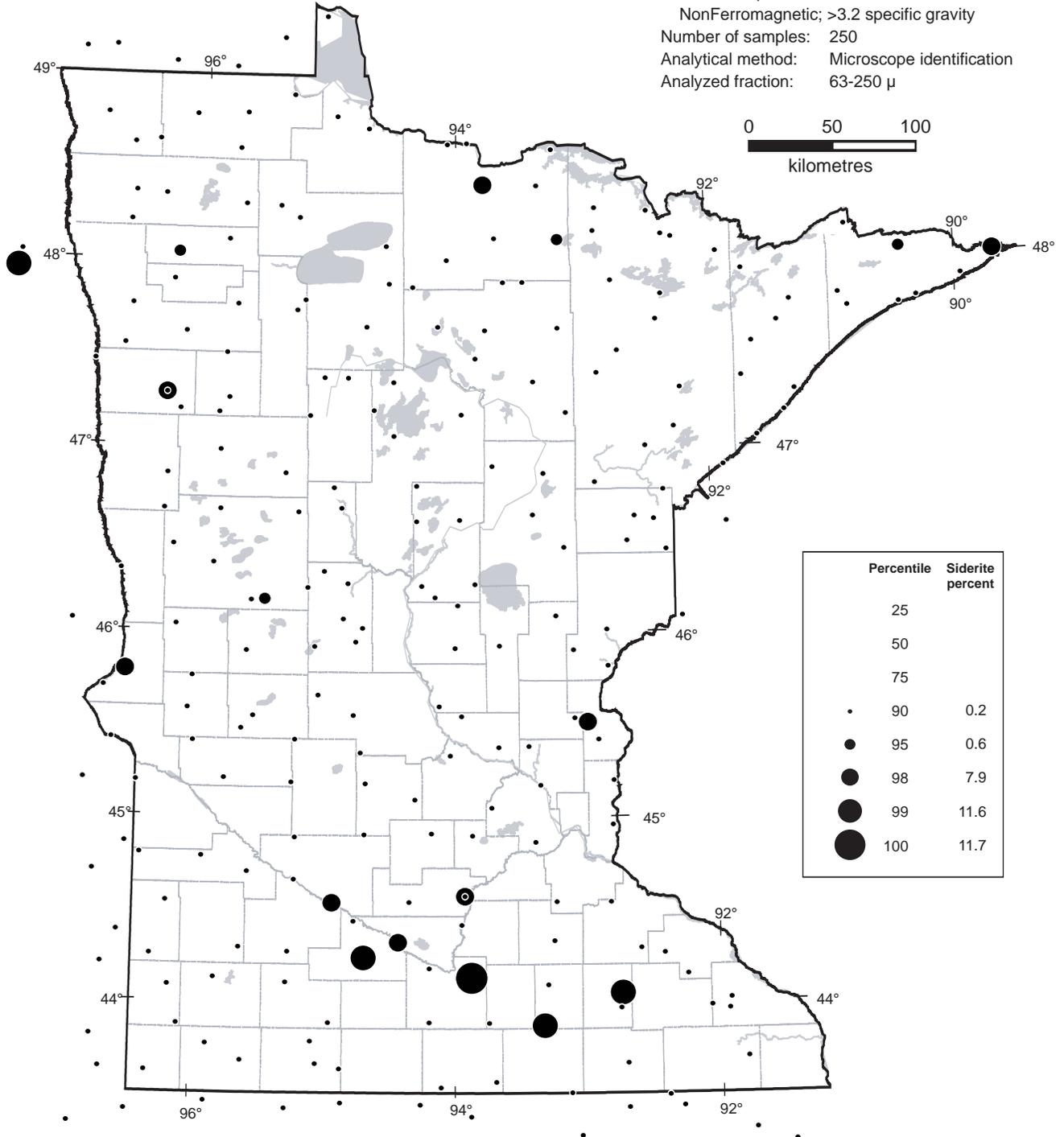


Fig. 13. Ilmenite in the 63 to 250 micron nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# 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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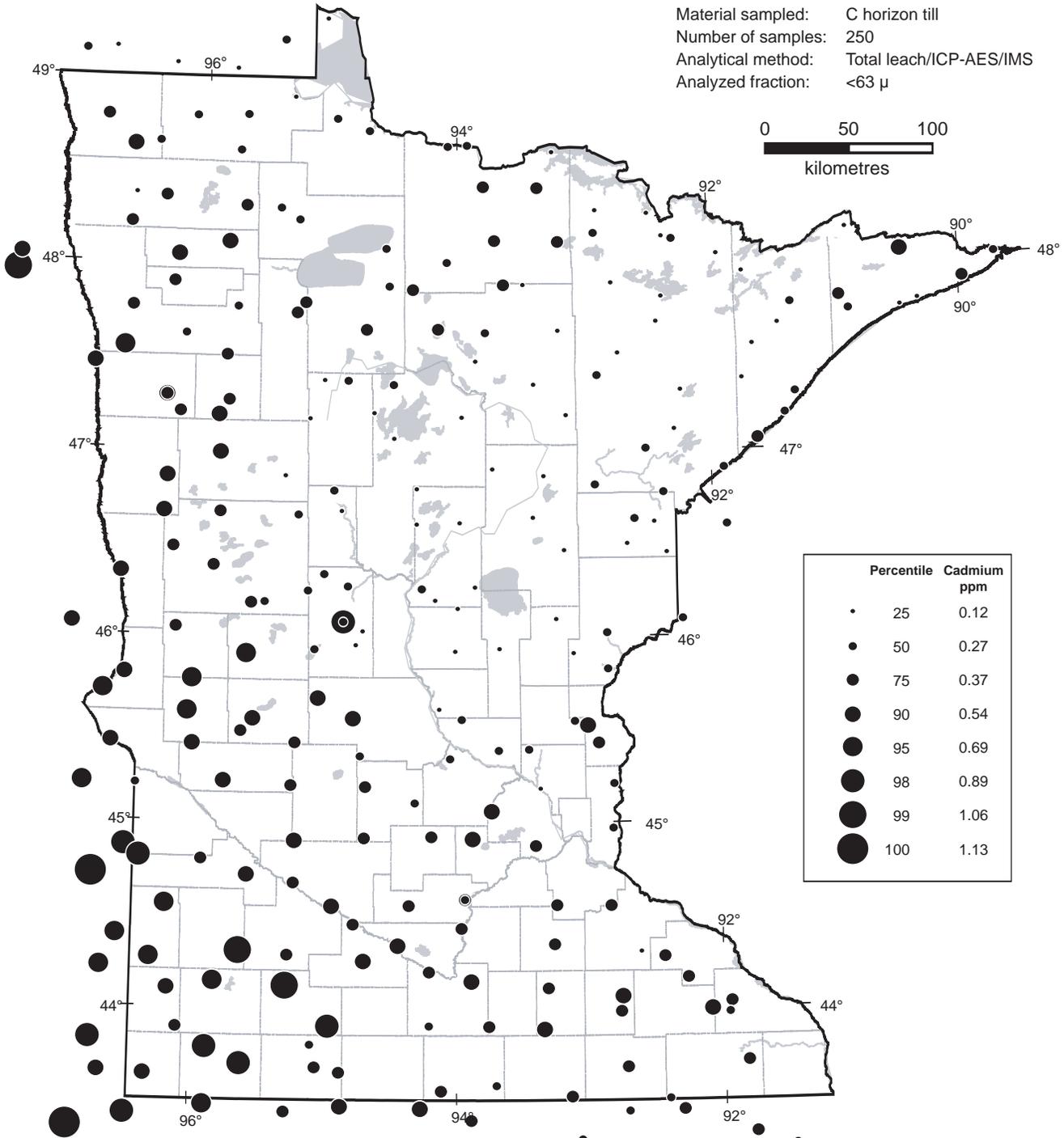
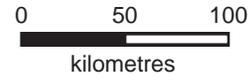


