SAMPLING IN THE MINERALS INDUSTRY

Introduction to Sampling Theory and Sampling Practice

By

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ACKNOWLEDGEMENTS

The authors would like to thank Mr Rolf Steinhaus of MULTOTEC © for his editing the relevant sections of this book and for contributions in terms of text and photographs he supplied in the sections dealing with the Delimitation Error, Extraction Error, Weighting Error and Preparation Error.

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1 PIERRE GY AND THE THEORY OF SAMPLING (TOS) – A TRIBUTE

The term Theory of Sampling (TOS) is attributable to Kim Esbensen who coined the term in his tribute to Pierre Gy "50 years of Pierre Gy's 'Theory of Sampling'-WCSB1": A tribute" that appeared in the in the Proceedings of the First World Conference on Sampling and Blending (WCSB1) (Chemometrics and Intelligent Laboratory Systems 74), in 2004, that was dedicated to Pierre Gy's special contribution to science and the advancement of knowledge. Esbensen (2004) suggested that the TOS be viewed as a representative discipline as well as 'a new, holistic approach to data analysis' that struggled to gain acceptance in the fields of analytical chemistry, process technology, industrial manufacturing, engineering, geology and medicine, etc., because of the perceived competition with an inadequate application of traditional statistical methods in the area of sampling. In bringing TOS to stature as a full blown discipline, it was realised that with the time available at the Sixth Scandinavian Symposium on Chemometrics in August 1999, it was impossible to do Pierre Gy's Theory of Sampling justice. 'The lack of knowledge of TOS in this and related scientific areas was simply too great' pg4. At this time the idea of a conference dedicated to a more complete introduction to the TOS was conceived and brought to fruition in the First World Conference on Sampling and Blending, WCSB1. At 75 years of age Pierre Gy compiled a new and updated introduction to the TOS that appears as the tutorial series in the proceedings of WSCB1 (2003). In addition Pierre Gy tells of his own personal history and the emergence of TOS in the paper entitled "50 years of Sampling Theory – a personal history".

The Theory of Sampling (TOS) is extremely significant in all branches of science, technology, and industry where *proper sampling* is the basis for meaningful generation of data and information from correct (representative) sampling. According to Esbensen (2004) there are some who have taken umbrage that "data quality" should be subject to scrutiny by TOS, and that there is consequently a call for analytical chemistry, process technology, data analysis, statistics, and chemometrics, etc to bear a greater share of the responsibility for practical sampling.

Proper scientific attention to TOS has become the order of the day in the southern African mining fraternity for example. While many may not have fully implemented the necessary procedures and protocols consistent with TOS, there is nodding assent and agreement that it is essential and that there is consistent working towards compliance with TOS in these matters. Courses that explain and apply the TOS are now presented by consultants, experts and academics, and are commonplace although there are some areas that remain refractory. The work of Pentti Minkkinen of Lappenranta University of Technology, Finland in academia, chemometrics and process technology is applauded since he has single-handedly taught the TOS at university level for more than 10 years.

The WCSB1 proceedings provide important reasons why a conference dedicated exclusively to sampling has been convened, and introduces the importance of proper sampling within a broad range of sciences and technological applications including analytical chemistry, mineral processing, process technology, engineering, pharmaceuticals, food, feedstock manufacturing, geology, geochemistry, clinical chemistry, and medicine amongst others.

Pierre Gy (extreme left), understands the importance of scientific interaction through meetings, symposia, conferences; he was 30 years old when this picture was taken in 1954. OECC Mission (North America). At the extreme right of picture: the Norwegian representative Prof. Magne Mortenson, Technical University of Trondheim.

PLEASE NOTE THAT DR FRANCIS PITARD IS A CO-AUTHOR OF THIS BOOK. Reference to his slides has been attached to all the relevant slides, but some may have been omitted by mistake.

2 STATISTICAL TERMS, DEFINITIONS SYMBOLS, AND NOTATION FOR THEORY OF SAMPLING

The Theory of Sampling has enabled order to be brought to this unique topic. The vocabulary and definitions for various fundamental terms in the Theory of Sampling are provided here.

Circumstantial property: A circumstantial property depends solely on chance or circumstance, i.e. upon properties of a material being sampled over which there is no human control. Examples include accuracy, the outcome of a bias test, or the outcome of a round-robin exercise, the segregation induced by a stacker-reclaimer system, a change in moisture content, a change in flow-rate, a change in particle size distribution, etc. There is not much one can do about these undesirable effects.

Structural property: Structural properties are intrinsic to the material being sampled. They are independent of the parts of the sampling problem over which there is no control. An example would be the heterogeneity of a material where it would be necessary to perform tests to quantify this. Another example would be the suitability of a sampling device on which it would be necessary to perform inspections to ascertain its correctness. A structural property in a system is the cause that gives rise to one or more consistent, permanent, guaranteed negative effects that cannot be controlled or changed. A structural property reliably delivers consistent sampling error or bias as an outcome that arises from the application of procedures or equipment that is thought to be correct, but is not. A poor sampling protocol, a faulty sampling device, an inappropriate analytical procedure, the faulty design of a storage silo, or stacker-reclaimer etc., are all structural properties that can be fixed once they are identified. Identifying structural properties that induce error and bias takes time and effort.

Bias: A bias is equal to the mean of the total sampling error when this mean is different from zero. A significant sampling bias is always introduced by incorrect sampling. Correct sampling is usually not biased to any significant degree. Correct sampling implies that IDE, IEE, IWE and IPE are negligible.

