A Brief Review of Heterogeneity Tests for Estimating the Variance of the Fundamental Sampling Error

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ABSTRACT

The fundamental sampling error (FSE) is generated whenever a sample with mass M_S is randomly selected, fragment by fragment, each with the same probability, from a particulate material lot with mass M_L. FSE represents the sampling error between the actual (but unknown) grade of a lot and the grade estimated from a selected sample. This is the smallest possible error for a sample selected under ideal conditions, hence the term "Fundamental Sampling Error". FSE is characterized by its variance, calculated relative to the measured grade of the lot, using the well-known "Gy's formula". The variance of FSE can either be calculated theoretically by applying a set of material factors or can be estimated experimentally by conducting empirical heterogeneity tests to estimate the constant factor of constitution heterogeneity, aka the "Intrinsic Heterogeneity of the Lot," IH_L. Several 'competing' ways to conduct heterogeneity tests and to calculate IH_L have been proposed historically, but a perennial question in the sampling community is: "Which procedure and formulation reveal the *actual* variance of the fundamental sampling error?" To this day, this question has not been answered to the satisfaction of everybody, because (to paraphrase Edward Deming) "without data, you're just another person with an opinion," and no study has so far proven superior validity of one method over another. This paper surveys and explains, in a simplified way, the main experimental methodologies and formulations for estimating the variance of the fundamental sampling error, highlighting the many remaining challenges of heterogeneity testing, which can be seen as a most fascinating topic, however, because of its complexity. It is possible that no singular best approach should be sought in view of the highly complex realm of economic geology and its many types of ore and mineralisation.

1. Introduction

The fundamental sampling error (FSE) is the only error defined in Pierre Gy's Theory of Sampling (Gy, 1967; 1979; 1992) that can never be eliminated and is related to the constitution or intrinsic heterogeneity (*IH*) of the material in question. To calculate the relative variance of the fundamental sampling error, s^2_{FSE} (Equation 1), for a certain sample taken from a certain fragmented lot, crushed to a certain size, the intrinsic heterogeneity of the lot (IH_L) must be estimated, which can be done theoretically applying the Gy's material-characterising factors, or experimentally performing heterogeneity tests.

$$s^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) cfgld^3 = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) IH_L \quad [1]$$

where s^2_{FSE} is the relative variance of the fundamental sampling error, M_S is the mass of the sample (given in g), M_L is the mass of the lot (given in g), *c*, *f*, *g*, and *l* are the four Gy's factors that characterise a specific material (dimensionless, except *c*, given in g/cm³), and *d* is the nominal top-size of the fragments or d_{95} (given in cm).

The AMIRA Metal Accounting Code of Practice (2007) states that there are three basic methods that can be used for determining the value of IH_L: (1) individual particle analysis, also known as the '50 (or more) piece analysis' method, (2) use of scanning electron microscope data from particle sections, and (3) multiple sample analysis. The Code warns that these methods all have their limitations.

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According to Chieregati (2024), there are also three main methods for experimentally determining the value of IH_L: the original heterogeneity test (Gy, 1988; Pitard, 1993) with its several variations, the sampling tree experiment (Minnitt et al., 2007), and the segregation free analysis (Minnitt et al., 2011), from whose results it is possible to calculate the variance of the fundamental sampling error.

The question that looms high is: Which test reveals the *true* s^2_{FSE} and which one reflects what happens in the daily *reality* of sampling processes?

The aim of this article is to present to the reader the existing methodologies as well as their simplified mathematical approaches, but deliberately not to answer the cardinal question above. Chieregati et al. (2023) proved that the tests yield different results, however, to this day, it has not yet been proven which of them allows for a more accurate estimation of IH_L and, consequently, of s^2_{FSE}. Despite considerable theoretical and practical efforts, the issue remains open (see sections Discussion and Conclusions for some reflections on why this may be the case).

2. Metodologies: to select individual fragments or to split the lot?

This section presents the experimental procedures of the main heterogeneity tests proposed over the years, starting with Pierre Gy, almost five decades ago... Note that different notations can be assigned to the same variable. This paper follows the notation from the original authors' work.

