

## Article

# A Comparison between the Standard Heterogeneity Test and the Simplified Segregation Free Analysis for Sampling Protocol Optimisation

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**Abstract:** Estimating the heterogeneity of base and precious metal mineralisation is a great challenge for mining engineers and geologists who undertake resource evaluation, grade control and reconciliation. The calculation of the minimum broken sample mass to represent a given lot of mineralisation at a given comminution size is based on the estimation of  $IH_L$ , the constant factor of constitution heterogeneity.  $IH_L$  can be derived by different heterogeneity testwork or calibration approaches. Three methodologies are well known in the mining industry: the standard heterogeneity test, the segregation free analysis, and the sampling tree experiment or duplicate sample analysis. However, the methodologies often show different results, especially when it comes to gold. These differences are due to many reasons. Assuming the variances added by sample preparation and analysis to be equivalent for all tests, the reasons for the differences may include the nugget effect (particularly the presence of coarse gold), the segregation effect and the procedure of collecting/splitting the samples when performing the tests. This paper analyses and compares two heterogeneity tests: the original heterogeneity test and the simplified segregation free analysis, both performed on mineralisation from different Brazilian operations. The results show clear differences between the tests, highlighting the complexity of estimating the heterogeneity of mineral deposits. The study reports the importance of using proper methodologies for constitution heterogeneity estimation so that minimum sample masses and relative standard deviations of the fundamental sampling error can be relied upon. It also provides recommendations for practitioners on the application of testwork/calibration studies.

**Keywords:** sampling optimisation; heterogeneity testing and calibration; segregation free analysis; constitution heterogeneity; fundamental sampling error; grouping and segregation error



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## 1. Introduction

Sampling—inclusive of sample collection, preparation and testwork and/or assaying—is a vital component of all stages of a mining project [1–5]. It includes the sampling of in situ material and broken rock for geological, metallurgical, geometallurgical and geo-environmental purposes. Sampling errors are additive throughout the sampling value chain and generate both monetary and intangible losses [1–5]. The aim is to collect representative samples to accurately describe the mineralisation in question. Field sample collection is followed by sample reduction in both mass and fragment size to provide a subsample for testwork or assaying. This process can be particularly challenging in the precious metal environment (e.g., gold and platinum) and may require special protocols.

Sampling errors are defined in the theory of sampling (TOS) as promulgated by the works of Dr Pierre Gy [4–6]. These errors are relevant to all sampling applications, including those of metallurgy, resource evaluation and grade control. A representative sample is taken by a procedure that is both precise (repeatable) and unbiased (accurate). Precision refers to the degree of reproducibility among sample grades (e.g., field or laboratory duplicates). Accuracy describes how close a sample grade is to the actual, true grade. Bias is the difference between the expected sample grade and the actual, true grade. An unbiased sample is likely to be more accurate than a biased sample.

A given sampling process is said to be probabilistic when all the units or fragments of the lot have a non-zero probability of belonging to the sample and are randomly selected. When the probability of selection is evenly distributed among all the units in the lot, the sampling process is called equiprobabilistic and the samples can be considered representative. To undertake representative sampling, practitioners need to optimise field samples to assay protocols by undertaking characterisation of the mineralised domains present [1–7].

Experimental approaches such as the heterogeneity test (HT) and the sampling tree experiment (STE) or duplicate series/sample analysis (DSA) are employed [8–11]. The HT approach uses the traditional “Gy” 50 single-piece or grouped tests [4,6,11], whereas the STE approach comprises the DSA [9,12] or the recently introduced segregation free analysis (SFA) based on consecutive crushing, screening and splitting [10].

Whichever method is applied, this testing may be referred to as “sampling calibration” or the “sampling for sampling” stage, where samples must be collected to provide inputs into sampling optimisation [13,14]. Timing for undertaking the calibration stage across projects is variable and likely to be reflected by one of the following:

- (i) The sub-optimal approach—no calibration ever undertaken, with little to no sample protocol optimisation, due to a lack of sampling expertise;
- (ii) The optimal approach—undertaken early in project development between advanced exploration and the pre-feasibility study, with the work undertaken either internally or by an expert;
- (iii) A sub-optimal approach, but better than nothing—during operation when something goes wrong or is deemed not to be optimal, with the work undertaken either internally or by an expert.

The reasons given for not undertaking calibration relate to perceived high cost, time required for proper undertaking, and complexity usually reflecting a lack of expertise. Such calibration should be undertaken early in project development, ideally during the resource development stage or during operation when a new domain is encountered.

This paper compares two heterogeneity/calibration tests: the standard HT and the simplified segregation free analysis (SSFA), performed on mineralisation from Brazilian mine operations and several different commodities (e.g., itabirite, bauxite, Pb-Cu-Zn, Cu, Ni and Au). The results are reported anonymously with respect to mine name and location. The authors also present a discussion on challenges for future work and make some recommendations for practitioners.

## 2. Background

### 2.1. The Nugget Effect

The heterogeneity of in situ mineralisation can be quantified by the nugget effect and has a direct link to TOS [15–17]. The nugget effect is a quantitative geostatistical term describing the inherent variability between samples (e.g., drill holes) at small separation distances. It is a random component of variability that is superimposed on the regionalised variable and is defined in a variogram as the percentage ratio of nugget variance to total variance. Deposits that possess a nugget effect above 50% are the most challenging to evaluate [17]. The magnitude of the total nugget effect relates to the following:

- Geological (geological nugget effect (GNE)) heterogeneity of the in situ mineralisation;
- Distribution of single grains or clusters of gold or sulphide-hosting gold particles distributed through the ore to larger continuous zones;
- Continuity of structures such as high-grade gold carriers within the main structure or veinlets within wallrocks;
- Sampling induced error variability (sampling nugget effect: SNE);
- Sample support (sample size–volume variance);
- Sample density (number of samples at a given spacing—information effect);
- Sample collection, preparation, testwork and assay procedures.

A clear indication of the GNE is obtained when two halves of a drill core (e.g., on the centimetre scale) are assayed and there is an order of magnitude or more difference in assay grades, or where two closely spaced face samples show an order of magnitude or more difference. A high GNE leads to high data variability, particularly where samples are too small, and protocols are not optimised.

The SNE component is related to errors induced by inadequate sample size, sample collection and preparation methods, and analytical procedures. Throughout the mine value chain, optimised sampling protocols aim to reduce the SNE. Calibration/heterogeneity tests are critical to this optimisation, providing they are undertaken as effectively as possible.

The critical output of any resource evaluation or grade control sampling, sample preparation and assay programme is to contribute to a variogram with a low total nugget effect (<50%) so that estimation variances are minimised [15,17]. To achieve low nugget variances, larger sample masses may be required, and subsequent sample splitting must be optimised for the fundamental sampling error (FSE). Programmes will then yield the best estimates of spatial correlation, which will provide optimal selective mining unit estimates.

## 2.2. Heterogeneity and Mass Optimisation

In the context of broken rock, the FSE is the smallest residual error that can be achieved even after homogenisation of a sample lot is attempted [5,6]. When FSE is not optimised for each subsampling stage, it often becomes a major component of the sampling nugget variance.

The FSE is dependent upon the constitution heterogeneity (CH), which relates to sample weight, mineral fragment size and shape, liberation stage of the metal-bearing phase of interest (e.g., native gold, chalcopyrite, galena), metal grade, and metal-bearing phase and gangue density. The FSE can be estimated before the material is sampled, provided the sampling characteristics or constants (e.g.,  $K$  and  $\alpha$ ) embedded in the FSE are determined. The heterogeneity tests lead to the calibration of  $K$  and  $\alpha$  [8–11,18].

A key step in optimal sampling is to guarantee that the process is representative, i.e., precise and accurate, through the reduction in the TOS errors (e.g., delimitation and extraction errors). The next step is the calculation of the minimum sample mass to represent a given lot at a given comminution size. This can be done by estimating the constant factor of CH or the intrinsic heterogeneity of the lot ( $IH_L$ ) using the so-called “heterogeneity test” (or variants thereof). In the same way as CH,  $IH_L$  is influenced by the grade, mineralogy, shape and size distribution of the fragments in the lot. Although  $IH_L$  is not a constant factor (it is a function of the fragments’ size distribution), it is called “constant” because, excluding the liberation factor, all factors in Pierre Gy’s original formula (Equation (1)) are constants for each type of ore or given lot [6].

$$s_{FSE}^2 = \left( \frac{1}{M_S} - \frac{1}{M_L} \right) f g c l d^3 \quad (1)$$

The estimation of  $IH_L$  allows the calculation of the FSE and subsequently the optimisation of sampling protocols. The François-Bongarçon-modified [19] FSE equation, based on Gy's formula, is given as follows (Equation (2)):

$$s_{FSE}^2 = \left( \frac{1}{M_S} - \frac{1}{M_L} \right) f g c d_1^b d^\alpha \quad (2)$$

where  $s_{FSE}^2$  is the relative variance of the FSE;  $f$  = shape factor;  $g$  = granulometric factor;  $d$  = nominal top-size (or  $d_{95}$ ) of the fragments in the lot (given in cm);  $c$  = mineralogical factor;  $l$  = liberation factor;  $d_1$  = liberation diameter (given in cm);  $b = (3 - \alpha)$ , where  $\alpha$  is determined experimentally (e.g., HT and DSA-based tests) and is a dimensionless sampling constant for each type of mineralisation investigated;  $M_S$  = sample mass (given in grammes); and  $M_L$  = lot mass (given in grammes) [4,6].

