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Counting a pot of gold: A till golden standard (AuGC-1)

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ABSTRACT

Determining the number of gold grains in till is a widely used exploration technique in glaciated terrain. Nonetheless, there is no existing reference material to assess the quality of either the recovery or the counting of gold particles in samples from glaciated sites. We manufactured a reference material (AuGC-1) by mixing a gold-free heavy mineral concentrate with gold grains recovered from mine tailings. The reference material contains 38 gold grains, with a standard deviation of 10 grains, in 1.5 g of heavy mineral concentrate. Of the 38 gold grains, 24 grains are >50 μm (optically determined), and 14 grains are <50 μm (using SEM). This grain concentration is considered optimal, well above Canadian Shield till background and sufficient for obtaining reproducible statistics. Gold grain reference materials can be improved further by ensuring that i) the grain size distribution of gold is similar to that of the matrix; ii) gold grains in the sample originate from different sources or a source having a great variation in gold grain composition; iii) a slightly larger number of gold grains are present in the samples (closer to 50 grains) to reduce the standard deviation; and iv) a larger sample size is used to improve mixing of the high-density gold grains with the mineral matrix.

1. Introduction

The chemical and mineralogical analysis of heavy minerals within glacial sediments is a commonly used exploration technique in areas where these sediments cover most of the landscape (Brundin and Bergstrom, 1977; McClenaghan, 2005). Large-scale mineral deposits have been found through the use of indicator minerals. Examples include diamonds, gold, platinum-group elements (PGEs), rare-earth elements (REEs), and base metal deposits (e.g., Plouffe et al., 2013; McClenaghan and Cabri, 2011; Averill, 2017; McClenaghan and Kjarsgaard, 2007; Averill and Zimmerman, 1986). Whereas till geochemistry is a useful technique in mineral deposit exploration (Shilts, 1984; McClenaghan and Paulen, 2018), indicator mineral counts provide additional information of the presence and proximity of ore deposits or related alteration, even when indicator minerals occur at low concentrations (McClenaghan, 2005; Gent et al., 2011; McClenaghan and Paulen, 2018). The indicator mineral technique requires sampling 5 to 40 kg of unconsolidated sediments, most commonly till or stream sediments (McClenaghan, 2011; Plouffe et al., 2013) and then extracting the heavy mineral concentrate (HMC) from which indicator minerals are identified, counted, and analyzed. Nevertheless, the reliability of this technique and the quality of the indicator mineral count data are difficult to evaluate because reference materials are unavailable. Currently, quality assurance–quality control (QA-QC) for indicator mineral surveys is limited to the use of blanks, duplicates, and spiked samples (Plouffe et al., 2013; McClenaghan et al., 2000).

When prospecting for gold deposits, there is little need for other indicator minerals, as gold grains are themselves concentrated in the HMC in a larger abundance than their associated indicators. Although gold could be easily quantified by chemical assaying, the characterization of gold grains (e.g., abundance, size, shape, individual grain chemistry) provides valuable information related to the type of ore deposit, the depth of erosion, and the transport distance (Townley et al., 2003; DiLabio, 1982, 1985, 1991; Shelp and Nichol, 1987; Averill, 2001; McClenaghan and Cabri, 2011). Therefore, the recovery, identification, and characterization of gold grains provide much more information than obtained through simple geochemical analyses.

The identification and counting of gold grains are commonly performed by visual sorting (i.e., picking) by trained professionals. The process can be tedious, time-consuming, and highly dependent on technician skill. The fine grain size of free gold produces the initial challenge for technicians. At the source, gold is found predominantly as

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particles sized at <50 μm, and 50% of the grains are commonly sized at <20 μm (Fig. 1a). Erosion and transport by glaciers do not significantly change particle size (Fig. 1b). The <10 μm grains, however, are absent from till data owing to either the difficulty of isolating these very fine grains when using a binocular microscope (Averill, 1988) or a recovery collapse for grains <20 μm (Girard et al., 2021). The second challenge for technicians is related to the possible occurrence of gold alloys involving other metals. These alloys give the gold grains different hues (Leuser, 1949), leading to possible confusion with other minerals such as sulfides and, therefore, lower gold grain counts.

During the last few years, several automated techniques have been developed to identify (Maitre et al., 2019) and count minerals in HMC (Lougheed et al., 2020; Cook et al., 2017; Crompton et al., 2019) and therefore limit the potential for human error while also decreasing sample processing time. Despite such improvements, there is still a lack of quantitative quality control of gold grain counting; no reference material exists to monitor the quality and uncertainties of automated and manual techniques. Therefore, all reported results remain debatable. It is common industry practice, as a quality control measure, to add known mineral grains to the samples, a technique referred to as "spiking," and then verify whether the added grains are recovered afterward. These spikes can be natural indicators inserted into a reputedly barren sample, laser-etched indicators that can be distinguished from natural grains, or artificial materials, such as brass chips, zirconium beads, or tinted heavy glass fragments (Michaud and Averill, 2009; Lamarche, 2016; Towie and Seet, 1995; Elrich and Haussel, 2002). The difficulty with gold relates to its small grain size (< 50 μm), which cannot be manipulated realistically to manufacture spiked doses containing an accurate number of grains. Therefore, well-characterized natural materials are required to produce a statistically robust reference material that is similar to a natural sample. Such a reference material would be a major development in the validation of gold grain recovery and counting data for till samples, not only scientifically but also legally because of the stringent QA-QC requirements for reporting in mineral exploration projects, regardless of the reporting code (e.g., National Instrument NI 43–101 (2011), JORC (2012), SAMREC (2016)).

Here we describe the steps taken to produce a reference material, named AuGC-1, which can be used for quality control for either automated or manual gold grain counting or as a spike that could be added to a till sample to test the efficacy of gold recovery and counting during indicator mineral studies.