Component: A component or ***constituent*** is the smallest elementary and autonomous part that can be separated and quantified by analysis. In particulate materials these are fragments, in liquids and gasses they are ions and molecules[1]. Components may be chemical or physical such as the silica content of an alumina shipment, or the proportion of the +63 micron particles in cement.[57]

Critical component: A chemical or physical *component of interest*, the proportion of which is relevant and must be estimated.

Content: Since it is always much easier to deal with dimensionless and relative values, the word “content” shall be used for the *proportion* of any given component - a_L should be expressed as a proportion of 1. For example, the 2% +63 micron particles in cement should be written 0.02 and 100ppm silica in pure alumina should be written 0.0001.

Critical content: Refers to the *content of the critical component* or component of interest.

Continuous selection model: Sampling is a selection process and continuity is a mathematical concept. Matter is essentially discontinuous although assumed continuous. Therefore, when studying a lot using the continuous model all discontinuities within the lot are voluntarily disregarded, except those properties at any given point of the entire lot.

Discrete model: Identifies a lot as a discrete set of units, such as individual particles or groups of particles. As far as sampling is concerned a lot is completely defined by the finite set of all particles or groups of particles characterised by their content and weight.

Estimate: The result obtained from the analysis by a laboratory of a sub-sample, for a given constituent of interest, is an estimate of the *true unknown* content of the original sample submitted. Assay results are always estimates of a true but unknown content. These results are always affected by various errors as there exists no exact measurements or analyses.

Estimator: The true unknown content of a sample is an *estimator* of the true unknown content of a corresponding lot. An estimate can be selected for representing the true unknown content of a sample and the true unknown content of a lot. It can also be selected for representing the true unknown content of a specimen, but not the true unknown content of a corresponding lot. Estimate and estimator of a specimen cannot be representative of the true unknown content of a corresponding lot.

Lot: Refers to a batch of raw material, the composition of which is estimated. Examples include a 50 000-ton ore stockpile, 3m HQ diamond core, an entire ore block, underground face sample or a laboratory sub-sample, etc. Gy[1]says the lot must be considered as a set of units, where the set can be either a population of non-ordered units e.g. a stationary stockpile, or a series of ordered units, e.g. a conveyor belt carrying units that are ordered chronologically and spatially. In addition the units can either be a single solid fragment, or it can be a group of fragments, i.e. a sample increment.

Increment: An increment is a group of neighbouring particles or fragments, or a certain amount of liquid or gas with suspended solids, extracted from a lot in a single operation of the sampling tool or device. Relevant numbers of increments taken from a lot in a systematic manner are composited to provide a sample.

Pastes: Pastes are mixtures of solids and liquids that are not easily separated i.e. filter cakes and sludge. Colloidal suspensions have similar handling properties. Problems in handling pastes include adhesion to and friction with surfaces of equipment, cracking of any paste into lumps and difficulty with pumping and conveying.

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[1] Gy, P. 2004. Sampling of discrete materials – a new introduction to the theory of sampling I. Qualitative approach. In: (Eds. K. H. Esbensen and P. Minkkinen) Special Issue: 50 years of Pierre Gy's Theory of Sampling. Proceedings: First World Conference on Sampling and Blending (WCSB1). Chemometrics and Intelligent Laboratory Systems, Vol. 74, Issue 1, 28 November 2004: 7-24.

Powders and fine solids: Powders and fine solids comprise materials in a range of 10 microns to 1 000 microns. Handling becomes more difficult with increasing moisture content, range of particle size distribution and range of density distribution, all of which induce segregation phenomena through separation of the finer material.

Sample: A sample is a part of a lot often obtained by the compositing of several increments or fractions of the lot and meant to represent it in further operations. A sample by definition is not just any part of a lot; it must be a representative portion of the lot. Its extraction must respect rules that sampling theory attempts to establish. Its extraction must be equiprobabilistic, which is every fragment in the lot must have the same chance as every other particle of being included in the sample. By definition samples are reliable representatives of the lot, they are correct.

Sampling protocol: A sampling protocol describes the steps involved in taking a correct sample, one that is representative of a lot and will involve one or more of the following steps:

Starting lot	**L**	
	↓	
Primary sample	**S1**	Primary rejects
	↓	
Primary preparation	**S'1**	
	↓	
Secondary sample	**S2**	Secondary rejects
	↓	
Secondary preparation	**S'2**	
	↓	
Laboratory sample	**S3**	Tertiary rejects
	↓	
Tertiary preparation	**S'3**	
	↓	
Analytical sub-sample	**S4**	Final rejects
	↓	
Analysis		Final result

The protocol describes nothing about the equipment used; assuming only that it is correctly designed and hence "perfect".

Selection: Selection describes either the sampling process or it may mean the increment selected from the lot; it can be either probabilistic or non-probabilistic. Non-probabilistic selection means not all fragments had an equal chance of being selected, while probabilistic means all constituents had an equal chance of being selected. Probabilistic selection is *correct* when all fragments have an equal chance of being selected during the sampling process and the integrity of the selected fragments is respected. A 'probabilistic selection' is said to be *incorrect* if the chance of a fragment being selected is a function of some other property such as size, density or shape.[1]

Slurries: Slurries can be divided into two categories:

i. Non-settling slurries or suspensions in which the solid particles are smaller than 20 microns: Random dispersion of solid particles in a liquid that can be maintained without inducing turbulence or mechanical agitation, and

ii. ***Settling slurries in which the solid particles are larger than 20 microns:*** Continuous mechanical agitation or the presence of turbulence is necessary in order to maintain a state of random dispersion. In laminar flow, and especially in horizontal ducts, large distribution heterogeneity by size, fraction and density layers can take place leading to difficult segregation problems during sampling and processing.

