Quality assurance and quality control measures applied to indicator mineral studies of glacial sediments at the Geological Survey of Canada

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There are a number of ongoing research projects at the Geological Survey of Canada (GSC) with the objective of enhancing exploration success through the identification of new indicator minerals hosted within, or associated with, a variety of mineral deposit types. These indicator minerals can be recovered from glacial sediments and traced back to their bedrock source as an effective mineral exploration method in glaciated terrain. As research on indicator minerals evolves, quality assurance and quality control (QA/QC) measures must be implemented to ensure that 1) in the field, samples are not contaminated from external sources or from other samples; 2) during sample processing and indicator mineral picking, loss of indicator mineral grains is minimized (close to zero), cross-contamination before and among sample batches does not occur, and minerals are correctly identified; and 3) all reported indicator mineral data include adequate metadata for future reference and comparison. To fulfill these needs, protocols have been developed for ongoing and future research projects at the GSC to ensure indicator mineral data are of the highest quality. These protocols satisfy the requirements of National Instrument 43-101 (2005), which specifies that technical information reported by exploration and mining companies in Canada (such as indicator mineral data) must include details of the quality assurance program being implemented.

These short course notes have been prepared specifically for short course SC07 *Application of Indicator Mineral Methods to Mineral Exploration* offered at the 26th International Applied Geochemistry Symposium, November 2013, in Rotorua, New Zealand. The notes have been presented in a similar workshop offered at the Prospectors and Developers Association of Canada (PDAC) Conference, March 2013 in Toronto, Canada (Plouffe *et al.* 2013) but have been revised based upon constructive comments received at PDAC workshop, and more recent results obtained as part of on-going GSC projects. These notes contain summary and key points addressed in Plouffe *et al.* (in press) and Spirito *et al.* (2011).

FIELD PROCEDURES

Detailed field procedures to accurately locate sample sites, record field notes, and identify the type of glacial sediments being sampled are addressed by Spirito *et al.* (2011). Metallic tools (shovels, picks, hammers, etc.) are generally used for the sampling of glacial sediments. At the onset of a field program, tools should be examined and thoroughly cleaned in order to reduce their role as potential sources of contamination. Paint, varnish, and other types of surface coatings should be removed as they are a potential source of contamination to heavy mineral concentrates. Sampling tools do wear with usage (Fig. 1), and produce metal shavings of various unnatural

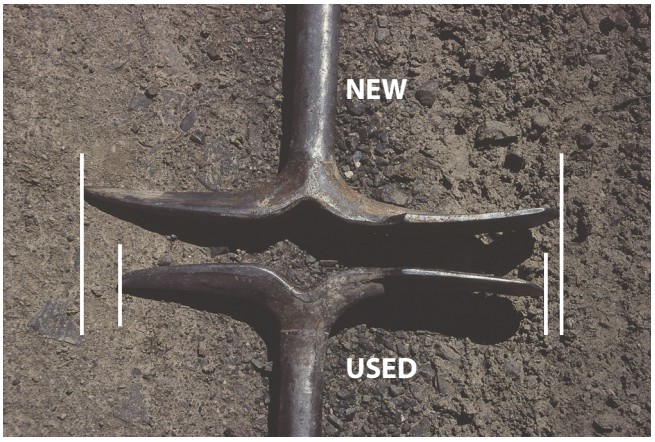

Fig. 1. Two hoe-picks of the same brand. The top one is nearly new and the bottom one has been used for three field seasons. Note that the old one has visibly lost metallic mass (distance between the white lines), which has, in part, ended up in sediment samples collected.

forms that should be expected and recognized in heavy mineral concentrates (Fig. 2). Glacial sediment samples collected from diamond drill core can be contaminated with industrial diamonds from the drill bit (Fig. 3). Tools should also be cleaned properly in between samples to avoid cross-contamination (Fig. 4A and 4B). As much as possible, samples should not be manipulated by bare hands or with dirty work gloves, which also represent a potential source of contamination (Fig. 4C). Finally, those collecting the samples should not wear hand jewellery because rings are known sources of contamination (Kontas 1991) and could impact geochemical analyses of heavy mineral concentrates.