2.1 Pierre Gy's 50-fragment method

The "50-fragment method" was proposed by Pierre Gy in his 1988 book, "*Hétérogénéité, Échantillonage, Homogénéisation*" (Gy, 1988), on which the first and second editions of Francis Pitard's books (1989a; 1989b; 1993) were based. Item 4.11 (p. 102) of Gy's book is titled "Experimental estimation of the intrinsic heterogeneity IH_L – the so-called 50/100 fragment method" (*Estimation expérimentale de l'invariant d'hétérogénéité* IH_L *– Méthode dite "des 50/100 fragments*", in French) and describes a method of experimentally estimating the intrinsic ('invariant') heterogeneity of the lot, IH_L.

The operational procedure proposed by Gy (1988) is similar to that presented in his previous works (Gy, 1975; 1982).

While the experimental procedure is practically the same, the interpretation of the results differs significantly.

The newest approach described by Gy (1988) is simpler than the one described earlier because it does not involve measurement of the volume of fragments, an operation little appreciated by practitioners and highly imprecise:

1. Collect randomly, one by one, at least 50, preferably 100 fragments F_i (i = 1, 2, ..., NF) belonging to the coarsest size class of the material lot under study. This can be done by operating with the material retained on the $d/2$ sieve (Figure 1) – if such sieving can be performed – or simply by visually selecting the coarsest fragments 'manually'. The set of all fragments F_i collected constitutes the lot E_1.
2. Wash the fragments (unless otherwise indicated) and dry them.
3. Weigh them dry, obtaining their individual masses M_i.
4. Analyse each fragment for all critical components (analytes): contents a_i, b_i, etc.

Figure 1: Coarsest size class of the material lot under study retained on the d/2 sieve.

According to François-Bongarçon (2024), it is advisable to collect fist-sized fragments so that the mass is adequate for preparation and chemical analysis. Otherwise, analysing a single fragment becomes unfeasible, leading to the method proposed as follows.

2.2 AusIMM's modified 50-piece test

The "Modified 50-piece test" suggests that it is the practitioner's decision to either select 50 individual fragments or 50 groups of individual fragments, each group composed of an equal number of fragments, selected one by one, randomly. The modified test presented by the AusIMM (2023) does not specify how many fragments each group should contain, so the professional has some leeway and may consider a number of fragments representing the mass of the subsample required for physical preparation in the laboratory.

The modified 50-piece test protocol, described below, essentially follows Gy's "50-fragment method" approach:

1. Select at least 50 individual (or subsamples consisting of groups of) fragments from the coarsest size class of a bulk sample with mass M.
   a. Note that the coarsest size class ranges from $d/2$ to d, with d being the nominal top size or d_{95}.
   b. Individual particles may be selected from the coarse size fraction after screening, or by visual estimate, which is often adequate in the case of ores with a large top size.
2. Dry the selected fragment/subsample separately.
3. Measure the dry mass M_j of each fragment/subsample.
4. Crush and pulverise each fragment/subsample separately to produce a pulp that is sufficiently fine (<150 µm) to serve as an analytical test portion.
5. Determine the concentration a_j of each fragment/subsample.

