Sampling – A key tool in modern process mineralogy☆

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ABSTRACT

The powerful modern toolbox of hybrid Process Mineralogy for flowsheet development uses best practice sampling as one of its tools. In this paper, the three key components of best practice sampling are reviewed with case studies. These three components are:

1. Minimum sample mass.
2. Rules of unbiassed sampled extraction.
3. The safety line.

These excellent models and rules are not commonly taught in undergraduate programmes. In this review paper, which is intended as an introductory reference for those practitioners in Process Mineralogy who have not had exposure to the sampling models, simple and practical explanations are presented for reference. It is shown that finer particle size distributions lead to smaller minimum sample mass requirements.

While sampling theory allows us to estimate the error involved obtaining a mass of sample for mineralogical analysis it is also useful to account for errors in the process mineralogy measurements themselves. Examples of the confidence intervals on liberation measurements made on high- and low-grade samples are provided to illustrate the importance of sample size—specifically measuring sufficient numbers of particles—in these analyses.

1. Introduction

The best practice of sampling in mining and metallurgical engineering places the project or operation on a sound platform with reliable data which may be used for business decisions. This best practice works on a basis of the criteria that divide "samples" from 'specimens'. In his 1979 seminal work on sampling of particulate materials, Gy stated that in most cases where a mining venture failed, the causes can nearly always be traced to unaccountable sampling errors because of the confusion between samples and specimens. He defined these two terms as:

- "**Sample**":
  ○ A part of the lot, often obtained by the reunion of several increments or fractions of the lot, and meant for representing it in further operations. A sample is not just any part of the lot; its extraction must respect certain rules that the theory of sampling intends to establish. The following key rules must be satisfied in order to declare material to be a sample:

- The mass of dry solids in a sample must be equal to or more than the minimum sample mass, as determined by Gy's minimum sample mass models, with sufficiently low fundamental variance.
- Any particle in the lot, of whatever size class, must be able to enter the primary sample with an equal probability of being excluded from the primary sample.

In other words, a sample differs from the lot only in mass. It has an identical composition to that of the lot.

- "**Specimen**":
  ○ A part of the lot obtained without respecting these (sampling) rules.

In short, best practice sampling amounts to three criteria:

1. How much primary sample must be taken
2. How the increments to that primary sample are extracted

3. How the primary sample is crushed and subsampled to a workable mass and size.

In a recently published monograph entitled "Process Mineralogy", numerous chapters describing relevant case studies in the practice of Process Mineralogy are provided (Becker et al., 2016). The reader is advised to add this reference to his or her reading.

This paper does not report any new material in the field of sampling. Its purpose is to set out a simple reference using prior published articles for the practitioner of Process Mineralogy, who probably has not had the opportunity of reading and understanding the basics of Gy's sampling models. Based on the observation of Minnitt, 2007, that details of Gy's sampling theories are only recently entering the undergraduate syllabi at universities, many professionals working in areas of the mining industry where sampling is important may not have been exposed to the details of these concepts. This view was supported by Holmes, 2010, who observed that the main reason sampling practice in an operation fails to meet with best practice is that the sampling responsibility is often left to people who do not have an appreciation of the significance and importance of sampling. It is acknowledged that since the seminal works of Gy, 1979, there has been a considerable amount of other excellent work performed by a number of workers, who have advanced his platform with suitable refinements describing relevant case studies. It is not the purpose of this paper to review this large volume of work in detail. Rather, a brief summary of these follows, to keep the article short and readable, for the reader's benefit.

A chapter entitled "Process Control" was written by Bartlett and Hawkins, 1987, in the SAIMM monograph entitled "The Extractive Metallurgy of Gold in South Africa". This chapter summarises the main components of the sampling models, and offers a few case studies. The entire sampling theory of Gy was re-explained in detail by Pitard, 1993. His book contains numerous case studies. A more recent publication summarises the essentials with case studies (Lotter, 2016).

1.1. Minimum sample mass M_s

One of the primary criteria for a sample is the minimum sample mass, or M_s. The entire set of sampling models written by Gy operate in the centimetre gramme second unit system (cgs). The primary sample mass must equal or exceed M_s for a selected fundamental variance, which is generally 8%, as recommended by Bartlett and Hawkins, 1987. The minimum sample mass and the fundamental variance are related to each other *by the sampling constant K as* in Equation (1).

$$M_s = \frac{K}{f_v} \tag{1}$$

where

M_s = minimum sample mass, grammes
K = the sampling constant, $g/\%^2$
f_v = the fundamental variance, $\%^2$

In this method, the fundamental variance f_v equates to the fundamental error. In order to relate this to the error resident in the mean assay grade $\bar{a}$, the square root of the fundamental variance is taken, providing the standard deviation. This standard deviation is then divided by the mean grade and expressed as a percentage of that mean error.