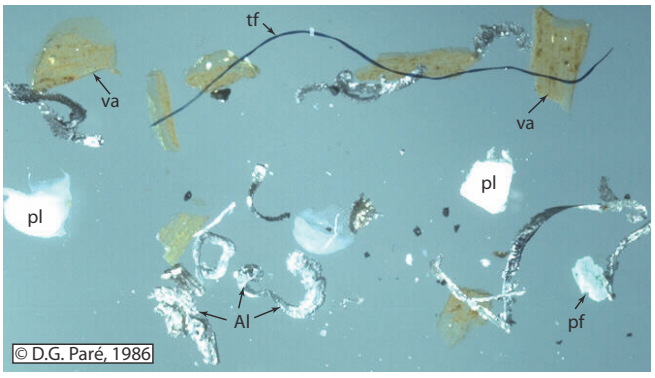

Fig. 2. Examples of contamination in heavy mineral concentrates including plastic from vial cover (pl), textile fibre (tf), aluminum shavings (Al), varnish flakes with traces of wood fibre (va), and paint flakes (pf); photographs from D.G. Paré, Consorminex Incorporated, Gatineau, QC (www.consorminex.com).

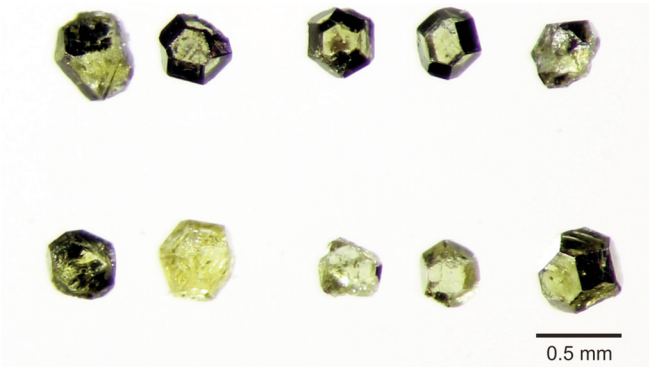

Fig. 3. Industrial diamonds recovered from the heavy mineral fraction of a till sample that was collected from diamond drill core.

Anthropogenic contamination of glacial sediments at sample sites may also affect the indicator mineral component and should be expected in areas proximal to present and past producing mines and related infrastructure (Bajc & Hall 2000; McMartin *et al.* 2002; Hozjan & Averill 2009; Michaud & Averill 2009). For example, gold spheres can be expected in samples collected close to gold mines with assay facilities (DiLabio *et al.* 1988). Although smelter particles can be small (for example 5 to 100 mm in Knight & Henderson 2006), larger particles (0.25 to 0.5 mm) can be expected near smelter sites (Fig. 5 and examples in Henderson *et al.* 1998; McClenaghan *et al.* 2013). At least in one instance, airborne smelter particles are suspected to have been introduced into till samples at the time of sampling (McClenaghan *et al.* 2013). Therefore, near anthropogenic sites, sample bags should be kept closed until the last moment of sample collection and samples should be collected at a minimum depth of 0.5 m (where possible) even if fresh unoxidized and undisturbed till is exposed at surface. All sites where anthropogenic contamination is suspected should be sampled with extreme caution and noted accordingly (Fig. 6). Near-surface till that appears to be undisturbed can actually be highly contaminated in these areas (McMartin *et al.* 1999). Knowledge of the sedimentological properties of till (cf. Evans *et al.* 2006) is essential to identify sediment genesis properly near anthropogenic deposits.

Samples should be collected in new plastic bags (>6 mil), plastic pails, or in metal pails lined with large plastic bags. Rice or cloth bags are not suitable for till or glaciofluvial sediment sampling as they are porous and easily torn, allowing the loss of fine-grained material and/or possible contamination of the sample. Care must be taken when transporting samples from field sites to the laboratory to avoid puncturing the sample bags which could also lead to loss of sample material and/or contamination. The chain of custody of all samples must be monitored and documented to prevent contamination or tampering.

Sample size suitable for recovery of indicator minerals is largely dependent on glacial sediment texture. To obtain an adequate number of sand-sized indicator mineral grains, a sample must contain an average of 5 to 10 kg of sand-sized material (0.063 to 2 mm) (Clifton *et al.* 1969; Averill 2001). Sample size may also be dictated by the range and type of analyses to be performed. In the field, a consistent sample size is collected based on volume, for example a full pail or full sample bag, knowing that a full pail or bag represents an approximate weight of material. The weight of the sediment will vary according to moisture content, sediment compaction, and composition. As a general guide, 10 to 20 kg of sandy-silty till, 20 to 40 kg of clayey till, and 12 to 25 kg of glaciofluvial sediment is required to obtain a representative heavy mineral sand-sized concentrate. At the GSC, samples are often not screened in the field to remove coarse clasts because silt and clay adhering to the larger clasts would be lost from the sample (in other words, potentially losing silt-sized indicator min-

Fig. 4. A) A dirty pick with sediment stuck to it after the collection of a sample, and therefore unsuitable for the collection of the next sample. **B)** A cleaned pick ready for the collection of a sample. **C)** Work gloves represent a potential source of contamination.