2. Gold grains in glaciated terrain and the characteristics of a targeted reference material

We use till samples from various formerly glaciated regions of Canada as material for this study. During the Pleistocene glaciations, ice covered approximately 80% of Canada, resulting in an extensive till cover throughout the country (Stokes et al., 2012; Fulton, 1995). Therefore, all geological provinces, and their respective ore deposits having gold as a major or trace constituent, potentially contributed gold grains to till deposits that overlie bedrock. A compilation of several regional till surveys reported by the Geological Survey of Canada (GSC) was used to calculate an overall till gold grain background to be considered when assembling our reference material.

We compiled a total of 4142 samples, and only those samples having counts below the 95th percentile were used to calculate the average background gold grain count; we considered samples in the upper 5th percentile as anomalies. These calculations suggest a Canadian average of 5 grains/10 kg with a standard deviation (σ) of 6 grains/10 kg. Applying the three-sigma (3σ) rule—a rule of thumb stating that 99.7% of the data fall within the three standard deviations of the mean—brings the estimated maximum background value to 23 grains/10 kg. Background calculations for individual surveys (Table 1) also highlight

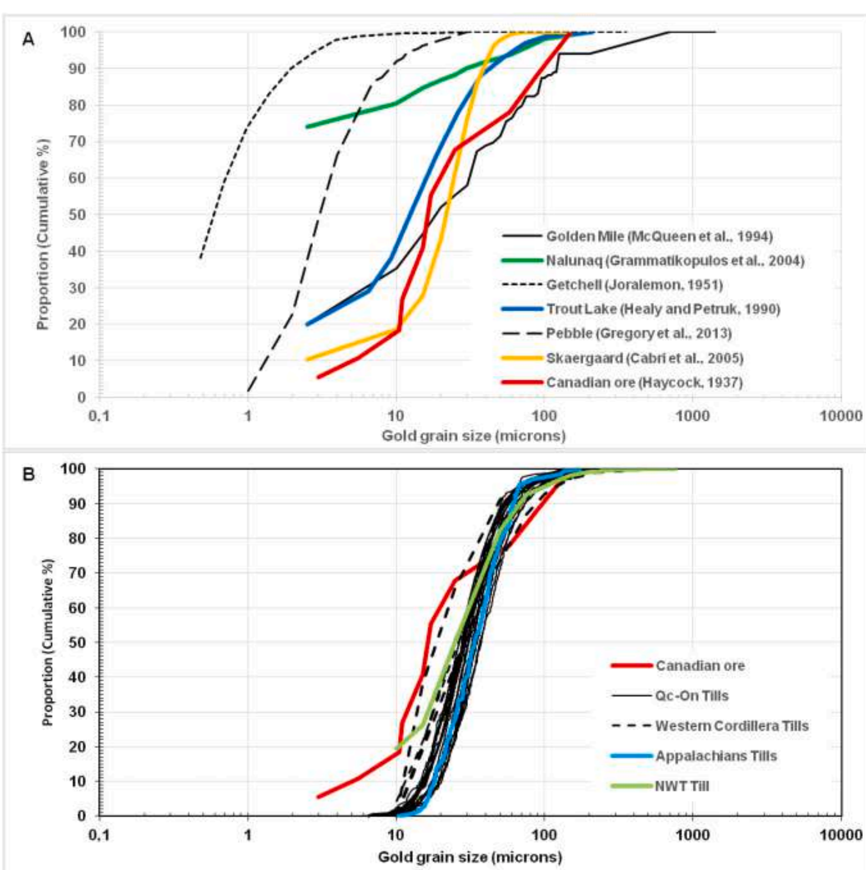

Fig. 1. Gold grain size distribution in A) orogenic gold deposits (Golden Mile and Nalunaq), a Carlin-type deposit (Getchell), a gold-bearing volcanogenic massive sulfide (VMS) deposit (Trout Lake), a porphyry deposit (Pebble), a gold-bearing platinum-group element (PGE) deposit (Skaergaard), and undifferentiated Canadian gold deposits. B) Cumulative gold grain size distributions for tills from different areas of Canada: Western Cordillera (Plouffe et al., 2013; Plouffe, 1995; Plouffe and Ferbey, 2016; Ferbey et al., 2016), Northwest Territories (Kerr, 2002), Quebec-Ontario and Appalachian tills (Girard et al., 2021). The Canadian ore size distribution of Haycock (1937) is included for comparison. Grain size for Getchell is reported as the diameter of spherical grains equivalent to the surface area reported by Joralemon (1951). Data for Pebble (Gregory et al., 2013) and Trout Lake (Healey and Petruk, 1990) are derived from histograms.

Table 1
Gold grains within tills covering various Canadian geological provinces, using the values below the 95th percentile. [a]Mean + three standard deviations (3σ). [b]There was no mention of sample weight; therefore, we set the sample weight at 7.5 kg (between 5 and 10 kg). [c]Duplicate samples were averaged. AB: Alberta, BC: British Columbia, NB: New Brunswick, NL: Newfoundland and Labrador, NS: Nova Scotia, NT: Northwest Territories, NU: Nunavut, ON: Ontario, QC: Québec, SK: Saskatchewan. Data for Borden include grain counts using the conventional method (CM) and ARTGold technology (AG). Data sources: 1: Plouffe et al. (2013), 2: Plouffe and Ferbey. (2016), 3: Ferbey et al. (2016), 4: Plouffe (1995), 5: McMartin et al. (2019); 6: McMartin (2000); 7: Henderson (1995), 8: Tremblay and Leblanc-Dumas (2015), 9: Kerr and Knight (2007); 10: Bajc (1991), 11: McClenaghan et al. (1998), 12: Girard et al. (2021), and 13: Plouffe et al. (2006).