The sampling constant is obtained for a particular ore by an experiment in which the particulate material to be characterised is sampled and sized into a series of bound size classes, then weighed and assayed per size class. At the time of taking this sample, the sampling equation is unknown, so it cannot be declared to be a true sample until the testwork is done, the sampling equation becomes known, and the minimum sample mass declared and quantified. The equation for K is deliberately written in a form that is sensitive to variance of the

paymetal grade in the sample. The form for the sampling constant K is shown in Equation (2).

$$K = \left(\left(\frac{g \cdot \bar{v}}{M \cdot \bar{a}^2} \right) \left(\sum_{i=1}^{n} \left((a_i - \bar{a})^2 \cdot \left(\frac{M_i^2}{v_i} \right) \right) \right) \right) \tag{2}$$

where

M_s = representative sample mass, grammes
$\bar{d}$ = weighted mean particle diameter (or d_{50} size), cm
M_i = the ith fractional mass corresponding to d_i
v_i = volume of the ith ore particle, cm^3
$\bar{v}$ = mean particle volume in sample lot, cm^3
M = the lot mass, grammes
$\bar{a}$ = weighted mean sample grade of paymetal, expressed as grammes per
tonne of ore, or percent metal in ore
a_i = the grade of metal in the ith size fraction
d_i = the ith particle diameter, cm
f_v = the fundamental variance
g = the size range factor

The size range factor is the ratio between the d_{50} size and the d_{95} size, as in Equation (3). Both values are easily estimated from a cumulative percent passing size plot. It is thus obvious that the domain of g is confined to $0 < g < 1$.

$$g = \frac{d_{50}}{d_{95}} \tag{3}$$

Note that all of these equations use the centimetre gramme second system of units (cgs).

In the case of metallurgical plant process streams, which have undergone comminution and classification to liberate the valuable mineral(s), Gy's fifty-piece experiment has to be modified in order to obtain estimates of variance and thus a sampling equation. In this modification, the metallurgical process stream sample tested is screened over an appropriate set of test sieves into bound size classes, then each size class weighed, assayed, and measured for dry solids density. Each size class is then regarded as a "piece" in the experiment.

1.2. Rules of unbiased sample extraction

The following is a summary of the typical sample extraction rules recommended by Pierre Gy in his 1979 publication:

For cross-stream sample cutters, which extract primary samples of slurry from a continuously flowing (process) slurry stream, these rules are:

1. The sample cutter must be non-restrictive and self-cleaning, discharge completely.
2. The geometry of the cutter opening must be such that the cutting time at each point in the stream must be equal. For linear-path cutters, the cutter edges must be parallel, while for cutters travelling in an arc or circle (e.g. Vezin cutters) the cutters must be radial.
3. No materials other than the sample must be allowed to enter the cutter, e.g. dust or slurry must be prevented from accumulating in the cutter when in the parked position.
4. The cutter must intersect the stream in a plane normal to the trajectory of the stream.
5. The cutter must travel through the stream at a uniform speed. In this regard electrically-driven cutters are best.
6. The cutter aperture must be not less than three times the nominal topsize of the stream being sampled, with a minimum size of 10 mm for slurries for cases where the topsize is less than 3mm.
7. The cutter must have sufficient capacity to accommodate the increment mass at the maximum flowrate of the stream.

8. For slurries, the loss of sample material due to dribbling is to be avoided.
9. The maximum cutter speed should not exceed 0.6 metres/s. This rule is taken from Gy, 1979, who conducted experiments to quantify the extent to which particles of known size bounce from the cutting edges of a horizontal cutter, either into or out of, the cutter with a measured gap and travelling at set speeds.
10. The mass of dry solids in a sample must be equal to or more than the minimum sample mass, as determined by Gy's minimum sample mass models. The increments taken into that sample must be taken independently.