Specimen: Part of a lot obtained without respect to the rules of theory of sampling, not based on the notion of equiprobabilistic selection. A specimen should *never* be used for representing a lot and should be labeled as such. Its purpose should only be qualitative.

3 INTRODUCTION

For any given set of measurements the spread of values around the mean is known as the variance. Values that are more or less equal have very little variability around the mean, whilst highly variable data will have large variances. The true, unknown variance of a population is referred to as σ^2, but all we have access to is s^2, the statistical estimator of σ^2, referred to as the 'estimated population variance' that is calculated from a limited number of values drawn from the population. There is a clear difference between σ^2, the error variance of the whole population, and s^2, the error variance that refers to a local pile of broken ore. The estimated population variance s^2 is given by the formula:

$$s^2 = \frac{1}{n-1}\left(\sum_{i=1}^{n} g_i^2 - n\bar{g}^2\right)$$

The variances s^2 of analytical sample values are additive and reflect the uncertainties and errors associated with primary and secondary sampling and analytical procedures. Errors could be introduced at any stage of the sampling and sub-sampling procedure and may be random with a mean of zero, random with a non-zero mean or accidental[2]. Each of these sources of error contributes to total variance through the variances acquired during each step in the process:

s^2total = s^2sampling + s^2subsampling + s^2analytical

In order to resolve these errors the variances for each step can be identified and calculated either by direct analysis or by difference. Analytical variance can be found in repetitive analyses of aliquots with known characteristics. Primary sampling variances can be estimated through evaluation of the sampling process. The difference between the analytical and primary variances is due to the heterogeneity of a lot. It should be noted that the relative variance and the precision are calculated from the descriptive statistics of the data whereas the bias, reproducibility and the representativeness are calculated from the descriptive statistics of the sampling error.

3.1 Global Estimation Error (GEE)

The following expressions of error components are provided by Gy[3]. Global Estimation Error (GEE): this is the relative difference between the analytical result a_R and the actual, well-defined but unknown, value of a_L:

$$GEE = \frac{(a_R - a_L)}{a_L}$$

[2] Neufeld, C. T. 2005. Guide to Sampling. Centre for Computational Geostatistics (CCG) Guidebook Series, Volume 2. Published by Centre for Computational Geostatistics, 3-133 Markin/CNRL Natural Resources Engineering Facility, Edmonton, AB, Canada T6G 2W2. http://www.uofaweb.ualberta.ca/ccg/

[3] Gy, P. 2004. Sampling of discrete materials – a new introduction to the theory of sampling I. Qualitative approach. In: (Eds. K. H. Esbensen and P. Minkkinen) Special Issue: 50 years of Pierre Gy's Theory of Sampling. Proceedings: First World Conference on Sampling and Blending (WCSB1). Chemometrics and Intelligent Laboratory Systems, Vol. 74, Issue 1, 28 November 2004: 7-24.

Global Estimation Error (GEE) = Total Sampling Error (TSE) + Total Analytical Error (TAE)

Total Sampling Error (TSE) = $TSE = \frac{(a_S - a_L)}{a_L}$

Total Analytical Error (TAE) = $TAE = \frac{(a_R - a_S)}{a_L}$

Total Sampling Error is a combination of the errors associated with the primary sampling event (PSE) and all subsequent errors associated with sub-sampling events (SSE), irrespective of how many there may be, including the selection of the final analytical aliquot (TAE), hence:

GEE = PSE + SSE + TAE

Because these components of error are independent of each other we have:

The Additivity of Biases: m(GEE) = m(PSE) + m(SSE) + m(TAE)

The Additivity of Variances: σ^2(GEE) = σ^2 (PSE) + σ^2 (SSE) + σ^2 (TAE)

Generally sampling errors associated with the primary and secondary sampling stages have been found to be very much larger that the analytical errors, with relative primary biases being up to 1000%, secondary biases being 50%, but analytical biases being only about 1%[3]. Until recently there has been an unintentional disconnect between the realms in which sampling events occur and the realm of analytical procedures that provide a result for the sampler. Postgraduate courses in Quality Assurance and Quality Control (QA/QC) provided by Scott Long of AMEC are helping to build a bridge between these disciplines. Chemical analysts are beginning to ask penetrating questions about the representativeness of the samples they are required to analyse because the outcomes impact heavily on internal laboratory QA/QC that is a reflection of the integrity and quality of the laboratory. The linkages between what is happening in the field during the primary and secondary sampling events is deepening and strengthening because of the beneficial commercial implications of integrated sampling and analytical processes.

3.2 Disaggregating the Total Sampling Error (TSE)

Gy[3] has shown that it is possible to disaggregate the TSE into the following two additional components, namely the Correct Sampling Error (CSE) and the Incorrect Sampling Error (ISE), such that TSE = CSE + ISE and because these are independent of each other it is possible to define biases and variances as follows:

m(TSE) = m(CSE) + m(ISE) and σ^2(TSE) = σ^2 (CSE) + σ^2 (ISE)

The Correct Sampling Error (CSE) is said to be a ‘structural property’ related to the structure of the material being sampled and is inevitable because it is a function of the Constitutional and Distributional heterogeneity’s.