2.3 Simplified 4-size-class heterogeneity test

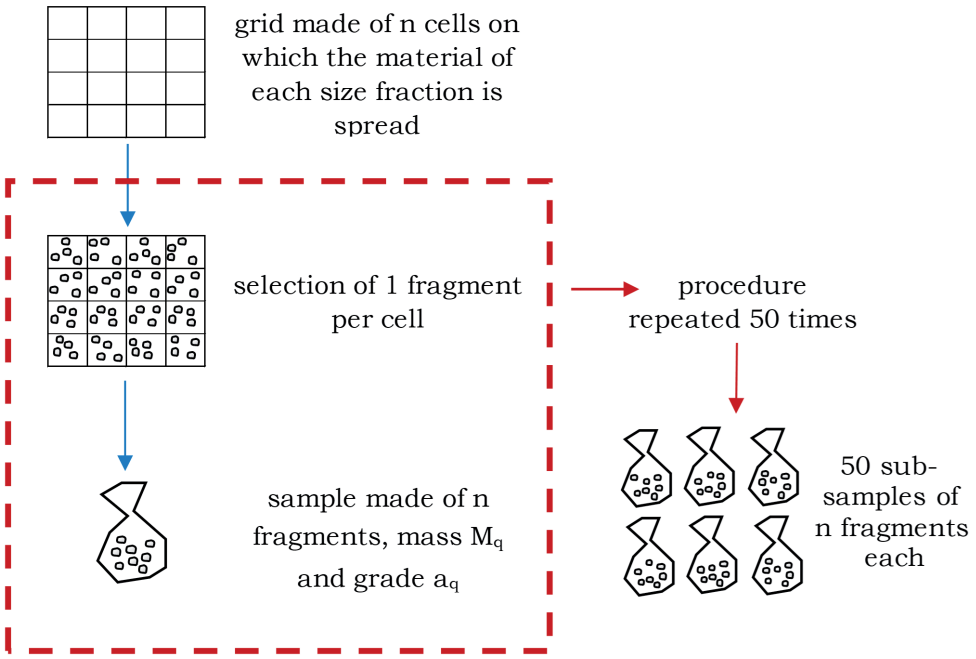
Also based on the "Modified 50-piece test", there are two ways to perform the heterogeneity test when aiming to obtain IH_L for more than one size fraction: (1) dividing the initial lot (ideally 250–500 kg) into four equal parts, crushing each part to a *different* top size, d, and then screening each part down to $d/2$; or (2) screening the entire lot (250–500 kg) into four different size fractions and performing the test for each size fraction separately. Because in the second method the lot is screened at the beginning, rather than being crushed into four different size classes and then screened, it was called the "simplified 4-size-class heterogeneity test" (SHT).

For both methods, the selection of subsamples must be done separately for each size class, as described below:

1. If the material is wet, dry the entire lot before starting the test.
2. Screen – or crush and screen – the lot into four size fractions, starting with the top size class ($-d_{95}+d_{95}/2$).
3. Spread the material of each size class evenly on a grid previously drawn with masking tape, ensuring that no fragment overlap with other fragments.
4. Select at least 50 subsamples, made of groups of n-fragments, from each size class. To give all fragments the same probability of selection, the subsamples are composed of one fragment randomly collected *from each cell* of the grid, making up 50 n-fragment subsamples, as n is the number of cells. Note that the cell sizes vary according to the particle size fraction.
5. Measure the dry mass M_q of each subsample.
6. Crush, pulverise, and split each subsample separately to serve as an analytical sample.
7. Determine the grade a_q of each subsample.

Figure 2 shows an example of n-fragment subsamples being produced from the −½"+¼" size class. In this example, n = 90 and each subsample will be made of 90 fragments (6 × 15 grid cells).

Figure 2 Fragments being collected during the simplified 4-size-class heterogeneity test (−½"+ ¼" size class).



Credit: A. C. Chieregati; used with permission.

Figure 3: Simplified heterogeneity test procedure for each size fraction (Chieregati et al., 2023).