1.3. The safety line

The general safety line proposed by Gy, 1979, is a relationship between topsize, or d_{95}, and permissible subsample mass that may be extracted at that size, as in Equation (4).

$$M_s = 125,000d^3 \qquad (4)$$

where

M_s = minimum sample mass, g
d = the topsize, cm

The safety line is a log-log plot of sample topsize in mm against sample mass in kg. The scales cover mass from 1000 kg to 1 gramme, and size from 100 mm to 100 µm. Any combination of sample topsize and sample mass will place the sample either to the left of, on, or to the right of, the safety line. If the point is located to the right of the line, it is unsafe, i.e. too coarse, to be sampled and must be crushed or pulverized to a size that relocates the point to the left of the line. If the sample point is located to the left of the line, it may be subsampled to an extent that relocates the point down to, but not crossing, the line. From that point it must be recrushed or repulverised to again relocate the sample point to the left of the line and allow vertical movement of the sample point downwards as the recrushed sample is again subsampled. A plot of this safety line is shown in Fig. 1. The sample starts at point A, which represents the topsize of 8 mm and a sample mass of 8 kg. Point A is located to the right of the Safety Line, therefore this material cannot be sampled unless it is crushed to some size smaller than 8mm. The sample is then crushed to a new topsize of 1.7 mm at B, which is located to the left of the Safety Line, and is safe to sample provided that the subsample mass extracted does not locate itself below the Safety Line, i.e. less than 700 grammes for the 1.7 mm topsize (approximately). The subsampling performed then takes a 2-kg subsample, which locates itself at C, to the left of the Safety Line. The 2-kg subsample is then rod-milled to a new topsize of 100 µm (0.1 mm) at D, which again is to the left of the Safety Line. The milled subsample of 2 kg is then subsampled to a 40-g assay subsample located at E, which again is to the left of the Safety Line.

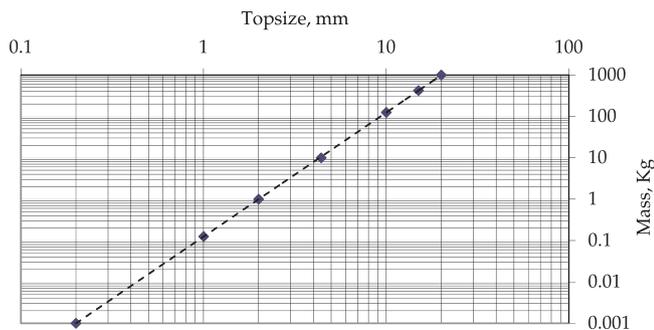Note that the coefficient of 125,000 is the general value recommended by Gy. In cases where precious metals are involved, he recommends a value of 250,000.

1.4. The semivariogram

One of the rules of unbiased sample extraction is that the increments taken cannot be autocorrelated, i.e. the increments are independent of each other. Because the ore mined in a 3-dimensional space in a stope or opencast bench is correlated (that is how the geostatisticians predict the metal grade of unsampled ore), as the ore is mined and delivered to the mill, the full correlation that existed at the mining face reduces to a residual correlation that must be acknowledged and managed in the sampling arrangements. This will affect the timing of the increments taken by a cross-stream sample cutter.

The semivariance of the paymetal is estimated by the semivariogram, and uses Eq. (5) (Clark, 1993).

$$\gamma_t = \frac{1}{2N} \cdot \sum_{1=1}^{N} (g_i - g_j)^2 \qquad (5)$$

where

N = the number of data
g_i = the metal grade at time point i
g_j = the metal grade at time point j
$\gamma(t)$ = the semivariance

A schematic of the semivariogram is shown in Fig. 2. Note that with real data sets, many variations from this format are seen, and the data and model should be interpreted with care.

From t = 0 to t = t_c, every extra spacing between g_i and g_j causes the semivariance to increase. After a spacing of t_c, i.e. for all t > t_c, the semivariance becomes constant. This area is called "the sill". Sample increments taken at any t > t_c are therefore independent of each other and satisfy the independence rule for sample increments. The intercept on the y-axis is an estimate of the total sampling error, as was recently demonstrated by Engström (2016). A good example of her interpretation follows in Case Study 2.

To construct a semivariogram for a flowing pulp stream, a series of timed samples is taken across a 60-min period at 1-min intervals. Each of the samples is dewatered and prepared for assay, e.g. % Cu. These assays are the values of g_i and g_j, and are arranged in a sequential array from t = 0 to t = 60. The first value of semivariance is calculated with a spacing of 1 min, then 2, and so on until a spacing of 30 is reached, at which point there are sufficient data to construct the model.