Fig. 14. Siderite in the 63 to 250 micron nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# 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

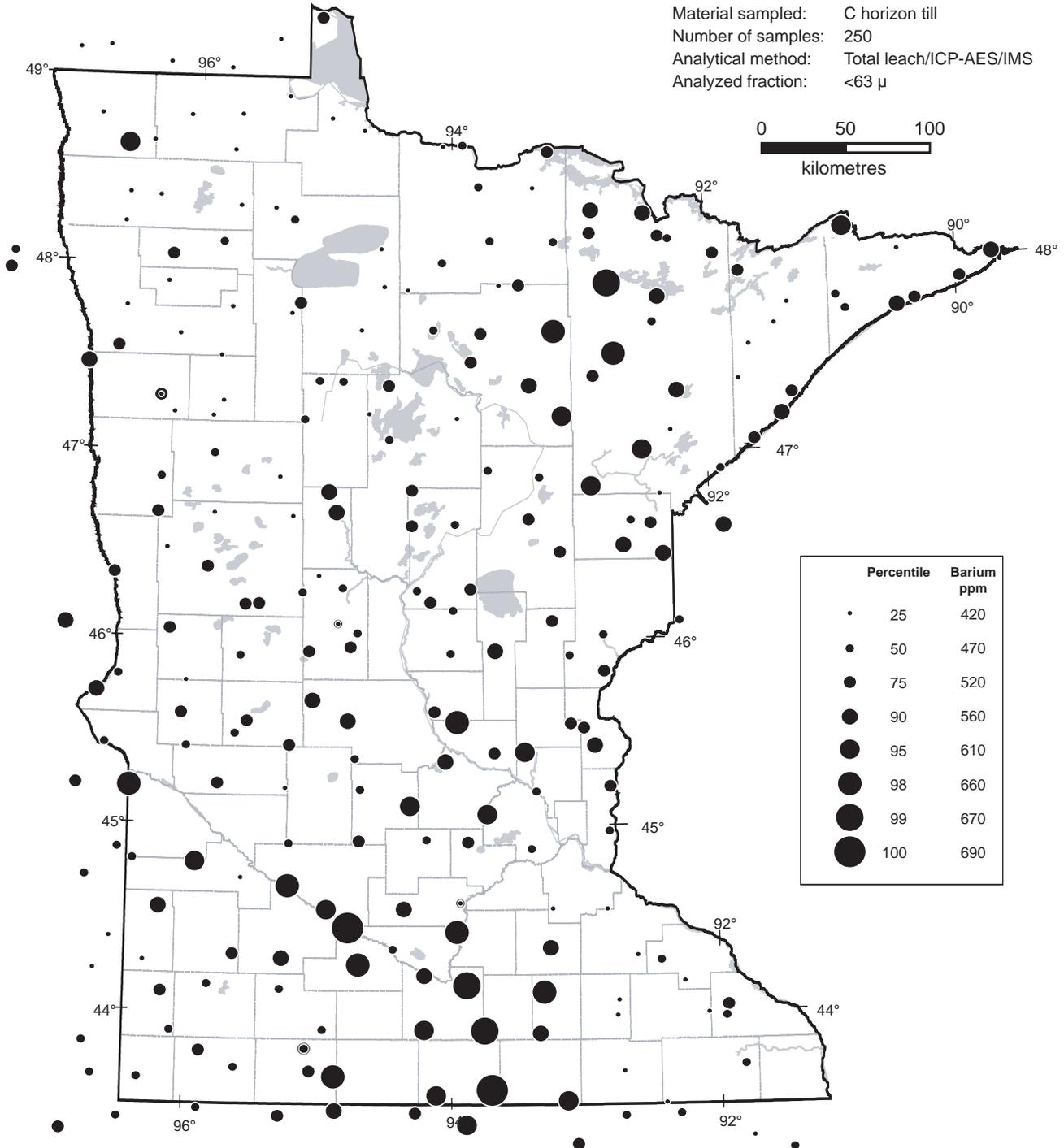
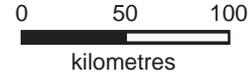


Fig. 15. Cadmium in the <63 micron fraction (from Thorleifson *et al.* 2007).

