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Sampling Errors and Control of Assay Data Quality in Exploration and Mining Geology

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1. Introduction

The fundamental cause of the errors of samples of rocks and minerals collected by geologists for evaluation of mining projects is heterogeneity of the sampled materials (Gy, 1982; Francois-Bongarcon, 1993; Pitard, 1993). Constitution heterogeneity and distribution heterogeneity (Pitard, 1993) both are important and cause geological sampling errors. The more heterogeneous the sampled material the more difficult it is to obtain a representative sample and infer characteristics of the geological object from samples. The current chapter overviews sampling theory explaining sampling error types and their likely causes, and also describes the practical approaches used in the mining industry for estimating sampling errors and monitoring them at an acceptably low level. It is based on numerous case studies by the author (Abzalov & Both, 1997; Abzalov, 1999, 2007, 2008; Abzalov & Humphreys, 2002; Abzalov & Mazzoni, 2004; Abzalov & Pickers, 2005; Abzalov et al., 2007; Abzalov & Bower, 2009) and also reviews of the recently published QAQC procedures used in the mining industry (Taylor, 1987; Vallee et al., 1992; Leaver et al., 1997; Long, 1998; Sketchley, 1998).

2. Types of sampling errors

Sampling errors are traditionally determined in terms of precision and accuracy of the data (Fig. 1). Precision, or repeatability, is a measure of how close sample values are to one another (Fig. 1) and accuracy is a measure of how close the sample value to the true grade (Fig. 1). Both of these parameters have to be estimated and strictly monitored during evaluation and the eventual exploitation of mineral deposits.

These errors can be generated at any stage of the samples extraction, preparation and the eventual analytical assaying. Depending on the factors causing sample errors they are grouped into three types (Eq. 1):

$$\text{TOTAL ERROR} = \text{Err. 1}^{\text{st}} \text{ Group} + \text{Err. 2}^{\text{nd}} \text{ Group} + \text{Err. 3}^{\text{rd}} \text{ Group} \quad (1)$$

Where:

Err.1st Group – are sampling errors related to a chosen sample extraction and preparation procedure, referred as sampling protocol. An example is poor repeatability of assays when sample sizes are disproportionately small in comparison with the degree of heterogeneity of

material. The main error of this type is known as Fundamental Sampling Error (Gy, 1982). It is always present and can not be fully eliminated as it is related to intrinsic characteristics of the sampled material, such as mineralogy and texture of mineralisation. The Fundamental Sampling Error (FSE) can be minimised through optimisation of the sampling protocols, which will be discussed in the next section. The first group also includes Grouping-Segregation error which is a consequence of the distribution heterogeneity of the sampled material (Pitard, 1993) and therefore this error also relates to the intrinsic characteristics of the sampled material.

Err.2nd Group – is the group of errors related to sampling practise, in other words the errors which depend on how rigorously the sampling protocol was developed, implemented and followed. The group includes delimitation, extraction, preparation and weighing errors. These errors are caused by incorrect extraction of the samples from a lot, their suboptimal preparation procedures, contamination and incorrect measurements. Human errors, such as mixed sample numbers, can also be included in this group. These types of errors can be minimised by upgrading practices of the samples extraction and preparation, which usually needs an improvement of the quality control procedures and often requires equipment upgrading.

Err.3rd Group – analytical and instrumental errors occurred during the analytical operations (Gy, 1982). The group includes assaying, moisture analysis, weighing of the aliquots, density analysis, precision errors and bias caused by suboptimal performance of analytical instruments. These errors are considered in the current study separately from the two first groups because of the different factors causing them.

