Centre for Computational Geostatistics (CCG)
Guidebook Series Vol. 2

Guide to Sampling

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Centre for Computational Geostatistics (CCG) Guidebook Series

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1 Introduction

In a perfect world when the assay of a sample is reported as 4.2% it is assumed that this figure is the content of the sample. However, the entire sample, which represents a much larger lot in itself, is not assayed, a small subsample of a few grams is selected and the assay refers only to the content of the small subsample. There will always be a discrepancy between the content of the lot, the original sample, and the assay sample. This discrepancy is the termed the sample error.

In sampling there are two forms of error: one that is present due to the intrinsic properties of the material being sampled and one that arises from improper sampling procedure and preparation. The goal of this guidebook is to review guidelines and procedures that are used in practice to design a sampling protocol that will minimize the errors introduced through improper procedure and to measure and use the "fundamental error" that is always present. This guidebook is a summary of Gy's sampling theory as put into practice by Francis Pitard and others.

2 Definitions and Basic Concepts

To help understand the concepts and ideas presented in this guide some definitions and basic concepts will be presented in this section. The notion of accuracy, precision, and repeatability will also be addressed.

2.1 Definitions

- **Fragment Size, d_α(cm):** Actual size of the fragment, or average size of the fragments, in the increment α. Not to be confused with the nominal fragment size defined below.
- **Nominal Fragment Size, d (cm):** Normally defined as the maximum fragment size in a lot, the nominal size is defined here as the square mesh size that retains no more than 5% of the oversize material. This removes potentially high fluctuations in d due to the oversize present.
- **Lot, L:** Amount of material from which increments and samples are selected. A lot of material should have well-defined boundaries: content of a bag, truck, railroad car, ship, etc. A lot is often referred to as a batch of material.
- **Increment, I:** An increment is a group of fragments extracted from a lot in a single operation of the sampling device. A sample that is often the reunion of several increments.
- **Sample:** A sample is a part of a lot obtained by the reunion of several increments and meant to represent the lot in further operations. A sample must respect certain rules that the theory of sampling will lay out. Sampling is often carried out by progressive stages: a primary sample is extracted from the lot, and then a secondary sample is extracted from the primary sample, and so on.
- **Specimen:** Part of the lot obtained without respecting the rules of delimitation and extraction correctness. A specimen does not represent the lot L.
- **Component:** Constituent of the lot that can be quantified by analysis. It may be a chemical of physical component such as: a mineral content, the water content, percent fines, sulphur content, hardness, etc.

  Critical Content, a: Proportion of a critical component that is to be estimated: the critical component of a lot L is denoted a_L, the critical content of a sample S is denoted a_S, etc.

$$\text{Critical content } a_L = \frac{\text{Weight of a critical component in the lot L}}{\text{Weight all components in the lot L}}$$

- **Probabilistic Selection:** A selection is said to be probabilistic when it is founded on the notion of selection probability that includes the intervention of some random element. A probabilistic selection is correct when the selecting probability is uniformly distributed among all units making up the lot and nil for material that does not belong to the lot. A selection is incorrect when one of these conditions is not satisfied. A selection is said to be nonprobabilistic when it is not founded on the notion of selecting probabilistically, such as grab sampling, for example. All forms of nonprobabilistic sampling should be avoided.

- **Heterogeneity:** The condition of a lot where not all elements are identical. There are two types of heterogeneity that we are concerned with: (1) the constitution heterogeneity and (2) the distribution heterogeneity.
- **Constitution Heterogeneity (CH):** Differences between the composition of each fragment within the lot. Contributing factors are the fragment shape, size, density, chemical composition, mineralogical composition, etc. Constitution heterogeneity generates the fundamental sampling error.
- **Distribution Heterogeneity (DH):** Differences from one group to another within the lot. Contributing factors are the constitution heterogeneity, spatial distribution, shape of the lot due to gravity, etc.
- **Errors:** Since samples, and the lots from which the samples are collected, are not exactly the same, errors are introduced in any estimation procedure. The magnitude of the errors can be used to validate the sampling methods and it is essential to differentiate the various sampling errors.
- **Sampling Protocol:** An agreed upon set of stages for sample taking and preparation meant to minimize errors and to provide a sample that is within certain standard controls.

2.2 Error Basics and Their Effects on the Sample Results

Errors may be introduced during the stages required for sampling and sample preparation. The errors can be random with a mean of zero, random with a non-zero mean, or accidental. Several different types of error will be discussed and their impact on the sampling results.

The "fundamental error (FE)" is the only error that cannot be eliminated using proper sampling procedures. It will be present even if the sampling operation is perfect. Fundamental error is a function of the constitution heterogeneity of the material being sampled and it can be quantified before sampling. The errors it generates are random with a mean of zero.

The "increment delimitation error (DE)" and "increment extraction error (EE)" are random errors but their mean is typically non-zero. Unlike the fundamental error delimitation and extraction error can be eliminated through proper sampling procedures. Delimitation error occurs when the shape of the volume for the increment extracted is not correct; for example not taking the entire cross section of a conveyor belt. Extraction error occurs when all of the fragments that belong in the correctly delimited volume for the increment do not end up in that volume. Since the mean of these errors is non-zero bias can be introduced to the sampling procedure.

Accidental errors that occur during sampling or preparation cannot be analysed using statistical methods as they are typically non-random events. Prevention of accidental errors is crucial for reliable sampling.

Since the errors are random variables and are independent in probability the following relationships are true:

- Total sampling error:

$$\mathrm{TE} = \mathrm{FE} + \mathrm{DE} + \mathrm{EE} + \ldots$$

- Average error:

$$\mathrm{E}\{\mathrm{TE}\} = \mathrm{E}\{\mathrm{FE}\} + \mathrm{E}\{\mathrm{DE}\} + \mathrm{E}\{\mathrm{EE}\} + \ldots$$

- Total error variance:

$$\sigma^2\{\mathrm{TE}\} = \sigma^2\{\mathrm{FE}\} + \sigma^2\{\mathrm{DE}\} + \sigma^2\{\mathrm{EE}\} + \ldots$$

The relationships above show that individual errors do not cancel out, but compound. This compounding effect emphasises the care and attention that sampling requires.

Accuracy and precision are very different concepts. When the mean of the sampling error, $\mathrm{E}\{\mathrm{SE}\}$, approaches zero the sample can be called accurate. The sampling error is a bias. A sampling selection is said to be precise when the variance of the sampling error, $\sigma^2(\mathrm{SE})$, is small, that is less than the standard required for a given purpose. It does not matter if the sample average is the same or significantly different from the lot.

In order to use these two measures of sample quality it is necessary to work with the square of the mean sampling error and the variance of the sampling error. When these two numbers are added they lead to the notion of representativeness:

$$\mathrm{r}^2\{\mathrm{SE}\} = \mathrm{m}^2\{\mathrm{SE}\} + \sigma^2\{\mathrm{SE}\} \le \mathrm{r}_\mathrm{o}^2$$

When the mean square of the sampling error, $\mathrm{r}^2(\mathrm{SE})$, is less than a standard threshold, r_o^2, the sample is considered representative.

3 Heterogeneity and the Fundamental Error

As mentioned above, sampling theory differentiates between two types of heterogeneity: (1) the distribution heterogeneity and (2) the constitution heterogeneity. Distribution heterogeneity will be dealt with later in this guide along with its impact on sampling. The constitution heterogeneity could be applied two different ways to sampling theory: the heterogeneity between the fragments making up a sample or the heterogeneity within the fragments of the sample. For our purposes, and sampling in general, the heterogeneity between the fragments is more important. The mathematical definition of constitution heterogeneity, how to practically estimate it, and the definition of fundamental error will be covered here.

3.1 Mathematical Definition of Constitution Heterogeneity

To define the constitutional heterogeneity we will start by examining the heterogeneity carried by one fragment within a lot and continue until we have defined the heterogeneity of the entire lot.