# 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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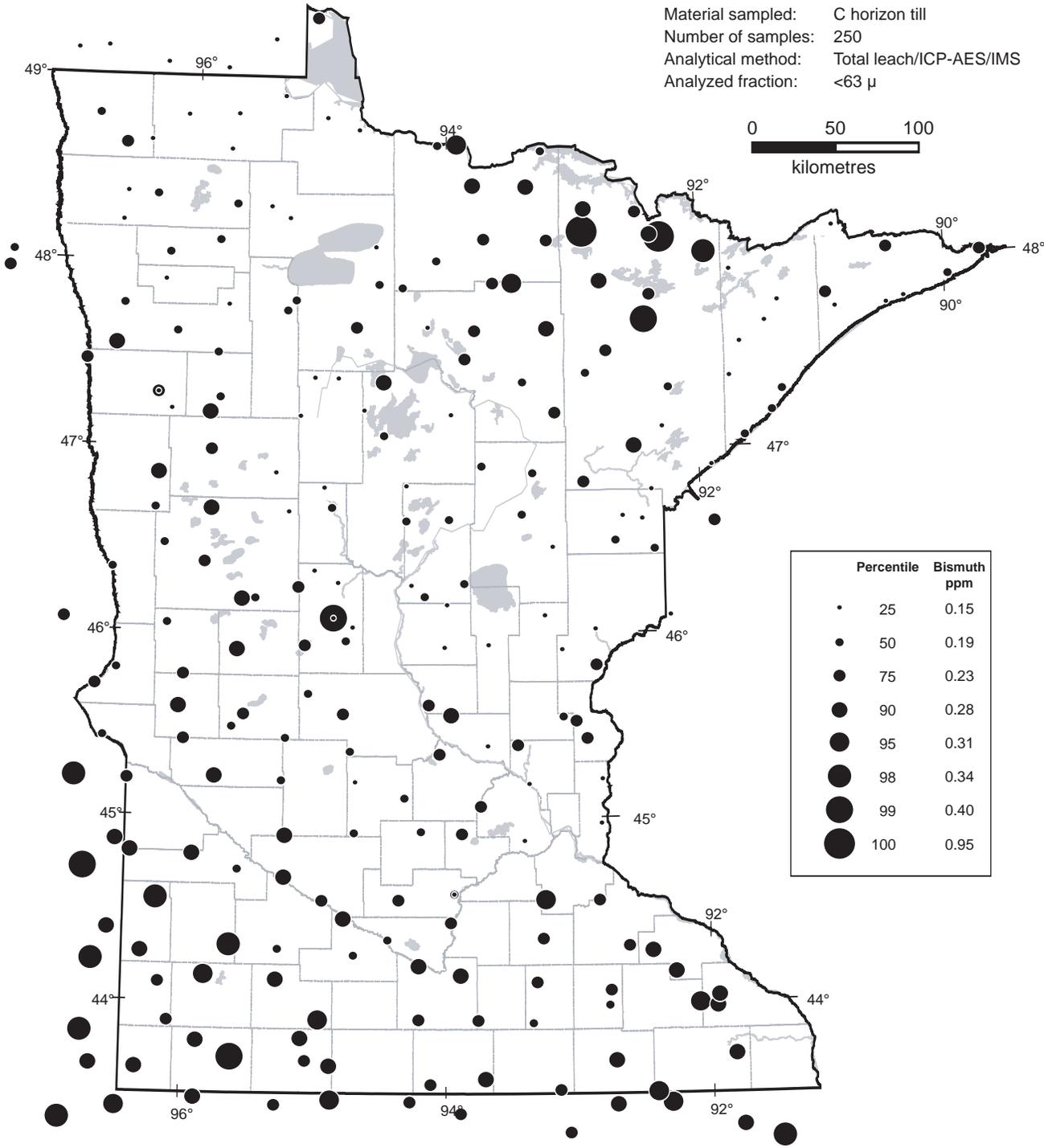
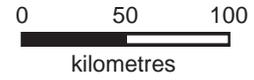


Fig. 16. Barium in the <63 micron fraction (from Thorleifson *et al.* 2007).