Geological province	Location	Background (grains/10 kg)		Samples (*n*)	Reference
		Mean	Maximum[a]		
Cordillera	Bonaparte Lake, BC	8	39	462	1
	Highland Valley, BC	3	9	108	2,3
	Mount Polley, BC	7	29	86	2
	Gibraltar, BC	2	9	94	2
	Woodjam, BC	3	10	90	2
	Central BC	1[b]	4[b]	173[c]	4
Rae	Wager Bay, NT	<1	3	129	5
Churchill	Meliadine Trend, NU	28	128	103	6
	Annabel Lake, SK	11	47	48	7
	Hall Peninsula, NU	0	3	111	8
Slave	Var. locations, NT	6	26	514	9
Superior	Fort Frances, ON	2	12	99	10
	Timmins, ON	4	15	138	11
	Borden, ON (CM)	1	4	833	12
	Borden, ON (AG)	9	29	1092	12
Int. Platform	Northwest AB	0	<1	61	13

significant variations between regions; for example, the Meliadine Trend in the Churchill Province has a maximum background of 128 grains/10 kg of till, whereas in other areas, even within the same geological province, have a background of <1 gold grain/10 kg of till. Given that AuGC-1 is a first attempt at producing a reference material, we chose to avoid a region-specific background but rather rely on an overall Canadian background.

For a reference sample to be effective in monitoring gold grain recovery and counting, it must have a sufficient number of gold grains to be statistically representative and easily discriminated from background samples. At 5σ (99.997%) and 8σ (highly improbable), gold grains in the sample add up to 35 and 53 grains, respectively. For convenience, we decided to target 50 grains for the reference sample.

Gold grain size is important when producing a reference material, as gold recoveries depend heavily on size (Girard et al., 2021). Fig. 1b shows the size distribution of gold grains in till and its relationship to gold particle size at the source. The reference material should have a similar gold grain size distribution; thus, the sizing is ideally between 15 and 250 μm with 80% of the grains finer than 50 μm.

A reference material that can be used to directly evaluate the efficacy of grain counting must have the gold grains present within a granular material similar to the HMC extracted from till. The mass of the reference material must be small enough that i) all grains can be examined within a reasonable amount of time, and ii) sample splitting is not required, therefore limiting potential grain loss due to superfluous sample handling. Nonetheless, the reference material mass must be sufficient to contain several thousand grains, as would a regular HMC extracted from till. From our experience, we chose 1.5 g as the reference material mass. Such a small mass would also enable this HMC reference material to spike a standard till sample and test gold grain recovery during sample processing. The addition of 1.5 g of reference material into 10 kg of sample is insignificant as far as mass is concerned. Because the reference sample matrix is a HMC, extra precautions must ensure that this HMC material is gold-free.

A major aspect of QA-QC is the reproducibility of the results. Moore (1979) proposed a series of equations to evaluate the coefficient of variation (CoV) for geochemical materials on the basis of the size of the contaminant particles (e.g., target elements/minerals). Using these equations, we calculated that a sample having 50 gold grains in a 1.5 g matrix would have a CoV of 14%, an acceptable value for the proposed reference material. Some assumptions must be made about grain size distribution that may differ slightly from those of Moore (1979), but his equations nonetheless serve as a guideline.

3. Sample preparation

3.1. Gold grains

The production of artificial grains to serve as a reference material is not easily achievable. Producing gold particles from man-made gold thread is difficult because gold's malleability prevents the use of any grinder. Also, ground gold grains can easily be visually confused with brass grains, a common contaminant if brass sieves are used. Gathering gold grains from till samples to use as reference material is risky, as the content is naturally variable and is not guaranteed even in gold-rich regions, such as the Abitibi region of Quebec, Canada. We collected gold grains for the reference material from the tailings of the closed Anacon (Tétrault) Mine located in Montauban-les-Mines, northwest of Quebec City, Quebec (Bernier et al., 1987). The mine produced 2.7 Mt. of ore between 1912 and 1966 (Turcotte et al., 2014). The deposit is a metamorphosed gold-bearing volcanogenic massive sulfide (Jourdain, 1993), and the tailings are known to contain a significant concentration of gold grains. A bulk sample of the tailings was preconcentrated on-site by the current deposit owner using a Wifley table. We used 1.5 t of the preconcentrate for this project.

The preconcentrate was wet-sieved at 1 mm, yielding about 80% of <1 mm material (Appendix A). The material was then processed for gold grain recovery using a fluidized bed, a proprietary technology used for till processing (Girard et al., 2021). This technique enables the efficient recovery of gold grains as fine as 5 μm. The 1200 kg of <1 mm material was reduced to 3.2 g of gold-rich heavy mineral concentrate (GC).

The GC was characterized at 40× using a stereomicroscope. The GC is composed, in order of abundance, of sphalerite, pyrrhotite, gold, hematite, galena, and magnetite. It also contains 5% of lesser minerals, including hypersthene, zircon, garnet, mica, amphibole, and pyrite. The occurrence of micas in the GC can occur because of particle adherence or dragging by heavier grains. Gold grain size varied between 35 and 640 μm.

To obtain an estimate of the number of available gold grains, we counted grains in the GC using computed tomography (CT) with a Bruker 1172 Skyscan from McGill University and applying the parameters listed in Table 2. Images were reconstructed using Bruker's NRecon software, and statistics were calculated from the results using a CT analyzer. We used microtomography (micro-CT) (which produce a 3D density model of the sample) to obtain realistic counts of gold grains to avoid opening the sample vial and the potential contamination or loss of gold grains.

The results from the micro-CT analysis were of poor quality. Preliminary testing suggested the large number of gold grains created significant interference, which affected the gold grain counting. Thus, the GC was diluted with a gold-free material (i.e., salt) and split into ten subsamples (diluted gold concentrate; dGC, e.g., dGC#1).

Table 2
Parameters used for data acquisition using Bruker's 1172 Skyscan micro-CT.

Parameter	Specification
Voltage	100 kV
Current	100 mA
Filter	Al–Cu
Number of rows	524
Number of columns	1000
Camera binning	4 × 4
Image pixel size	7.95 μm
Object to source	48.130 mm
Camera to source	280.121 mm
Filter	Al–Cu
Rotation step (total rotation)	0.680° (180°)
Scan duration	00:18:55

We acquired six 3D images of sample dGC#1 (sample mass = 1.95 g). Gold grain counts of the sample averaged 6062 (SD ± 1214) (Table 3). The variability between counts resulted from interference owing to high grain abundance. At high concentrations, gold grains touch each other, which reduced the precision of grain counts by micro-CT, and the ability to discriminate touching grains becomes highly dependent on the CT settings. This variability in grain counts and the interpreted interference observed in the original GC sample highlighted the difficulty counting gold grains in a GC. It also clearly demonstrated the difficulty in identifying adjacent grains of similar density using micro-CT (Kyle and Ketcham, 2015). No images were acquired for dGC#2 to dGC#10.