Since we are interested in the critical content of the fragment, a_i, the critical content of the lot, a_L, and the heterogeneity carried by that fragment within that lot, h_i, it seems reasonable that the heterogeneity would be proportional to the difference between a_i and a_L. To simplify the process, h_i is standardized by a_L allowing us to work with dimensionless numbers leading to:

$$h_i \propto \frac{(a_i - a_L)}{a_L}$$

Now that we have a relationship for the heterogeneity based on the critical content we must understand the effect that one fragment will have on the entire lot, L. To do this we use the mass of the fragment, M_i, the average fragment mass, $M_{\bar{i}}$, the mass of the lot, M_L, and the number of fragments, N_F, contained in the lot:

$$h_i = \frac{(a_i - a_L)}{a_L} \cdot \frac{M_i}{M_{\bar{i}}}$$

$$M_{\bar{i}} = \frac{M_L}{N_F}$$

$$h_i = N_F \frac{(a_i - a_L)}{a_L} \cdot \frac{M_i}{M_L}$$

The average of the heterogeneities carried by the fragments of a lot:

$$m\{h_i\} = \sum_i \frac{h_i}{N_F} = 0$$

Taking the heterogeneity of a lot as a random variable the variance can be defined as:

$$\begin{aligned}\sigma^2(h_i) &= E^2\{h_i - m(h_i)\} \\ &= \frac{1}{N_F}\sum_i (h_i - 0)^2 \\ &= \frac{1}{N_F}\sum_i \left(N_F \frac{(a_i - a_L)}{a_L} \cdot \frac{M_i}{M_L} \right)^2 \\ CH_L &= N_F \sum_i \frac{(a_i - a_L)^2}{a_L^2} \cdot \frac{M_i^2}{M_L^2}\end{aligned}$$

This is termed the constitution heterogeneity, CH_L, of the lot.

Due to the difficult nature in calculating the number of fragments within the lot and still requiring the characteristics of the material within the lot, modifications to the constitution heterogeneity formula are needed. The first step is to multiply the constitution heterogeneity by the average fragment mass. Doing this removes the need to calculate the number of fragments in the sample and defines the constant factor of constitution heterogeneity, or the intrinsic heterogeneity, IH_L:

$$\begin{aligned} IH_L &= \frac{CH_L M_L}{N_F} \\ &= \sum_i \frac{\left(a_i - a_L\right)^2}{a_L^2} \cdot \frac{M_i^2}{M_L} \end{aligned}$$

Because the constitution heterogeneity is dimensionless the constant factor of constitution heterogeneity has the units of mass. The units of mass become useful when using the fundamental error for sampling protocol optimization presented later.

Using some approximations the constant factor of constitution heterogeneity can be easily calculated regardless of the sample size. Experience has shown that there is generally a strong correlation between the critical content of a fragment and its density, while the correlation between the critical content of a fragment and its size is not as important. If the lot is separated into fractions based on size and density the average fragment, $F_{\alpha\beta}$, in the lot, $L_{\alpha\beta}$, can be characterized by:

- Volume: v_α.
- Density: λ_β.
- Mass: $M_{F\alpha\beta} = v_\alpha \lambda_\beta$.
- Critical Content: $a_{\alpha\beta}$.

Approximating the constant factor of heterogeneity using groups of similar fragments instead of individual fragments is as follows:

$$\begin{aligned} IH_L &= \sum_i \frac{\left(a_i - a_L\right)^2}{a_L^2} \frac{M_i^2}{M_L} \qquad \text{but: } M_{F_{\alpha\beta}} = \frac{M_{L_{\alpha\beta}}}{N_{\alpha\beta}} = v_\alpha \lambda_\beta \\ &= \sum_\alpha \sum_\beta N_{\alpha\beta} \frac{\left(a_{\alpha\beta} - a_L\right)^2}{a_L^2} \frac{M_{F_{\alpha\beta}}^2}{M_L} \\ &= \sum_\alpha \sum_\beta \frac{\left(a_{\alpha\beta} - a_L\right)^2}{a_L^2} \frac{M_{F_{\alpha\beta}} M_{L_{\alpha\beta}}}{M_L} \\ &= \sum_\alpha v_\alpha \sum_\beta \lambda_\alpha \frac{\left(a_{\alpha\beta} - a_L\right)^2}{a_L^2} \frac{M_{L_{\alpha\beta}}}{M_L} \end{aligned}$$

Using the above formula the constant factor of constitution heterogeneity can be determined accurately for any material. Doing so requires separating the material into a two-dimensional grid of volume and density fractions and the assaying and weighing each of the fractions. Although the above formula is simple and complete it is still too cumbersome to be used outside of a laboratory and requires a simplified form for practical use.

3.2 Estimation of IH_L

Splitting the sample into different size fractions allows IH_L to be determined for the different fractions and the summed to give the value of IH_L:

$$IH_L = (IH_L)_{\alpha 1} + (IH_L)_{\alpha 2} + \cdots + (IH_L)_{\alpha k}$$

where:

$$(IH_L)_\alpha = \frac{v_\alpha}{a_L^2 M_L} \sum_\beta \lambda_\beta (a_{\alpha\beta} - a_L)^2 M_{L_{\alpha\beta}}$$

Using observed features from both laboratory results and field experience this formula can be further simplified with two hypotheses.

First: It has been shown that the critical content of the fraction, $a_{\alpha\beta}$, varies much more from one density fraction to the next than from one size fraction to the next. It follows that all values of the critical content, $a_{\alpha\beta}$, may be replaced with the average critical content, a_β, of the density fraction, L_β.

Second: The ratio, $M_{L\alpha\beta}/M_{L\beta}$, has been shown to vary little from one density fraction to the next. Therefore replacing the ratio, $M_{L\alpha\beta}/M_{L\beta}$, with an average value, $M_{L\alpha}/M_L$, is assumed to be a good approximation and we can write:

$$\begin{aligned} IH_L &= \left(\sum_\alpha \frac{v_\alpha}{a_L^2 M_L}\right)\left(\sum_\beta \lambda_\beta \left(a_{\alpha\beta} - a_L\right)^2 M_{L_{\alpha\beta}}\right) \qquad M_{L_{\alpha\beta}} = \frac{M_{L_\beta} M_{L_\alpha}}{M_L} \\ &= \left(\sum_\alpha \frac{v_\alpha M_{L_\alpha}}{M_L}\right)\left(\sum_\beta \lambda_\beta \frac{\left(a_\beta - a_L\right)^2}{a_L^2} \frac{M_{L_\beta}}{M_L}\right) \\ &= XY \end{aligned}$$

The two factors remaining, X and Y, are related to the size fraction and density fraction respectively and now we will learn how to estimate them.

Shape Factor, f

The shape factor, also called the coefficient of cubicity, is a measure of the fragment shape deviation from a cubic shape. Since the calculation of X requires knowing the volume, v_α, of the fragment and since the volume is equal to the product of the shape factor and the cubed fragment size, $f_\alpha d_\alpha^3$, the shape factor is a correction factor taking into account the fragment size, d_α, and the shape of the fragment to determine its volume.

- If the fragments are perfect cubes, $f_\alpha = 1$.
- If the fragments are perfect unit spheres with $r = 1$, the volume of the sphere would be $4/3\ \pi r^3 = 0.523$, therefore with shape factor $f = 0.523$.

It is a dimensionless unit that has been experimentally determined with most minerals having a shape factor approximately equal to 0.5: coal = 0.446, iron ore = 0.495 to 0.514, pure pyrite = 0.470, quartz = 0.474, etc.

There are a few exceptions though:

- Flaky materials, such as mica, have a shape factor around 0.1.
- Soft solids submitted to mechanical stresses, such as gold nuggets, have a shape factor around 0.2.
- Acicular, or long and pointed, minerals, asbestos, have a shape factor greater than 1 and may be as large as 10. They are difficult to sample.

Examination of the fragments under a microscope is usually sufficient to estimate the shape factor using observations along with the rules of thumb above. Now X can be re-written using the volume definition from the shape factor:

$$\begin{aligned} X &= \sum_{\alpha} \frac{v_{\alpha} M_{L_{\alpha}}}{M_L} \\ &= \sum_{\alpha} \frac{f_{\alpha} d_{\alpha}^3 M_{L_{\alpha}}}{M_L} \end{aligned}$$

Experience has shown that the shape factor is relatively constant for the different size fractions giving us:

$$X = f \sum_{\alpha} \frac{d_{\alpha}^3 M_{L_{\alpha}}}{M_L}$$

Although simplified there is more work to be done for X.

Granulometric Factor, g

The definition for X so far requires knowledge of all the different size fractions contained within a sample even though it is difficult to determine. Using the granulometric factor, g, and the nominal fragment size, d, the fragment size distribution can be accounted for. The granulometric factor is a measure of the range in fragment sizes in the sample:

- Noncalibrated material, crusher product, it is around 0.25.
- Calibrated material, between two screens, it is around 0.55.
- Naturally calibrated material, cereals or beans, it is around 0.75.