The key output from heterogeneity tests, the  $IH_L$ , comprises the following components of the FSE equation at a given  $d$  value (Equation (3)):

$$IH_L = f g c d_1^b d^\alpha = K d^\alpha \quad (3)$$

$IH_L$  can thus be substituted into the FSE equation (Equation (4)):

$$s_{FSE}^2 = \left( \frac{1}{M_S} - \frac{1}{M_L} \right) IH_L = \left( \frac{1}{M_S} - \frac{1}{M_L} \right) K d^\alpha \quad (4)$$

Using this formula, it is possible to (a) calculate the error for a given sample size split from the original lot or (b) calculate what subsample size should be used to obtain a specified variance at a given reliability.

There is more than one methodology to estimate the constants  $K$  and  $\alpha$ : the original HT [18], the DSA/STE [9] and the SFA [10].

Several questions arise regarding the results of heterogeneity tests, especially for gold mineralisation. These questions may include the following:

- (i) Which test is best suited to the estimation of  $IH_L$  and FSE?
- (ii) Do all the tests really isolate the CH of the lot, or do they include both the CH and the distributional heterogeneity (DH)?
- (iii) Which one better reflects what is happening in daily reality in sampling processes?
- (iv) Is it better to perform the standard heterogeneity test that attempts to isolate the FSE, or to perform an alternative heterogeneity test (e.g., DSA) that estimates the quality fluctuation error ( $QFE_1$ ), i.e., the FSE plus the grouping and segregation error (GSE)?

Debates have taken place addressing the limitations of some approaches and the true nature of variability measured in heterogeneity experiments [20]. To date, there is no agreement on which approach is correct or best. However, for some heterogeneous ores, particularly of gold, a more integrated approach using various inputs (e.g., metallurgical testwork, mineralogy/mineral department and field observations) may be appropriate [7,13,14,21–24]. The motivation of this study was to answer some of the fundamental questions on the calibration of  $K$  and  $\alpha$ .

### 3. Methodology

For confidentiality reasons, the mines are here addressed as numbers from 1 to 9 (Table 1).

**Table 1.** Mines, types of mineralisation and mineral/element analysed. ROM: run of mine.

Mine	Mineralisation	Mineral/Element	Approx. ROM Grade
Mine 1	Itabirite type 1	Fe	39%
Mine 1	Itabirite type 1	SiO <sub>2</sub>	40%
Mine 1	Itabirite type 2	Fe	39%
Mine 1	Itabirite type 2	SiO <sub>2</sub>	40%
Mine 2	Bauxite	SiO <sub>2</sub>	4%
Mine 2	Bauxite	Fe <sub>2</sub> O <sub>3</sub>	14%
Mine 2	Bauxite	Al <sub>2</sub> O <sub>3</sub>	46%
Mine 3	Polymetallic mineralisation	Pb	10%
Mine 3	Polymetallic mineralisation	Cu	0.2%
Mine 3	Polymetallic mineralisation	Zn	15%
Mine 4	Copper mineralisation	Cu	5%
Mine 5	Niobium mineralisation	Nb <sub>2</sub> O <sub>5</sub>	>0.5%
Mine 6	Low-grade gold mineralisation	Au	0.7 gt <sup>-1</sup>
Mine 7	Low-grade gold mineralisation	Au	0.9 gt <sup>-1</sup>
Mine 8	High-grade gold mineralisation	Au	2.6 gt <sup>-1</sup>
Mine 9	High-grade gold mineralisation	Au	7.9 gt <sup>-1</sup>

In all cases, approximately 500 kg of sample was selected at the plant feed; a composite of 5 to 8 cuts was taken on the stopped conveyor belt after the secondary or tertiary crushing stage, crushed below 25 mm (where necessary) and screened in four size fractions. Next, each size fraction was split in half using a riffle splitter to generate the sample for the standard HT and the simplified segregation free analysis (SSFA).

### 3.1. Standard Heterogeneity Test (HT)

For the HT, the samples were screened to the following size fractions:

- –25.4 mm + 12.7 mm;
- –12.7 mm + 6.35 mm;
- –6.35 mm + 4.75 mm;
- –4.75 mm + 3.35 mm.

Although it is known that the fines are richer for most of the mineralisation, the –3.35 mm fraction was discarded. This decision was because it is impossible to collect superfine fragments one by one at random; therefore, the HT data would not exist for this size fraction, and the results of the comparison with the SSFA data would be biased. It is important to emphasise that, despite the true nature of variability measured in this experiment, the objective was to present a comparison between heterogeneity tests, which could not be conducted on the –3.35 mm particles. The material of each size fraction was then evenly spread on a grid previously drawn using tape. The cell sizes varied according to the particle size fraction (Table 2 and Figure 1).

**Table 2.** Cell sizes for each size fraction.

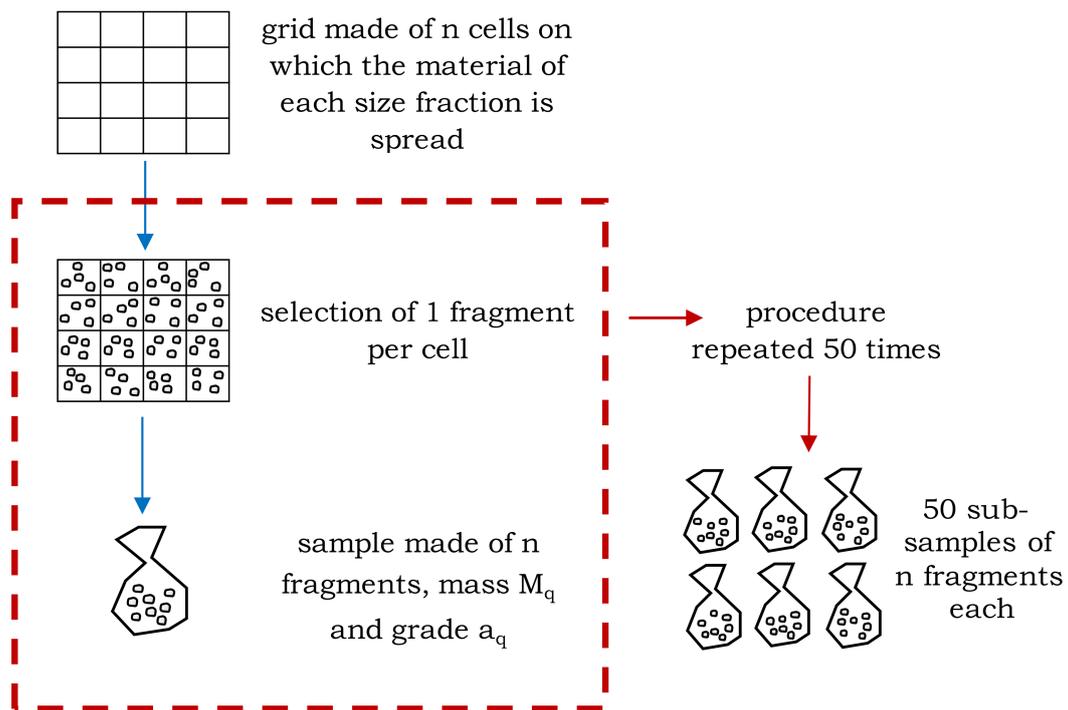
Size Fraction	Cell Size (mm)
–25.4 mm +12.7 mm	300 × 300
–12.7 mm +6.35 mm	200 × 200
–6.35 mm +4.75 mm	100 × 100
–4.75 mm +3.35 mm	100 × 100

**Figure 1.** Material spread on the grid for the HT, size fraction –12.7 mm +6.35 mm.

The subsamples were composed of one fragment randomly collected from each cell of the grid, making up n-fragment subsamples, as n is the number of cells. Taking Figure 1 as an example, each subsample would be made of 90 fragments (6 × 15 grid).

This procedure was repeated 50 times, generating 50 subsamples of n fragments for each of the four size fractions (Figure 2). The total number of subsamples for each HT was 200.

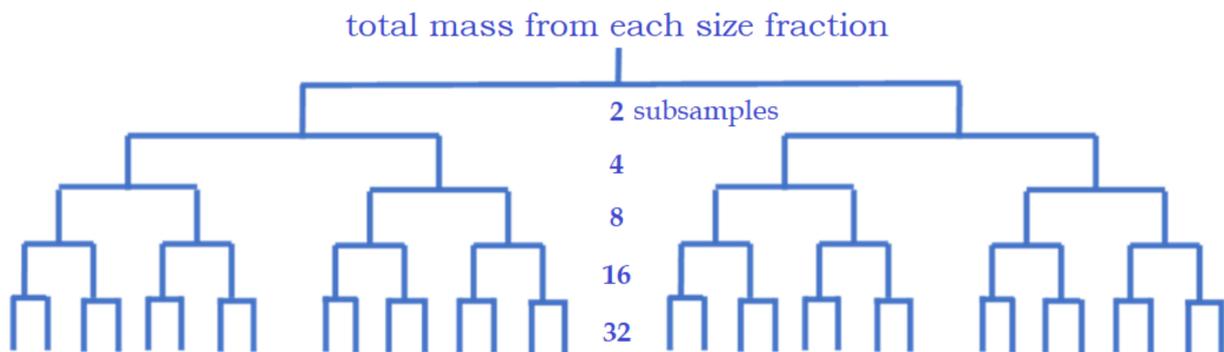
Finally, the 200 subsamples were sent to the relevant mine laboratory for preparation and analysis. Each mine proceeded with its routine protocol of sample preparation and chemical analysis. None of the samples were assayed to extinction, which would be impossible considering the local protocol. Most were assayed in duplicate or triplicate, particularly the gold mineralisation. Note that although the variances added by preparation sub-sampling stages (FSE), preparation error (PE) and chemical analysis (AE) should be considered in the FSE calculations, since this is a comparative study and the samples from both tests were equally prepared and analysed, the authors decided not to address this issue. In any case, the degree of PE should be minimal to nil assuming correct laboratory procedures.