# 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  $\mu$



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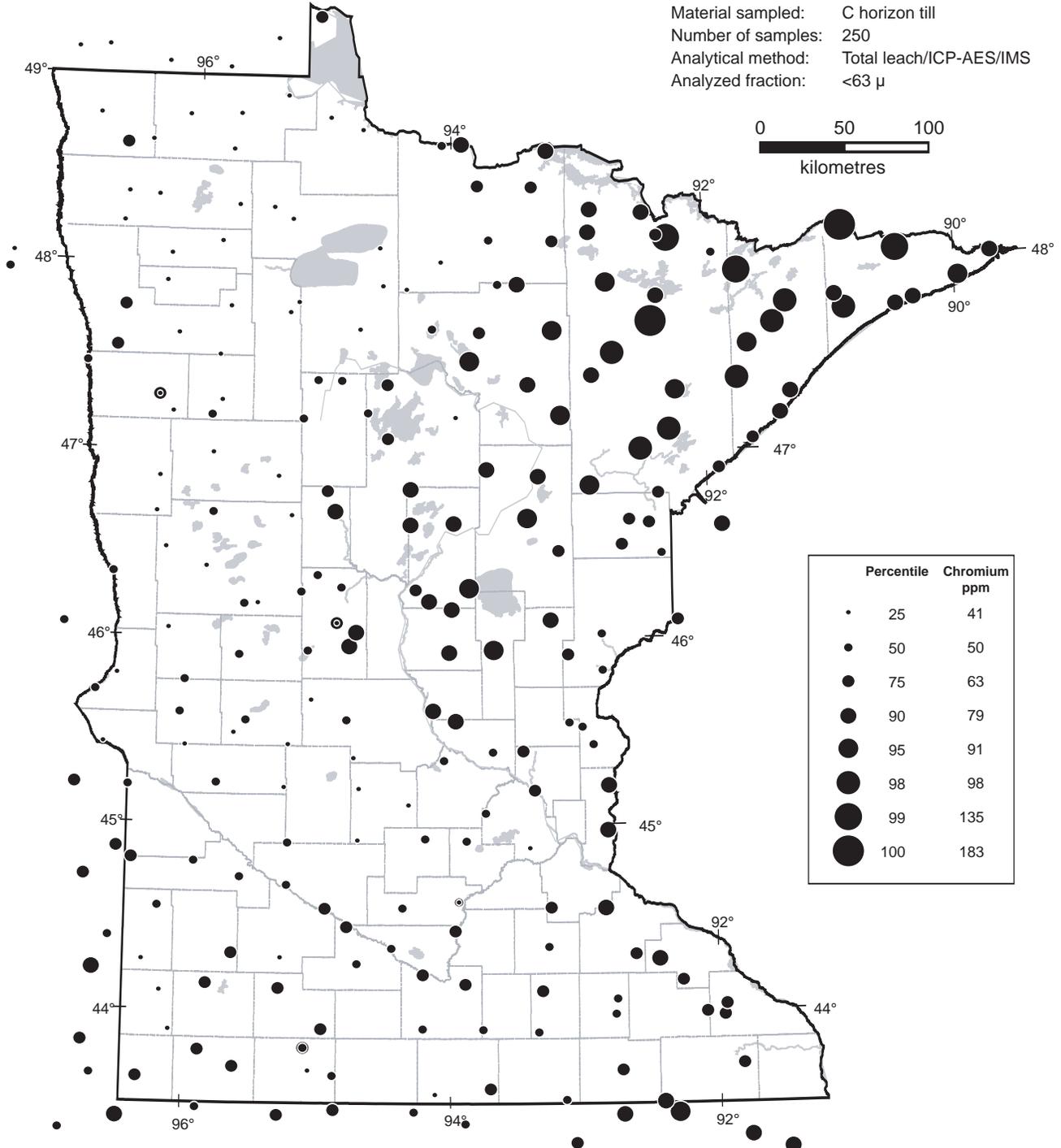
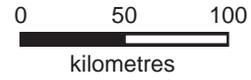


Fig. 17. Bismuth in the <63 micron fraction (from Thorleifson *et al.* 2007).

# 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  $\mu$



Percentile	Chromium ppm	
•	25	41
•	50	50
•	75	63
•	90	79
•	95	91
•	98	98
•	99	135
•	100	183