Variations to the model shown in Fig. 2 are known. Some comments on semivariograms for concentrators follow. There are three types of variation:

Fig. 1. The safety line, showing the correct stages of size and mass reduction from the mill feed sample down to the assay sample.
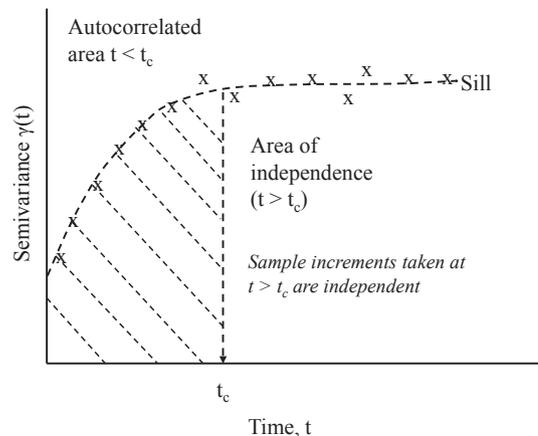

Fig. 2. The semivariogram.

1. A short-range term, mainly random, discontinuous at every instant, reflecting the random nature of constitution heterogeneity within one increment.
2. A long-range term, nonrandom, mainly continuous, representing trends between units.
3. A cyclic term, continuous, such as cycles introduced by reclaiming operations or corrective actions to maintain parameters between two known limits. Tailings streams commonly demonstrate this feature because of the circulating load behaviour in the cleaner circuit.

Case study 2 provides an actual example of a semivariogram using the percent mass retained in the topsize of a particle size distribution of pelletised iron ore.

1.5. Estimating errors in process mineralogy measurements

While sampling theory provides the means to estimate the error involved in taking a mass of particulate material as a sample for mineralogical analysis, other techniques can be applied to estimate the error present in the process mineralogy measurements themselves. The choice of method applied to estimate the error depends on the type of data being collected by the process mineralogist. Some of the earliest methods for assessing measurement errors in mineralogical measurements were developed by Chayes (1944, 1945) and van der Plas and Tobi (1965) who published methods to assess the error in measurements of mineral grade made on particles in polished section.

The more widespread use of automated mineralogy systems over the last 20 years has provided users with access to a range of detailed mineralogical data such as mineral grain size and mineral liberation distributions. Researchers have published methods which allow the end user to estimate the error in these measurements. Leigh et al. (1993) developed a method to estimate the error in mineral liberation distribution measurements based on an analytical solution that requires the liberation data to be expressed in a cumulative liberation yield format. More recently Evans and Napier-Munn (2013) and Mariano and Evans (2015) applied a statistical approach based on the bootstrap method (Efron, 1987) to estimate the error in measurements of mineral grain size and mineral liberation distribution respectively. As would be expected, as the sample size (measured as number of particle sections observed) increases the confidence in the data increases.

Leigh et al. (1993) proposed the following equation to estimate the 95% confidence interval at particle composition C on the cumulative liberation distribution for a mineral:

$$CI_c \approx 1.96 * \left(1.25 Y_c^2 (1 - Y_c)^2 \left(\frac{1}{N_0} + \frac{1}{N_1}\right)\right)^{0.5} \tag{6}$$

where

Y_c = value of cumulative liberation distribution at particle composition C (as a proportion)
N_0 = Number of particles in composition classes with composition lower than C
N_1 = Number of particles in composition classes with composition greater than or equal to C

Case study 3 below provides an example of the application of the method of Leigh et al. for high grade and low grade samples. The reader is cautioned that the estimates arising from Leigh's method are approximate and not exact.

2. Case study 1: Merensky ball mill feed

A crushed ball mill feed stream of Merensky ore was sampled and sized into size classes (Lotter, 1995a,b). Each size class was weighed

Table 1
Merensky mill feed.