We had used micro-CT to estimate the number of gold grains in the GC in order to add the appropriate quantity of HMC to produce a reference material with approximately 50 gold grains per 1.5 g of sample. Therefore, we did not use more time-intensive means to obtain more precise micro-CT results. Applying a low estimate of 4848 gold grains (average – SD) for dGC#1 (Table 3), we calculated that dGC#1 (1.95 g) contained sufficient grains to produce 97 vials of reference material, each containing on average 50 grains. Targeting a mass of 1.5 g per dose thus required diluting dGC#1 with 143.55 g of gold-free HMC matrix. This added matrix material must be similar to that obtained through regular till processing.

3.2. Till (matrix)

Although complex in-ice processes control particle transport (Hooke et al., 2013), till composition is related to the bedrock located up-ice from the till sample site. Because there are no known gold deposits in the Lac-Saint-Jean area (Quebec, Canada), we sampled till at Saint-David-de-Falardeau, located just beyond the lacustrine deposits of the Laflamme Sea (Leduc, 2016). The HMC extracted from this till served as the matrix to which the GC was added to produce the reference material. The occasional presence of a gold grain in this material would not significantly influence the counts of the reference material.

The 10 kg till sample was first wet-sieved at 1 mm, and the fine material was then processed using a shaking table to produce the HMC.

Table 3
Estimates of the number of gold grains in sample dGC#1. Avg: average, SD: standard deviation.

Count trial	Grains (*n*)	Avg. grain size (μm)	Mode (μm)
1	5087	93	45
2	5033	110	55
3	7139	84	40
4	5964	85	45
5	5220	85	40
6	7930	81	40
Average	6062	89	40
Avg −1 SD	4848	–	–
Avg +1 SD	7276	–	–

We used about 200 g of this HMC material. Stereomicroscope examination of the HMC indicated that the sample was composed of hornblende, amphiboles, pyroxenes, several other accessory heavy minerals, and a few grains of quartz and feldspar, but no gold or sulfides. Inductively coupled plasma–mass spectrometry (ICP-MS) analyses of three 0.5 g aliquots of the HMC, dissolved in aqua regia at the Earth Material Laboratory (LabMaTer) of the Université du Québec à Chicoutimi, yielded a gold concentration of 6.33 ± 0.67 ppb. If all gold occurs as free grains, such a grade could be caused by the presence of a single 7 μm diameter gold grain in the sample. Assuming that the HMC is homogeneous and that all the gold is present as free particles, we should expect a maximum of 400 gold grains of 7 μm diameter to be found in 200 g of this HMC. Because gold grains are rarely coarser than the HMC (Fig. 1), it is reasonable to assume that the entire 10 kg of collected till might contain up to 400 gold grains. At first glance, this value would seem concerning. However, if the 400 grains are truly present, i) they would be split between the 97 aliquots (approximately 4 grains per aliquot), and ii) 7 μm diameter gold grains are too small to be detected by visual counting techniques (Averill, 1988). For these reasons, we can consider the collected till sample and the extracted HMC as being gold-free. For the worst-case scenario in which the HMC adds a few gold grains, the final gold grain count will include these few added grains.

3.3. Blending the gold and the HMC of the collected till

A major challenge when creating a reference material for grain counting is ensuring that gold grains are evenly distributed within the HMC matrix. We blended the 1.95 g of spike (dGC#1) and 143.55 g of matrix by combining both in a mixing bottle and then shaking the bottle for 45 min, regularly changing the bottle's orientation to prevent settling. Applying a bed-blending methodology (Gy, 1981), we then poured the material into a V-shaped container over multiple passes. We scooped aliquots from the V-shaped container using a laboratory spatula and poured the 1.5 g aliquots into individual vials amenable for X-ray microtomography. We reserved 56 of the 97 aliquots for future studies. Rotary splitters have not been used because, to our knowledge, no rotary splitter can handle such small size samples.

3.4. Characterizing the AuGC-1 reference material

3.4.1. Number of grains

We attempted grain counting on 21 aliquots using computed tomography (micro-CT). Although this technique does not require unsealing the vials, thus preventing the loss of gold grains during manipulation, it is a time-intensive approach. Therefore, voxel (i.e., a 3D pixel) resolution must be reduced to accelerate analyses. Consequently, the success of this method becomes highly sensitive to technical parameters and user skill (Kyle and Ketcham, 2015). As we observed great variability in our counting based on micro-CT, we determined this approach to be unsuitable for this stage of material characterization.

We then counted gold grain abundance in the aliquots by direct sorting using the automated SEM-based ARTGold™ method (Girard et al., 2021). The aliquots were sieved at 50 μm using a disposable single-use mesh. We visually sorted the >50 μm fraction using a high-magnification research-grade stereomicroscope, whereas the <50 μm fraction was scanned by SEM backscattered electron imaging. Coarse (>50 μm) grains must be removed for BSE scanning because of the shadowing of finer grains under the electron beam. Both visual and SEM counts were summed and considered as a reliable total count.

The ARTGold™ method provides reliable counts down to a grain size of 2 μm (Girard et al., 2021). Visually sorted grains are of sufficient size for reliable identification, and a second mineralogist re-examined the initial counts. The 21 aliquots (Table 4) yielded an average count of 38 gold grains (Table 4), fewer than expected based on the micro-CT data. Nonetheless, as discussed above, the micro-CT results served only as a guideline and may not be very accurate. The occurrence of 38 gold

Table 4
The number of gold grains in each of the 21 analyzed aliquots of the AuGC-1 reference material as determined using ARTGold™ technology. Note: coarse grains were counted visually using a stereomicroscope, and fine grains were counted using a SEM. CoV: coefficient of variation.