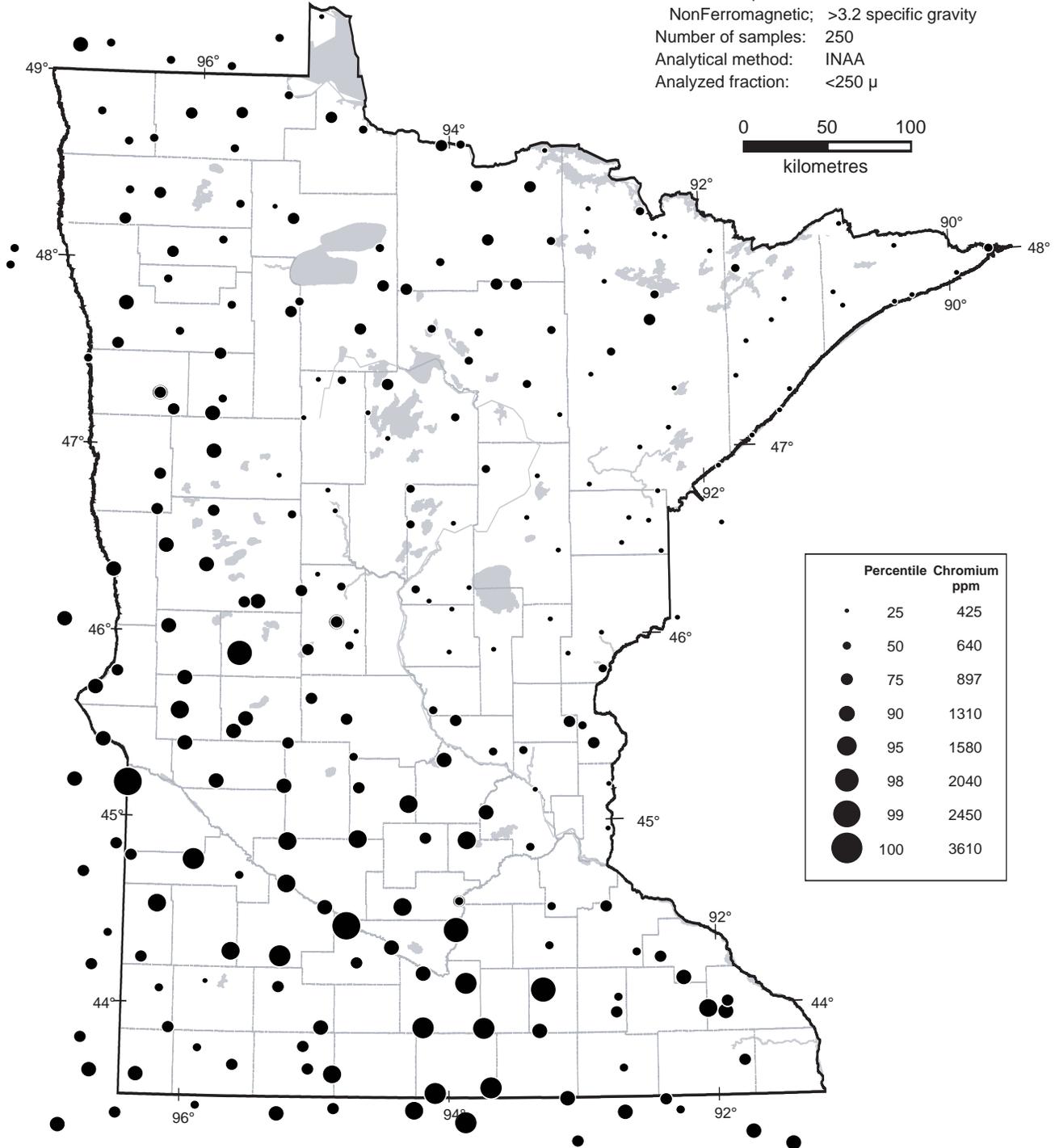


Fig. 18. Chromium in the <63 micron fraction (from Thorleifson *et al.* 2007).

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

## Chromium

Year of collection: 2004  
 Material sampled: C horizon till;  
 NonFerromagnetic; >3.2 specific gravity  
 Number of samples: 250  
 Analytical method: INAA  
 Analyzed fraction: <250 μ



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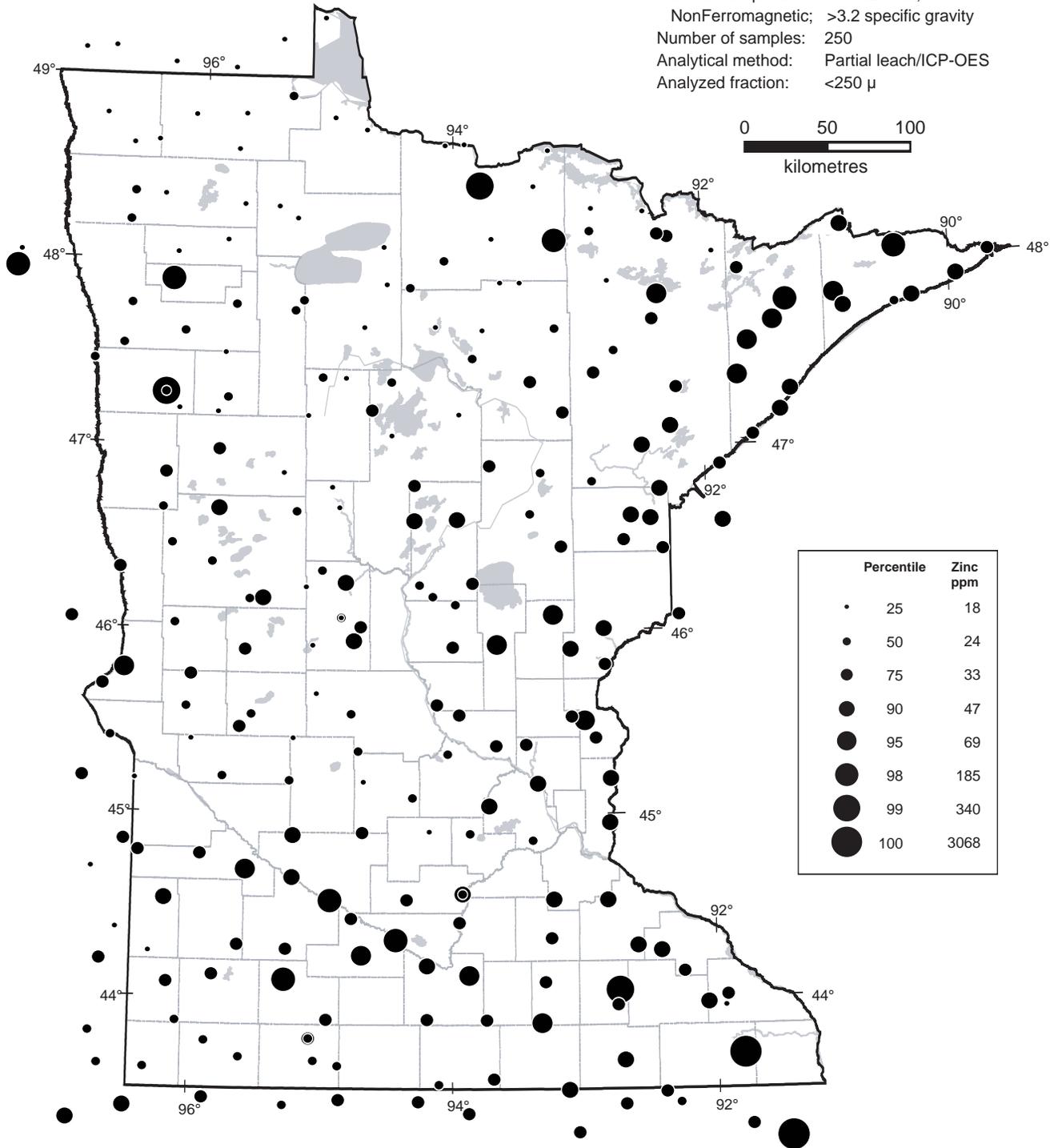