**Figure 2.** HT procedure for each size fraction (adapted from Fernandes, Chierigati and Vargas [25]).

3.2. Simplified Segregation Free Analysis (SSFA)

The SSFA [26] is a simplification of the SFA, where only 4 size fractions are used instead of the 14 proposed by Minnitt, Francois-Bongarçon and Pitard [10]. Each size fraction was riffle split into a series of 32 subsamples, from five splitting stages: 2, 4, 8, 16, and 32 subsamples (Figure 3).

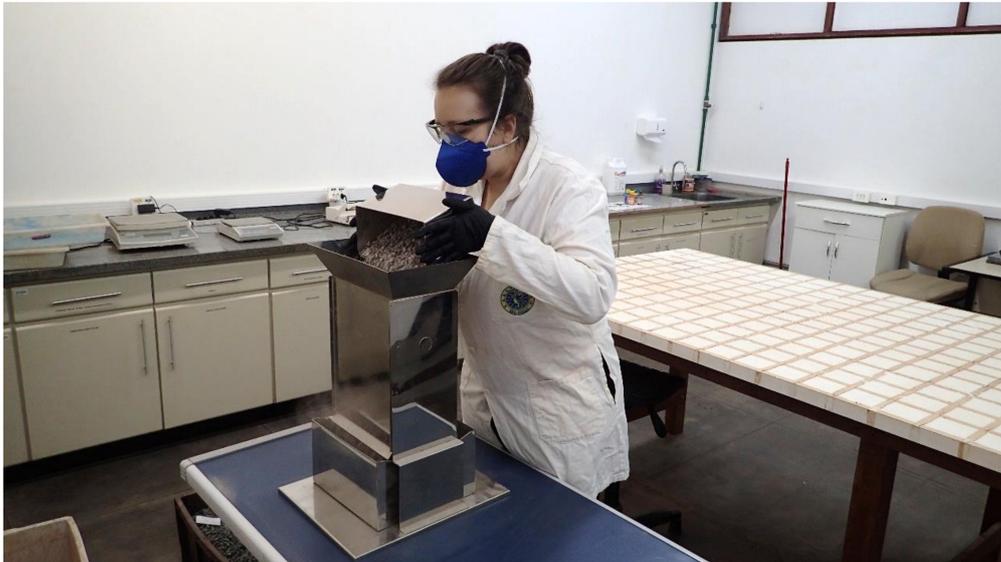


**Figure 3.** SSFA procedure for each size fraction (adapted from Minnitt et al. [9]).

Two riffle splitters were used: one for the largest size fraction and one for the smaller size fractions (Figure 4).

This study compared two types of heterogeneity tests; therefore, the same size fractions were used for both tests:

- −25.4 mm + 12.7 mm;
- −12.7 mm + 6.35 mm;
- −6.35 mm + 4.75 mm;
- −4.75 mm + 3.35 mm.



**Figure 4.** Material being split during the SSFA procedure.

Consequently, the total number of subsamples for each SSFA test was 128, which were sent to the relevant mine laboratory for preparation and analysis.

### 3.3. $IH_L$ Estimation

$IH_L$  was estimated in the same way for both the HT and SSFA, using the simplified method described by Pitard (Chapter 11) [18]. In this approach, there is no need to calculate the liberation factor. Once the samples were weighed and analysed, the mass  $M_q$  (given in grammes) and grade  $a_q$  (given in decimal) for each group of fragments, as well as the average mass  $M_Q$  (given in grammes) and weighted average grade  $a_Q$  (given in decimal), were calculated according to Equation (5).

$$a_Q = \frac{1}{M_Q} \sum_q a_q M_q \quad (5)$$

The estimated constant factor of constitution heterogeneity, EST  $IH_L$  (given in grammes), was then calculated for each size fraction according to Equation (6).

$$EST\ IH_L = g \sum_q \frac{(a_q - a_Q)^2 M_q^2}{a_Q^2 M_Q} \quad (6)$$

The variable  $g$  is the granulometric factor of “Gy’s formula”, considered as 0.55 (dimensionless) since the material was screened before the tests were conducted [18]:

- $g = 0.25$  for uncalibrated material;
- $g = 0.55$  for calibrated material;
- $g = 0.75$  for naturally calibrated materials.

The regression line equation, based on the different values of  $IH_L$  calculated for each size fraction and plotted in a bi-log graph, allowed the estimation of the sampling constants  $K$  and  $\alpha$ . Considering the regression line equation,  $y = ax^b$ , “ $x$ ” is  $d$ , “ $y$ ” is  $IH_L$ , “ $a$ ” is  $K$  and “ $b$ ” is  $\alpha$  (Equation (7)). Refer to the work of Pitard (1993) [18] for a more detailed step-by-step calculation of EST  $IH_L$ .

$$IH_L = Kd^\alpha \quad (7)$$

The average particle diameter  $d$  for screened material is calculated as follows:

$$d = \sqrt[3]{\frac{d_1^3 + d_2^3}{2}} \quad (8)$$

where  $d_1$  and  $d_2$  are the upper and lower screen openings of each size fraction.

#### 4. Results

According to Minnitt, François-Bongarçon and Pitard [10], the identification of outliers or groups of outliers is important for the adjustment of the experimental line. Although cutting high values slightly changes the distribution from typically lognormal to normal, Minnitt et al. [9,10] affirm that the process is essential to ensure a reasonable fit of the data.

Gold mineralisation usually presents higher variability (nugget effect) than base metals, especially when conducting a standard fire assay for gold content determination. This effect occurs because the coarse gold particles ( $>150 \mu\text{m}$ ) do not comminute well, and the procedure of collecting a 30–50 g analytical sample may or may not select all or some of the gold particles present [17]. This results in grades that show high variance. The potential negative, albeit transient, effect of the GSE when handling pulps containing liberated gold cannot be overstated [27].

However, an “outlier” value may represent a true result reflecting the presence of one or more coarse gold particles. Therefore, the authors decided not to cut or modify any value that could represent an outlier to the detriment of a more accurate calibration of the sampling constants for gold, aiming to avoid an inappropriate alteration of the original results.

##### 4.1. Heterogeneity Test

The following graphs (Figures 5–20) show the calibration of the sampling constants ( $K$  and  $\alpha$ ) for the 16 chemical elements or minerals derived from the heterogeneity tests (Equation (7)).  $IH_L$  is plotted on the y-axis and  $d$  is plotted on the x-axis. To derive  $K$  and  $\alpha$  directly from the regression line equations, the data are plotted as bi-log graphs. The square of Pearson’s coefficient ( $R^2$ ) indicates the goodness of fit. For reasons already addressed, a lower Pearson’s coefficient is expected for the elements or minerals with greater variability in grades (e.g., SNE). For base metal deposits, the  $R^2$  is usually  $>0.80$ , whereas for gold it can be  $<0.80$ .

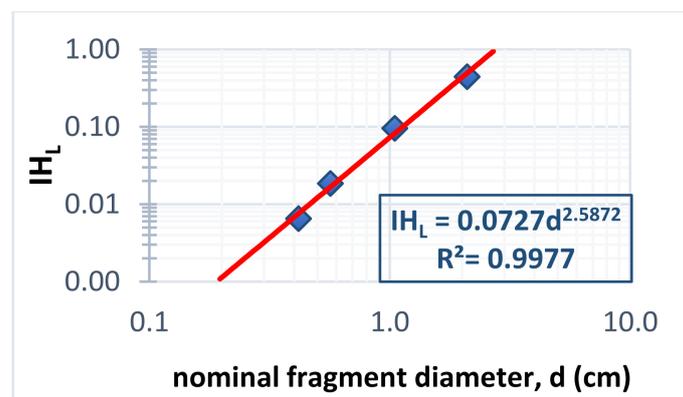


Figure 5. HT Mine 1—Itabirite 1—Fe.

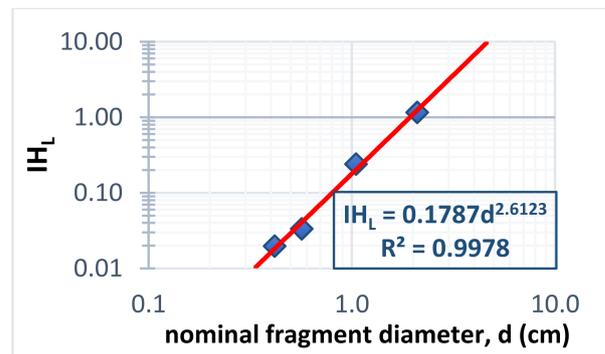


Figure 6. HT Mine 1—itabirite 1— $SiO_2$ .

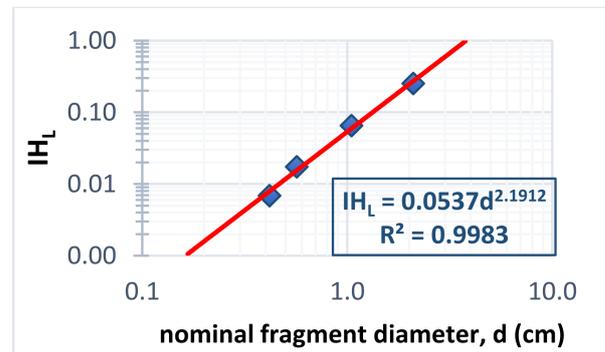


Figure 7. HT Mine 1—itabirite 2—Fe.