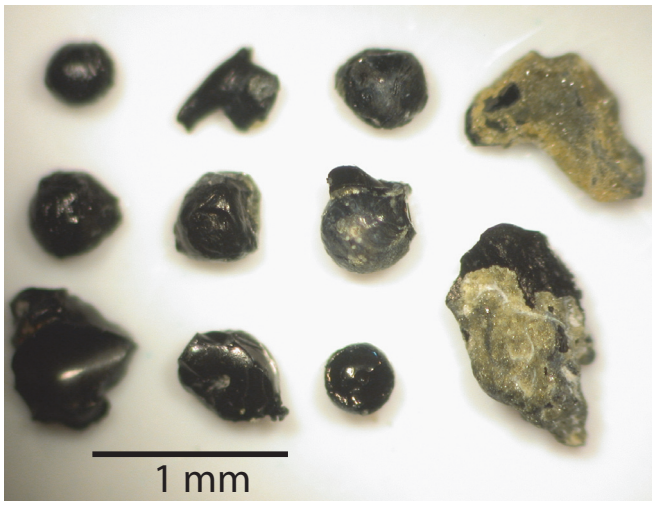

Fig. 5. Smelter particles recovered from till samples collected proximal to the Thompson Ni-Cu mine site in central Canada (McClenaghan *et al.* 2013).

erals such as gold and platinum group minerals), and sieves may represent an additional source of cross-contamination.

Field duplicate samples may be collected to serve two purposes. First, they provide some indication of sediment heterogeneity and site variability. In this case, the duplicate sample can be collected 5 to 10 m away from the original sample hole (site variability) (Fig. 7) or from the exact same sample hole (sediment heterogeneity). For example, duplicate samples collected 300 m down ice of the Izok Lake Zn-Cu-Pb-Ag volcanogenic massive sulphide deposit in Nunavut, Canada, demonstrate till heterogeneity near mineralization (McClenaghan *et al.* 2012). The original sample (09-MPB-016: 12.2 kg of <2 mm processed) contains no sphalerite grains and the duplicate sample (09-MPB-058: 11.8 kg of <2 mm processed), collected 5 m from the original site, contains 1500 sphalerite grains in the 0.25 – 0.5 mm fraction. Such a significant difference in mineralogical composition between samples could be a result of either 1) a weathered sphalerite-rich clast in the duplicate sample that was disaggregated during sample processing, or 2) sampling of a discontinuous layer of metal-rich till at the duplicate sample collection. On the other hand, at six field stations not located close to known mineralization, duplicate and matching original samples, collected approximately 10 m apart, were found to contain comparable mineralogy (McMartin *et al.* 2013). The sampling procedures for the duplicate samples need to be clearly stated with the reported results.

Second, field duplicate samples can be used to measure precision for the entire laboratory process — from heavy mineral separation to identification — if both samples (the original and duplicate) are completely homogenized. However, field or laboratory methods for homogenizing unconsolidated sediment samples that will then be used to measure laboratory precision for indicator mineral analysis need to be tested. Glacial sediment heterogeneity and site variability (as exemplified above with till), especially near mineralization, eliminates the possibility of using unmixed samples to measure laboratory precision. An incremental sampling methodology (www.itrcweb.org/ISM-1/Executive_Summary.html; see also their list of references) consisting of taking small sample increments and placing them alternatively in two separate sampling bags, as utilized for the sampling of contaminated soils, could be further tested as a means of collecting homogenized sediment samples for indicator mineral study.