The Incorrect Sampling Error (ISE) is said to be a 'circumstantial property' that arises because the principles of correct sampling (uniform selecting probability, P) are not respected or upheld during the sampling process.

3.3 Disaggregating the Correct Sampling Error (CSE)

It is necessary here to refer to the dimensions of sampling and distinguish the zero-dimension and one-dimension sampling models.

Zero-dimension model: In strict mathematical terms a zero-dimension sampling event requires that two conditions be met, namely that the principle of correct sampling, (all fragments have the same probability of being selected in the sampling process), be upheld, and secondly, that there is no restriction on accessing any increment in the lot, i.e. each increment is presented to the sampling event independently of the preceding or following increments. If these two conditions are met then the only components comprising CSE are the Fundamental Sampling Error (FSE) and the Grouping and Segregation Error (GSE), and we write:

CSE = FSE + GSE.

One-dimension model: At the scale of increments the zero-dimension model applies, but Gy identified another error specific to the one-dimensional lot (particulate material on a conveyor belt), that he termed the Point Selection Error (PSE). Cross-belt or cross-stream samples are point increments taken from a flowing stream of material, weighed, prepared and assayed. These estimates replace the integral of the grade function, which is the proportional to the true grad of the lot a_L and this replacement is what Gy defines as the PSE and we can write:

CSE = PSE + FSE + GSE

3.4 Disaggregating the Incorrect Sampling Error (ISE)

The sampling operation is broken up into three independent steps, each incurring their own error:

Increment Delimitation Error: This is the difference between the correctly defined size, shape, geometry and morphology of the increment that should be extracted, and the actually defined of the size, shape, geometry and morphology of the increment. If the cutter geometry and cutter velocity through the stream meet specific conditions IDE is zero.

Increment Extraction Error: This is the difference between the intended size, shape, geometry and morphology of the increment to be extracted and the actual size, shape, geometry and morphology of the increment extracted because of the mechanical interaction of the sampling device and the material being sampled. If the width of the cutter (W) is three times the coarsest fragment in the stream, and the cutter velocity through the stream does not exceed 0.45cm/sec, then the model has a good chance of being the actual physical increment.

Increment Preparation Error: This error is introduced during recover and handling of the increment during subsequent procedures, e.g. transport, drying, crushing, grinding etc. This is usually the sum of six components, namely, contamination of the sample, loss of material, alteration of chemical and physical composition, negligence in handling the sample, or deliberate, non-random interference with the sample.

Hence in general terms, ISE = IDE + IEE + IPE, and we can summarise by writing:

TSE = CSE + ISE = PSE + (FSE + GSE) + IDE + IEE + IPE

3.5 Statistics of the Data

3.5.1 Accuracy and Precision

The calculated statistics of data sets are no better than the quality of the parent input data with embedded errors and bias due to incorrect processes and procedures while gathering the data. A specific decision indicated by correct statistical analysis may be incorrect because the parent data are flawed. High levels of analytical precision in the assay laboratory supported by technically advanced instrumentation, is not matched by high levels of accuracy. Only if bias is eliminated by correct sampling procedures before the sample arrives at the laboratory can acceptable levels of accuracy be achieved.

Firstly correct raw material characterisation ensures an optimised sampling protocol that minimises the inherent variability of the ore. Secondly correctly implementation of the protocol ensures that bias at each sampling stage of the in the process is eliminated or at least minimised.

3.5.2 Accuracy

In the context of the Theory of Sampling accuracy is a measure of how close sample measurements are to the true unknown value of the lot. Repeated sampling is compared to bullet holes in a target (Figure 4.1). Accuracy describes the closeness of the holes to the bull's-eye at the centre of the target. Bullet holes closer to the bull's-eye are considered more accurate than those farther away. The closer the average of the results is to the true value of the lot, the more accurate the samples are considered to be. An estimate may be accurate or not depending on the numerous factors which affect the manner in which measurements are made. However, the resultant estimate, which is an average of all taken measurements, is only ever an estimate of the true value. It is impossible to define true value because measurements deviate either randomly or systematically and these deviations are called errors.

The relative distribution of the bullet holes around the bull's-eye should also be taken into consideration. Widely scattered holes may have an average exactly equal to the same number of tightly clustered holes, but the measure of accuracy is much more reliable if the precision is good (Long, 2007). Accuracy is a measure of the degree to which bias is absent. Better accuracy means less bias. However, even accurate sampling techniques and processing and measuring methods yield results which, while approaching the true value of their average, are scattered over a relatively wide range.

With regard to accuracy the difference between the mean of the samples and a reference material is referred to as the bias (Figure 4.1). Accuracy is a measure of the degree to which bias is absent so better

accuracy means less bias. In order to correctly calibrate an instrument it is essential to determine and correct for the bias. However, even accurate sampling techniques and processing and measuring methods yield results which, while approaching the true value of their average, are scattered over a relatively wide range. Delimitation, extraction and preparation of the sample are the principle sources of bias in the sampling protocol and everything possible should be done to eliminate these biases.