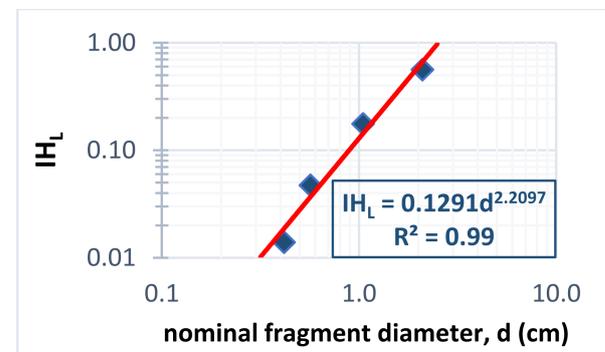


Figure 8. HT Mine 1—itabirite 2— $SiO_2$ .

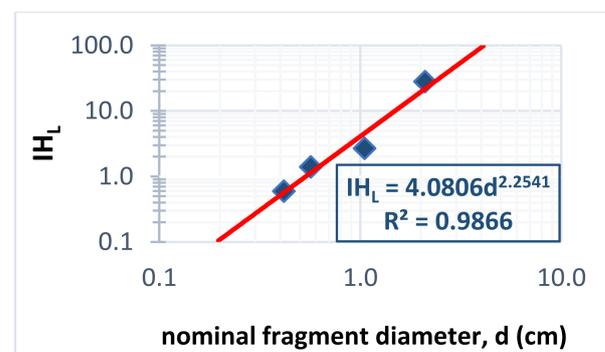


Figure 9. HT Mine 2—bauxite— $SiO_2$ .

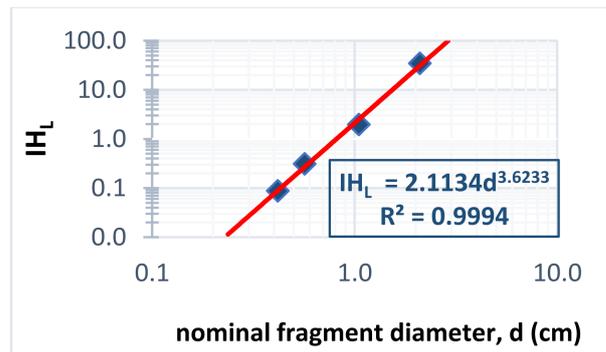


Figure 10. HT Mine 2—bauxite— $Fe_2O_3$ .

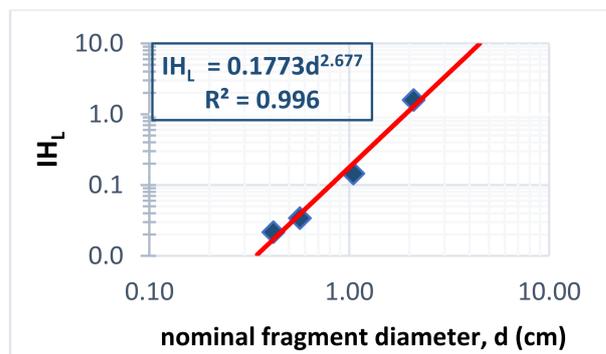


Figure 11. HT Mine 2—bauxite— $Al_2O_3$ .

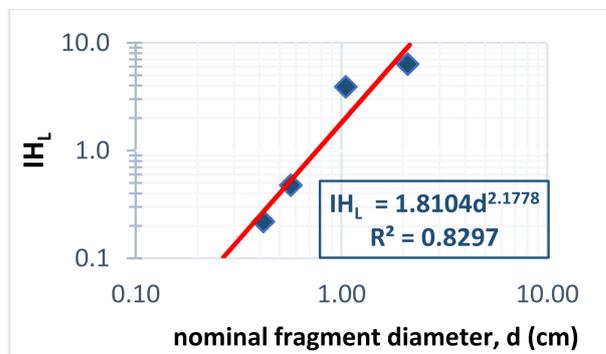


Figure 12. HT Mine 3—polymetallic mineralisation—Pb.

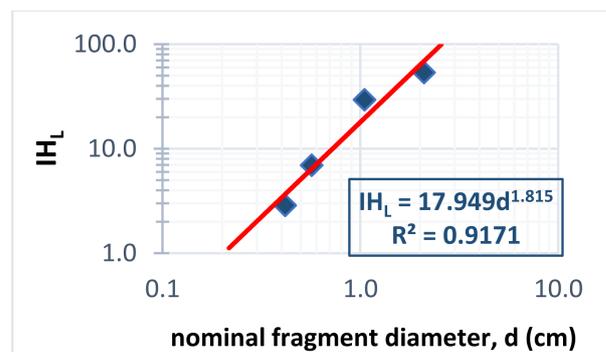


Figure 13. HT Mine 3—polymetallic mineralisation—Cu.

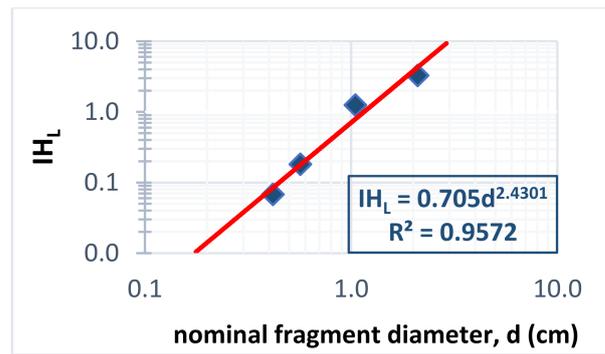


Figure 14. HT Mine 3—polymetallic mineralisation—Zn.

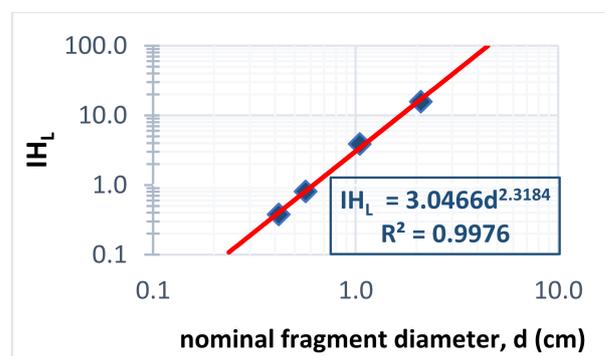


Figure 15. HT Mine 4—copper mineralisation—Cu.

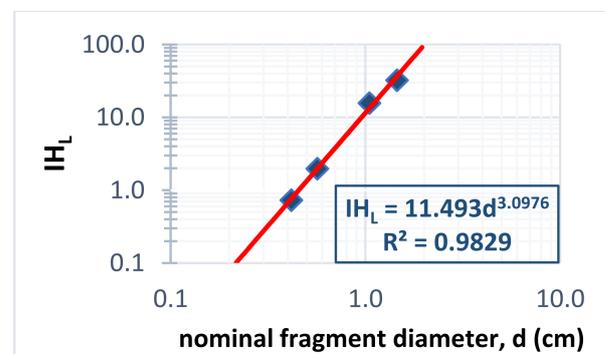


Figure 16. HT Mine 5—niobium mineralisation— $Nb_2O_5$ .

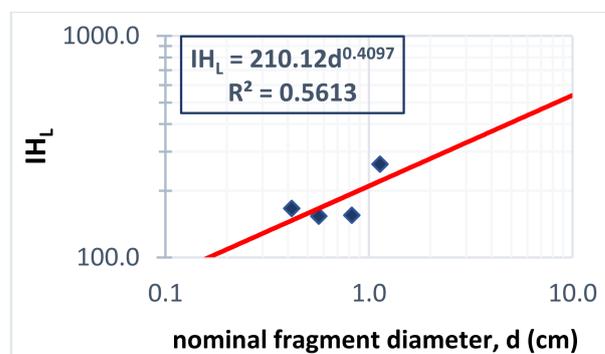


Figure 17. HT Mine 6—low-grade gold mineralisation—Au.

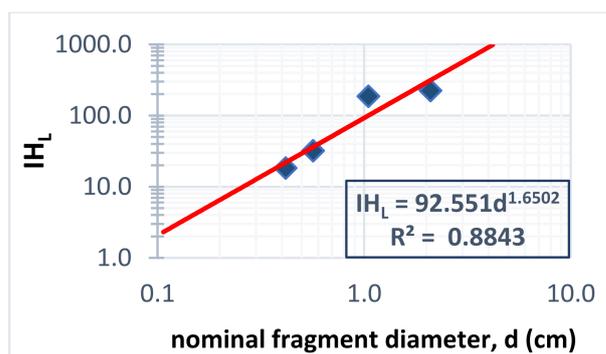


Figure 18. HT Mine 7—low-grade gold mineralisation—Au.

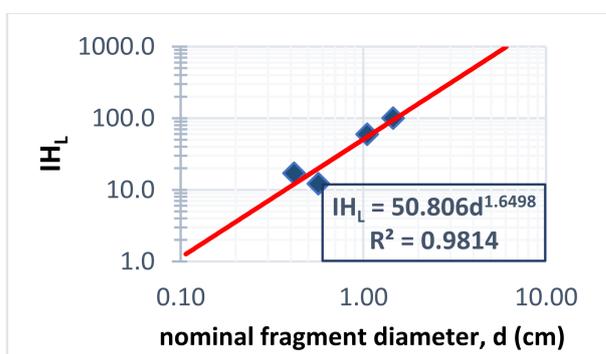


Figure 19. HT Mine 8—high-grade gold mineralisation—Au.

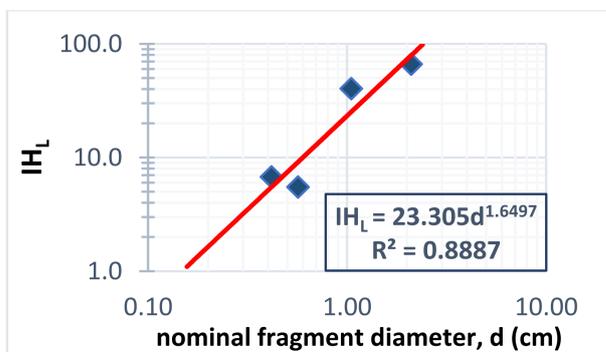


Figure 20. HT Mine 9—high-grade gold mineralisation—Au.