Now that we have the nominal size and the granulometric factor, both very easy to obtain, X becomes:

$$\begin{aligned} X &= f \sum_{\alpha} \frac{d_{\alpha}^3 M_{L_{\alpha}}}{M_L} \\ &= fgd^3 \end{aligned}$$

X takes into account the shape, size, and size distribution for the fragments in the sample.

Mineralogical Factor, c

Now, we simplify Y. As it is defined now, Y is the sum proportional to $(a_{\beta}-a_L)^2$ over all of the density fractions. It is a minimum of 0 when the material is completely homogeneous and a maximum when the mineral is completely liberated. When the mineral is completely liberated we assume that there are only two constituents remaining: the mineral and the gangue. When this occurs there is the density fraction, L_M, that contains the pure mineral with a density of λ_M, and the other density fraction, L_g, that is the gangue fraction with a density of λ_g. Furthermore, the mineral critical content $a_M=1$, the gangue critical content $a_g=0$, the critical content of the lot $a_L=M_M/M_L$, and the mass of the lot $M_L=M_g+M_M$. Placing these into the equation for Y yields an equation for Y_{max} that is also called the mineralogical factor c:

$$Y = \sum_{\beta} \lambda_{\beta} \frac{\left(a_{\beta} - a_L\right)^2}{a_L^2} \frac{M_{L_{\beta}}}{M_L}$$

$$\begin{aligned} Y_{max} &= \lambda_M \frac{\left(a_M - a_L\right)^2}{a_L^2} \frac{M_M}{M_L} + \lambda_g \frac{\left(a_g - a_L\right)^2}{a_L^2} \frac{M_g}{M_L} \\ &= \lambda_M \frac{\left(1 - a_L\right)^2}{a_L^2} a_L + \lambda_g \frac{\left(0 - a_L\right)^2}{a_L^2} \frac{\left(M_L - M_M\right)}{M_L} \\ &= \lambda_M \frac{\left(1 - a_L\right)^2}{a_L} + \left(\lambda_g \frac{{a_L}^2}{a_L^2}\right)\left(1 - a_L\right) \end{aligned}$$

$$c = \lambda_M \frac{\left(1 - a_L\right)^2}{a_L} + \lambda_g \left(1 - a_L\right)$$

In many practical cases the final equation can be simplified to one of two forms depending on a_L:

- When a_L is less than 0.1 (10%):

$$c = \frac{\lambda_M}{a_L}$$

- When a_L is greater than 0.9 (90%):

$$c = \left(1 - a_L\right)\lambda_g$$

Both of these provide reasonable estimates of c when a_L is within its respective limits.

It may seem odd that the critical content of the lot, a_L, is present in the formula for the mineralogical factor. Although this is the value that we are trying to determine its expected value, or a close approximation, is most likely known, and if not then an iterative approach can be used. It is worth mentioning that the critical content, a_L, is a proportion, not a percentage or parts per million number, and it is the proportion of a physical element, not the chemical element. For example: if a concentrate is said to be 33% copper that is the percentage of the element and it must be converted to the proportion of the copper hosting mineral for use in determining the mineralogical factor.

Liberation Factor, ℓ

As Y was defined it has a minimum of 0 and a maximum of c. The liberation factor, ℓ, will be used to specify where Y is between 0 and c:

$$Y = c\ell$$

$$0 \leq \ell \leq 1$$

When the material is perfectly homogenous ℓ=0 and when the mineral is completely liberated ℓ=1. Most materials can be classified according to their degree of heterogeneity:

- Very heterogeneous material, ℓ=0.8.
- Heterogeneous material, ℓ=0.4.
- Average material, ℓ=0.2.
- Homogeneous material, ℓ=0.1.
- Very homogeneous material, ℓ=0.05.

These categories lack reliability and should not be used for analytical purposes. Also the liberation factor varies considerably and it is difficult to assign an accurate average to it.

Calculation of the liberation factor is the next step. The first method shown is based on the liberation size of the critical component. The method has changed since the second edition of *Pierre Gy's Sampling Theory and Sampling Practice* by F. Pitard was published [5]. D. François-Bongarçon and P. Gy wrote a paper, [2], with an improved method of determining the liberation factor that corrected some of the common problems associated with the previous calculation and use of the liberation factor. Only the corrected method will be presented here and those interested in the progress of the liberation factor are urged to consult F. Pitard's book. The second method is based on the mineralogy of the material.

Liberation Size Method

Estimation of the liberation factor can be done using the nominal fragment size of the sample, d, the liberation size, d_ℓ, and an additional parameter, b, that can be adjusted to specific deposits based on experience and experimental results. As the fragment size is reduced it reaches a point when the mineral becomes liberated from the gangue: the liberation size. When the fragments are larger than the liberation size ℓ<1, and when all of the fragments are smaller than the liberation size ℓ=1. The liberation factor may be estimated with the following formula:

$$\ell = \left(\frac{d_\ell}{d}\right)^b$$

The new parameter, b, may be initially confusing but there are a few rules governing its use and some suggested values that help with its application. First of all, b will never have a value less than 1. A value of 1 is the least favourable and if used will result in an overestimation of Y and therefore the constant factor of constitution heterogeneity. With analysis of the sampling results, b can be increased to a value appropriate for each deposit. Due to the fact that it is a new concept there have not enough experiments and studies performed for reliable trends in the value of b to be determined. Experiments have shown that gold has a b value of approximately 1.5.

Mineralogy Method

The mineralogy method for calculating the liberation factor requires the critical content of the lot, a_L, and the critical content of the largest fragment, a_{MAX}. The liberation factor can be estimated using the following formula:

$$\ell = \frac{a_{MAX} - a_L}{1 - a_L}$$

The available information will help determine which method should be used to calculate the liberation factor.

Final Form of the IH_L Estimation Equation

Assembling all of the factors gives us one simple equation that can be used for the estimation of IH_L. The required factors are:

- **f:** the shape factor accounts for the shape of the fragments and how they deviate from a perfect square; it is a dimensionless number.
- **g:** the granulometric factor accounts for the size differences between the fragments; it is a dimensionless number.
- **d:** the nominal fragment size; d has units in centimetres.
- **c:** the mineralogical factor accounts for the maximum heterogeneity condition that can be present in the sample; units in grams per centimetre cubed or specific gravity.
- **ℓ:** the liberation factor accounts for the degree of liberation in the sample; dimensionless number.

Resulting in the final equation:

$$IH_L = c\ell f g d^3$$

The constant factor of constitution heterogeneity has units of mass, specifically grams, and is used to relate the fundamental error to the mass of the sample.

3.3 Fundamental Sample Error, FE

The fundamental sampling error, FE, is defined as the error that occurs when the selection of the increments composing the sample is correct. This error is generated entirely by the constitution heterogeneity. Gy has demonstrated that the mean, m(FE), of the fundamental error is negligible and that the variance, σ^2_{FE}, can be expressed as:

$$\sigma^2_{FE} = \frac{1-P}{PM_L} IH_L$$

Where P is the probability of selection for any one fragment within the lot and:

$$M_S = PM_L$$

Substituting this into the variance equation gives us:

$$\sigma^2_{FE} = \left(\frac{1}{M_S} - \frac{1}{M_L} \right) IH_L$$

and when $M_L >> M_S$:

$$\sigma^2_{FE} = \left(\frac{1}{M_S} \right) IH_L$$

These are very practical and useful formulae for designing and optimizing sampling protocols. The application of this formula is the focus of the next section.

4 The Nomograph

To make use of the constitution heterogeneity and its effects on the sampling process we have to relate the state that the sample is in, fragment size and mass, to the state that we want the sample to be in, smaller mass with a smaller fragment size. The error produced during these stages is measured and minimized using the constitution heterogeneity of the material being sampled and the nomograph.

4.1 Nomograph Construction

A nomograph is a base 10 log-log plot with the sample variance on the ordinate axis and the sample mass on the abscissa axis. To plot the variances versus sample size the formula must be converted to logarithmic space:

$$\begin{aligned}
\sigma^2_{FE} &= \left(\frac{1}{M_S} \right) IH_L \\
&= \frac{1}{M_S} c\ell fgd^3 \\
\log\left(\sigma^2_{FE}\right) &= \log\left(\frac{1}{M_S} c\ell fgd^3 \right) \\
&= \log(c\ell fg) + 3\log(d) - \log(M_S) \\
&= C + 3\log(d) - \log(M_S)
\end{aligned}$$

Plotting changes to the sample using this method allows easy visualization of changes made to the sample. These changes are actual steps in the sample preparation and can be either a reduction in fragment size through comminution, crushing or grinding, or a reduction in the sample mass through splitting.