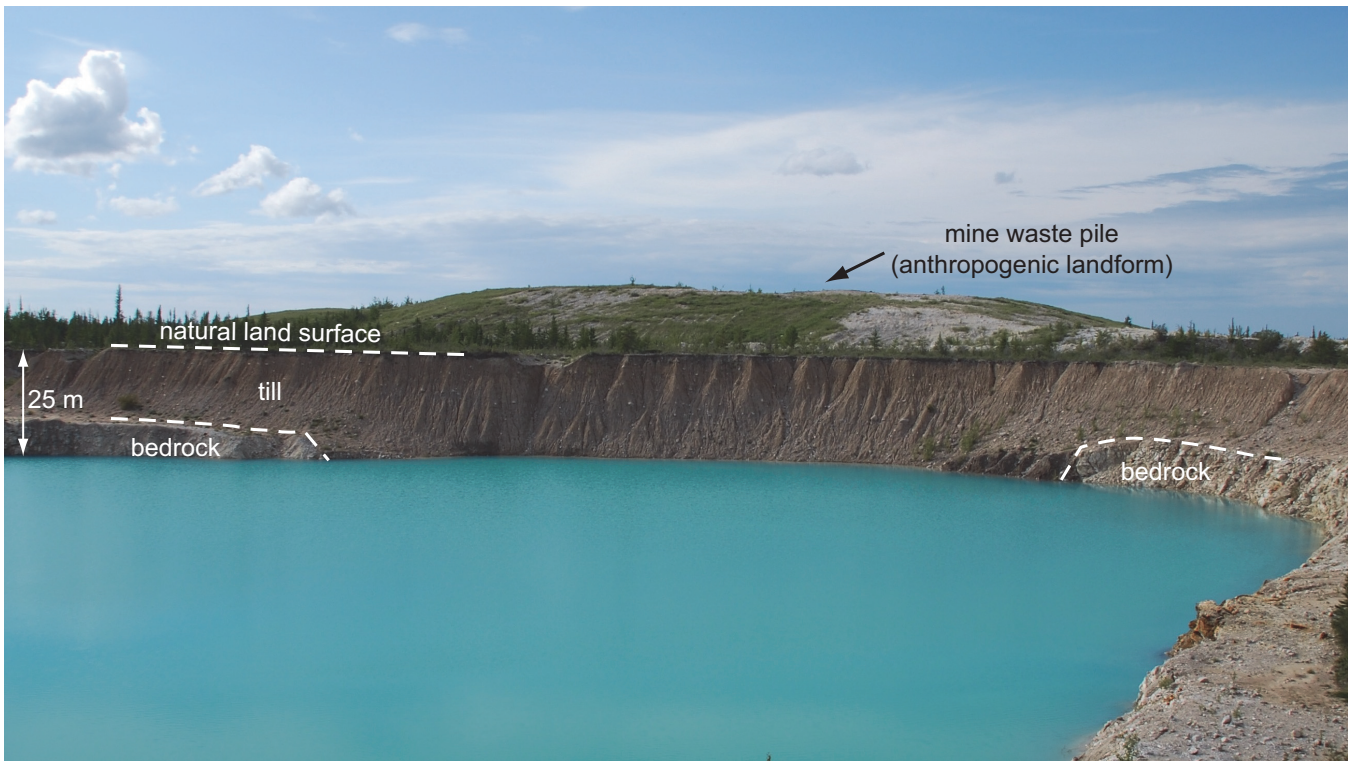

Fig. 6. A till sample site in a region heavily disturbed by anthropogenic activity at the old Pine Point Pb-Zn Mississippi Valley-type mining district, Northwest Territories. Till samples were collected in a former open pit mine, below the original natural land surface (Rice *et al.* 2013), and away from mine waste piles, which consisted of till excavated from the former open pit, mixed with other mine debris.

Fig. 7. Example of a till sampling site with a routine (black arrow) and field duplicate (white arrow) sample collected in the same sedimentary unit and at approximately the same depth.

Fig. 8. A) Sample site of the unconsolidated weathered Silurian-Devonian granite (grus) used for GSC blank heavy mineral samples. **B)** Close-up view of the blank sample material. **C)** Close-up view of the 2–4 mm fraction of the blank material.