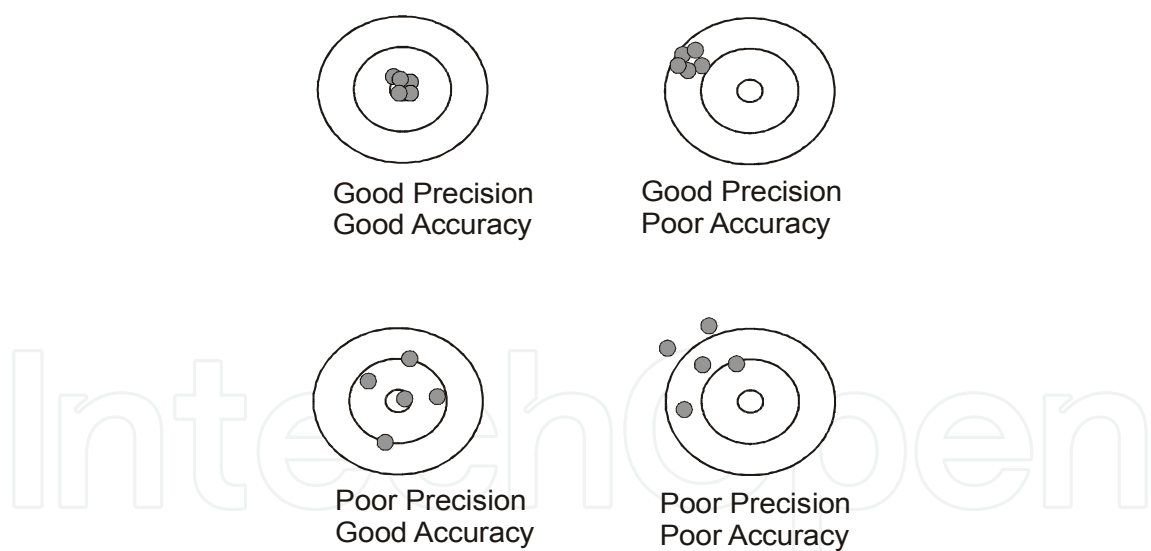


Fig. 1. Sketch explaining precision and accuracy of the data

2.1 Fundamental Sampling Error

Fundamental Sampling Error (FSE) is error related to constitution heterogeneity of the sampled material. It depends on the shape and size of the particles which constitute the sampled material, the size at which the critical components are liberated and also on mineralogy and density of gangue and valuable components. It is the only error which can be theoretically determined as it directly related to the constitutional characteristics of the sampled materials.

a. Theoretical background.
The theoretical approach for estimating the FSE was proposed by P.Gy (1982) and further developed by F.Pitard (1993) and D.Francois-Bongarcon (1993, 1998, 2005). The theory states that FSE, representing precision of the samples expressed as their relative variance, can be estimated as follows (Eq. 2):

$$\sigma_{FSE}^2 = fgcd_N^3 \left(\frac{1}{M_s} - \frac{1}{M_L} \right)$$

(2)

σ_{FSE}^2 - Fundamental Sampling Error, representing relative variance of the precision error;
 f - shape factor. This parameter represents geometry of the particulate materials. It is a dimensionless factor varying from zero, when particles are ideal cubes, to one, when they represented by ideal spheres. Most types of mineralisation have shape factor varying in a narrow range from 0.2 (gold or mica flakes) to 0.5 (isometric grains).
 g - granulometric factor, which is also called a particle size distribution coefficient or size range factor. This factor is dimensionless and taking into account the fact that fragments do not have the same size (d). If all fragments have had exactly the same size the factor (g) would be equal to 1. This theoretically is possible only in an ideal case when studied material is perfectly sorted. In practice it never happens, therefore the (g) factor is less than one and can be as small as 0.1 when particles show a wide range of distribution. Default values of (g) factor are summarised in Table 1. In the mining industry the value of 0.25 is usually used as default value as it suits for most types of mineralisation and corresponds to a case when 95% of particles pass the nominal mesh size.

Type	Explanation	Default (g) value
Non sorted material	Output of jaw crusher	0.25
Sorted material	Material between two consecutive screen openings	0.55
Naturally sorted material	Grains, e.g. rice	0.75

Table 1. Default values of the granulometric factor (Pitard, 1993)

d_N - a nominal particle size in centimetres. This is size (diameter) of a mesh retaining the upper 5% of particles.
 M_s - mass of sample in grams.

M_L - mass of lot in grams.

c - mineralogical composition factor (g/cm³) calculated using (Eq. 3)

$$c = \left(\frac{1-t_L}{t_L} \right) \times (\rho_M (1- t_L) + \rho_G t_L)$$

(3)

where:
 t_L - absolute grade of a lot expressed as decimal proportions of ore mineral, it changes from 0 to 1 (e.g. 1g/t = 0.000001), ρ_M - specific gravity of ore minerals, ρ_G - specific gravity of gangue.

The formula (Eq. 3) can be simplified (Francois-Bongarcon, 1998) and represented by it's concise version (Eq. 4).

$$c = \left(\frac{1-t_L}{t_L} \right) \times \left(\frac{\rho_M \times \rho_G}{\rho} \right) \quad (4)$$

In the equation (Eq. 4) (ρ) denotes the average specific gravity of mineralisation at a given grade (t_L), other variables are the same as in Eq. 3.