Fig. 19. Chromium in the <0.25 mm nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

# 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



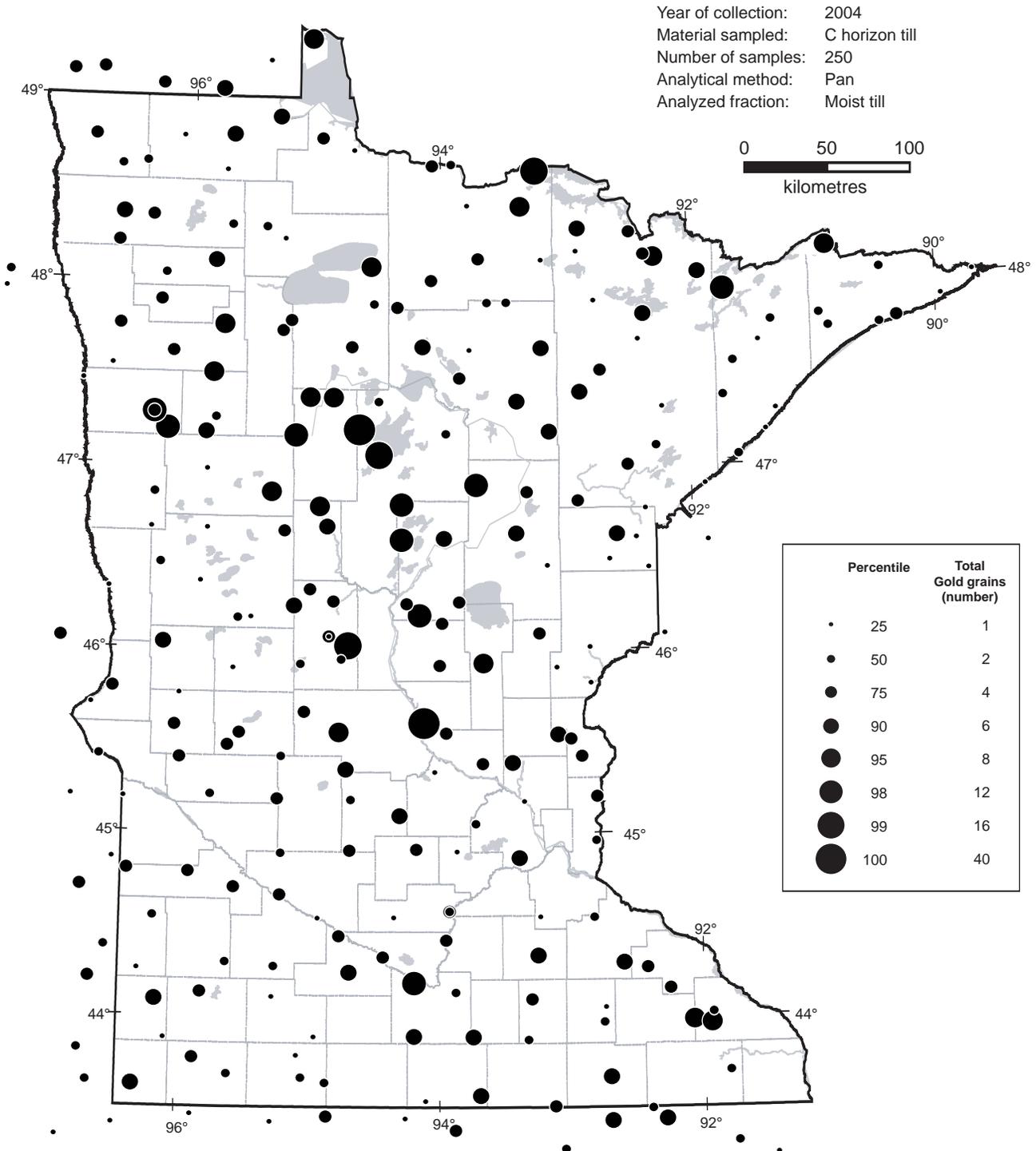
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Fig. 20. Partial zinc in the <0.25 mm nonferromagnetic heavy mineral concentrate (from Thorleifson *et al.* 2007).