3.5.3 Precision

Precision is a measure of how closely repeated experimental values agree, and is expressed in terms of the standard deviation (σ) of the experimental results. Consequently, a good sampling and analysis process has high accuracy (bias is small) as well as good precision (the results of repeated experiments fall within an acceptable range). The size of the cluster of bullet holes is a measure of the precision and corresponds to the concept of reproducibility. Tightly grouped bullet holes are referred to as precise since they all hit close to the same spot; this may not necessarily be near the bull's-eye.

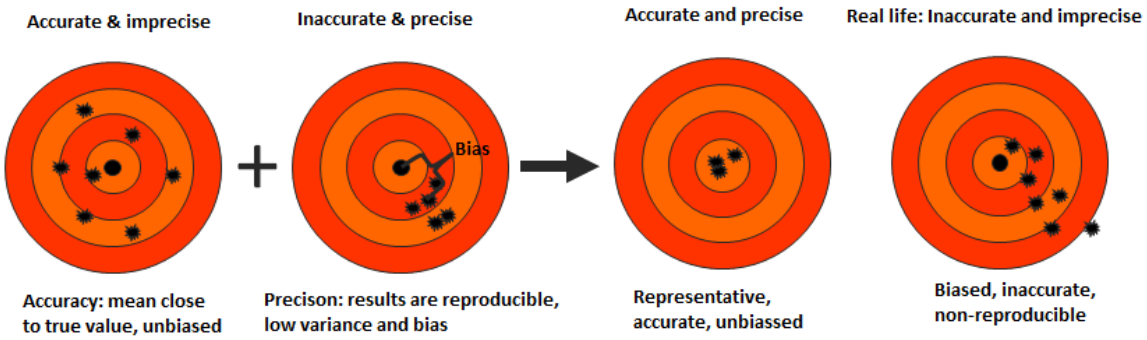

Figure 3.1: Accuracy, bias, precision and representativeness

The measurements are precise, though not necessarily accurate. Sample values from a 10g/t ore body that consistently return 9.5g/t gold, may be said to be precise, but the measurement is inaccurate. However if the values range from 4 to 16g/t they could return an average value close to the true value of the lot, but the precision would be poor. Sample values that are all close to and tightly clustered around a known value would be both accurate and precise. Repeated measurements of a known reference material or standard should be both accurate and precise.

Provided there are sufficient samples and provided they are drawn from a Normal distribution, the associated errors will also be normally distributed. Typically the errors then can also be represented by a normal distribution meaning that at one standard deviation the area under the curve is 68.3% of the measurements. This means that a typical sample value will be equal to the mean plus one standard deviation 68.3% of the time, it will be equal to the mean plus two standard deviations 95.4% of the time and it will be equal to the mean plus three standard deviations 99.7% of the time.

Precision describes the 'reproducibility' of estimates and generally the person doing the investigation, defines a standard of precision at some appropriate level. Geologists sampling Cu may use ±10%, while others sampling Au may use ±15% (if they do this well it will be a miracle!). Metallurgists at a floatation plant should use ±5% or better for metallurgical accounting whilst the sales department should aim for ±1% (the

financial consequences of very small differences must be taken into account). Ultimately precision depends on the DQO set for the operation. In nomograms constructed for the purpose of estimating the change in precision with changes in sample mass and the degree of comminution, precision is set at 10% (equivalent to the 'Safety Line' in a nomogram) and means that the variance of the selection error σ^2(SE) or the relative variance, is not greater than 0.01. **Note:** Variance (σ^2) rather than precision, is often used to describe how closely experimental values agree, because variances for individual sampling stages are additive and provide an estimate of the overall variance. This simplifies the equations and arithmetic considerably. According to Deardorff[4] (2000) precision is often reported quantitatively by using relative or fractional uncertainty. For example, the gold content, *g* = 14.5 ± 0.5 g/t has a relative uncertainty of

$$\text{Relative uncertainty} = \left|\frac{\text{Uncertainty}}{\text{Measured quantity}}\right|$$
$$= \left|\frac{0.5}{14.5}\right|$$
$$\text{RU} = 0.0345 = 3.45\%$$

Furthermore he suggests that accuracy is often reported quantitatively by using relative error. For example if the expected value for g is 17.0 g, then the relative error is:

$$\text{Relative error} = \frac{\text{Measured value} - \text{expected value}}{\text{expected value}}$$
$$= \frac{14.5 - 17.0}{17.0}$$
$$\text{RE} = -0.147 = -14.7\%$$

1.1.1 Relative Variance

Relative variance and relative standard deviation (precision) are used to compare different sets of analyses with one another on an equal basis and to compare the results of different sampling experiments. Relative variance is calculated as:

$$\text{Relative Variance} = \sigma^2 = \frac{\text{Variance}}{\text{Mean}^2}$$

Some analysts prefer to use the *percent* relative variance obtained by multiplying the relative variance value by 100.

Adding the variances and then taking the square root is the correct way to combine errors; it is incorrect to simply add standard deviations together. The RSD for the protocol shown in ***Table 4.1*** is 30.4%, calculated by adding the variances of each step and then taking the square root of the result. The table also shows the

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[4] Deardorff, D. 2000. Introduction to Measurements & Error Analysis. The University of North Carolina, Chapel Hill, Department of Physics and Astronomy. http://www.physics.unc.edu/~deardorf/uncertainty/UNCguide.html. Last revised: August 10, 2000 by Duane Deardorff, Director of Undergraduate Laboratories.
[4] International Organization for Standardization (ISO), ``*Guide to the expression of uncertainty in measurement''*, Geneva, Switzerland, 1993.
4 http://www.measurementuncertainty.org/mu/guide/index.html?content_frame=/mu/guide/uncertainty.html

FSE associated with the analytical procedure (fire assay) is only about 4% whereas that associated with the primary crushing process is 24%.