For low-grade ores, a mineralogical factor (c) can be further simplified and approximated as ratio of the density of the mineral of interest by the average grade of the studied material (Eq. 5):

$$c = \frac{\rho_M}{t_L} \quad (5)$$

The mineralogical factor (c) relates the sampling variance given by formula (Eq. 2) to the grade of mineralisation (lot) being sampled. D.Francois-Bongarson and P.Gy (Francois-Bongarson & Gy, 2001) have noted that 'any use of the formula, or any sampling nomogram derived from it, only makes sense when the grade level at which it is established is duly stated'.

l - liberation factor, estimated as ratio of liberation size to a nominal particle size (Eq. 6).

$$l = \left(\frac{d_L}{d_N} \right)^A \quad (6)$$

where: d_N - a nominal particle size in centimetres, d_L - liberation size in centimetres, representing a liberation diameter of a mineral of interest, A - exponent.

Substituting liberation factor (Eq. 6) to equality defining FSE it becomes (Eq. 7):

$$\sigma_{FSE}^2 = fgc \left(\frac{d_L}{d_N} \right)^A d_N^3 \left(\frac{1}{M_S} - \frac{1}{M_L} \right) \quad (7)$$

If the exponent (A) is expressed as ($3 - \alpha$) and after reduction of (d_N^3) the FSE formula becomes (Eq. 8)

$$\sigma_{FSE}^2 = f g c d_L^{3-\alpha} d_N^\alpha \left(\frac{1}{M_S} - \frac{1}{M_L} \right) \quad (8)$$

Product of ($f g c d_L^{3-\alpha}$) is known as the sampling constant (Francois-Bongarson, 1993; De Castilho et al., 2005) and usually is denoted as (K).

$$K = f g c d_L^{3-\alpha} \quad (9)$$

Substituting sampling constant (K) to equality (Eq. 2) leads to formula of FSE (Eq. 10) which is most commonly used in practice.

$$\sigma_{FSE}^2 = K d_N^\alpha \left(\frac{1}{M_S} - \frac{1}{M_L} \right) \quad (10)$$

The value of exponent (α) changes depending on (d_N). When (d_N) is smaller than liberation size (d_L) the exponent (α) is equal 3. Above the liberation size (d_L) the exponent (α) can be smaller, within the range of 1 to 3.

Equality (Eq. 10) can be further simplified by removing the ratio ($\frac{1}{M_L}$) which becomes negligibly small when the mass of a lot (M_L) is significantly larger than sample mass (M_S), which leads to concise version of the FSE formula (Eq. 11)

$$\sigma_{FSE}^2 = \frac{K d_N^\alpha}{M_S}$$

(11)

Equality (Eq. 10) and its concise version (Eq. 11) are practically the most convenient tools for experimental definition of the FSE because parameters (K) and (α) can be calibrated experimentally (Francois-Bongarson, 1993, 2005). Methods of calibration are discussed in the next section of the book. When calibrated parameters are not available D.Francois-Bongarcon (1993) has suggested default (K) and (α) values for low-grade mineralisation, such as gold veins, which are $K = 470$ and $\alpha = 1.5$. However, great care should be taken as actual values of the sampling constant (K) can significantly differ from the default value (Sketchley, 1998).

b. Experimental calibration of sampling constants.

Several techniques have been proposed (Gy, 1982; Pitard, 1993; Francois-Bongarson, 1993, 2005; Bartlett & Viljoen, 2002; Minkinen & Paakkunainen, 2005; De Castilho et al., 2005; Minnitt et al., 2007) for experimental determination of sampling constants. The most common approach is the technique developed by Francois-Bongarson (2005), representing a modified version of the ‘sampling tree experiment’ (Francois-Bongarson, 1993), and ‘heterogeneity test’ of Pitard (1993). ‘30-Pieces Experiment’ developed by D.Francois-Bongarson (1993) has many similarities to above mentioned ‘heterogeneity test’ (Pitard, 1993) representing a simplified version of it.

‘Sampling Tree Experiment’ was first proposed by D. Francois-Bongarcon in 1993 and then modified in 2005 (Francois-Bongarson, 2005). The modified version represents analysis of the series of the duplicate samples (Fig. 2) cut from a lot at various comminution degrees (Table 3) allowing to experimentally obtain the (K) and (α) parameters of the Fundamental Sampling Error (Eq. 11).