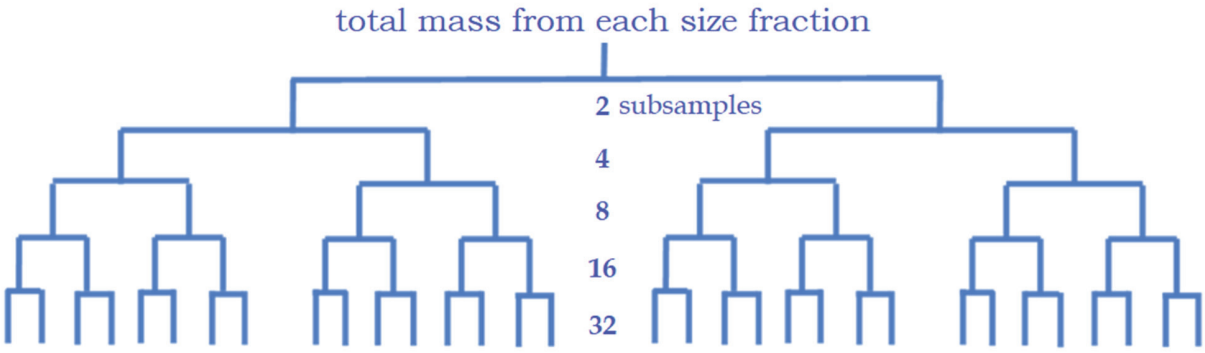
The simplified heterogeneity test procedure is schematised in Figure 3, where 50 *n*-fragment subsamples are generated. Consequently, the total number of subsamples will be 200 (4 size fractions × 50 subsamples). It is important to emphasize that the test can also be performed using three size fractions instead of four, indeed also with an even higher number of size fractions – where and when deemed necessary (of course at a greatly increased workload). The idea is to have 3, or 4 points (or even more) in a graph to calibrate the sampling constants, which will be detailed in the next section.

2.4 Sampling tree experiment and segregation free analysis

The sampling tree experiment (STE) was proposed by François-Bongarçon (1993; 1998; 2008) and is well described with a practical example by Minnitt et al. (2007). The segregation free analysis (SFA) was proposed by Minnitt, François-Bongarçon and Pitard (2011).

The experimental procedure of both methods is similar and is based on the binary sampling tree. The difference is the way each size class is prepared and the number of size classes to be tested: (1) in the STE, the initial lot (approx. 60 kg) is divided into four equal parts, after which each part is crushed to a different top size to be tested; (2) in the SFA, the initial lot (approx. 200 kg) is screened in fourteen different size fractions to be tested. The following steps are common for both tests:

1. After preparing the four or fourteen size classes, riffle split each size class material into a series of 32 subsamples, resulting from five splitting stages and forming the binary sampling tree shown in Figure 4.
2. For the STE, two subsamples can be chosen at random from each size fraction for granulometric analysis to check the d_{95}, leaving 30 subsamples per size fraction for chemical analysis.
3. Measure the dry mass M_s of each subsample.
4. Crush, pulverise, and split each subsample separately to serve as an analytical sample.
5. Determine the grade of each subsample.



Credit: A. C. Chieregati; used with permission.

Figure 4: STE and SFA sampling tree procedure for each size class (adapted from Minnitt et al., 2007).

Figure 5 shows the STE/SFA riffle splitting procedure, where 32 subsamples from each size fraction are generated, two for granulometric analysis (STE) and the remaining 30 (STE)/ 32 (SFA) for chemical analysis. Consequently, the number of subsamples will be 120 (4 size fractions × 30 subsamples) for the STE and 448 (14 size fractions × 32 subsamples) for the SFA. It is important to emphasize that the SFA can be performed using a different number of size fractions. Chieregati et al. (2023) used only four size fractions and called the modified test "simplified segregation free analysis" (SSFA).

Credit: A. C. Chieregati; used with permission.

Figure 5: Material of the −½"+ ¼" size class being riffle split during the STE/SFA procedure.

3. Formulations: to calculate or to calibrate the sampling constants?

This section presents the simplified mathematical approach on which each heterogeneity test described in the previous section is based. Note that different notations can be assigned to the same variable. Out of respect, this paper follows the notation in the original authors' work.

3.1 Pierre Gy's 50-fragment method

With the results of the mass and grade determined for each of the 50 selected fragments, and using Pierre Gy's original formula (Gy, 1988, p. 360):

$$s^2_{EF} = \frac{1-P}{P\,M_L}\,IH_L = \left(\frac{1}{M_E} - \frac{1}{M_L}\right) IH_L \quad [2]$$

where s^2_{EF} is the relative variance of the fundamental error, P is the selection probability, M_E is the mass of the sample (given in g), M_L is the mass of the lot (given in g), and IH_L is the intrinsic ('invariant') heterogeneity of the lot (given in g), it is possible to calculate s^2_{EF} after the experimental estimation of IH_L described as follows:

1. Calculate the mass M_{E1} of the lot E_1:

$$M_{E1} = \sum_i M_i \quad [3]$$

2. Calculate the grade a_{E1} of the lot E_1:

$$a_{E1} = \frac{\sum_i a_i M_i}{M_{E1}} \quad [4]$$

3. Calculate the unbiased random estimator $EST\,[IH_{E1}]$ of the intrinsic heterogeneity of the lot E_1:

$$EST[IH_{E1}] = \sum_i \frac{(a_i - a_{E1})^2}{a^2_{E1}} \cdot \frac{M_i^2}{M_{E1}} \quad [5]$$

4. Evaluate the proportion M_{L1}/M_L of the class. If the lot E_1 is obtained by sieving a lot E with a mass M_E, the estimator $M_{L1}/M_L = M_{E1}/M_E$ can be used. In the absence of objective information, the average value of 0.30 can be adopted.

5. Calculate the estimator of $[IH_L]_1$, which is the standard estimator of IH_L when $[IH_{L1}]$ approaches $[IH_{E1}]$:

$$EST[IH_L] \approx EST[IH_L]_1 \approx EST[IH_{L1}]\frac{M_{L1}}{M_L} \approx EST[IH_{E1}]\frac{M_{E1}}{M_E} \quad [6]$$

According to Gy, the validity of this method depends mainly on the 'invariance' of the intrinsic heterogeneity IH_{L1}, which is a random function of the mass M_{L1}.

3.2 AusIMM's modified 50-piece test

With the results of mass and grade of each of the 50 fragments/subsamples, the following procedure should be carried out for the estimation of IH_S and s^2_r:

1. Calculate the combined dry mass M_S of all fragments/subsamples as:

$$M_S = \sum M_j \quad [7]$$

2. Calculate the combined concentration a_S of all fragments/subsamples as:

$$a_S = \frac{\sum a_j M_j}{M_S} \quad [8]$$

3. Calculate the parameter IH_S as:

$$IH_S = \sum \frac{(a_j - a_S)^2}{a^2_S} \cdot \frac{M_j^2}{M_S} \quad [9]$$

4. Evaluate the mass proportion M_A/M, where M_A is an estimate of the ore weight retained in the size class $d/2$ to d. For example, if the +12.5 mm size fraction in a <25 mm mill feed stream constitutes 25% of the total material flow, the ratio M_A/M would be expressed as 0.25.

5. Calculate the constitution heterogeneity IH of the ore as:

$$IH = IH_S \cdot \frac{M_A}{M} \quad [10]$$

6. The relative variance V_r or s^2_r is calculated as:

$$V_r = s^2_r = \frac{IH}{M_{sample}} \quad [11]$$

3.3 Simplified 4-size-class heterogeneity test

With the results of mass and grade of each of the 50 subsamples per size fraction:

1. Calculate the combined mass M_Q of all subsamples:

$$M_Q = \sum_q M_q \quad [12]$$

2. Calculate the weighted average grade a_Q of all subsamples:

$$a_Q = \frac{\sum_q a_q M_q}{M_Q} \quad [13]$$

3. Calculate the unbiased random estimator *EST IH*$_L$ of the intrinsic heterogeneity of the lot. Note that Pierre Gy's Equations 5 and 6 can be rewritten as Equation 14 (refer to Pitard, 1993, p. 176):

$$\text{EST IH}_\text{L} = g \sum_i \frac{(a_q - a_Q)^2}{a_Q^2} \cdot \frac{M_q^2}{M_Q} \quad [14]$$

4. Use Gy's granulometric factor *g* (0.25 for uncalibrated material and 0.55 for calibrated material) as the mass proportion of the top size fragments for each size class.