When the sample is split there is no change in the nominal fragment size so all of the terms in the sample variance equation are constant and the variance becomes directly proportional to $-\log(M_S)$. Changes to the mass of the sample, and the variance of the sample, by splitting will follow a line on the nomograph with a negative one slope. This allows lines representing the different nominal fragment sizes during the sample preparation to be plotted on the nomograph. During comminution the mass of the sample stays constant and the other terms of the equation will change. Comminution results in a reduction of the sample variance due to the reduced fragment size, and on the nomograph this will be a vertical line from the larger nominal fragment size line straight down vertically to the lower nominal fragment size line.

Figure 4-1 shows the size lines for six different nominal fragment sizes, a sample mass reduction step, and a comminution step. The size lines, for the nominal fragment sizes d_1 decreasing to d_6, are shown as the thin lines that extend beyond the upper and left edges of the nomograph boundary. When the sample was split the nominal size stayed the same but the mass decreased resulting in an increase in the sample variance. This is shown as the thick line from point A to point B. During the comminution phase the sample mass stayed the same and the nominal fragment size decreased resulting in the sample variance dropping from its position on the larger size line down to a point on a lower size line corresponding to the nominal fragment size produced from the comminution cycle. This is shown as the thick line from point B to point C.

4.2 Sampling Fundamental Error

The fundamental error that occurs during the sampling protocol is:

$$\sigma_{FE}^2 = \left(\frac{1}{M_{S2}} - \frac{1}{M_{S1}} \right) IH_L$$

From this formula it is apparent that comminution of the sample, where the mass remains constant, does not introduce any error. That means that the sampling error occurs when the sample is split from a large mass, M_{S1}, to a smaller mass, M_{S2}. Over several sample preparation stages the fundamental error is the sum of the error variances during the individual stages:

$$\begin{aligned}\sigma_{FE}^2 &= \sum_i \sigma_{(FE)i}^2 \\ &= \sigma_{(FE)1}^2 + \sigma_{(FE)2}^2 + \cdots + \sigma_{(FE)n}^2\end{aligned}$$

This is a simple method for calculating the fundamental error introduced during sample preparation. Using the nomograph for sampling protocol optimization is explained in Section 10.

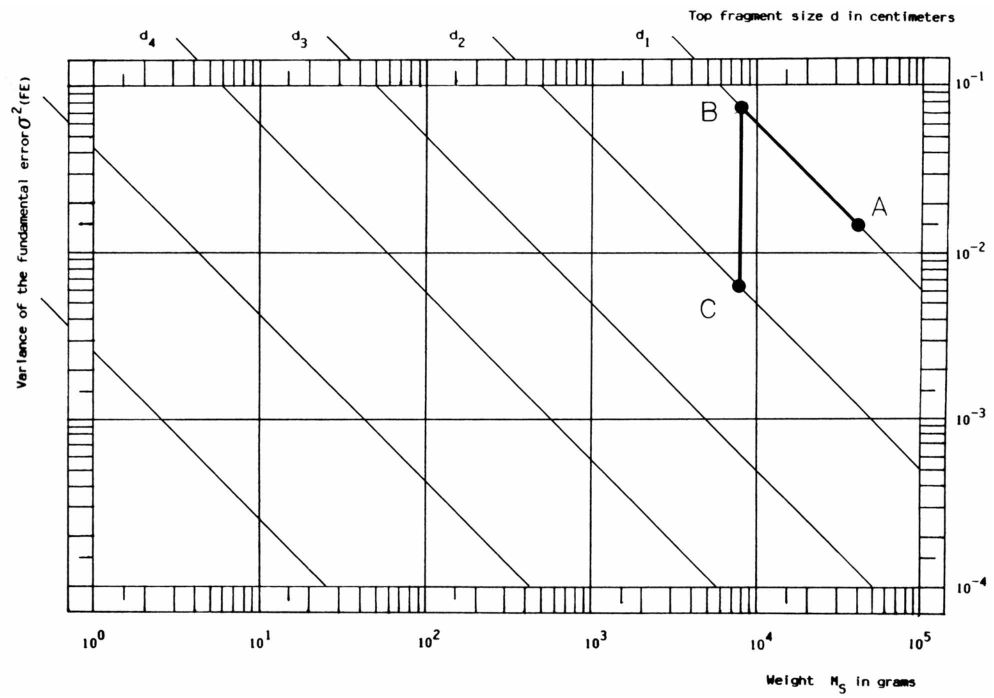

Figure 4-1: Example nomograph showing the nominal size lines, a sample splitting cycle, and a comminution step [5].

5 Segregation or Distribution Heterogeneity

Now that we have discussed the heterogeneity between the different fragments within the material that is being sampled we will discuss briefly the heterogeneity between groups of fragments within the sample lot. The distribution of groups of fragments within the lot is called distribution heterogeneity or segregation. Segregation can occur during the geological development of the deposit or during handling of the material once it has been mined. This segregation of material in a lot is due to differences in the fragment size, shape, density, mass, angle of repose, etc. Figure 5-1 and Figure 5-2 show two different cases of segregation and how the material properties contribute to the segregation. Short scale distribution heterogeneity, like those shown in Figure 5-1 and Figure 5-2, must be understood and the sampling protocol designed to remove any influence that the segregation may have.

Larger scale regional segregation that occurs as part of a deposit has a minimal impact on the sample quality due to the fact that the small local area being sampled is representative of that area. Trends are an example of segregation that can occur on a large scale.

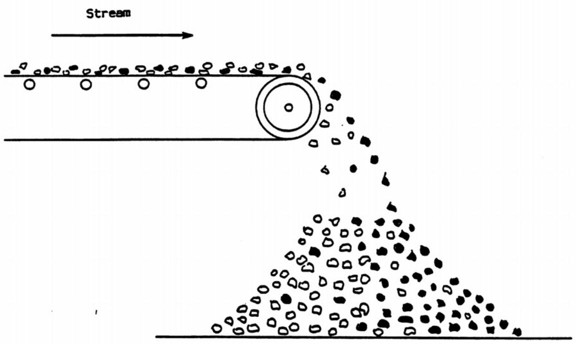

Figure 5-1: Segregation due to differences in the fragment density (higher density fragments carry to the far side of the pile while the less dense fragments remain on the near side) [5].

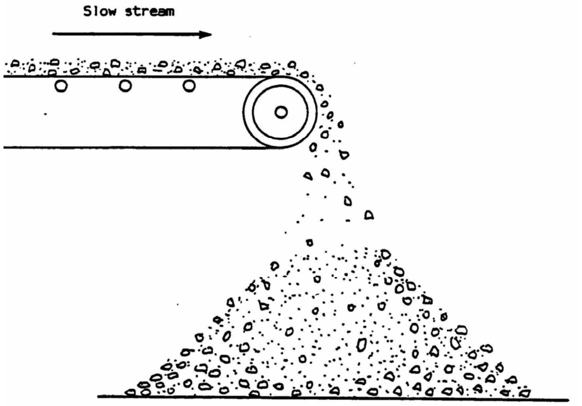

Figure 5-2: Segregation due to differences in the fragment size (coarse fragments roll down the pile concentrating themselves at the edge of the pile while the fine fragments remain near the centre) [5].

6 Lot Classification

Theoretically a lot will always have three dimensions but usually one or two of the dimensions can be regarded as having only secondary importance. Simplifying lots based on their geometric configuration greatly reduces the complexity of the sampling process. In practice it will be common to encounter the following:

- **Three-dimensional lots:** content of a ship, truck, railroad car, bag, jar, etc., as long as one of these three-dimensional objects is considered as the whole lot. It could also be a compact solid such as a block inside a mineral deposit.
- **Two-dimensional lots:** where the thickness becomes negligible because it is very small compared to the two other dimensions. An example of a two-dimensional lot is a three-dimensional lot that has been flattened or a seam of a coal deposit.
- **One-dimensional lots:** continuous and elongated piles, material on conveyor belts, streams, etc., or a series of trucks railroad cars, bags, jars, etc. The thickness and width of the lot are insignificant compared to the length.

Each of the different dimensions has an ideal sample shape. Delimitation error is introduced when the shape of the increment selected from the lot is not appropriate for the type of lot being sampled, but it can be eliminated through proper practices.

6.1 Three-Dimensional Lots

The isotropic module for correct delimitation of a three-dimensional lot is a sphere. A sphere ensures that all fragments the same distance from the centre point of the sample have and equal probability of being sampled.