Aliquot	Coarse grains (>50 μm) (*n*)	Fine grains (<50 μm) (*n*)	Total (*n*)
910-04	32	27	59
910-05	30	28	58
910-07	15	19	34
910-09	26	16	42
910-14	27	15	42
910-16	18	13	31
910-19	22	10	32
910-20	27	18	45
910-24	21	11	32
910-27	25	14	39
910-30	26	10	36
910-31	18	22	40
910-32	27	15	42
910-34	18	4	22
910-43	18	7	25
910-44	20	4	24
910-47	34	15	49
910-48	17	5	22
910-51	23	9	32
910-55	23	7	30
910-56	28	16	44
Average	24	14	38
St. dev.	5	7	10
CoV	22	50	28

grains in a sample closely matches the average Canadian background plus 5σ, an abundance appropriate for reference material. There is a distinct difference, however, when comparing the coefficient of variation (CoV) for the coarse fraction, which is identified by a trained mineralogist, and the one for the fine fraction, which is identified by SEM routine.

3.4.2. Grain size distribution

Grain size is one of the main characteristics affecting the efficacy of both manual and automated counting techniques. The gold grain reference material should have a similar grain size distribution to the HMC extracted from till. The reference material aliquots share a relatively similar size distribution (Fig. 2), although the proportion of grains at 50 μm varies between 9% and 59%. Sample 91020034 is clearly coarser than the other samples, whereas sample 91020007 is slightly finer. Removing these two samples brings the cumulative proportion at 50 μm to between 18% and 48%, a more reasonable range.

3.5. Grain chemistry and color

The color of gold grains could affect visual counting. The specific color depends on chemical composition (Fig. 3). Considering that color variability has been noted during the stereomicroscopic identification of the coarse fraction from the GC (Fig. 4), composition using XRF-EDS were assessed under SEM to further characterize the gold grains of the reference material.

All grains examined from in the 21 aliquots are copper-free electrum, plotting close to the Au-Ag axis of the Leuser gold color chart (Fig. 3): whitish to pale greenish yellow. None of the grains in the reference material plot in the reddish fields, an observation that is expected because only traces of copper are found in gold grains in nature, whereas Ag-Au alloys (e.g., electrum) are common (Townley et al., 2003). The few grains having a higher Cu content could be gold grains on which small particles of Cu-bearing sulfides are attached. A brownish–blackish luster, however, was observed on several gold grains. This tarnish is

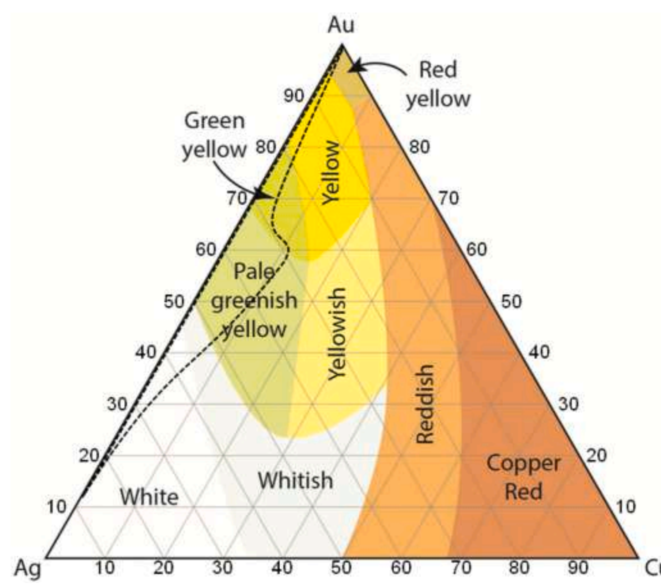

Fig. 3. Distribution of the gold grains from the reference material (polygon highlighted by dashed line) within the gold color triangle (modified from Leuser (1949)). These colors refer to grains with pristine luster, whereas our study material grains were heavily tarnished toward the brownish hues. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

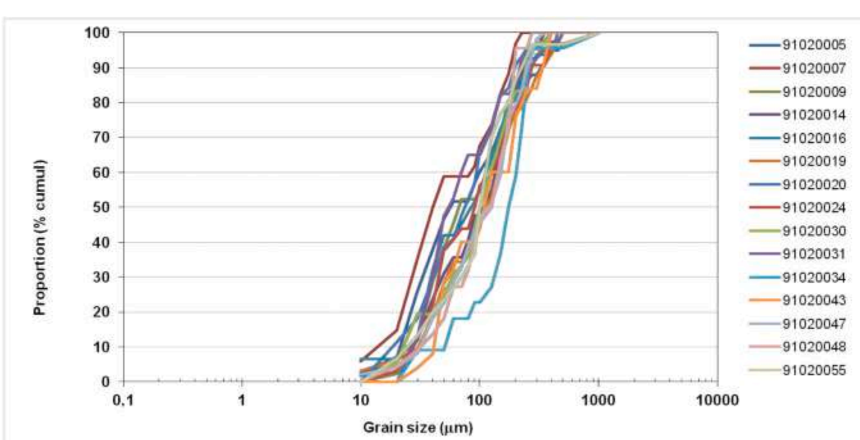

Fig. 2. Grain size distribution for 15 aliquots of the AuGC-1 reference material. Grain size was measured using SEM images of both fine (<50 μm) and coarse (>50 μm) grains.