PREPARING SAMPLES PRIOR TO INDICATOR MINERAL PROCESSING

Blank and spiked samples should be introduced into a sample batch prior to being shipped to an indicator mineral processing laboratory. Blank samples consist of unconsolidated earth materials devoid of indicator minerals of interest. Currently, the GSC utilizes as a blank a weathered granite (grus) collected from a Silurian-Devonian intrusion of the South Nepisiguit River Plutonic Suite (Wilson 2007) located approximately 66 km west of Bathurst, New Brunswick (Fig. 8). A blank is introduced as the first sample in a batch to monitor cross-contamination potentially derived from previously processed samples. A blank sample should also be introduced immediately after a sample known to contain large abundances of indicator minerals of interest (e.g. collected near known mineralization). In a large sample batch (N>200), a blank sample can be introduced randomly every 50 samples. Blank samples serve to detect cross-contamination, but cannot necessarily prevent it. For example, a blank sample of weathered granite introduced at the beginning of a sample batch at an external processing laboratory, detected the presence of pyrite contamination (10 grains) from another client's previously processed samples (McClenaghan *et al.* 2012). Similarly, contamination (chromite, ruby corundum, cinnabar, pyrite, and chalcopyrite) was detected in quartz blanks introduced at the beginning and throughout a batch of bedrock samples crushed by an electric pulse disaggregator and processed for indicator minerals (Normandeau & McMartin 2013). Again, the contamination was derived from another client's previously processed mineralized samples.

Spiked samples consist of base material into which spiking grains are voluntarily introduced. They are used to quantitatively monitor the effectiveness of a processing laboratory at recovering and identifying specific indicator minerals (Michaud & Averill 2009). These spiked samples are the equivalent to the secondary standards with known elemental concentrations used to monitor accuracy of geochemical analyses (McClenaghan *et al.* in press). The base material should be similar in texture to the rest of the routine samples and its mineralogy known as a result of repetitive indicator mineral separations and analyses. Currently, the GSC uses till recovered from a borrow pit near Almonte, Ontario as its base material for spiking. The material is texturally typical of till derived from the southern Canadian Shield and has an established average mineralogical composition based on repetitive indicator minerals analyses (Plouffe *et al.* in press). The spiking grains should be mineral grains of interest that have been either laser etched (Whiteford 2003) or photographed so that they can be recognized. The spiking grains should be carefully selected to ensure that they are not fractured or well-cleaved, as they could break into smaller particles during the sample processing (Hozjan & Averill 2009; Michaud & Averill 2009). If possible, spiking grains should be ones that have been recovered from other sediment samples. Spiking grains from crushed or disaggregated bedrock may differ in morphology compared to natural grains present in the sample and could bias the assessment of processing and picking recovery. Artificial density beads or cubes with specific size ranges and densities are commercially available and can also be used for spiking (Baumgartner 2006; Gent *et al.* 2011; McClenaghan 2011) with some limitations outlined in Plouffe *et al.* (in press). As part of the protocols implemented at the GSC, it is recommended that 2% of a sample batch be spiked samples.

Blank, spiked, and duplicate samples should have numbers similar to the routine samples so that they cannot be easily recognized by the processing laboratory as quality control inserts. A processing order for all samples should be communicated to the laboratory. For instance, to avoid carry-over contamination, samples known to potentially contain large amounts of indicator minerals of interest (e.g. collected near known mineralization) should be processed last.

LABORATORY PROCEDURES FOR THE RECOVERY OF INDICATOR MINERALS

Towie & Seet (1995), Gent *et al.* (2011), and McClenaghan (2011) provide a summary of several processing methods available for the recovery of indicator minerals from unconsolidated sediments. It should be emphasized that different processing methods will produce concentrates optimized for varying mineral species. Therefore, it is important to ensure that the method used in the laboratory is appropriate for the recovery of the target minerals. Furthermore, as part of the laboratory selection, Doherty (2009) recommends visiting the facility to identify potential steps in the processing where contamination might occur or mineral grains may be lost.

Plouffe *et al.* (in press) describe the heavy mineral processing methods adopted by the GSC. These methods have been utilized by the GSC in various geological settings with glacial sediments of varying textures for more than 25 years with satisfactory results. GSC methods include 1) pre-concentration of the <2 mm size fraction of a large sediment sample using a shaking table (material >2 mm is retained for the identification of clast lithologies); 2) micro-panning of the table concentrates for the counting and determination of the size of small mineral grains with high density (e.g. gold grains, platinum group minerals, uranium, and sulphide minerals); 3) further density concentration of the table concentrate with heavy liquids (typically specific gravity of 3.2 but can also be done at 2.8 and 3.0 depending on the minerals of interest); 4) extraction of the ferromagnetic fraction from the heavy mineral concentrates with a hand magnet; and 5) examination and identification of indicator minerals in three different size fractions: 1.0–2.0 mm, 0.5–1.0 mm, and 0.25–0.50 mm. Electromagnetic separations at precise amperages are used to further separate minerals in the 0.25–0.50 mm fraction as per their magnetic properties that facilitates mineral identification (e.g. McClenaghan 2011). The ferromagnetic fraction can also be examined for the presence of specific indicator minerals, such as pyrrhotite, and the recovery of magnetite (e.g. McMartin *et al.* 2011). Sample weights should be recorded at each step of the processing. Visual identification of potential indicator minerals is carried out using a binocular microscope and is aided by using a scanning electron microscope (SEM) and ultraviolet light.