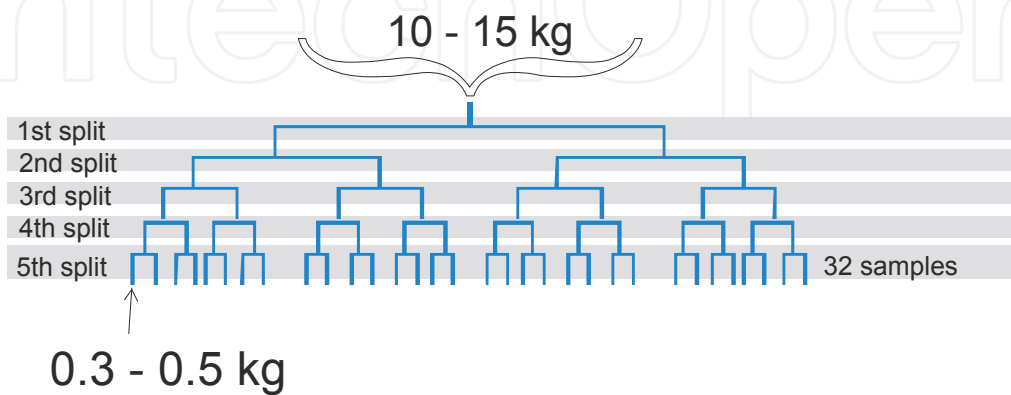


Fig. 2. Flow sheet of the ‘Modified Sampling Tree Experiment’ (MSTE). The shown binary sampling tree is applied to each of the four nominal size fractions (Table 2)

Theoretical background of this method is as follows. Firstly the formula (Eq. 11) can be logarithmically transformed to the equality Eq. 12:

$$\text{Ln} \left(M_s \sigma_{FSE}^2 \right) = \alpha \text{Ln}(d_N) + \text{Ln}(K)$$

(12)

According to this expression the values of $[\text{Ln}(M_s \sigma_{FSE}^2)]$ are plotted against the particle sizes $[\text{Ln}(d_N)]$ as a straight line because the equality (Eq. 12) represents equation of the line ($Y = AX + B$). Tangent of the angle between this line and the abscissa axis (A) is equal to exponent (α) in the equality Eq. 12 and constant (B) is equal to $[\text{Ln}(K)]$. The objective of the ‘MSTE’ is to deduce parameters (A) and (B) of a linear function describing relationships between $(M_s \sigma_{FSE}^2)$ values and the particle sizes (d_N) . In practise, to infer parameters of a linear function $[\text{Ln}(M_s \sigma_{FSE}^2) = \alpha \text{Ln}(d_N) + \text{Ln}(K)]$ it is sufficient to experimentally obtain several points which are plotted onto the diagram $\text{Ln}(M_s \sigma_{FSE}^2)$ vs. $\text{Ln}(d_N)$ and then a linear function is inferred by a suitable best fit algorithm.

‘MSTE’ method is based on collecting a representative sample of 40-60kg which is then dried, successively crushed and split following the flow sheet shown on the Fig. 2. The nominal particle sizes for the four groups of subsamples depend on mineralogy and texture of the mineralisation. Examples of the particle sizes that have been used at the ‘MSTE’ are shown in Table 2 which can be used as a reference when ‘MSTE’ is planned; however, best practise is to determine experimentally the sample weight and the nominal particle size of each sampling series.

		Sampling Series				Elements of Interest	Reference
		First	Second	Third	Forth		
Deposit type	Orogenic Gold	2.5	0.3	0.1	0.05	Au, As	a, b
	Ni-S: Komatiitic-type	3	1	0.5	0.1	Ni, Cu, As	b
	Cu-Au-U: IOCG-type	2.5	0.5	0.1	0.05	Cu, U, Au, S	b
	U: unconformity-type	2.5	1	0.1	0.01	U	b
	Bauxite	3	1	0.5	0.1	Al ₂ O ₃ , SiO ₂ , Fe, LOI	b
	Iron ore: BIF-derived	3	1	0.5	0.1	Al ₂ O ₃ , SiO ₂ , Fe, LOI, P	b
	Cu-Au: porphyry-type	2.5	1	0.1	0.05	Cu, Mo, Au, As	b
LOI – Loss on Ignition Data used: a – (Minnitt et al., 2007); b – Abzalov, M. (unpublished data).							