#### 4.2. Simplified Segregation Free Analysis

The following graphs (Figures 21–36) show the calibration of the sampling constants ( $K$  and  $\alpha$ ) for the 16 chemical elements or minerals derived from the SSFA (Equation (7)).  $IH_L$  is plotted on the y-axis,  $d$  is plotted on the x-axis, and the data are plotted as bi-log graphs, to derive  $K$  and  $\alpha$  directly from the regression line equations. Again, the  $R^2$  indicates the goodness of fit.

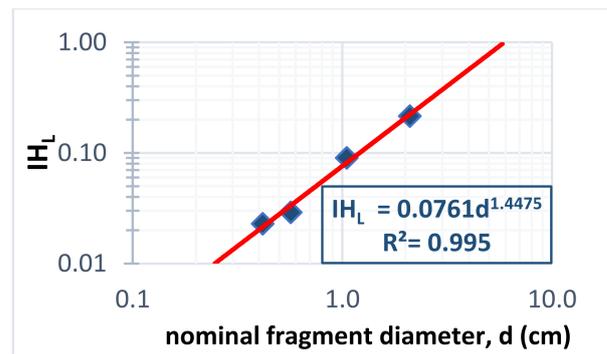


Figure 21. SSFA Mine 1—itabirite 1—Fe.

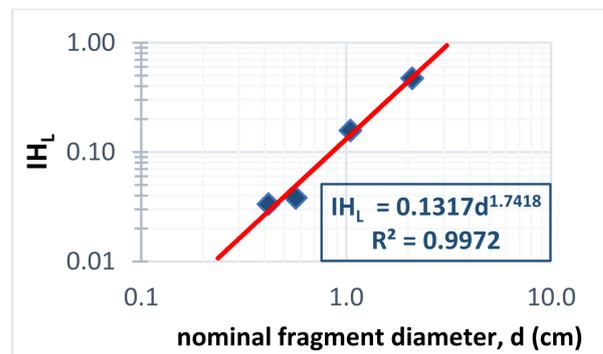


Figure 22. SSFA Mine 1—itabirite 1—SiO<sub>2</sub>.

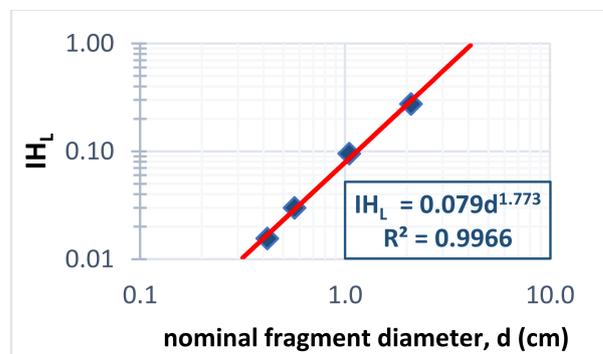


Figure 23. SSFA Mine 1—itabirite 2—Fe.

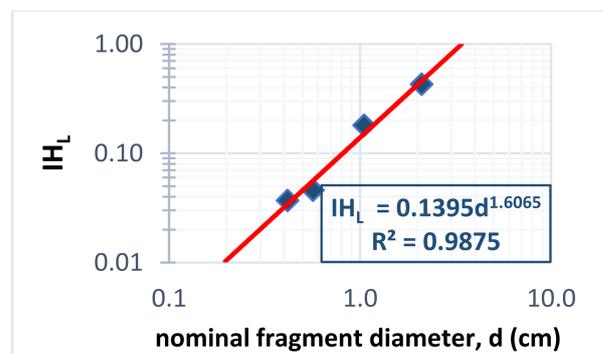


Figure 24. SSFA HT Mine 1—itabirite 2—SiO<sub>2</sub>.

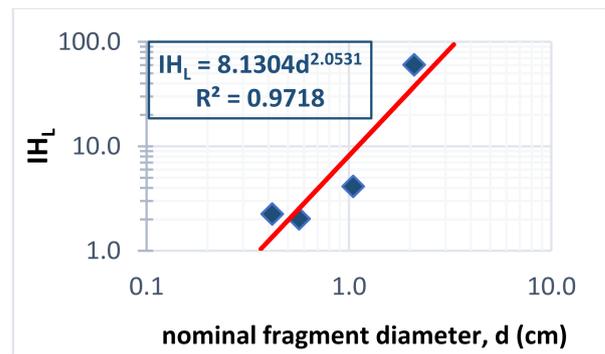


Figure 25. SSFA Mine 2—bauxite— $SiO_2$ .

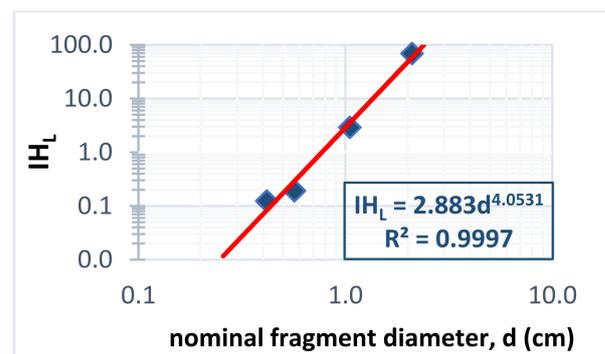


Figure 26. SSFA Mine 2—bauxite— $Fe_2O_3$ .

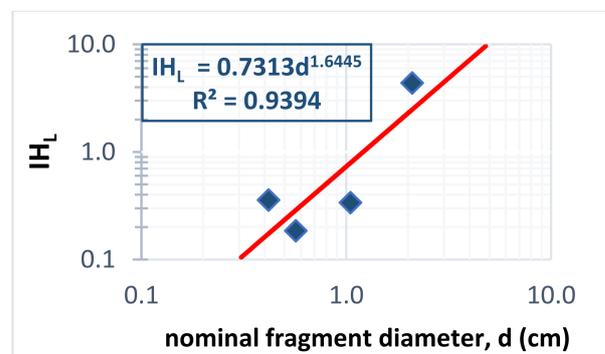


Figure 27. SSFA Mine 2—bauxite— $Al_2O_3$ .

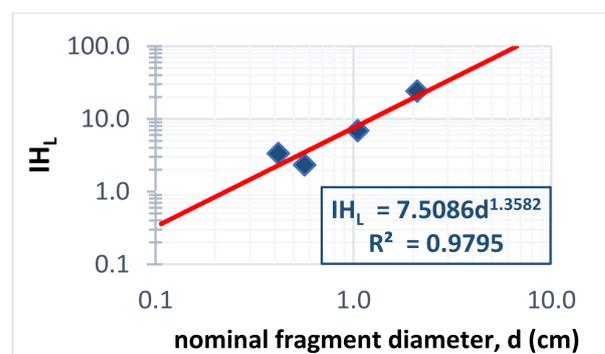


Figure 28. SSFA Mine 3—polymetallic mineralisation—Pb.

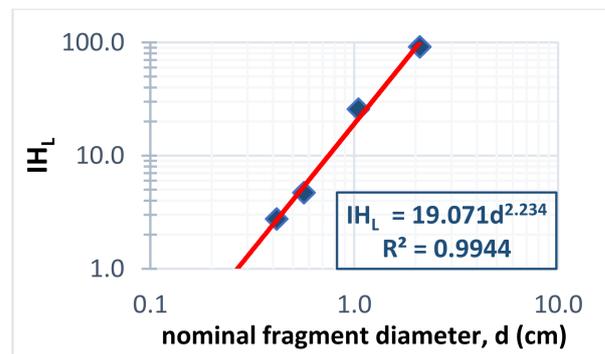


Figure 29. SSFA Mine 3—polymetallic mineralisation—Cu.

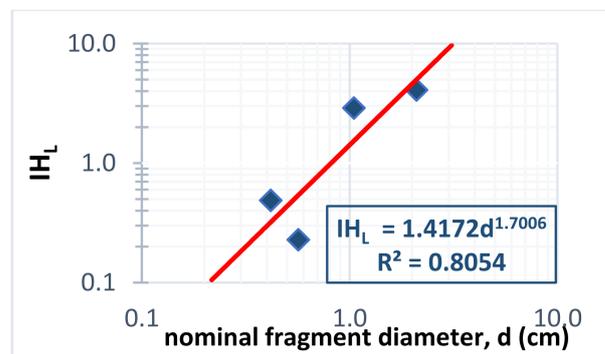


Figure 30. SSFA Mine 3—polymetallic mineralisation—Zn.

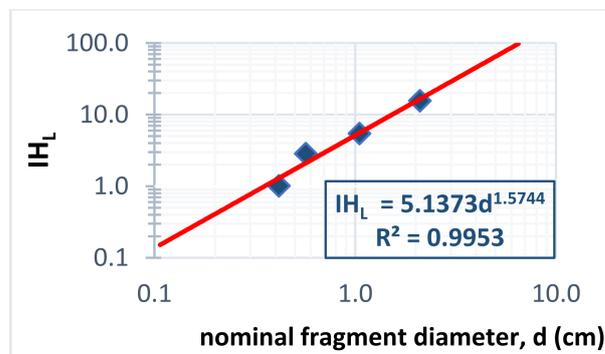


Figure 31. SSFA Mine 4—copper mineralisation—Cu.