Spherical samples are nearly impossible to retrieve as access to the selected point is often impractical, the sampling device alters the material integrity before reaching the point, and the collection of a sphere of material is not realistic. Only liquids and very fine suspended solids in liquid can be sampled correctly with a spherical sampling device. All other material cannot be sampled in this way and therefore we must rely on two-dimensional samples such as coring.

6.2 Two-Dimensional Lots

Two-dimensional lots are slightly easier to sample than three-dimensional lots as the ideal shape for correct sample delimitation is a uniform cylinder with a constant cross section taken through the entire thickness of the deposit. Drilling for either solid core or cuttings is the best method for obtaining a representative sample.

Using augers, thief probes, or triers can be representative for the majority of the lot but they are incapable of sampling the bottom correctly. Chip sampling by hand can be representative but the cross section has the potential to be highly variable.

6.3 One-Dimensional Lots

Correct sampling of one-dimensional lots is easy to achieve. Ideally the sample will be extracted inside two parallel planes where the distance between the planes remains constant and the lot is entirely crossed by the sampling device.

7 Delimitation Error and Extraction Error

Improper sample shape, delimitation error, and incorrect methods for obtaining the sample, extraction error, yield errors that are very detrimental to correct sampling. The errors produced generally have non-zero means and introduce a bias to the results. After the correct shape of the increment has been determined its extraction must follow the centre of gravity rule. This rule states that all fragments with their centre of gravity inside the increment belong in that increment and all of the fragments with their centre of gravity outside the increment do not belong.

7.1 Delimitation Error

It is very difficult to properly sample three-dimensional lots since the ideal sample shape is a uniform sphere. Liquids and fine powders are the only materials that can be sampled effectively for this shape. In practice core and rotary drilling samples are taken.

Two-dimensional lots are easier to sample than three-dimensional lots with the ideal sample being a cylinder that extends from the top to the bottom of the deposit. These types of samples are very easy to obtain using core or rotary drilling. It is important to ensure that the entire thickness of the deposit is sampled.

One-dimensional lots are the easiest to sample and correct delimitation of the sample is very easy to achieve. Ideally the sample will be extracted inside two parallel planes where the distance between the planes remains constant and the lot is entirely crossed by the sampling device. Figure 7-1 and Figure 7-2 show proper delimitation of stream sampling.

Proper delimitation of blasthole cuttings is a constant problem for those involved with sampling at producing mines. The best and most practical method uses a sectorial sampler placed at the collar of the hole. It should be pie shaped and be deep enough so that it will not overflow before the end of the drilling. Figure 7-3 shows an illustration of a sectorial sampler.

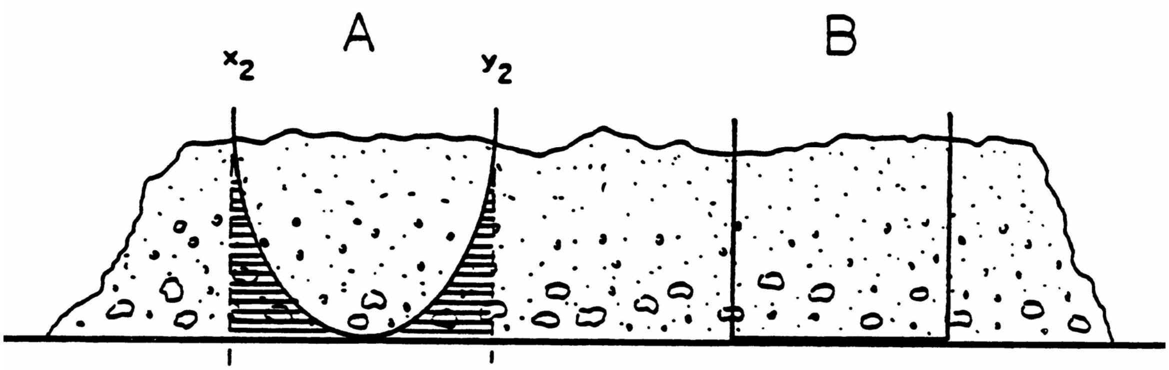

Figure 7-1: Incorrect and correct delimitation introduced by round and square section scoops respectively. It is important that the scoops being used have sides that are tall enough to contain the sample or some material may fall from the scoop and be lost [5].

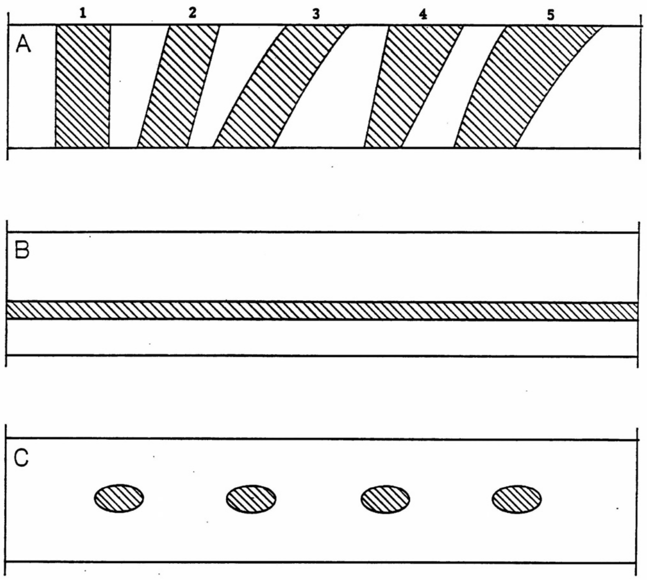

Figure 7-2: (A) Correct samples of the stream are shown as 1, 2, and 3 taken part of the time while 4, and 5 are incorrect. (B) and (C) Taking part of the stream all of the time or only some of the time is incorrect [5].

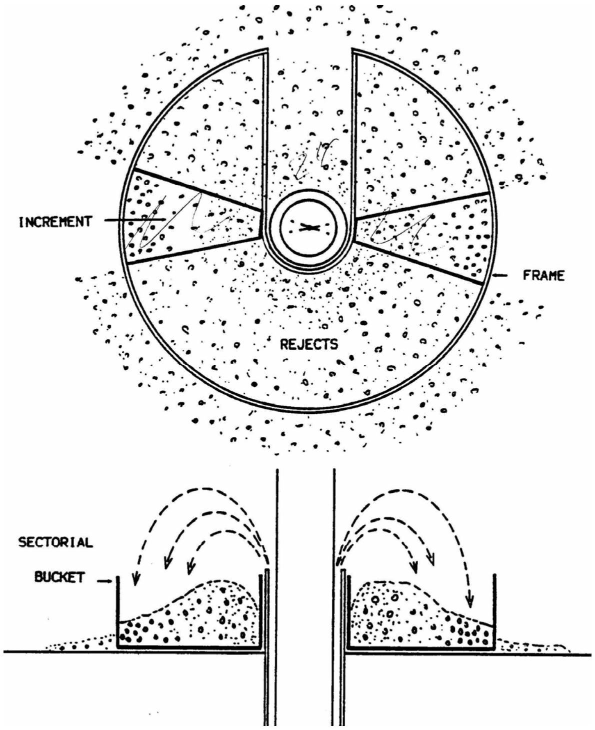

Figure 7-3: Sectorial sampler for sampling blast hole cuttings [5].

7.2 Extraction Error

Since drilling provides the majority of samples used in ore reserve estimation it is important to understand how errors can be introduced by the different drilling methods. Once the samples have been obtained they are generally split. The splitting should also follow proper extraction rules.

Drilling

Core drilling is used when the material is competent enough to provide solid sections of core or at deep depths. When drilling competent material, the bits will cut through the fragments and the introduction of extraction error will be minimal. If soft material is being drilled the error will be minimal as long as no large fragments are encountered. Any large fragments that are encountered may be pushed downwards by the cutting edge of the bit and block recovery of core material.

Rotary and percussion drilling fracture all of the material and extracts only cuttings as the sample. The high water and air pressure required could lead to caving of the hole walls and including material that should not have been. Figure 7-4 shows the possible deviation from ideal for a reverse circulation drill hole.