Table 3.1: Accumulating errors in a sampling protocol and the correct way of calculating the errors

Steps in the Protocol	RSD (%)	RSD (decimal)	Relative Variance
Primary Crush 1/4" (6.35mm)	24%	0.24	0.058
Split	11%	0.11	0.0121
Roll crush	1%	0.01	0.001
Split	12%	0.12	0.0144
5g of pulp	8%	0.08	0.0064
Fire assay	4%	0.04	0.0016
Totals	**60% (incorrect)**	**Do not add these values**	**0.0925**
		Relative standard deviation (decimal)	**0.304**
		Precision = (√0.0925) x 100 (√0.0925) x 100 =	**30.4%**

For the above example the precision, or RSD, is 30.4%.

$$\text{Precision} = (\text{Relative Std Deviation} \times 100) = \sqrt{\frac{\text{Variance}}{\text{Mean}^2}} \times 100 = \sqrt{0.0925} \times 100 = 30.4\%$$

Provided the variances are small, something less than 0.02, it is possible to use the approximation that precision, which is defined as the variance of the selection error, is 100 x RSD, and it is possible to calculate the relative variance from the Precision (RSD) as follows:

$$\text{Relative Standard Deviation} = \sigma_{\text{RSD}} = \frac{\text{Precision}}{100}$$

$$\text{Relative Variance} = \sigma_{\text{R}}^2 = \left(\frac{\text{Precision}}{100}\right)^2$$

3.5.4 Representative Sampling

The most important objective in the sampling process is to obtain a representative sample, something that can only be achieved through random sampling. A sample is therefore representative when it is at the same time both correct (control of bias) and reproducible (control of variance), both of which are functions of the sampling process and not the material itself[3]. Once the increment has been selected and extracted it is impossible to determine if it is representative. A random sample must be one unbiased. However easy it is to demonstrate that a bias exists, it is theoretically impossible to demonstrate its absence. Sampling is always biased to some extent, and therefore any claim that a sample is "non-significantly biased" means that the average of estimates for a given constituent content from many random samples taken in the same

lot will estimate the true, unknown content of the lot with a sufficient pre-selected accuracy. Taking a random sample provides a:

- Statistically accurate enough result, and
- Statistical estimate of the precision of the result.

Random sampling means that “each and every particle in a lot has an equal chance of appearing in the chosen sample”. The application of this principle is far more difficult to implement than it appears. In order to be representative the sample must also be reproducible i.e. it must have a low variance. Ideally a fully representative sample is one that can be expressed as follows:

Representative = Sample is accurate enough (correct according to DQO) + Sample is reproducible enough (precise, according to DQO).

Sampling, sample processing and analysis yield a result for a given characteristic (e.g. gold content) which is equal to the true (but unknown) value plus an experimental error that varies randomly each time the routine is carried out. Bias is the difference between the true and average values of a variety of experimental values **(*Figure 4.1*)**. It can be reduced through the elimination of poor sampling techniques, processing and measurement practices as well as incorrect sampling systems.

3.6 Statistics of the Sampling Error

3.6.1 Sampling Error (SE)

All sampling events are affected by errors, which could be either random, non-random variables, negligible or systematic. Without a clear understanding of the differences between these various categories of error, it is not possible to understand the complicated notion of heterogeneity and the capital notion of sampling correctness. Sampling plans such as those recommended by regulatory agencies or standards committees, often involve a simple analysis of the variance of values obtained from an experiment without taking into consideration the many components responsible for this resulting variance. It is essential to know connection between each component of error and its source. Some errors affect precision, while others affect both accuracy and precision. Many analyses of sampling data highlight the resulting overall error, without any attempt to analyse the components of this error and their respective sources. Since variances are additive, the overall variance bears very little resemblance to its components; only the theory of sampling disaggregates these variances providing the information necessary to minimise the impact of systematic errors on the total error. Systematic biases, another component of the overall errors, can only be identified using statistical approaches.

According to Gy (1996[5]) the sampling error variance for any stage of sampling is given by:

$$SE = \sigma_{SE}^2 = \frac{a_s - a_L}{a_L}$$

Errors are random variables and are independent in probability which means that the total sampling error is a linear combination of all the different sources of error. Because sampling and analytical errors (AE's) are additive the total sampling error is given by:

$$T_{SE} = NE + FE + GSE + DE + EE + ... + AE$$

The average error in this case is:

$$Ave[T_{SE}] = Ave[NE] + Ave[FE] + Ave[GSE] + Ave[DE] + Ave[EE] + ... + Ave[AE]$$

The Total Error Variance is:

$$\sigma_{T_{SE}}^2 = \sigma_{NE}^2 + \sigma_{FE}^2 + \sigma_{GSE}^2 + \sigma_{DE}^2 + ... + \sigma_{\text{Analytical error}}^2$$