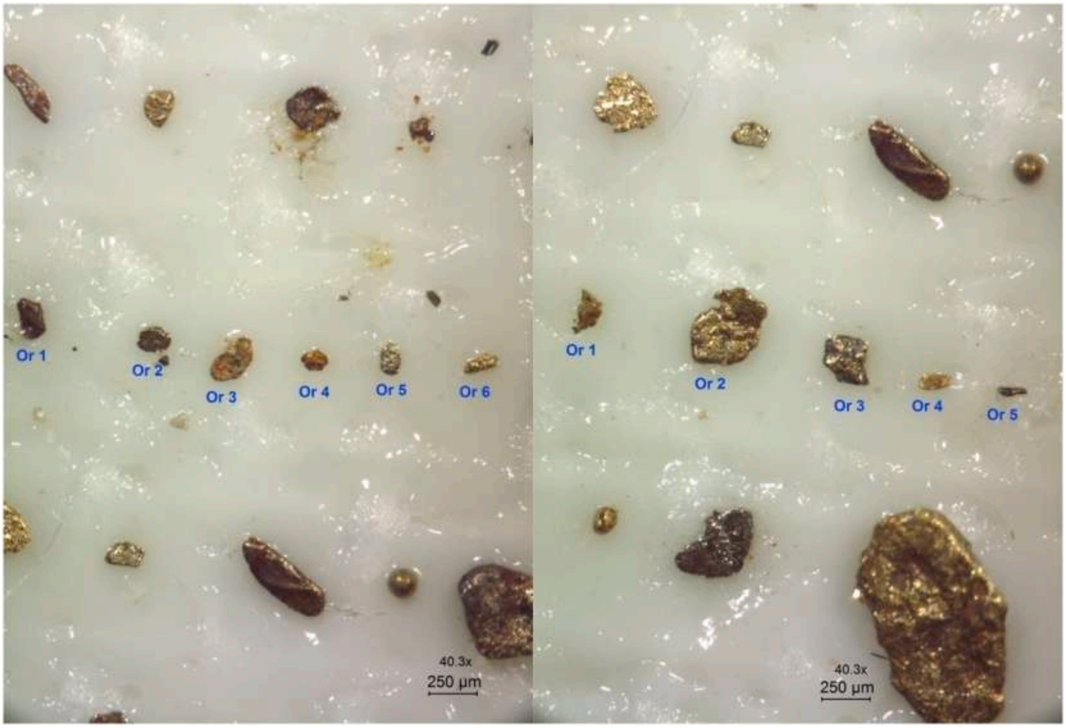

Fig. 4. Stereomicroscope (40×) images of gold grains from aliquots #16 (left) and #21 (right) showing the variability in color. All grains were confirmed as gold by XRF-SEM. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

produced by an iron coating, which is expected because the gold grains come from an oxidized sulfide-rich material. Such tarnishing may fool less experienced mineralogists undertaking the visual sorting. This is important as the counting technique must be sufficiently flexible to recognize gold grains that vary in color to avoid any bias related to a particular chemistry (and possible ore type).

4. Discussion

There are two aspects of the AuGC-1 reference material that require attention. The first is the number of gold grains counted within the reference material, and the second is the grain size distribution.

Our average of 38 gold grains (SD = 10) was calculated according to a Gaussian distribution. The 21 aliquots that were examined appear, however, to be distributed according to Erlang's k-distribution (Fig. 5); the calculated Erlang-based mean (38) and standard deviation (9) are similar to our calculation. The coefficient of variation is either 28% or 25%, depending on the selected probabilistic distribution (Gaussian or Erlang, respectively). The Erlang scale parameter ($\mu = 2.22$) suggests that few variables control this parameter.

A possible controlling factor is the distribution of the finer gold grains within the sample matrix. Table 4 and Fig. 2 show a noticeable difference in size distribution and CoV for grains <50 µm (50%) compared with coarser grains (22%), meaning that the finer grains are

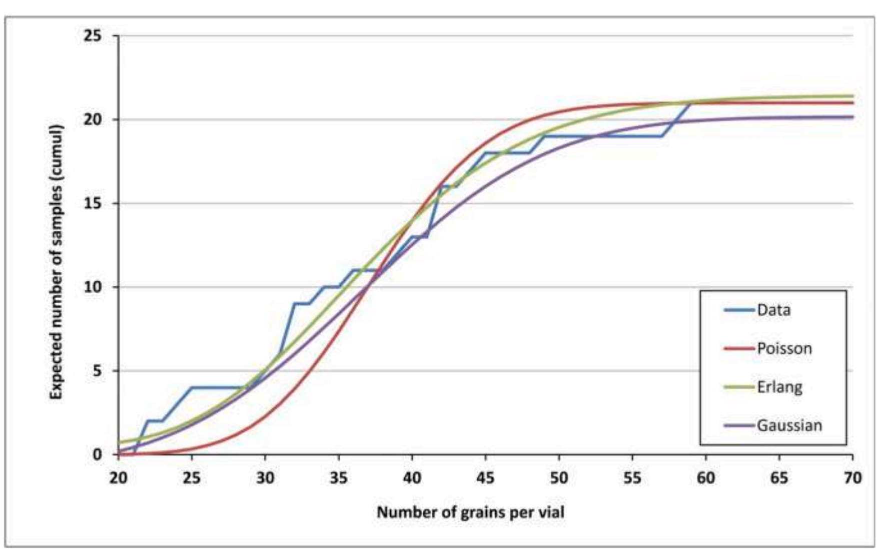

Fig. 5. Probabilistic distribution modeling of the grain count results. The expected number of grains per sample is based on the 21 prepared aliquots. The Poisson model used a mean (λ) of 38, and the Erlang was modeled using a shape (k) of 17 and a rate (λ) of 0.45 (scale (μ) of 2.22).

more heterogeneously distributed within the sample matrix.

The grain size distributions of gold grains and the matrix material form two clearly distinct curves (Fig. 6). The grain size distribution of the reference material matrix HMC, measured by a laser Fraunhofer diffraction device (Fritsch Analysette 22), shows a bimodal distribution, with a population around 20 μm, a second around 350 μm, and almost no grains in the 20–125 μm range. The grain size distribution of the gold grains, measured from the SEM images, ranges from 6.7 to 950 μm. Approximately 55% of the gold grains plot within the 20–100 μm size. This obvious dichotomy in grain size, caused by using two different concentration techniques (a fluidized bed and a Wifley table for gold and heavy minerals, respectively), can affect material homogenization. In such mixtures, gold grains would be expected to slide between the coarser matrix grains at any stage of the sample preparation and create grain size distributions similar to the inverse grading observed in grain flow (Dasgupta and Manna, 2011), which is further amplified by the high density of gold. A similar grain size distribution for the gold and matrix would help material homogenization.