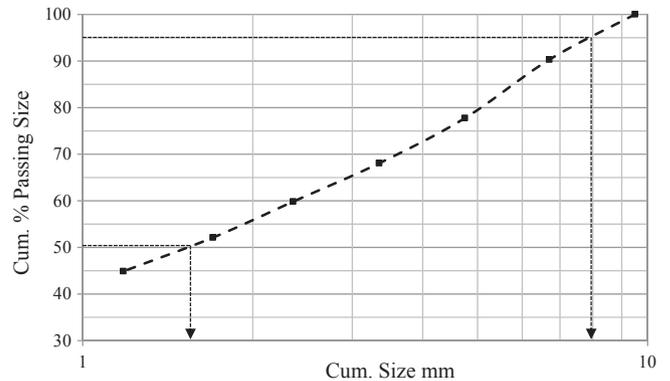| Size class, mm	Mass, g	Mass, %	Grade, g/t 4E	Solids density, g/cc
+6.70–9.50	4981.0	9.71	5.41	3.03
+4.75–6.70	6432.7	12.54	4.53	3.33
+3.35–4.75	4965.0	9.68	4.89	3.04
+2.36–3.35	4219.0	8.23	4.06	3.04
+1.70–2.36	3969.0	7.74	3.68	3.04
+1.18–1.70	3712.0	7.24	4.10	3.11
+0.85–1.18	3967.0	7.20	4.24	3.10
+0.60–0.85	3779.0	7.37	2.29	3.10
+0.00–0.60	15538.0	30.29	5.27	3.08

Fig. 3. Cumulative percent passing size curve for the mill feed.

and assayed for 4E PGE (total of Pt, Pd, Au and Rh), and was measured for solids specific gravity. The results are shown in Table 1.

A cumulative particle size distribution from these data (Fig. 3) shows the d_{50} and d_{95} sizes to be 1.3 and 8.0 mm, so g = 0.16.

From the rest of the data, the parameters for the sampling constant formula are as follows:
where

$\bar{d}$ = weighted mean particle diameter, cm (the d_{50} from Fig. 3) = 1.3 mm
M_i = the ith fractional mass corresponding to d_i
v_i = volume of the ith ore particle, cm^3
$\bar{v}$ = mean particle volume in sample lot, cm^3 = 2565.04 cm^3
M = the lot mass, grammes = 51562.7 g
$\bar{a}$ = weighted mean sample grade of paymetal = 4.55 g/t
a_i = the grade of metal in the ith size fraction
d_i = the ith particle diameter, cm
g = the size range factor = 0.16

To obtain M, we sum each ith M_i, viz. from Table 1: M_1 = 4981.0 g; M_2 = 6432.7 g, and so on, all adding up to M = 51562.7 g.

For $\bar{a}$, which is the weighted mean metal grade, or the equivalent of an overall assay for the whole sample if it had been pulverised and assayed as a whole, this is easily calculated by multiplying each size fraction mass by the corresponding metal assay for that size class. For example, from Table 1 the coarsest size fraction, which is −9.5 + 6.7 mm, weighing 4981.0 g, has a corresponding assay of 5.41 g/t PGE. The product of these two values is (4981.0)(5.41) = 26947.21 g^2/t. The second size class, which is −6.7 + 4.75 mm, weighs 6432.7 g, and assays 4.53 g/t, so the product is (6432.7)(4.53) = 29140.31 g^2/t, and so on to the smallest size class. Each of these products is then added up to a total of 234679.70 g^2/t. We then divide this total by the total mass M, which is 51562.7 g, to obtain $\bar{a}$, thus:

$\bar{a}$ = (234679.70/51562.7) = 4.55 g/t PGE.

For the size fraction volume v_i, we divide the size fraction mass by

Table 2
Summary of calculations for the minimum sample mass sampling constant K.

Size class, mm	Mass, g M_i	Grade g/t 4E PGE	Solids s.g, g/cc	Mass × Grade	$(a_i-\bar{a})^2$	M_i^2	v_i, cc	$\frac{M_i^2}{v_i}$
+6.7 − 9.5	4981.0	5.41	3.03	26947.21	0.74	24810361	1643.89	11162.36
+4.75 − 6.7	6432.7	4.53	3.33	29140.13	0.00	41379629	1931.74	8.56
+3.35 − 4.75	4965.0	4.89	3.04	24278.85	0.12	24651225	1633.22	1744.82
+2.36 − 3.35	4219.0	4.06	3.04	17129.14	0.24	17799961	1387.84	3079.47
+1.7 − 2.36	3969.0	3.68	3.04	14605.92	0.76	15752961	1305.59	9132.57
+1.18v1.7	3712.0	4.10	3.11	15219.2	0.20	13778944	1193.57	2337.73
+0.85 − 1.18	3967.0	4.24	3.10	16820.08	0.10	15737089	1279.68	1181.81
+0.60 + 0.85	3779.0	2.29	3.10	8653.91	5.11	14280841	1219.03	59835.02
+0.0 − 0.60	15538.0	5.27	3.08	81885.26	0.52	241429444	5044.81	24809.09