None of the individual sampling errors are compensating; all of them are additive and give rise to large biases unless care and attention is paid to sampling events. Accuracy and precision are two measures of sample quality. As the mean of the sampling errors approaches zero $m(SE) \Rightarrow 0$ the samples can be said to be accurate. This means sampling errors, which are sometimes positive and other times negative, will balance out and the estimate will be unbiased. Practically speaking, in order to achieve accuracy the modulus of the mean of sampling errors must be less than a predetermined per cent relative bias or standard mean of errors required for a given purpose: $|m(SE)| \leq m_0$. When the variance of the sampling errors becomes very small, again less than a standard required for a given purpose, the sample can be said to be precise, irrespective of the difference between the sample average and the grade of the lot. However it should be noted that better precision suggests that an accurate result is more believable. Accuracy, represented by the square of the mean sampling error and precision, represented by the variance of the sampling error, can be combined into a single statistic that leads to the notion of representativeness[6]. Therefore:

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[5] Gy, P. 1998. Sampling for Analytical Purposes. The Paris School of Physics and Chemistry, Translated by A.G. Royle. John Wiley and Sons Ltd, New York.153p.

[6] Neufeld, C. T. 2005. Guide to Sampling. Guidebook Series Volume 2, Centre for Computational Geostatistics (CCG), 3-133 Markin/CNRL Natural Resources Engineering Facility, University of Alberta, Edmonton, AB, Canada. T6G 2W2. p. 30. Website at http://www.uofaweb.ualberta.ca/ccg/

Additivity of Means (biases) gives rise to $m^2(SE) = m^2(SE_1) + m^2(SE_2) + m^2(AE)$

Additivity of Variances gives rise to $s^2(SE) = s^2(SE_1) + s^2(SE_2) + s^2(AE)$

Representativeness = $r^2(SE) = m^2(SE) + s^2(SE)$

Generally sampling variances can be considerably greater than analytical variances. The equation for the sampling error (SE) implies that the mean of sampling errors can only be calculated if there is a value accepted as a known true value (which is impossible to know). However, for the sake of this exercise assume that the known true value of copper is 1.45%CuO and two labs return the copper analyses listed in ***Table 4.3.*** The sampling errors of each analysis from the two labs must be listed in columns 3 and 4. From these it is possible to calculate the mean and variance of the sampling errors.

3.6.2 Bias

Bias is the mean of the sampling errors; it is the average sampling error variance and provides an indication of how far the sample average is form the true value. $\text{Bias} = m^2(SE)$

3.6.3 Reproducibility

The concept of precision (Wikipedia, 2007) may be refined using two further terms:

Repeatability – over short time periods: the variation arising when all efforts are made to keep conditions constant by using the same instrument and operator, and;

Reproducibility – over longer time periods: the variation arising using the same measurement process among different instruments and operators.

Good reproducibility means there is a low dispersion of sample values about the mean i.e. the variance of the sampling errors (SE) is less than the total variance, where s^2_0 is some predetermined value: $s^2(SE) \leq s_0^2$.

It is incumbent upon the sampler to define the acceptable RSD. (~10% is the usual limit with anything below being acceptable). 40 samples or more would be needed to achieve this.

3.6.4 Representativeness (r)

The combination of accuracy and reproducibility is the sum of the sampling error $s^2(SE)$ and the squared mean of error $m^2(SE)$: $r^2(SE) = m^2(SE) + s^2(SE) \leq s_0^2$. This is a reflection of the dispersion around the centre of the target.

Practical representativeness: Errors of representativeness are less than the errors of variance plus the mean of squared errors: $r^2(SE) \leq m_0^2 + s_0^2$.

Example: Using the analytical results shown for two laboratories given in Table 4.2 calculate the Relative variance, the precision, the bias and the representativeness for the two laboratories. Which laboratory would you choose?

A representative sample must be a microcosm of the block of ore to be broken if the true grade is to be found. In actual fact the sample should have the same grade as the block. It may well be asked, “What are the characteristics of a representative sample?” Remember that any analytical result is a consequence of successive sampling events. Errors associated with each event accumulate in the sampling process. The sampler himself must quantify and eliminate these errors as far as possible during the sampling process. In this text the errors are referred to as σ2 with a subscript that explains the type of error being referred to.

Truly representative samples must be taken in accordance with a sampling protocol that ensures the sample is representative (the physical sampling methods must minimise the bias) and consistent (the theoretical variation between samples must be minimised). The bias, variance and representativeness are shown in ***Table 4.3.*** You must be able to reproduce these results. Remember a_L = 1.45 %Cu and that you use the population variance because the lot is not sampled to extinction.

Table 3.2: % CuO analyses from two labs (true value = 1.45 %CuO)

	Analyses		Sampling Errors	
	1	2	3	4
Sample	LAB 1	LAB 2	LAB 1	LAB 2
1	1.46	1.33		
2	1.43	1.33		
3	1.4	1.33		
4	1.48	1.34		
5	1.46	1.34		
6	1.49	1.34		
7	1.48	1.34		
8	1.45	1.34		
9	1.46	1.34		
10	1.47	1.34		
11	1.44	1.34		
12	1.43	1.34		
13	1.47	1.34		
14	1.44	1.34		
15	1.45	1.35		
16	1.46	1.35		
17	1.48	1.35		
18	1.49	1.35		
19	1.41	1.36		
20	1.43	1.36		
21	1.46	1.37		
22	1.45	1.38		
23	1.46	1.39		
24	1.43	1.45		
25	1.45	1.45		
26	1.41	1.46		
27	1.42	1.47		
28	1.49	1.55		
29	1.45	1.6		
30	1.43	1.75		
Mean				
Variance				

Table 3.3: Relative variance, precision, bias, reproducibility, and representativeness for the analytical results shown in Table 3.2

	Analyses		Sampling Errors	
Sample	LAB 1	LAB 2	LAB 1	LAB 2
Mean	1.451	1.391		
Variance	0.001	0.009		
Rel Var	0.0003	0.0047		
Precision	2%	7%		
Bias			0.021	-1.228
Reproducibility			0.000	0.004
Representativeness			0.021	-1.223
Representivity			2%	-122%

8. If an acceptable mean of errors (relative bias) is 5%, Lab 1 at 2% is within the practical accuracy limit, Lab 2 at -122% is outside an acceptable range. Consequently, with regard to the practical limit of accuracy, bias and representativeness, Lab 1 is preferred above Lab 2.