The bed-blending procedure itself (see Section 3.3) may not be adequate to produce the degree of homogeneity required during sample preparation. Gy's (1981) work indicates that the number of beds is irrelevant for bed-blending as long as this number is greater than 100. For the current material, we only poured 17 beds owing to the small amount (150 g) of reference material; a bed-blending involving more than 100 beds would be impractical for producing >30 cm long beds with only 1.5 g of material.

5. Using the reference material

Proper use of a reference material is critical (Jenks and Zeisler, 2000), as improper use can produce erroneous results or, even worse, false confidence. The new reference material AuGC-1 can be used for method development (evaluating and verifying the precision and accuracy of protocols, evaluating field methods, and developing new or improved protocols or approaches) and evaluating and ensuring measurement compatibility—intra- and interlaboratory quality assurance, demonstration of the integrity and performance of a gold counting system (Jenks and Zeisler, 2000). Users should ensure that the precision and accuracy of the AuGC-1 reference material fit the purpose of their experiment (Bédard and Barnes, 2010). AuGC-1 should be used for gold grain counting and not for geochemical determination, as some gold is held within sulfides and the number of gold grains of different size cannot simply be related to a gold concentration. It was not designed to be used as a reference material for other minerals, e.g., sulfides and silicates. Although it was not evaluated, our reference material should have a very long shelf life (>10 years). AuGC-1 should, however, be stored in a dry environment, ideally inside a desiccating vessel to minimize the oxidation of sulfides. Once the material has been extracted out of the vial, the reference material is considered modified, and it should not be reused.

Reference material AuGC-1 could be used to train mineralogists or counting systems. It could also be used to test the precision and accuracy of gold grain counting, i.e., the “analytical instrument,” by simply counting the number of gold grains from a vial. Minerals should be poured onto a clean surface, and the user should ensure that there are no mineral grains left inside the vial (taking care for static electricity). Gold grain counting can be done visually, with optical photography with or without artificial intelligence (Maitre et al., 2019) or under SEM (Girard et al., 2021). AuGC-1 could also be used to spike field-collected samples to assess the combined quality of recovery and gold grain counting

6. Conclusions

We produced a reference material (AuGC-1) for the counting of gold grains in till. This reference material contains an average of 38 grains, with a standard deviation of 10 grains, in 1.5 g of heavy mineral concentrate. It is difficult to determine whether the high variance in gold grain counting relates to material preparation or the intrinsic variability caused by an Erlang distribution. As well, reproducibility is of concern when working with low grain abundances.

This study identified the challenges and traps to avoid when manufacturing a reference material for counting gold grains, or any other mineral, unless the exact number of grains in the aliquot is counted directly during production.

1 Commonly used counting techniques are effective for grains >15 μm. Therefore, the gold grain size in the reference material should range between 15 and 1000 μm. Increasing the number of gold grains that are <30 μm would be more representative of the size range in the mineralized bedrock; however, identifying these grains under optical stereomicroscope is not reliable.
2 The grain size distribution of gold should be similar to that of the matrix to limit material sorting during handling. When gold grains are finer than the matrix, as observed in this study, the gold grains will tend to sink to the container's bottom during manipulation, which could compromise sample homogeneity.

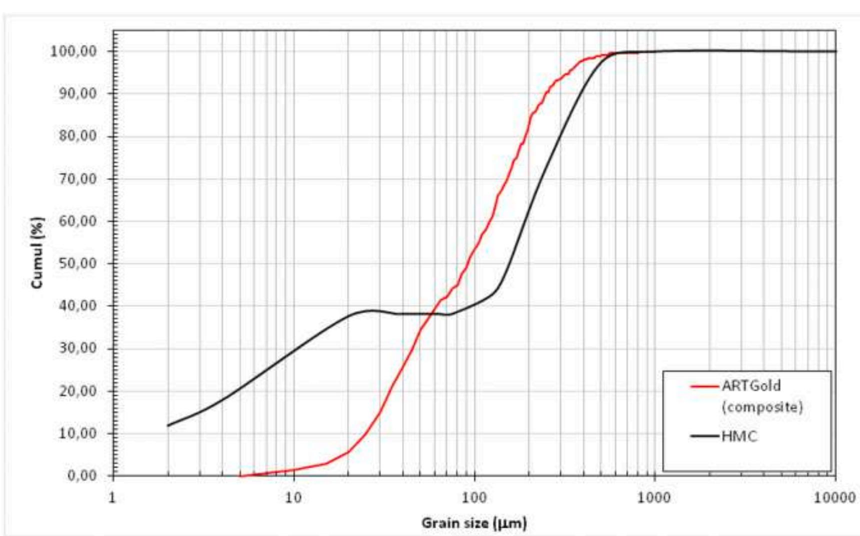

Fig. 6. Grain size distribution of the heavy mineral concentrate (HMC) prior to its mixing with the gold grains, measured using the Fritsch Analysette 22 and gold grain size measured by SEM (ARTGold; combined results of 15 aliquots).

3 Microtomography to identify and count all gold grains is not practical at this scale and requires further testing before use for certifying aliquots.
4 Ideally, gold grains in the reference material should originate from different sources or a single source having a great variation in gold grain composition. Therefore, potential variations in chemistry (and color) and shape render the reference sample better adapted to various geological settings.
5 The number of grains per sample should be significantly higher than that of the background. Increasing the number of grains per aliquot would reduce the CoV but increase the sorting time and effort. Targeting 50 grains within a reference material is adequate for most Canadian geological provinces; however, an average of 38 grains (5.5σ) in our prepared material is clearly above background even for gold-rich regions of Canada, except for a few areas in the Churchill Province, which is important for spiking till samples. As the development of an appropriate reference material improves, specific grades could be produced for different areas.

The high-density contrast between gold grains and the matrix mineral requires careful mixing, pouring, and aliquoting to avoid segregation. This study's reference material mass (150 g) does not permit the bed-blending of more than 100 beds as required by Gy's model (1981). A larger mass is needed to attain such a number of beds.