Sample Splitting

Only true splitting devices and methods will be presented here. Riffle splitters use symmetry of the sampling system to prevent extraction error. The splitters usually consist of 8-12 chutes that discharge on alternating sides of the splitter. When the sample is passed through equal portions should be present on each side. To ensure proper sample splitting the riffle splitters should be constructed of smooth material, fed slowly into the centre and perpendicular to the plates not parallel to them

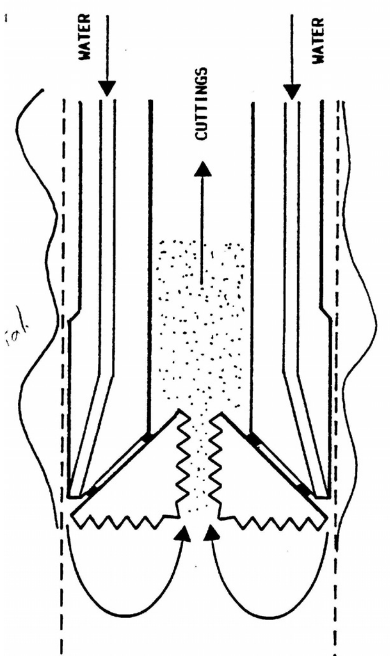

Figure 7-4: Reverse circulation drilling with caved walls where some of the cuttings recovered do not belong to the sample The dashed line is the ideal wall profile while the solid line is the actual [5].

Sectorial splitters are the best choice for sample splitting as they are unlikely to introduce extraction error due to the symmetry of the system but they are not practical for field use. The angles of the chute and constant material feed per rotation are required for proper splitting to occur.

Cone and quartering is a method for splitting samples by hand. First the material must be piled into a cone. Once the pile is built dragging the material down towards the edge with the shovel flattens the cone leaving a flat circular cake. The cake is then divided into four identical quarters. This method is not recommended, as the alternate shovelling method is more effective.

Alternate shovelling is similar to cone and quartering but works better for large samples. The pile of material is built in the same way as for the cone and quartering method but the division of the pile is different. A shovelful of material is removed at a time and placed into alternating piles making sure that each shovelful is the same size, each heap is built with the same number of shovels.

Fractional shovelling is identical to the alternate shovelling method except that instead of having the sample broken into two portions there are N portions when the sample splitting is complete.

Figure 7-5 show how increments can be selected from a lot and the combined to give a sample that is representative.

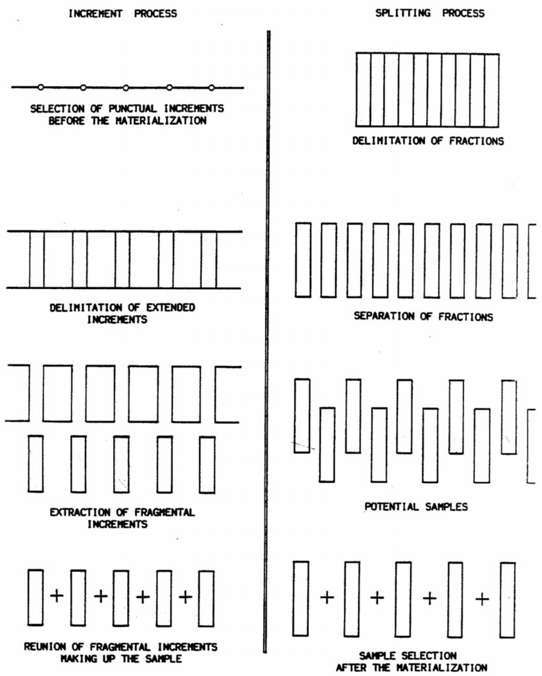

Figure 7-5: Selection or increments from a lot and combining to obtain a sample [5].

8 Preparation Error

As soon as the insitu material is disturbed to the moment that it is assayed it is possible to introduce a wide range of possible preparation errors. Due to the non-random nature of these errors and the long time frame over which they can occur, it is important to have a good sampling protocol that outlines handling and preparation of the sample material so that the samples are representative. The errors can come from contamination, losses, changes in the chemical or physical composition, unintentional mistakes, and fraud or sabotage.

Contamination of the sample can be in the form of dust falling on the sample, material left over in the sampling circuit, abrasion on the sampling equipment, or corrosion. These errors can either increase or decrease the critical content for the component of interest and once the sample has been contaminated there is nothing that can be done remove the contamination.

Losses may occur as fines in dust, material left in the sampling circuit, or smearing of the critical component. Usually the component being sampled is a minor portion of the overall material and it is significantly different in composition from the gangue material. This means that losing a specific fraction of the sample, the fine or coarse fraction, will result in the sample no longer being representative.

Changes in the chemical composition of the sample must be avoided. Some potential changes are sulphide oxidation, or fixation of water or carbon dioxide. Alterations in the chemical nature of the material will impact the assay results and the expected recovery of the material as it is processed. Physical composition changes are generally less important than chemical changes and may be moisture content gain or loss, or a change in the fragment size. Since this is normally done during the sample preparation these changes are acceptable; unless of course the goal is to determine the water content of the size fractions within the sample.

One time human errors have the potential to be significant and it is very hard to determine the source of the error. Dropping the samples, mixing of samples, improper labelling, poor maintenance of the equipment, contamination, etc, are mistakes that may introduce error. Care and attention to detail as well as following the sampling protocol will ensure that errors of this type are minimal.

Fraud and sabotage are the intentional alteration of the samples used to increase, or decrease, the value of a sample for personal or corporate gain. Inflation of the mineral content in the deposit to increase share value has occurred in the past and as a result the requirements for sampling quality and documentation of the sampling undertaken are becoming more stringent.

9 Special Considerations in Sampling

9.1 Gold

Gold presents some interesting complications to the sampling process. When the gold is not liberated or it is associated with another mineral it can be treated similar to other particulate matter. Due to its very high specific gravity, segregation is very strong as soon as the gold becomes liberated from its host material. Additionally, gold does not comminute very well resulting in the gold smearing and coating the sampling equipment. These two phenomena of gold require special attention and have even resulted in a modified formula for the fundamental error.

$$\sigma_{FE}^2 = \left(\frac{1}{M_{S2}} - \frac{1}{M_L} \right) \frac{0.8}{a_L} d_{Au}^3$$

Another important consideration is that gold can occur at very low levels and still be economic to mine. Again more care is required to prevent any sampling error other than the fundamental error

9.2 Coal

Coal is slightly different than most other minerals. Coal is generally the predominant component in the lot and the sampling is being done to determine the contaminants present in the lot.

All of the regular rules for sampling still apply but the coal is treated as the gangue material and the contaminant as the critical component. Sampling is normally done to determine the moisture content, ash content, sulphur content, and the amount of organic volatiles.

9.3 Oilsands

The oilsands of Northern Alberta are unique. Bitumen is the mineral of interest that normally occurs at roughly 10-12%. Sand, water, and clay comprise the remainder of the host material. All of these factors impact how the material behaves during processing and are all determined during the sampling.

9.4 Diamonds

Diamonds present a very unique sampling problem. The chance of intersecting a gem quality stone with core drilling is miniscule while the value of the deposit is highly dependant on the quality of the gemstones.

Exploration is usually done for geological structures, kimberlite pipes, and indicator minerals. Bulk sampling is required for an accurate estimate of the gem content and quality that is extremely important for the value of the deposit.

As more and more data becomes available relationships can be determined for the gemstone content versus the fine diamond content. This is useful for production sampling whether through dedicated drilling or blast hole sampling.

10 Sampling Protocol Optimization

Understanding and minimizing all of the above mentioned errors is essential to designing and maintaining an effective sampling protocol. The general stages involved are (1) taking the primary sample, (2) selecting a subsample either by splitting or comminution then splitting, and (3) repeating this until the sample is small enough that it can be sent to the lab. Figure 10-1 shows an example sampling protocol.