Table 2. Examples of the nominal particle sizes (cm) of the samples at the “Modified Sampling Tree Experiment”

- Procedure of the ‘Modified Sampling Tree Experiment’ is as follows (Fig. 2):
- Representative sample of 40-60kg is collected and dried;
- The whole sample is crushed at jaw crusher to a nominal size of 95% passing the mesh size chosen for Series 1 (Table 2);
 - One-quarter of the sample (lot) is split out and forms the first subsampling series;
 - Remaining material is crushed to a nominal size of 95% passing the mesh size chosen for Series 2 (Table 2);
 - One-third of these secondary crushed material is split out and forms the second subsampling series;

- Remaining two fractions are recombined and crushed to a nominal size of 95% passing the mesh size chosen for Series 3 (Table 2);
- The crushed material is split by riffle splitter onto two equal subsamples, one of them split out and forms the third subsampling series;
- The remaining material is crushed to a nominal size of 95% passing the mesh size chosen for Series 4;
- Using a riffle splitter each of these portions is now split into 32 samples (Figure 2). Each of the produced samples is weighed, pulverised and assayed. Minnitt et al. (2007) recommends to use 2 samples for granulometric analysis. These samples are randomly chosen from the group of 32 and remaining 30 samples are assayed and used for statistical inference of the (K) and (α) parameters (Fig. 3).

This approach produces 4 groups of 32 samples. Each group includes samples of the same nominal size of the particles and approximately of equal weight.

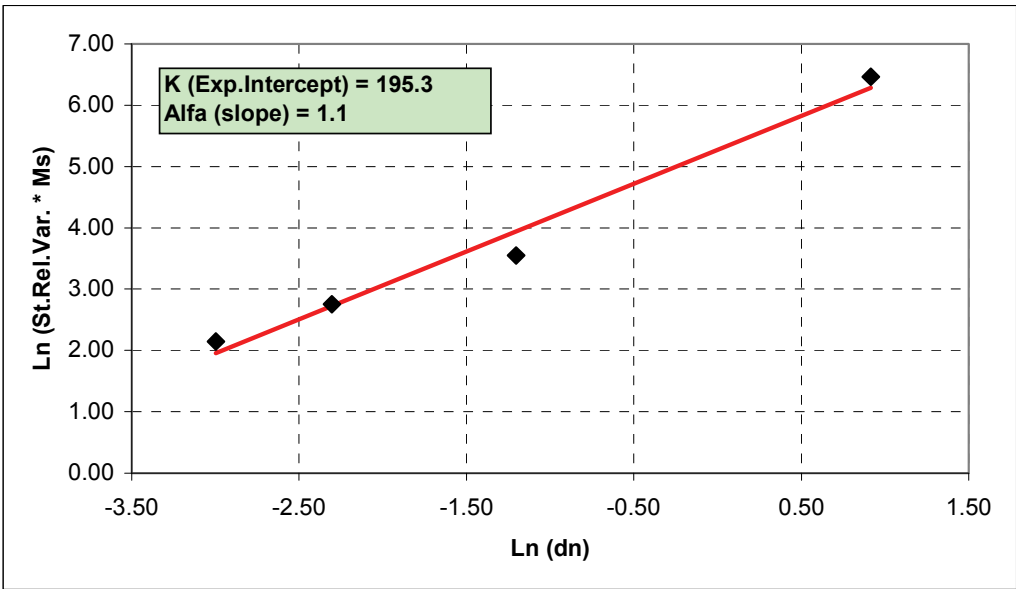


Fig. 3. Experimental calibration of the (K) and (α) parameters of the Eq. 12

c. Sampling nomogram.

Variance of the fundamental sampling error (FSE) can be graphically expressed as a function of sample weight (M_s) and the nominal particle size (d_N). Diagram representing relationships between these parameters is called nomogram (Fig. 4). This is achieved by plotting the FSE vs. the given sample mass (Pitard, 1993; Francois-Bongarcon, 1993). For practical reasons all values are plotted on the nomogram in the logarithmic coordinates (Fig. 4).

On the sampling nomogram (Fig. 4) the crushing and grinding stages, which do not contribute to sampling variance, are represented by vertical lines. The sample reduction stages, when a smaller sample is extracted from a larger sample, in other words a sample mass reduction at constant rock particle size, are represented on the diagram as a path along the straight lines of a slope -1. The actual position of the line depends on particle size (d_N) and also sampling constants (α) and (K) therefore only one line can be constructed for

each sub sampling stage at the given sample particle size (

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