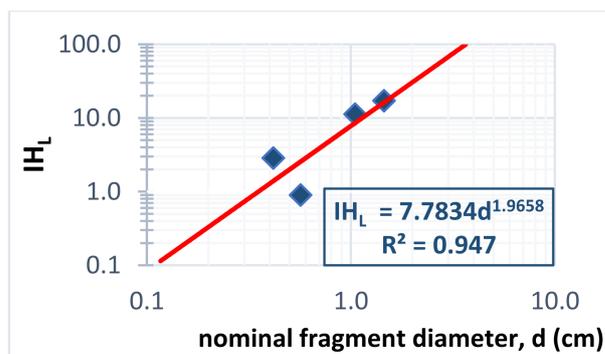


Figure 32. SSFA Mine 5—niobium mineralisation— $Nb_2O_5$ .

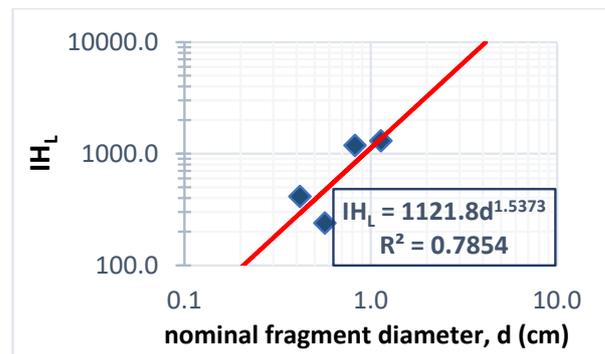


Figure 33. SSFA Mine 6—low-grade gold mineralisation—Au.

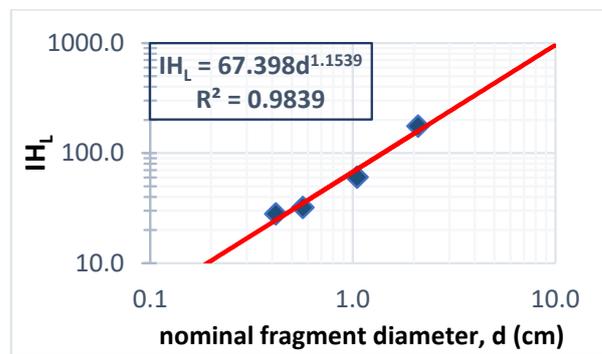


Figure 34. SSFA Mine 7—low-grade gold mineralisation—Au.

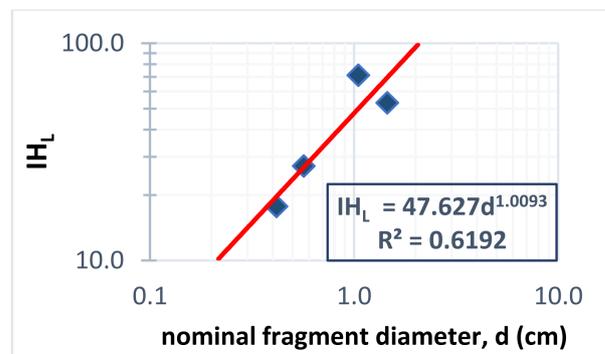


Figure 35. SSFA Mine 8—high-grade gold mineralisation—Au.

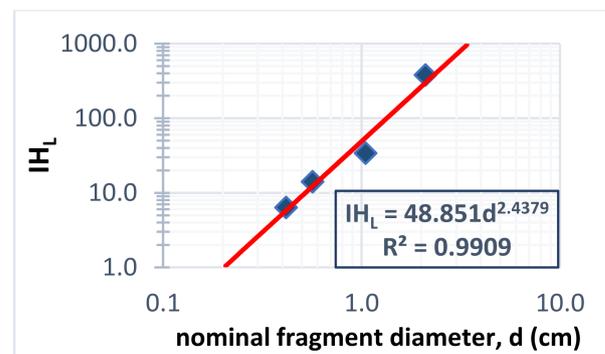


Figure 36. SSFA Mine 9—high-grade gold mineralisation—Au.

### 4.3. Sampling Protocols and Results

Figure 37 provides the general sampling protocol for the plant feed at the case study mines. Based on Equation (4), the total relative standard deviation of the FSE ( $s_{FSE}$ ) was calculated for the 16 elements or minerals. Two examples of  $s_{FSE}$  calculation are shown in Tables 3 and 4 (itabirite type 1—Fe) and in Tables 5 and 6 (high-grade gold mineralisation).

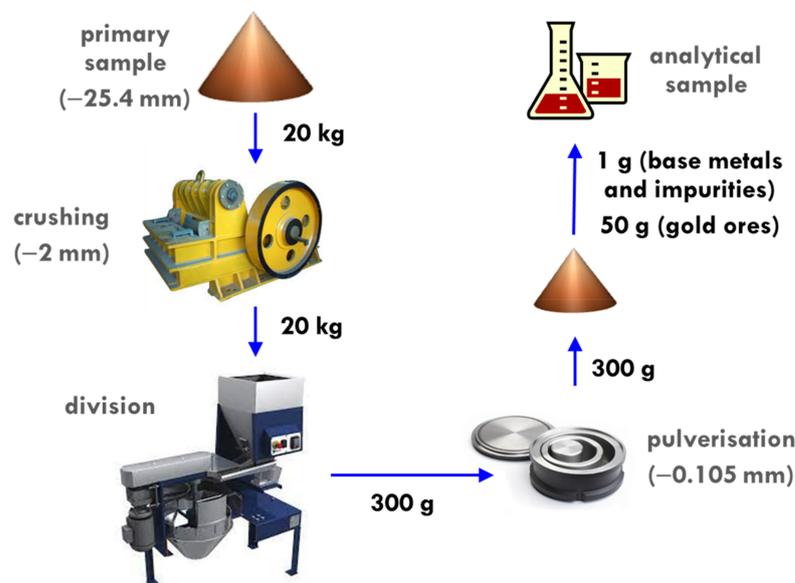


Figure 37. General plant feed sampling protocol.

The 500-t lot mass ( $M_L$ ) for each case (Tables 3–6) represents the belt load over a period, usually 1–2 h during which the 20 kg ( $M_S$ ) primary sample was collected.

Table 3. FSE calculated based on the HT results (Mine 1: itabirite 1—Fe).

Stage	$M_L$ (g)	$M_S$ (g)	d (cm)	$I_{HL}$ (g)	Rel var ( $s^2_{FSE}$ )	Rel dev ( $s_{FSE}$ )
1. Primary sampling	500,000,000	20,000	2.54	0.81	0.000041	0.64%
2. Crushing	20,000	20,000	0.20	0.0011	0.000000	0.00%
3. Division	20,000	300	0.20	0.0011	0.000004	0.19%
4. Pulverisation	300	300	0.0105	0.0000006	0.000000	0.00%
5. Selection of analytical sample	300	1	0.0105	0.0000006	0.000001	0.07%
<b>Total</b>					<b>0.000045</b>	<b>0.67%</b>

Table 4. FSE calculated based on the SSFA results (Mine 1: itabirite 1—Fe).

Stage	$M_L$ (g)	$M_S$ (g)	d (cm)	$I_{HL}$ (g)	Rel var ( $s^2_{FSE}$ )	Rel dev ( $s_{FSE}$ )
1. Primary sampling	500,000,000	20,000	2.54	0.29	0.000015	0.38%
2. Crushing	20,000	20,000	0.20	0.0074	0.000000	0.00%
3. Division	20,000	300	0.20	0.0074	0.000024	0.49%
4. Pulverisation	300	300	0.0105	0.00010	0.000000	0.00%
5. Selection of analytical sample	300	1	0.0105	0.00010	0.000104	1.02%
<b>Total</b>					<b>0.000143</b>	<b>1.19%</b>

**Table 5.** FSE calculated based on the HT results (Mine 8: high-grade gold).

Stage	M <sub>L</sub> (g)	M <sub>S</sub> (g)	d (cm)	IH <sub>L</sub> (g)	Rel var (s <sup>2</sup> <sub>FSE</sub> )	Rel dev (s <sub>FSE</sub> )
1. Primary sampling	500,000,000	20,000	2.54	236.49	0.011824	10.87%
2. Crushing	20,000	20,000	0.2	3.57	0.000000	0.00%
3. Division	20,000	300	0.2	3.57	0.011724	10.83%
4. Pulverisation	300	300	0.0105	0.03	0.000000	0.00%
5. Selection of analytical sample	300	50	0.0105	0.03	0.000460	2.15%
<b>Total</b>					<b>0.024008</b>	<b>15.49%</b>

**Table 6.** FSE calculated based on the SSFA results (Mine 8: high-grade gold).

Stage	M <sub>L</sub> (g)	M <sub>S</sub> (g)	d (cm)	IH <sub>L</sub> (g)	Rel var (s <sup>2</sup> <sub>FSE</sub> )	Rel dev (s <sub>FSE</sub> )
1. Primary sampling	500,000,000	20,000	2.54	122.03	0.006101	7.81%
2. Crushing	20,000	20,000	0.2	9.38	0.000000	0.00%
3. Division	20,000	300	0.2	9.38	0.030810	17.55%
4. Pulverisation	300	300	0.0105	0.48	0.000000	0.00%
5. Selection of analytical sample	300	50	0.0105	0.48	0.007989	8.94%
<b>Total</b>					<b>0.044900</b>	<b>21.19%</b>

## 5. Discussion

Tables 7–9 show the values of the constants K and  $\alpha$  and the total relative standard deviation of the FSE (total s<sub>FSE</sub>) obtained from the HT and the SSFA tests. The tables also show the relative differences between the results for the same chemical element or mineral (calculated based on the HT results), where the values that did not follow the main tendency are highlighted in grey.