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

## Total Gold Grains



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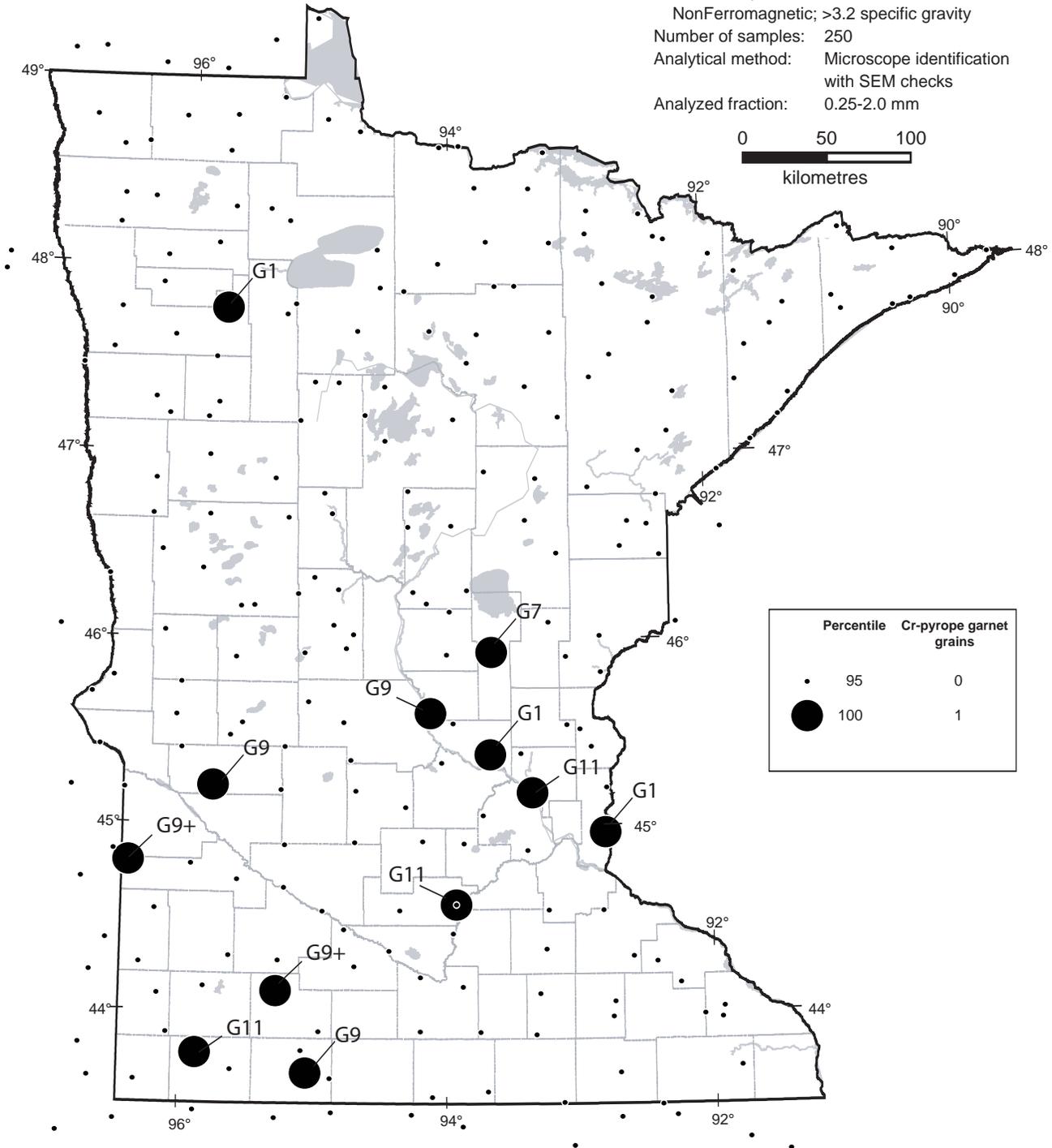
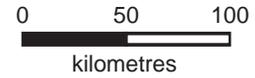


Fig. 21. Total gold grain count (from Thorleifson *et al.* 2007).

# 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



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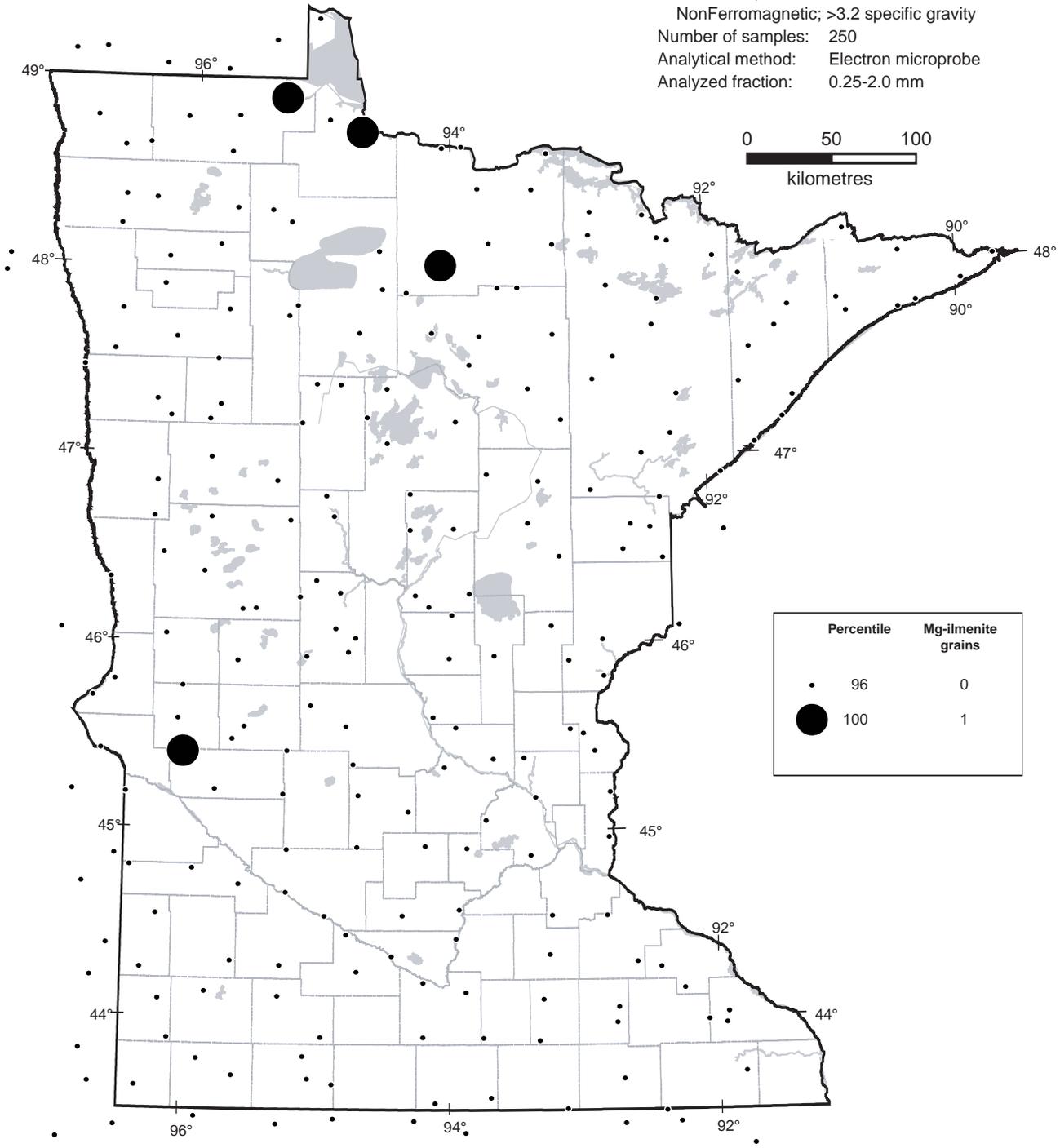