the solids specific gravity (s.g) of that same size class, for example the first size class, which is −9.5 + 6.7 mm, from Table 1, weighs 4981.0 g and reports a solids s.g. of 3.03 g/cm^3. The size fraction volume is thus v_i:

$$v_i = 4981.0/3.03 = 1643.89 \text{ cm}^3.$$

In like manner, the size class −6.7 + 4.75 mm has a weight of 6432.7 g and a solids sg of 3.33 g/cc, so $v_i = 1931.74$ cm^3, and so on through the rest of the size classes. The sum total of all these volumes is 16639.36 cm^3. To obtain the weighted mean volume $\bar{v}$, we multiply each size class volume v_i by its mass m_i, which for the first size class −9.5 + 6.75 mm is 1643.89 times 4981.0, or 8188237.95 g^2/cm^3, and so on down to the smallest size class, obtaining a total product of 132260569.00 g^2/cm^3. This figure is then divided by the total mass M to obtain the weighted mean volume $\bar{v}$, thus:

$$\bar{v} = 132260569.00/51562.7 = 2565.04 \text{ cm}^3.$$

A summary of these calculations is shown in Table 2.

Thus the sampling constant K = 44.65 g/%^{2}, and the sampling equation resolves from Eq. (2) to

$$M_s = \frac{44.65}{f_v}$$

For an 8% fundamental variance, the minimum samples mass resolves to 6976 g, or 6.976 kg, thus:

$$M_s = \frac{44.65}{(0.08*0.08)} = 6976 \text{ g}$$

The next problem is how to reduce this primary sample mass of 6.976 kg to a smaller mass for analysis and mineralogical measurement, as well as flotation testing. Here the Safety Line plays a key role (Fig. 4).

3. Case study 2: Construction of a semivariogram

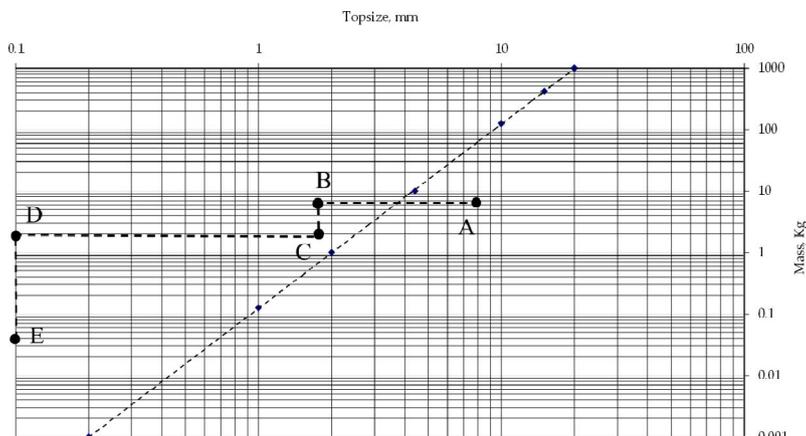A semivariogram was constructed for an iron ore pellets in a

Table 3
Raw data of 12.5–16.0 mm topsize for semivariogram of process feed in an iron ore plant. (10 out of a total of the 220 data points used for the calculation are shown (due to proprietary reasons).

Date and time	% Mass topsize
2017/01/18 00:17	7.18
2017/01/18 00:44	7.14
2017/01/18 01:39	7.62
2017/01/18 02:07	7.02
2017/01/18 03:02	6.55
2017/01/18 03:29	7.56
2017/01/18 04:24	7.34
2017/01/18 04:52	7.61
2017/01/18 05:50	6.87
2017/01/18 06:17	7.70

pelletising plant in Sweden. In this case, the particle size distribution of the stream is the measure of interest in this variographic characterisation. The particle size class 12.5–16.0 mm was selected as the variable to be analysed. A part of the raw data used for construction of the semivariogram is presented in Table 3, and reports the % mass retained in this size class at time intervals of 26 min.

The entire data set is first analysed with a data spacing of 1 unit using 5 to estimate the semivariance for a spacing of 1 time unit (26 min). Referring to Table 3, the first data pair is 7.18 and 7.14% mass retained in this size class, so the difference of (7.18–7.14) = 0.04%, when squared, amounts to 0.002%^{2}. The next data pair is 7.14 and 7.62, so the difference of (7.14–7.62) = −0.48%, which when squared, amounts to 0.23%^{2}, and so on. There are 219 such data pairs from this spacing. Summing the squares of each difference, we obtain 142.37, so the semivariance for this spacing is 142.37/(2 ∗ 219) = 1.30. In like manner, the next spacing of 2 time units starts with the data pair 7.18 and 7.62%; the following data pair at this spacing is 7.14 and 7.02, and so on.