5. Calculate the nominal diameter of the fragments, where d_{MAX} and d_{MIN} are the openings (in cm) of the upper and lower screens of each particle size fraction, respectively:

$$d_N = \sqrt[3]{\frac{d_{MAX}^3 + d_{MIN}^3}{2}} \quad [15]$$

6. Plot $IH_L \times d_N$ on a log-log graph and the power regression line (example in Figure 6). In the regression line equation $y = ax^b$, y represents IH_L, a represents K, x represents d_N, and b represents α of the IH_L calibrated formula, K and α being the sampling constants:

$$\text{EST IH}_\text{L} = K d_N^{\alpha} \quad [16]$$

7. The relative variance of the fundamental sampling error, s^2_{FSE}, is then calculated as:

$$s^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \text{EST IH}_\text{L} \quad [17]$$

It is important to emphasize that calibrating the sampling constants *K* and *α* (François-Bongarçon, 1998; Minnitt et al., 2007; Minnitt et al., 2011; Ganguli et al., 2017; Bortoleto et al., 2019; Chieregati et al., 2023) is manifestly not unanimously accepted as the best alternative among sampling experts, between which rather adverse attitudes have been prevalent at times. However, a preliminary study on aluminium ores (Marques and Chieregati, 2023) shows a significant correlation between the theoretical IH_L calculated using Gy's material factors and the experimental IH_L calculated using the calibration of *K* and *α* through the simplified 4-size-class heterogeneity test. This is encouraging as it shows the way forward for more studies in an incredibly complex mineral realm (see sections Discussion and Conclusions).

The calibration proposition based on the simplified 4-size-class heterogeneity test suggests that a log-log plot be constructed with the nominal fragment size d_N on the x-axis and the corresponding values of *EST* IH_L on the y-axis. By plotting the power regression line of the four points on the graph, estimates of the parameters *K* and *α* from Equation 16 are obtained, where *K* is a constant factor representing the product of all Gy's material factors, and *α* is the exponent of the nominal fragment size, equal to 3 in Gy's original formula and determined by the slope of the regression line on the heterogeneity graph.

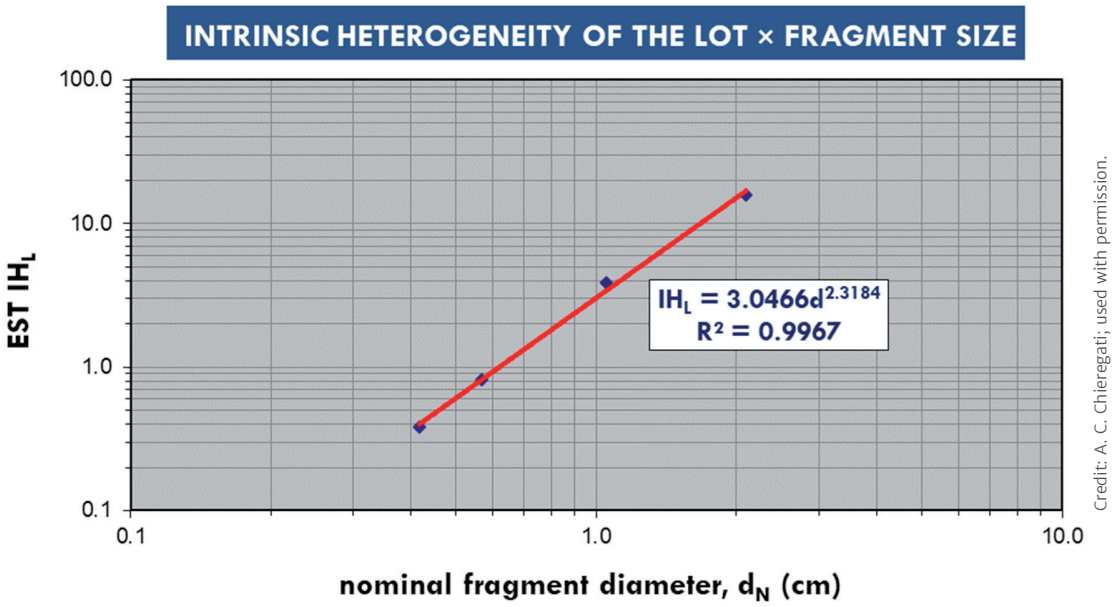


Figure 6: Example of sampling constant calibration using the simplified 4-size-class heterogeneity test.