There is a definite need for gold grain reference material from a scientific, industrial, and legal perspective—however, the challenges related to the efforts and techniques for preparing such a reference material remain. Continued efforts will eventually produce a commercially distributed certified reference material for monitoring the accuracy and precision of gold grain counting. The next steps in this research are to 1) ensure that the produced HMC has a proper grain size distribution vis-à-vis the targeted till sample, and 2) increase the resultant HMC mass and the number of gold grains to produce more reference material. A greater amount of material would render bed-blending more effective and result in decreased intersample variability.

Although we did not meet several characteristics required for a certified reference material, such as those described by Kane et al. (2003, 2007), the reference material produced in this study can be used to evaluate the efficacy of HMC recovery and gold grain counting procedures on the condition that statistics are maintained to monitor any deviation over time.

CRediT authorship contribution statement

Jean-François Boivin: investigation, formal analysis, resources, visualization, and writing (early draft). L. Paul Bédard: conceptualization, methodology, validation, writing (review and editing), supervision, project management, and funding acquisition. Hugues Longuépée: writing (draft, review, and editing), validation, formal analysis, and visualization.

Declaration of competing interest

Jean-François Boivin was a student at Université du Québec à Chicoutimi (UQAC) under the supervision of Paul Bédard, held an internship at IOS Services géoscientifiques (named IOS thereafter). JFB subsequently worked for IOS but he is currently out of the field; he works at the municipal library. Paul Bédard has no financial interest in IOS. Hugues Longuépée undertook this research as a postdoc at UQAC under the supervision of Paul Bédard and is now employed by IOS.

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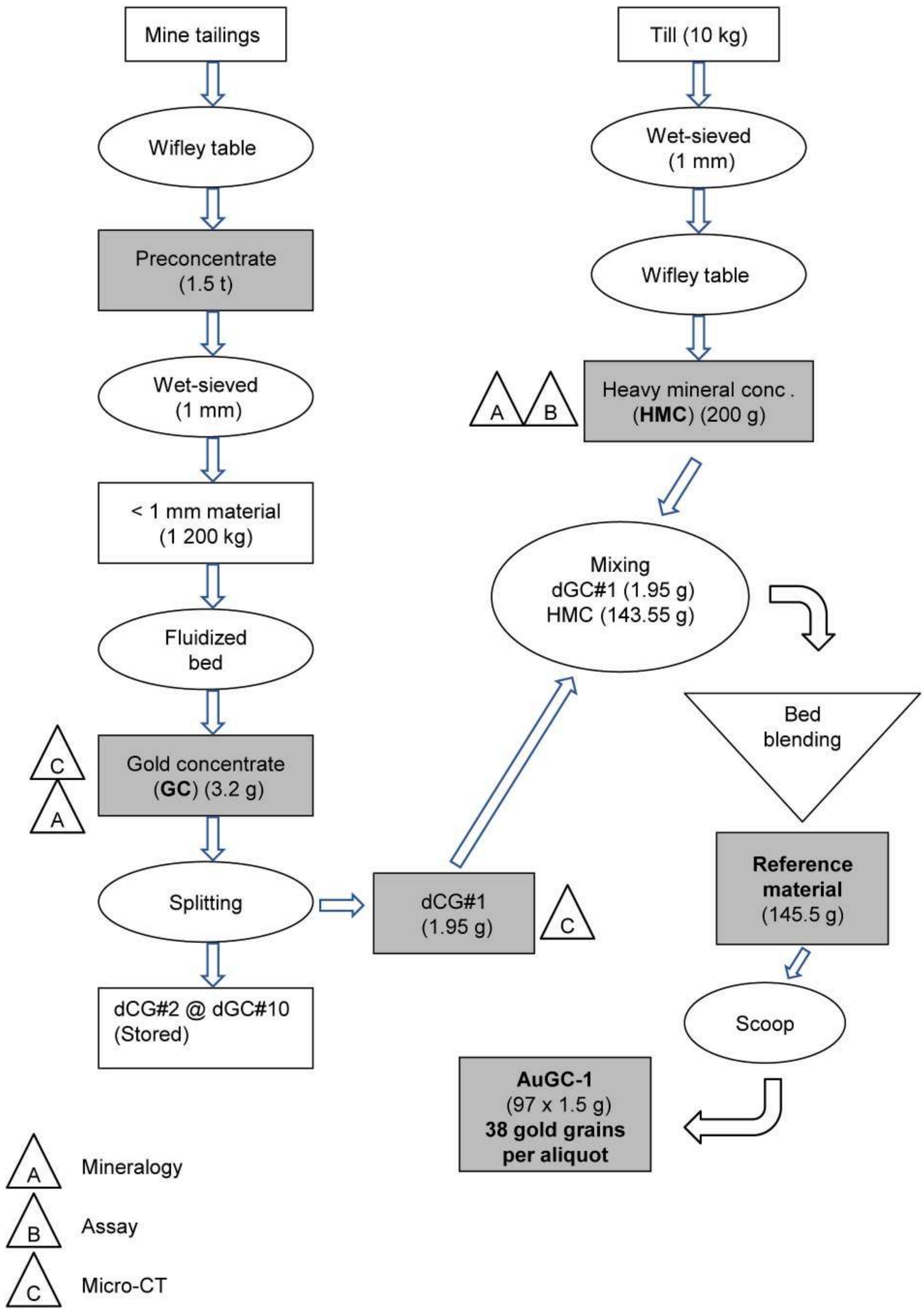
Appendix A. Reference material preparation flowchart


Mine tailings
Wifley table
Preconcentrate (1.5 t)
Wet-sieved (1 mm)
< 1 mm material (1 200 kg)
Fluidized bed
Gold concentrate (GC) (3.2 g)
Splitting
dCG#2 @ dGC#10 (Stored)
dCG#1 (1.95 g)
Till (10 kg)
Wet-sieved (1 mm)
Wifley table
Heavy mineral conc . (HMC) (200 g)
Mixing dGC#1 (1.95 g) HMC (143.55 g)
Bed blending
Reference material (145.5 g)
Scoop
AuGC-1 (97 x 1.5 g) 38 gold grains per aliquot
A Mineralogy
B Assay
C Micro-CT

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