Fig. 4. The safety line showing the apprpriate size and mas reduction stages from the mill feed sample to the assay subsample.
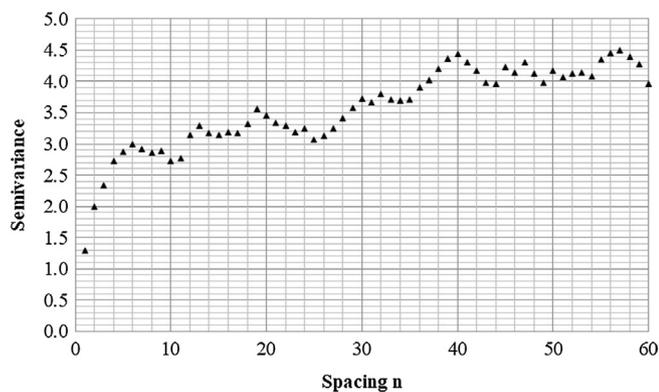
Fig. 5. Semivariogram for the Topsize 12.5–16.0 mm for iron ore pellets.

The values of semivariance are plotted on the y-axis, and the lag (time spacing between data points) on the x-axis. The semivariogram for pellet size 12.5–16 mm presented in Fig. 5 shows a classic increasing variogram with a small periodicity. The sill is reached at lag 36 or approximately 72 h. The sill of the semivariogram is calculated as the average of all data points and is approximately 4.0. The total sampling error described by the nugget effect is approximated by the extrapolation of the semivariogram to the y-axis. In this case the nugget effect is estimated to 0.75. This means that the important nugget to sill ratio for the measurement system of 12.5–16 mm pellet size is approximately 18%.

The measurement system for iron ore pellet size is an automated linear cross stream sampler collecting primary increments every ten minutes. The primary increments are combined into 30–60 min analytical aliquots (depending on belt load) and analysed on an automated sieving machine. The semi-variogram for 12.5–16 mm is indicating a nugget effect to sill ratio of less than 20%. This shows that the measurement system for pellet size is fit for purpose to enable successful process control. The semivariogram is additionally showing a weak periodicity of approximately 8 lags. As the pellet size is dependent on several process parameters it is impossible to identify one single cause for the present periodicity, but natural fluctuations in balling and sieving process are likely to introduce the variability in the process. As long as the periodic variability is as small as the semivariogram is indicating, it will not affect the overall end quality of the product. The semivariogram is however a powerful tool to monitor all types of variability (including periodicity) to ensure that quality parameters does not exceed customer specifications.

The intercept to the semivariance axis for a zero spacing is estimated at 0.75, so the measurements of topsize have an error of (0.75/4.0) or 18% approximately.

4. Case study 3: Estimating confidence intervals for mineral liberation data

The liberation distribution of chalcopyrite has been measured in a concentrate and tailings streams and to provide the end-user of the data with information on the errors present in the data confidence intervals have been calculated using the analytical approach of Leigh et al. (1993). The estimation of these confidence intervals is approximate only and should be regarded as such. Tables 4 and 5 show the measured data required for the calculation, intermediate calculations of N_0 and N_1 and the estimates of the confidence intervals.

The cumulative liberation distributions of the two streams are shown in Fig. 6, together with the calculated values of the confidence intervals. It is clear that the level of confidence in the liberation data for the lower grade sample from the tailing stream (Cu grade 0.11%) would be much less than the confidence in the higher grade concentrate sample (Cu grade 15.5%). The low grade nature of the tailing stream results in much lower numbers of particles of interest being present in each particle composition class and leads to the wide confidence intervals. The graphs clearly show why automated mineralogy systems have developed specialised measurement routines for low grade samples to specifically search for and measure particles containing the minerals of interest. These specialised routines maximise the number of particles containing the minerals of interest that are measured in the available measurement time when analysing low grade samples and increase the confidence in the resulting liberation data.

5. Further work

It is recommended that, as part of some further work, a team plan an integrated study using these models so that additive properties can be harnessed and used to assess the cumulative error in the liberation measurement, starting from minimum sample mass, then the semivariogram, and ultimately the liberation measurements. That way, the true error levels in this measurement will be seen.