3.4 Sampling tree experiment and segregation free analysis

Although the STE and SFA experiments involve more complex data processing, including removal of outliers, reduction of the analytical data, calculation of the standardised variance, calculation of the liberation size, etc., the author chose to present a simplified formulation, focusing solely on the calibration of the sampling constants *K* and *α*. For a detailed description of all data processing steps, please refer to François-Bongarçon (1993; 1998; 2008) and Minnitt et al. (2007; 2011).

With the results of mass and grade of each of the 30-32 subsamples per size fraction:

1. Calculate the total relative variance σ^2 of the data for each size fraction.

2. Calculate the residual relative variance σ_R^2 for each size fraction, subtracting the analytical variance σ_A^2 from the total variance:

$$\sigma_R^2 = \sigma^2 - \sigma_A^2 \qquad [18]$$

Note: According to Minnitt et al. (2011) and François-Bongarçon (2024), this adjustment to the variances is necessary, because it has influence on the values for the slope *α* and the intercept *K*, and probably affects the series with the smaller d_N. The variance derived from the 30-32 chemical analyses of each size fraction is a multi-stage variance that includes both the pulp variance and the analytical variance.

The authors state that it is important that the variances from the analytical (pulverised) stage are subtracted from each of the respective multi-stage variances (sets of 30-32 analyses) to provide an unencumbered single-stage variance.

3. Rearrange the simplified Gy's formula (Equation 19) to give a linear equation in logarithmic graph (Equation 20):

$$\sigma_R^2 = \frac{Kd_N^\alpha}{M_S} \qquad [19]$$

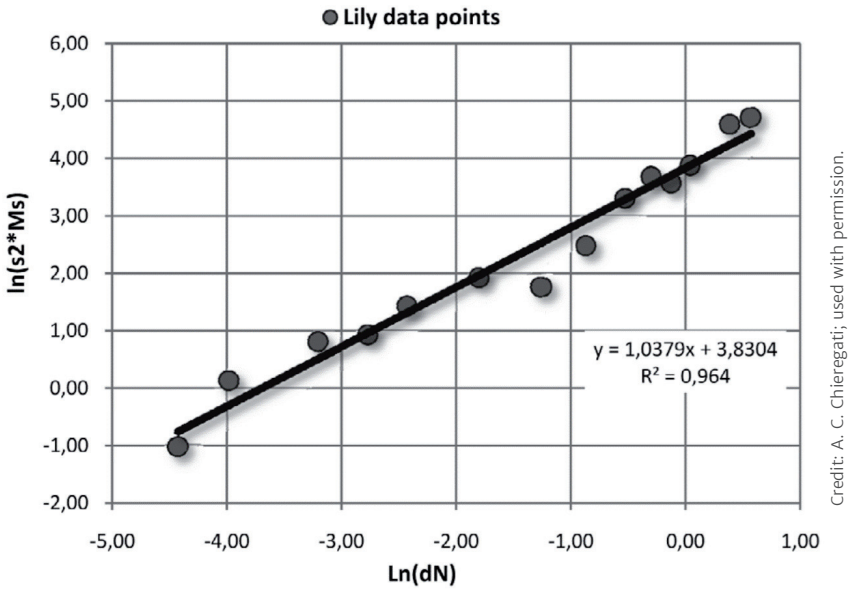
$$\ln(\sigma_R^2 * M_S) = \alpha \ln(d_N) + \ln(K) \qquad [20]$$

4. Plot $ln(\sigma_R^2 * M_S) \times ln(d_{N/MAX})$ on a graph and the linear regression line (example in Figure 7). For the STE, plot each point for its nominal top size d_N; for the SFA, in turn, plot each point for its d_{MAX} (upper screen opening of the size class), not the average diameter.

5.The slope of the line provides a value for *α*, while the constant is the intercept on the y-axis and provides an estimation of a value for *K*. The regression line in Figure 7 shows $\alpha = 1.0379$ and $K = e^{3.8304} = 46.08$ (as for Equation 20).