**Table 7.** HT and SSFA comparison—constant K.

Comparison between HT and SSFA			Constant K		
Mine	Mineralisation	Ore Type	HT	SSFA	Rel. Difference (%)
Mine 1	Itabirite type 1	Fe	0.073	0.076	4.7
Mine 1	Itabirite type 1	SiO <sub>2</sub>	0.179	0.132	−26.3
Mine 1	Itabirite type 2	Fe	0.054	0.079	47.1
Mine 1	Itabirite type 2	SiO <sub>2</sub>	0.129	0.140	8.1
Mine 2	Bauxite	SiO <sub>2</sub>	4.081	8.130	99.2
Mine 2	Bauxite	Fe <sub>2</sub> O <sub>3</sub>	2.113	2.883	36.4
Mine 2	Bauxite	Al <sub>2</sub> O <sub>3</sub>	0.177	0.731	312.5
Mine 3	Polymetallic mineralisation	Pb	1.810	7.509	314.7
Mine 3	Polymetallic mineralisation	Cu	17.949	19.071	6.3
Mine 3	Polymetallic mineralisation	Zn	0.705	1.417	101.0
Mine 4	Copper mineralisation	Cu	3.047	5.137	68.6
Mine 5	Niobium mineralisation	Nb <sub>2</sub> O <sub>5</sub>	11.493	7.783	−32.3
Mine 6	Low-grade gold mineralisation	Au	210.120	1121.80	433.9
Mine 7	Low-grade gold mineralisation	Au	92.550	67.398	−27.2
Mine 8	High-grade gold mineralisation	Au	50.806	47.627	−6.3
Mine 9	High-grade gold mineralisation	Au	23.3050	48.851	109.6

**Table 8.** HT and SSFA comparison—constant  $\alpha$ .

Comparison between HT and SSFA			Constant $\alpha$		
Mine	Mineralisation	Ore Type	HT	SSFA	Rel. Difference (%)
Mine 1	Itabirite type 1	Fe	2.5872	1.4475	−44.1
Mine 1	Itabirite type 1	SiO <sub>2</sub>	2.6123	1.7418	−33.3
Mine 1	Itabirite type 2	Fe	2.1912	1.7730	−19.1
Mine 1	Itabirite type 2	SiO <sub>2</sub>	2.2097	1.6065	−27.3
Mine 2	Bauxite	SiO <sub>2</sub>	2.2541	2.0531	−8.9
Mine 2	Bauxite	Fe <sub>2</sub> O <sub>3</sub>	3.6233	4.0531	11.9
Mine 2	Bauxite	Al <sub>2</sub> O <sub>3</sub>	2.6770	1.6445	−38.6
Mine 3	Polymetallic mineralisation	Pb	2.1778	1.3582	−37.6
Mine 3	Polymetallic mineralisation	Cu	1.8150	2.2340	23.1
Mine 3	Polymetallic mineralisation	Zn	2.4301	1.7006	−30.0
Mine 4	Copper mineralisation	Cu	2.3184	1.5744	−32.1
Mine 5	Niobium mineralisation	Nb <sub>2</sub> O <sub>5</sub>	3.0976	1.9658	−36.5
Mine 6	Low-grade gold mineralisation	Au	0.4097	1.5373	275.2
Mine 7	Low-grade gold mineralisation	Au	1.6502	1.1539	−30.1
Mine 8	High-grade gold mineralisation	Au	1.6498	1.0093	−38.8
Mine 9	High-grade gold mineralisation	Au	1.6497	2.4379	47.8

**Table 9.** HT and SSFA comparison—total  $s_{FSE}$  for the plant feed samples from 500 t lot to assay charge (refer to Figure 37).

Comparison between HT and SSFA			Total FSE Relative Standard Deviation ( $s_{FSE}$ )		
Mine	Mineralisation	Ore Type	HT	SSFA	Rel. Difference (%)
Mine 1	Itabirite type 1	Fe	0.67%	1.19%	78.5
Mine 1	Itabirite type 1	SiO <sub>2</sub>	1.06%	1.03%	−2.5
Mine 1	Itabirite type 2	Fe	0.53%	0.77%	45.5
Mine 1	Itabirite type 2	SiO <sub>2</sub>	0.83%	1.26%	52.1
Mine 2	Bauxite	SiO <sub>2</sub>	4.65%	6.66%	43.2
Mine 2	Bauxite	Fe <sub>2</sub> O <sub>3</sub>	5.58%	7.95%	42.4
Mine 2	Bauxite	Al <sub>2</sub> O <sub>3</sub>	1.08%	2.73%	153.3
Mine 3	Polymetallic mineralisation	Pb	3.09%	13.95%	351.2
Mine 3	Polymetallic mineralisation	Cu	11.24%	10.05%	−10.6
Mine 3	Polymetallic mineralisation	Zn	1.99%	3.54%	77.9
Mine 4	Copper mineralisation	Cu	4.05%	7.99%	97.2
Mine 5	Niobium mineralisation	Nb <sub>2</sub> O <sub>5</sub>	10.29%	6.72%	−34.7
Mine 6	Low-grade gold mineralisation	Au	95.59%	74.99%	−21.6
Mine 7	Low-grade gold mineralisation	Au	20.91%	22.42%	7.2
Mine 8	High-grade gold mineralisation	Au	15.49%	21.19%	36.8
Mine 9	High-grade gold mineralisation	Au	10.49%	16.40%	56.2

The results show that the SSFA, compared to the HT, tends to overestimate K and the total  $s_{FSE}$ , as 12 of the 16 results (75%) present higher values of K and of the total  $s_{FSE}$ . On the other hand, SSFA tends to underestimate  $\alpha$ , as 12 of the 16 results (75%) presented lower values of  $\alpha$ .

The elements/minerals that did not show overestimation of K are as follows:

- $\text{SiO}_2$  (Mine 1—itabirite type 1);
- $\text{Nb}_2\text{O}_5$  (Mine 5);
- Low-grade Au (Mine 7);
- High-grade Au (Mine 8).

The elements/minerals that did not show underestimation of  $\alpha$  are as follows:

- $\text{Fe}_2\text{O}_3$  (Mine 2—bauxite);
- Cu (Mine 3);
- Low-grade Au (Mine 6);
- High-grade Au (Mine 9).

The elements/minerals that did not show overestimation of the total  $s_{FSE}$  are as follows:

- $\text{SiO}_2$  (Mine 1—itabirite type 1);
- Cu (Mine 3);
- $\text{Nb}_2\text{O}_5$  (Mine 5);
- Low-grade Au (Mine 6).

Note that of the eight elements or minerals that did not follow the tendencies, two are impurities ( $\text{SiO}_2$  for iron ore and  $\text{Fe}_2\text{O}_3$  for bauxite) and four are gold. These usually present lower grades, higher nugget effect and often more erratic grade distributions. Thus, it can be stated that the differences for both K and  $\alpha$  values are systematic.

## 6. Conclusions

### 6.1. Overview

This paper compares the sample division using a riffle splitter with the random collection of individual fragments to make up a sample for the calibration of the constants K and  $\alpha$  and the calculation of the FSE. The results of the different mineralisation types show that the SSFA includes variability in addition to the FSE. According to Pitard and François-Bongarçon [20], two types of heterogeneity tests can be performed:

- (1) Tests to estimate exclusively the variance of the FSE;
- (2) Tests to estimate the variance of the  $QFE_1$ , which includes the FSE and the GSE.

The first type estimates exclusively the intrinsic CH of the lot, because the samples are generated by collecting fragments one by one at random—the only condition under which the GSE will cancel. The second type of estimation includes the DH between replicate splits or groups of fragments. It should also be remembered that all types of test result in the inclusion of AE, and potentially PE.

Both the SSFA and the STE (or DSA) are classified as the second type of test; i.e., they include part of the GSE in their results. Therefore, the overestimation of the FSE is more likely since these tests estimate the variance of  $QFE_1$ . According to Pitard and François-Bongarçon [20], the estimation of  $QFE_1$  better reflects what is happening in daily reality in sampling protocols, although segregation is a transient phenomenon that varies from one second to another as the material flows and depends on local conditions that can be many. In other words,  $QFE_1$  varies according to different conditions, where some of them are uncontrollable, and, consequently, a fixed value cannot be attributed to it.

When comparing the HT and the SSFA results, the values of the constants K and  $\alpha$  are disparate. The HT represents the first type of test, estimating the CH alone, while the SSFA represents the second type of test, estimating a combination of both the CH and DH.

## 6.2. Discussion of Results

The results of this study confirmed the overestimation of  $K$  by a type-2 heterogeneity test (e.g., SSFA). The constant  $K$  is expected to be overestimated by the SSFA since part of GSE is added to the results and the variance of the FSE in Gy's formula is directly proportional to the constant  $K$  (Equation (4)). Overestimation of the total FSE relative standard deviations by the SSFA is in line with this statement.

As for the underestimation tendency of  $\alpha$ , a more careful analysis should be conducted to reach a more accurate conclusion about its cause. Dominy et al. [7] affirm that the value of  $K$  depends on the microscopic geostatistical properties of the minerals and varies with gold grade and its liberation diameter ( $d_1$ ). For gold mineralisation in particular, the effect of gold particle clustering at various scales cannot be understated [7,24,28]. Similarly, the underestimation of  $\alpha$  by type-2 heterogeneity tests might also be explained from a geological point of view.