6. Concluding remarks

This paper offers a short review and three case studies on the subject of sampling ore and the associated implications for microscopic measurement of liberation of minerals. With the common use of modern automated scanning electron platforms to measure liberation, it is appropriate to use best practice sampling to present the sample for mineralogical characterisation, as well as to declare the associated confidence limits of these measurements.

Table 4
Confidence interval estimation for a concentrate sample ($-53 + 38$ μm, 15.5% Cu).

Mean grade of composition class	Cumulative distribution of mineral	Number of particles in class	N_0	N_1	95% CI	Lower CI	Upper CI
Barren	100.00	6185					
5	100.00	2624	6185	10518	0	100.0	100.0
15	97.2	1484	8809	7894	0.09	97.1	97.3
25	91.8	1222	10293	6410	0.26	91.6	92.1
35	84.4	1006	11515	5188	0.48	83.9	84.9
45	75.5	906	12521	4182	0.72	74.8	76.3
55	65.8	685	13427	3276	0.96	64.8	66.7
65	56.3	580	14112	2591	1.15	55.1	57.4
75	46.6	489	14692	2011	1.30	45.3	47.9
85	36.0	400	15181	1522	1.36	34.7	37.4
95	26.2	235	15581	1122	1.31	24.8	27.5
100	19.9	887	15816	887	1.20	18.7	21.1

Table 5
Confidence interval estimation for a tailing sample ($-53 + 38$ μm, 0.11% Cu).

Mean grade of composition class	Cumulative distribution of mineral	Number of particles in class	N_0	N_1	95% CI	Lower CI	Upper CI
Barren	100.0	19908					
5	100.0	62	19908	102	0	100.0	100.0
15	82.1	12	19970	40	5.10	77.0	87.2
25	72.9	4	19982	28	8.19	64.7	81.1
35	62.7	4	19986	24	10.46	52.3	73.2
45	53.7	6	19990	20	12.19	41.5	65.9
55	41.2	2	19996	14	14.19	27.0	55.4
65	35.1	4	19998	12	14.41	20.7	49.5
75	25.7	2	20002	8	14.80	10.9	40.5
85	23.7	0	20004	6	16.16	7.5	39.8
95	23.7	2	20004	6	16.16	7.5	39.8
100	14.7	4	20006	4	5.10	1.0	28.4

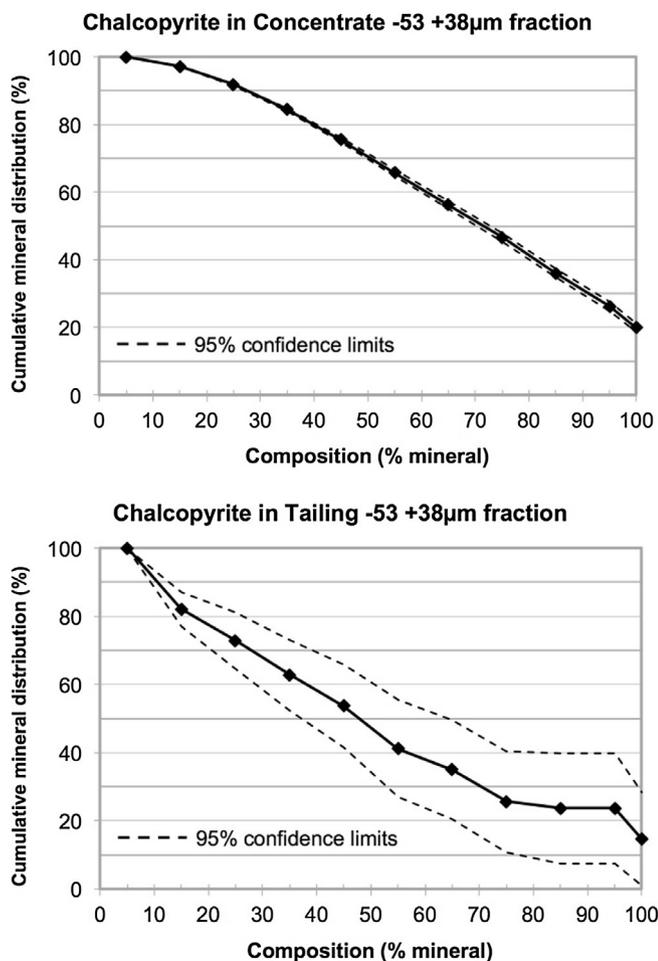**Fig. 6.** Comparison of the confidence intervals on chalcopyrite liberation in concentrate and tailing samples.

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