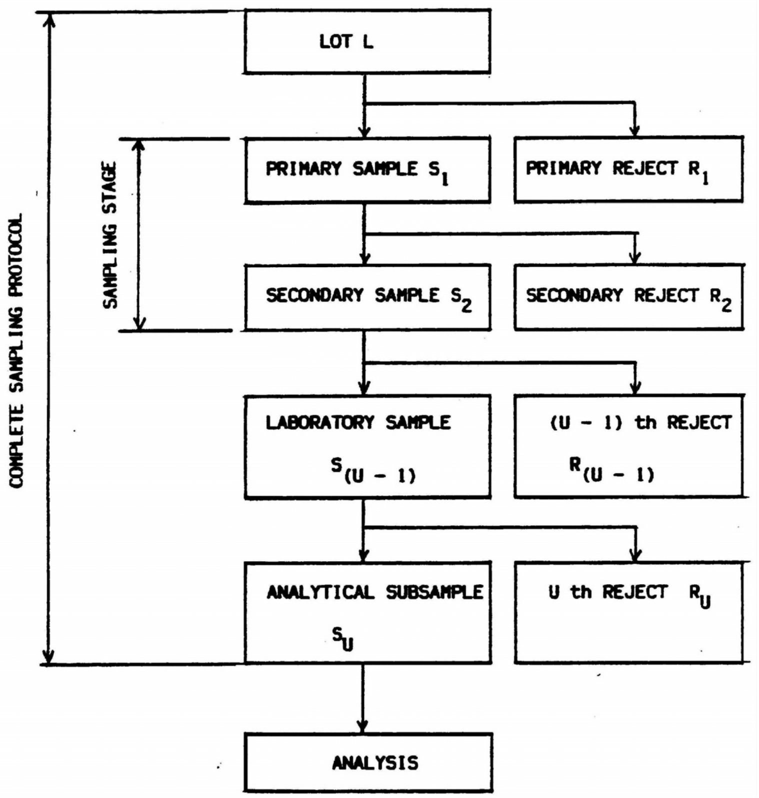

Figure 10-1: Example sampling protocol [5].

Experience has shown that the fundamental error, measurable using IH_L and the nomograph, constitutes only half of the total error found in the sample when everything is said and done. Doubling the variance is a more accurate estimation of the sample error. Accounting for this in the design stages, as well as using all of the error reduction tools available, helps to ensure the sampling is correct for its intended purpose.

A step-by-step example showing how to construct the nomograph, the steps required to design a sampling protocol, and measuring the sample error is shown in section 12. Design and optimization of the sampling protocol is an iterative process. Choosing a protocol and calculating the final error and then modifying the protocol based on available equipment will result in the best protocol suited to the local conditions.

11 Sampling Standards

Different tolerances in sampling are required for the many various applications sampling is used for. In pit sampling of ore versus a sales contract for concentrate require drastically different degrees of confidence in the sampling.

Some accepted standards are:

- 15% for internal process control and exploration.
- 5% for contract sales or compliance.
- 0.5% for commercial sales.

Achieving the targeted sample error cannot always be done using a single sample and may require taking multiple samples and averaging. The fundamental error variance will decrease as follows provided that the samples are independent:

$$\sigma^2 = \frac{1}{n}\sigma_s^2$$

Sampling to set targets is achievable. Sampling protocols should have set quality control targets that are checked on an ongoing basis.

12 Example Construction and Use of a Nomograph

This example, modified from [5], illustrates the steps and equations required to construct the sampling nomograph and to calculate the fundamental sampling error produced during the sample preparation. Characteristics of the material being sampled are:

- Molybdenum deposit occurring as MoS_2.
- Expected mineral content; a_L=0.100%
- Mineral density; λ_M=4.7.
- Gangue density; λ_g=2.7.
- Liberation size; d_ℓ=500μm
- 40 kg core sample.
- Half of the core will be assayed.
- Reduce the sample through comminution and splitting to a size of 1g that is appropriate for assaying

First the constant factor of constitution heterogeneity, IH_L, must be calculated for all the possible nominal sizes that the sample could have during the preparation process. The nominal sizes are dependant on the equipment being used. For example: blasted rock will be coarse and have a target size for the crusher, crushed rock produced from the crusher will be screened for milling, etc.

To estimate IH_L all of the required factors must be chosen from a table or calculated. The following values were used and the rational given:

$$f = 0.5 \quad (\text{standard value for most materials})$$

$$g = 0.25 \quad (\text{unclassified material})$$

$$\lambda_M = 4.7 \quad (\text{rock property})$$

$$\lambda_g = 2.2 \quad (\text{rock property})$$

$$a_L = \frac{0.100\%}{100} \cdot \frac{160}{96} \quad \begin{pmatrix} \text{160 is the atomic weight of } MoS_2 \\ \text{96 is the atomic weight of Mo} \end{pmatrix}$$

$$= 0.00167 \quad (\text{Molybdenum metal content changed to mineral content})$$

$$\ell_d = \left(\frac{0.05}{d}\right)^1 \quad \begin{pmatrix} \text{the liberation factor was calculated in the table below} \\ \text{and a conservative value of 1 was chosen for b} \end{pmatrix}$$

$$c = \frac{\lambda_M}{a_L} = \frac{4.7}{0.00167} = 2814 \quad (\text{since } a_L \text{ is less than 0.1 an approximation was used})$$

The calculation and plotting of IH_L for the possible fragment sizes can now be done. To determine the coordinates of the point being plotted a mass was chosen for each of the nominal sizes. The error for each point was calculated assuming that $M_L >> M_S$ with the results summarized in the Table 12-1:

Table 12-1: Calculation of IH_L for multiple nominal fragment sizes.

d (cm)	ℓ	IH_L (g)	size	σ^2(FE)
2.5000	0.020	110	1.00E+05	1.10E-03
0.9500	0.053	15.9	1.00E+04	1.59E-03
0.4750	0.105	3.98	1.00E+04	3.98E-04
0.2360	0.212	0.982	1.00E+03	9.82E-04
0.1700	0.294	0.509	1.00E+03	5.09E-04
0.1000	0.500	0.176	1.00E+02	1.76E-03
0.0710	0.704	0.0888	1.00E+02	8.88E-04
0.0425	1.000	0.0271	1.00E+01	2.71E-03
0.0250	1.000	0.00551	1.00E+00	5.51E-03
0.0150	1.000	0.00119	1.00E+00	1.19E-03
0.0106	1.000	0.000420	1.00E+00	4.20E-04

Plotting these points on the nomograph and drawing lines with a slope of negative one through them gives us the size lines that will be used to decide on the splitting and comminution stages during the sample preparation.

An additional constraint is that during the sample preparation no single operation will introduce more error than σ_{FE}=5%. To plot this threshold on the nomograph, the error limit has to be converted to a variance σ^2_{FE}=0.025. Figure 12-1 shows the nomograph up to this stage. The points calculated for the size lines are shown on the nomograph as red bullets and the size lines as thin black lines running through the circles. Each of the lines is labelled with its size, in centimetres, at the upper left end of the line. The variance design line is shown as the dashed blue line with its value at the right edge of the line.

After splitting the initial core sample of 40kg in half 20kg of intact core is left. Deciding what size to crush the core to is the initial step in the sample preparation procedure. First locate the 20kg, or 20 000g, mass on the horizontal axis and choose an intersection with one of the size lines that is below the variance design line. Let's pick 0.475cm as the nominal size of the first comminution. Place a point on the 0.475cm size line for a sample mass of 20kg; the fundamental error variance for the sample can now be read off of the vertical axis. The next step is to split the sample in preparation for the next comminution. To decide how small of a subsample to take follow the size line to the left and upwards until it is just below the variance design line and stop there. After following the size line it is decided to split the sample from 20kg down to 2kg, the point on the size line is below the variance design line but that's all right. Then continue with comminution and splitting until the final sample mass is 1g. The nomograph shown in Figure 12-2 was used to determine each step of the sampling protocol. Table 12-2 summarizes the sampling protocol showing the sample size reduction during each splitting stage, the fragment size reduction during each comminution stage, the fundamental sample error introduced during each splitting stage, the sample error due to fundamental error, and the final sample error.

You can see now how useful nomographs are in understanding the impact that the different sample preparation stages have on the final sample error and how this can be used to plan the sampling protocol to achieve the best possible sampling results.

If the sampling error of 9.7% is too great; then, (1) the protocol can be modified to reduce the error introduced due to the fundamental error or (2) multiple samples can be used and averaged.

Table 12-2: Summary of the sampling protocol.