Two hypotheses were outlined by the authors for the observed differences in the constants  $K$  and  $\alpha$  in HT and SSFA:

**Hypothesis 1:** *The GSE increases when minerals are liberated, particularly if the mineralisation contains sulphide or gold, which present high densities. For fragments smaller than 1 cm, especially when they are close to the liberation diameter ( $d_1$ ), if  $\alpha$  is smaller than 1, the variance increases since the variance of the FSE in Gy's formula is directly proportional to  $d^\alpha$  (Equation (4)). Hypothesis 1 suggests that if GSE is included in the SSFA results, the standard deviation of the FSE would increase in the following stages:*

- (i) *The protocol stages where  $d$  is greater than 1 cm due to the increase in  $K$ ;*
- (ii) *The protocol stages where  $d$  is smaller than 1 cm due to the decrease in  $\alpha$ .*

**Hypothesis 2:** *The subsample mass ( $M_q$ ) is squared in the EST  $IH_L$  formula (Equation (6)), and the subsample masses generated by the SSFA are greater than the subsample masses generated by the HT. Therefore, Hypothesis 2 suggests that the greater mass generated by the SSFA would be a relevant factor for the increase in  $K$  and would cause, comparatively with the results of the HT, the following:*

- (i) *The minimisation of the nugget effect impact on the results, particularly for gold mineralisation;*
- (ii) *The reduction in the variance of the 32 subsample grades considering each granulometric fraction;*
- (iii) *The reduction in the relative differences between the four  $IH_L$  values referring to the four granulometric fractions, causing a reduction in the slope of the " $IH_L \times d$ " regression line and, therefore, a decrease in  $\alpha$ .*

Based on the above, it can be concluded that regardless of the real cause of the observed tendencies, the sample division method (SSFA) tends to generate sampling protocols with larger FSE relative standard deviations, which is advantageous as it results in a conservative evaluation of the protocol.

On an individual basis, the high-grade gold "Mine 8" (Tables 5 and 6) displays calculated FSE values of  $\pm 15.5\%$  and  $\pm 21.2\%$  for the HT and SSFA, respectively. Pitard recommends that calculated FSE values are less than  $\pm 16\%$  [29]. The HT-derived FSE value may be deemed acceptable, whereas the SSFA-derived value is high. The itabirite iron ore "Mine 1" (Tables 3 and 4) FSE values are different (0.7% versus 1.2%), though both are acceptable. It is postulated that for base metal mineralisation by virtue of their higher grades compared to gold, the difference between HT and SSFA values is immaterial.

Minnitt, Rice and Spangenberg [9] note that "one of the main objections to the use of the Sampling Tree Method for determining the sampling parameters  $K$  and  $\alpha$ , is the way that these results carry a bias introduced from the total error plus the analytical error that is not accounted for. As a result, the fundamental error will be overestimated." There

are variables other than the ones coming exclusively from the FSE that may inflate the variance, such as the nugget effect (e.g., presence of coarse gold), sample preparation and chemical analysis. However, they are present in both the HT and SSFA results; therefore, the comparison in this study is valid.

## 7. Challenges and Future Work

### 7.1. Challenges

As the sampling community has realised, estimating the heterogeneity of mineralisation, particularly of gold is a challenge. It is known that the results of all heterogeneity test approaches can be used to develop optimal sampling protocols and calculate minimum representative sample masses. However, it is important to keep in mind that protocol optimisation and sample mass calculation will only be effective if the primary sample selection and preparation procedures are equiprobabilistic and unbiased (e.g., representative). In other words, representativity is always dependent upon the successful elimination of systematic errors and minimisation of random errors in all sampling and sample preparation stages.

Of importance is the mass of material taken for any calibration test (e.g., HT or SSFA). For these tests to produce robust results, the primary composite taken must be representative of the mineralisation type or domain. What is appropriate for oxide mineralisation may not suit fresh/sulphide mineralisation. Within a given mineralisation type, the sampling characteristics of low grade versus high grade may also be different [7,23,24]. To evaluate the required representative sample mass, a priori information is required, particularly the key grades and related  $d_1$  values and degree of clustering [7,21,22,24,28]. Yet this is unknown and a required outcome for the testwork/calibration programme.

Another issue is the cost and complexity of calibration testwork, which usually results in a single test of any type (e.g., HT or DSA) being undertaken. This provides no validation of the method. Dominy and Xie [11] note differing results between standard HT tests particularly in the presence of coarse gold. The ultimate calibration will come from multiple tests yielding consistent results, which may be impractical to achieve.

This study was based on the comparison between the estimated sampling constants coming from 50 subsamples made of the same number of fragments (HT) and from 32 subsamples made of approximately the same mass (SSFA). As a recommendation for future work and aiming to validate—or not—the results presented in this paper, the authors suggest comparing heterogeneity tests using the same number of subsamples with approximate masses in each size fraction. For example, for Mine 1 (itabirite type 1), the average HT subsample masses for the first, second, third and fourth size fractions (smallest to largest) were 62.0 g, 91.4 g, 191.5 g and 777.6 g, and the average SSFA subsample masses for same size fractions were 434.8 g, 449.2 g, 1059.2 g and 917.1 g. The HT subsample masses depend on the average fragment mass and the number of cells in the grid, whereas the SSFA subsample masses depend on the amount of mass retained in each screen.

### 7.2. Future and Practical Work

This study indicates that HT and SSFA calibration experiments on the same mineralisation are likely to provide different results for both base and precious metal mineralisation. Various authors recommend integrated calibration of  $K$  and  $\alpha$  through characterisation, where mineralisation logging/mapping, metallurgical and mineralogical testwork, and calibration experiments (e.g., HT or DSA) are applied in support of each other [7,21–23].

The key consideration with the integrated characterisation approach is that it provides various data streams that contribute to developing total deposit knowledge, and it supports the calibration of  $K$ . The determination of  $\alpha$  only results from HT or DSA testwork, though it can be assumed [19,30].

Recommendations are as follows:

- Calibration of mineralisation by an integrated approach should start as soon as it is encountered by outcrop (surface or underground) and/or drilling. At brown-

field sites, a review of historical information, including core, mapping and metallurgical data, can provide information to drive the initial sampling and assaying campaigns [7,13,14,23,24]. Preliminary  $d_1$  values may be identified, and potential worst-case scenarios may be investigated across the following:

- (i) The mineralisation indicator grade;
  - (ii) The mining cut-off grade(s);
  - (iii) The ROM grade;
  - (iv) The high mine grade.
- Thought must be given to the source of material to be used for calibration. In its simplest sense, this will be from one of the following:
    - (i) In situ material (e.g., collected from surface or underground outcrops or composited from drill core);
    - (ii) Broken material (e.g., coarse material from surface or underground stockpiles or conveyed material).
  - Consideration must also be given as to whether the composite is a true composite collected, say over a 2 to 4 h period, from plant feed or a variability sample collected, for example, from an individual underground blast round. If an “average” value is required, then the composite is appropriate. If, however, some measure of local value is required, then the variability sample is optimal.
  - For precious metal mineralisation, in particular gold, initial assaying campaigns should commence with screen fire assaying to identify the presence of coarse gold (if any). This must be supported by core/exposure logging/mapping to identify visible gold and gold associations. For base metal mineralisation, similarly, logging of key minerals to establish particle size and distribution is also important.
  - When mineralisation is physically accessible or enough core or RC residues are available, undertake characterisation bulk (or mini-bulk) sampling and testwork and integrate them with mineralogical and metallurgical programmes. Sample mass requirements will need to be reviewed based on domains, grade distribution and liberation diameter. In gold deposits, a representative mass may be on the scale of tonnes [24], whereas in base mineralisation, a few 100 kg or less may be needed.
  - Characterisation bulk sample protocols should be reviewed on a grade-mineralisation scenario basis and accounting for potential correlation of grade with liberation diameter [23,24]. As a protocol progresses through sample comminution, consideration should be given to the change in liberation diameter. Optimisation around the mineralisation indicator grade and likely breakeven mining cut-off grade must be investigated as they may be the worst-case scenario. Any bulk sample proposed protocol will require validation during initial implementation. This should include the following:
    - (i) Review of practical application;
    - (ii) Collection of field and laboratory duplicates to monitor precision;
    - (iii) Optimisation of grade control during sampling and comparison with any a priori estimates;
    - (iv) Reconciliation with a priori estimates.
  - If HT or DSA testwork is undertaken, then planning is required. The DSA approach is likely to be the better option, given that it evaluates the  $QFE_1$  rather than solely the FSE [20]. Multiple tests must be undertaken across key mineralised domains. Optimally, a master composite from each domain should be collected, preferably from a belt covering an appropriate period, though consideration must be given to the blend of feed source(s) during the collection period. Three or more variability composites should be collected from locations within the domain to investigate variability. QA/QC should support all calibration assays. Laboratory procedures should remove PE. All results, from whichever calibration method is applied, should be reviewed for reality against geological/mineralogical observations [7,21,22].

- A representative sample is unlikely to exist, since extensive and often heterogeneous geological entities are being evaluated. Characterisation/calibration studies to support sampling design are unlikely to fully represent the mineralisation unless excessive expenditure is made. It is therefore important for practitioners to understand any limitations of their work and seek validation of HT or DSA study outputs through other means [7,21,22]. Several integrated characterisation programmes have led to optimised sampling and improved operational performance [13,14,23,24].

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## Abbreviations

AE	Analytical error
CH	Constitution heterogeneity
DH	Distribution heterogeneity
DSA	Duplicate series/sample analysis
FSE/S <sup>2</sup> FSE	Fundamental sampling error/Variance of the FSE
GNE	Geological nugget effect
GSE	Grouping and segregation error
HT	Heterogeneity test
IH <sub>L</sub>	Constant factor of CH or Intrinsic heterogeneity of the lot
K and $\alpha$	Sampling constants
PE	Preparation error
QA/QC	Quality assurance/quality control
QFE <sub>1</sub>	Quality fluctuation error (component 1)
ROM	Run of mine
SFA	Segregation free analysis
SNE	Sampling nugget effect
STE	Sampling tree experiment
SSFA	Simplified segregation free analysis
TOS	Theory of sampling

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