6. The relative variance of the fundamental sampling error, σ^2_{FSE}, is finally calculated as:

$$\sigma_{FSE}^2 = \frac{Kd_N^\alpha}{M_S} \qquad [21]$$



Credit: A. C. Chieregati; used with permission.

Figure 7: Example of sampling constant calibration using the SFA (Minnitt et al., 2011).

4. Discussion

After the brief review presented in this paper, the question "which procedure and formulation reveal the *actual* variance of the fundamental sampling error?" remains unanswered, despite ongoing studies. Chieregati et al. (2023; 2024) studied different types of ore and concluded that the simplified segregation free analysis, compared to the simplified 4-size-class heterogeneity test, tends to underestimate the sampling constant α and to overestimate both the sampling constant K and the total s_{FSE} of the sampling protocol. Two thirds of the 16 chemical elements analysed in these studies presented lower values of α and higher values of K and s_{FSE}. These trends are partially explained by Pitard and François-Bongarçon (2011), who state that there are two main types of heterogeneity tests: (1) to estimate exclusively the variance of the fundamental sampling error (FSE), or (2) to estimate the variance of the quality fluctuation error, component 1 (QFE_1), which includes both the fundamental sampling error and the grouping and segregation error (GSE). The first type of test estimates exclusively the intrinsic constitution heterogeneity of the lot because the samples are composed by collecting individual fragments one by one at random, the only condition under which GSE will cancel; the second type includes the distribution heterogeneity between extracted replicate splits or groups of fragments. According to these authors, the variance of QFE_1 better reflects what is happening in daily reality in sampling protocols.

Based on all available results from empirical studies that can be found in the open literature, there does not seem to be conclusive systematic patterns for a 'best' heterogeneity test behaviour representing specific types of ore or mineralisation across the mining and exploration industry. Rather there is a strong analogy to the findings of Engström (2017) and Engström and Esbensen (2017) in the study of blast hole sampling versus reverse circulation drilling, which found a similar lack of correlation with respect to specific ore types. Each case is best served with being evaluated individually.

One might be tempted to speculate that a two parameter (K, α) mathematical relationship may be *too simple* a formalism for covering the extremely complex realm of Geology. With so many different types of mineralisation and ores, there is perhaps no reason to expect a singular universal best practice.

Pierre Gy himself once wrote (Gy, 1982, p. 279): "*[...] the method which was developed 25 years ago, breaking up as it does the fundamental variance into a product of simple factors, precises remarkably well the influence of the various characteristics of the material to be sampled.*" According to Gy, then, applying the set of four material-characterising factors (Equation 1) for each type of ore may still be the best option for calculating the relative variance of the fundamental sampling error.

5. Conclusions

Even though a much greater discussion and detailing about heterogeneity studies could be made – and in fact has already been done by François-Bongarçon (2008; 2024) –, the aim of this paper is only to present the general outlines of different experimental procedures and data processing. There will always be a need for carefully planned and meticulously executed empirical characterisation of the material for which a quantitative heterogeneity characterisation is needed, either to estimate s^2_{FSE} or to calculate realistic optimal sample masses.

The most important issue is to keep in mind that depending on how the heterogeneity test is conducted and how the data is processed, different results can be obtained and, consequently, different conclusions will be drawn. According to François-Bongarçon (2024), *designing* the heterogeneity experiment may be the most important step which, when poorly done, can trigger irreversible damage to the conclusions of the study. The author addresses the main problems of heterogeneity studies in detail and affirms that "*on-going recipes and publications are unclear and often false, [...] articles are never supposed to be recipes to follow blindly, instead they should be viewed at most as enlightened suggestions*" (François-Bongarçon, 2024, p. 21).

This is exactly the didactic purpose of this paper: to bring the complex heterogeneity tests to the readers' attention, so they can reflect on them, study the different methods in greater depth, and draw their own conclusions to conduct their own studies, rather than claiming that one approach has superior validity over another. And perhaps one day the cardinal question will have a definitive answer.

The seed has been sown... who would like to take on this challenge?

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