AFTER RECEIVING DATA AND INDICATOR MINERALS FROM A PROCESSING LABORATORY

Once data are received from the mineral processing and identification laboratory, all QA/QC results should be examined and evaluated. The QA/QC results should then be communicated to the laboratory. Satisfactory results indicate to the lab-

oratory that their procedures are adequate for the recovery and identification of the reported minerals. In the case of perceived errors, laboratory procedures may need to be adjusted.

To verify the precision of the indicator mineral identification, approximately 10% of the mineral concentrates should be re-submitted (Doherty 2009; McClenaghan 2011). Picked mineral grains are usually not recombined with the heavy mineral concentrates, especially if they will be used for further analyses. Those samples should be re-labelled so that they cannot be identified by laboratory personnel.

The chemical composition of indicator minerals provides key information about the genesis of the mineralization, alteration, or bedrock lithology and, in some cases, the mineral fertility of a potential deposit (e.g. diamond grade of a kimberlite). A number of instruments and analytical methods are available to assess and/or determine the composition of mineral grains, including scanning electron microscopy equipped with an energy dispersive X-ray spectrometer (SEM-EDS), electron microprobe (EMP) analysis, laser ablation-inductively coupled plasma mass spectrometry (LA ICP-MS) (Jackson 2009), and secondary ion mass spectrometry (SIMS). Regardless of the selected analytical method, certified mineral reference standards and duplicate grain analyses should be used to monitor analytical accuracy and precision (de Souza 2006; Doherty 2009).

At the GSC, all indicator mineral data including original laboratory reports and mineral chemistry, as well as sample heavy mineral concentrates, unmounted picked grains, and grain mounts are permanently archived, using specific guidelines, for future reference (Spirito *et al.* 2011, 2013).

REPORTING INDICATOR MINERAL DATA

As a minimum, all GSC published reports with indicator mineral data include the following (from Plouffe *et al.* 2013, in press; Spirito *et al.* 2013):

- Sample medium: till, glaciofluvial sediments, stream sediments, etc.;
- Name of the processing laboratory;
- Name of the mineral identification laboratory (if different from the processing laboratory);
- Weights of material processed for recovery of indicator minerals (original sample weight, weight of table feed - <2 mm);
- List of blank samples and their type;
- List of duplicate samples and sampling methodology;
- List of spiked samples and their spiking grain content (size, morphology, and mineralogy);
- Pre-concentration method (e.g. panning, hydro-separator, shaking table, dense media separator, Knelson concentrator, jig, rotary spinal concentrator, other);
- Heavy liquid separation: name of liquid, and density;
- Magnetic separation: type of magnet used (e.g. hand magnet, Frantz, roll magnet, or other methods) and amperages if an electromagnet is used;
- List of all size and density fractions prepared and their individual weights;
- Weight and size range of fraction(s) examined for indicator minerals and percentage of concentrate examined for each sample if the complete concentrate was not examined;
- Mineral identification or characterization method: visual scan under the binocular microscope, MLA, quantitative evaluation of material by scanning electron microscopy (QEMSCAN), SEM-EDS, cathodoluminescence (CL), or other methods;
- Mineral chemistry determination method, machine operating conditions, and laboratory name for EMP, SEM-EDS, LA ICP-MS, other;
- Raw indicator mineral count data as reported by the picking laboratory;
- Indicator mineral count data as confirmed by EMP, SEM-EDS, or other methods;
- Indicator mineral count data as values normalized to total sediment weight processed (e.g. number of grains per 10 kg table feed of the <2 mm fraction).

Note: the total indicator mineral grain counts for an individual sample are never added together and reported as one number; they are reported separately for each size fraction.

CONCLUSIONS

Indicator mineral sampling surveys represent a significant investment and, as such, implementation of proper QA/QC measures at all stages of surveys, from field to archive, will ensure that the data generated are of the highest quality possible. The GSC protocols presented in these short course notes will continue to be improved over time as more samples are processed, and as mineral separation and identification procedures at commercial laboratories evolve and improve.

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