Position in Protocol	Point on Nomo-graph	Sample Mass M_S (g)	Fragment Size d (cm)	Fundamental Error σ^2	Fundamental Sample Error σ^2	Sample Error % σ
Primary Crushing of Core	A	20000	0.475	1.99E-04		
Split for First Subsample	A-B	2000	0.475	1.99E-03	1.79E-03	4.23
Secondary Crushing	B-C	2000	0.071	4.44E-05		
Second Split	C-D	50	0.071	1.78E-03	1.74E-03	4.17
Grinding of Sample	D-E	50	0.015	2.38E-05		
Split for Lab Assay	E-F	1	0.015	1.19E-03	1.17E-03	3.42
Sample FE					**4.69E-03**	**6.85**
Total Sample Error					**9.38E-03**	**9.69**

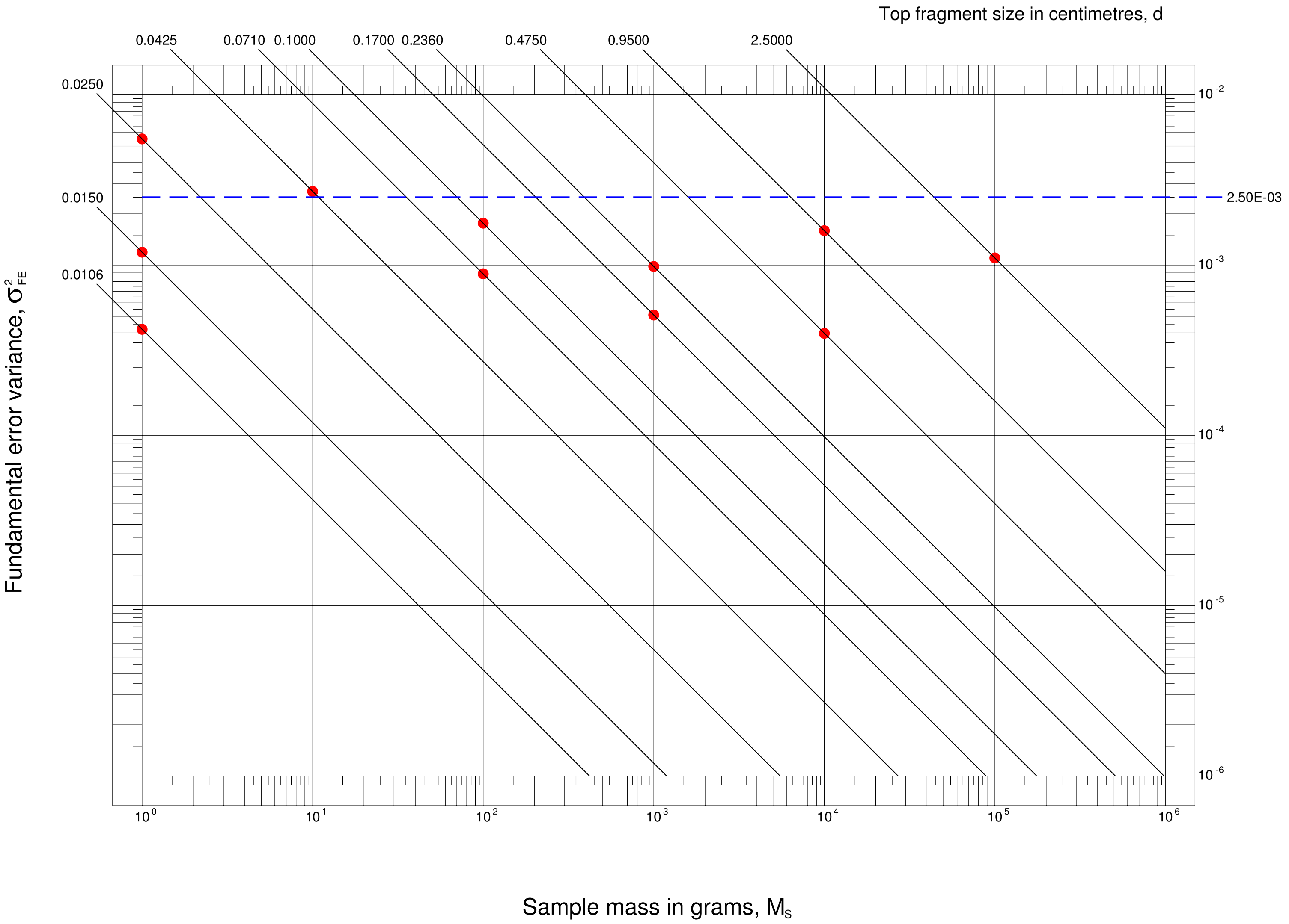

Figure 12-1: Nomograph showing the points to place the size lines (red dots), the size lines (black line), and the variance design line (dashed blue line).

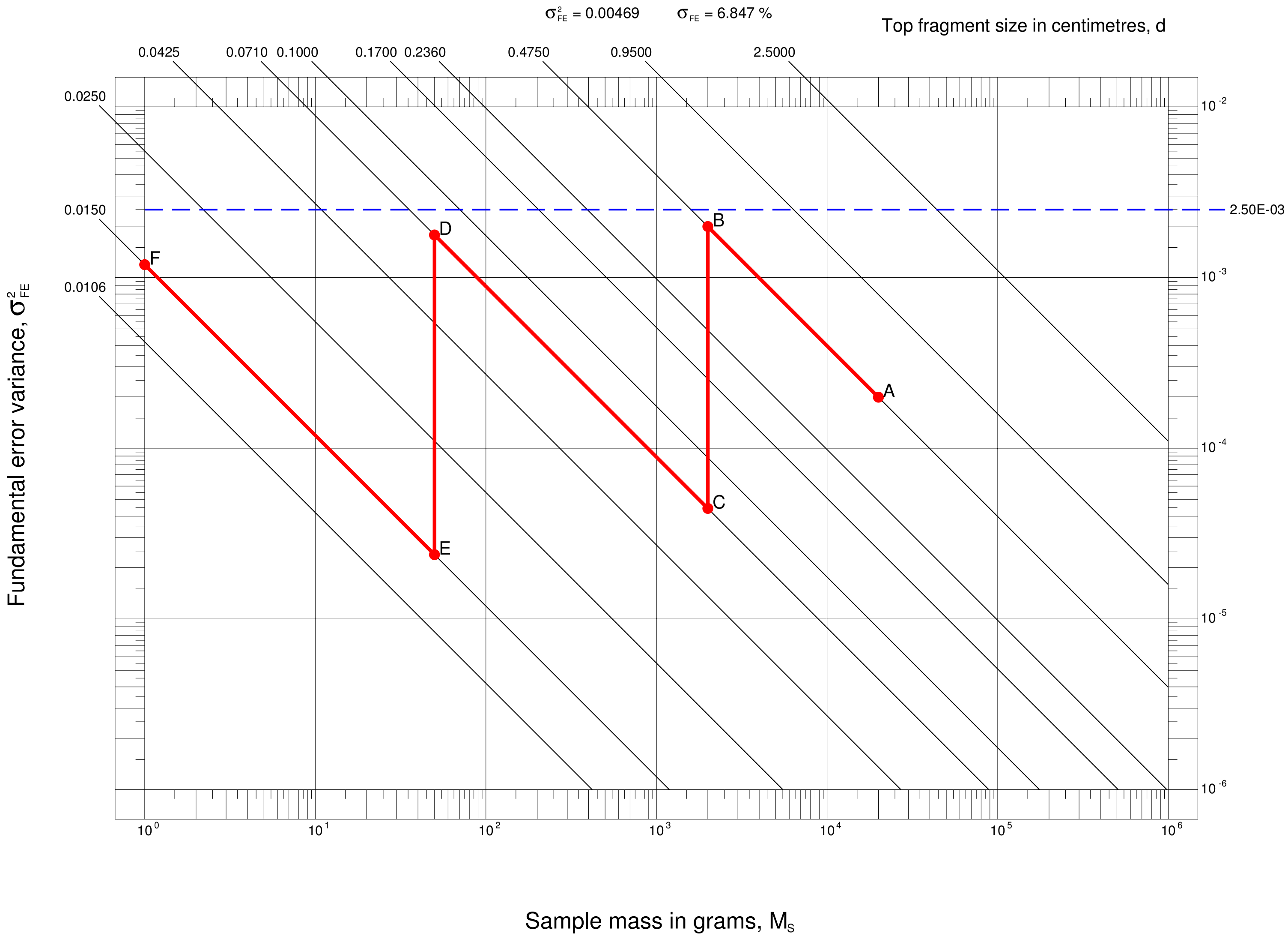

Figure 12-2: Nomograph showing the sampling protocol and the final sample error variance and standard deviation.

13 Conclusion

When first encountered the theory and basis of sampling appears very complex. As your understanding grows the practically of employing Pierre Gy's theory to everyday sampling become apparent. Designing sampling protocols and monitoring the long-term reliability/accuracy of a sampling program are just two problems that can be taken on using the methods presented here.

One unfortunate, but intriguing, facet of sampling is that local conditions tend to dominate compared to the expected norm. Due to this variability it is difficult to provide a step-by-step procedure that will be valid for all cases. Readers are urged to consult *Pierre Gy's Sampling Theory and Sampling Practice* by F. Pitard for an expansion on the theory presented here. For specific mineral or rock sampling problems the best sources of information are published papers or conference proceedings.

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