Fig. 22. Cr-pyrope in the 0.25 to 2 mm fraction (from Thorleifson *et al.* 2007).

# 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



Percentile	Mg-ilmenite grains
• 96	0
● 100	1

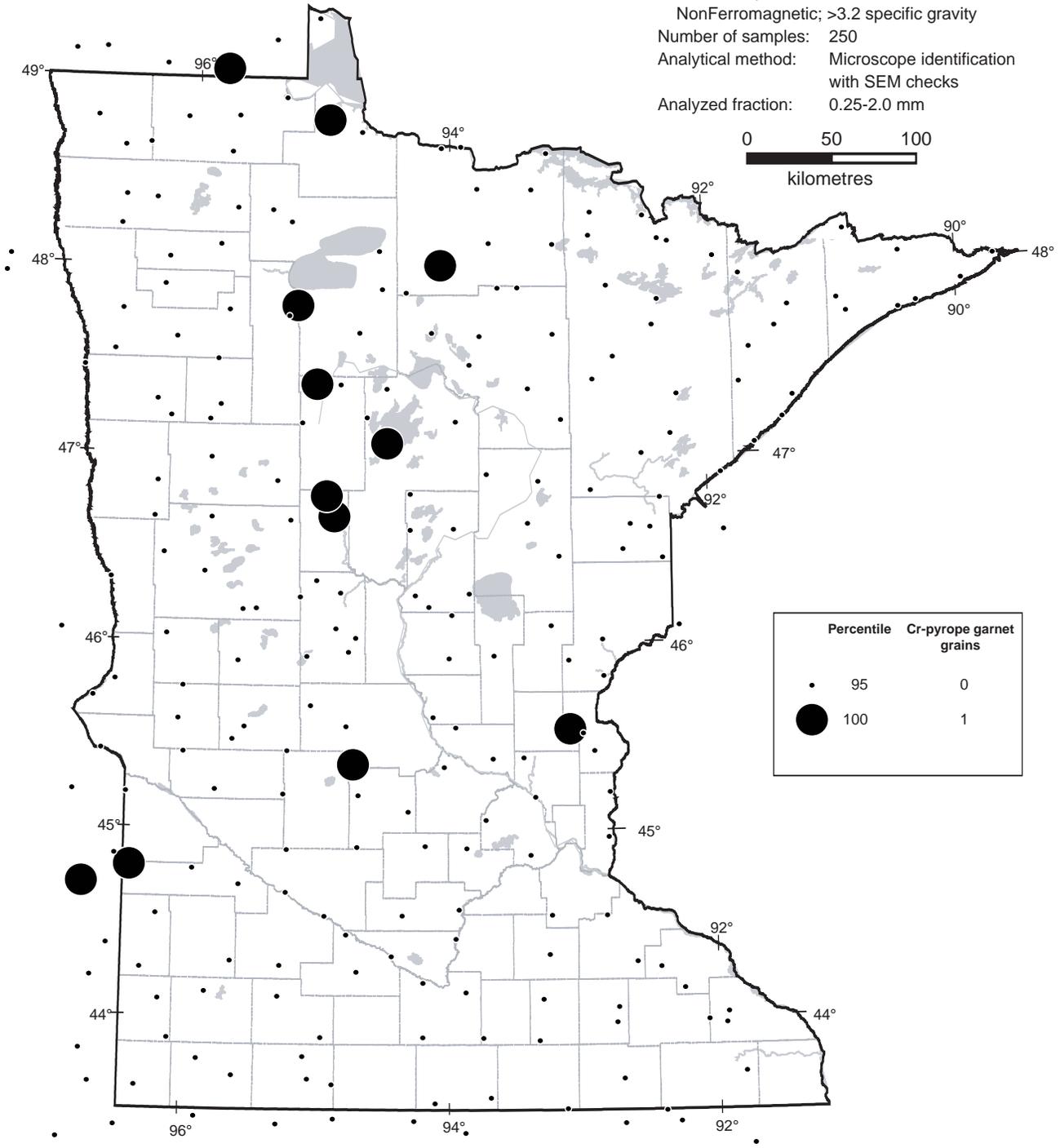


Fig. 23. Mg-ilmenite in the 0.25 to 2 mm fraction (from Thorleifson *et al.* 2007).

# 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



Percentile	Cr-pyrope garnet grains
• 95	0
● 100	1

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

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 north-eastern 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.

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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