3.7 Correct Sampling

With regard to sampling, *sample correctness* is the structural guarantee of achieving sufficient accuracy. *"Randomness in the selection of original increments is indeed the number one problem in the sampling of bulk material"*. The principles governing correct sampling are that:

i. ***Every part of a lot has an equal chance of being sampled***
ii. ***The integrity of the sample is preserved during and after the activity***

It is important to:

i. ***Define the sample taken (Increment Delimitation Error)***
ii. ***Physically obtain the sample that has been correctly defined (Increment Extraction Error)***

If these two requirements are met the integrity of the sample is preserved during and after its collection. Unbiasedness and correctness are not equal. In fact they are two totally different concepts. If a sample is biased it cannot have been taken correctly but may still be accurate enough. If a sample is taken several times and the average of the sampling error is not zero, then the sample is considered biased.

Bias is a characteristic of the mean because it is only spoken of in a distribution. It is a property of the distribution of errors. Correctness can be controlled and is therefore a tool for ensuring unbiasedness. There is no way of identifying, detecting or verifying unbiasedness. If a sample is correct then it will be unbiased. If not, then it will almost certainly be biased. However, there is no sure way of knowing. If the sampling error is systematic on one side this will give rise to a bias. Any sample that does not correctly represent the granularity of the lot is incorrect, yet may still be accurate enough.

Correctness is not to be confused with representativeness. A sample is representative when it is both accurate (unbiased) and reproducible (precise). Once a sample is taken it is too late to determine if it is not incorrect nor whether it is a good or bad sample. Correctness must first be attained which will in turn take care of unbiasedness. Samples for ore body delineation; metallurgical assessment, process control and quality control are generally taken from a range of locations including blast holes, relevant feed product streams and ship loading at the port. The collected samples are used for determining chemical composition and moisture content as well as determining a range of physical characteristics such as particle size distribution.

1.1.2 Examples of good practice

The following examples will ensure good sampling practice:

i. Mix the material before sampling;
ii. Take several increments and composite them to form the sample;
iii. Crush, grind or pulverise the material before sampling, and;
iv. Sample frequently enough to identify process cycles

Rather than trying to remember rules, establish a *protocol* which will ensure that the sample is representative. Three criteria must be met in order to achieve this. A sampling protocol would need:

i. A clear definition of DQO
ii. An understanding of the component parts of the sampling problem
iii. Some basic principles that can be applied to any sampling situation

Pierre Gy identified and explained the causes of variation and showed that the major contributors to errors can be reduced. His theory addresses all the aspects around the sampling of particulate materials, including:

i. Sampling variation and bias
ii. A method for determining the required mass of a sample
iii. Process variation and its relation to sampling
iv. Physical sampling and the sampling procedure
v. The principle of correct sampling

3.8 Sampling Dimensions of the Lot

The shape, physical geometry, and mobility of a lot, stationary or moving, present the greatest challenge to the extraction of representative samples. Objects in three-dimensional space are easily represented in models having fewer dimensions. For example a three-dimensional cube could represent a building, a two-dimensional plane could represent a soccer field, and a one-dimensional line could represent a conveyor belt, and a cluster of balloons could represent a zero-dimensional group. The spatial arrangement of sampling opportunities often presents themselves in ways that can be interpreted as zero-, one- two- or three-dimensional arrays. The sampling dimensions of the lot are defined by the way a sampling instrument moves through or across the lot and because the chances of taking a correct sample are improved by reducing the number of sampling dimensions where possible, any sampling procedure that reduces the number of dimensions also reduces the sampling error. Correct sampling of three-dimensional lots such as a stockpile is not practical so sampling across one or two dimensions is the best solution. For example a stockpile can be drilled, or it may be spread out on a surface in two dimensions and sampled. The correct sample should be defined by using the correct geometry for the sampling dimension; a cylinder for two-dimensional sampling, and a slice for one-dimensional sampling. Furthermore correctly using the right sampling equipment will ensure extraction of the sample as correctly defined. The ideal sampling arrangement is the extraction of an increment from a falling stream of material using a cross-stream sampler.

3.8.1 Zero-dimensional sampling

Consider 27 individual blocks as shown in Figure 3.2a, each representing a single sample, all well-defined and discrete units, each being as easily accessible as the next so every block has an equal chance of being sampled. Zero-dimensional sampling (3.2a) occurs where there is no restriction or constraint on the access

to any increment of the lot; all lots are equally accessible and all can be extracted without bias. There is no difficulty in extracting a block and there is no problem with handling the sampling. Such sampling situations are usually presented as theoretical constructs to complete the concept of the dimensions of sampling.

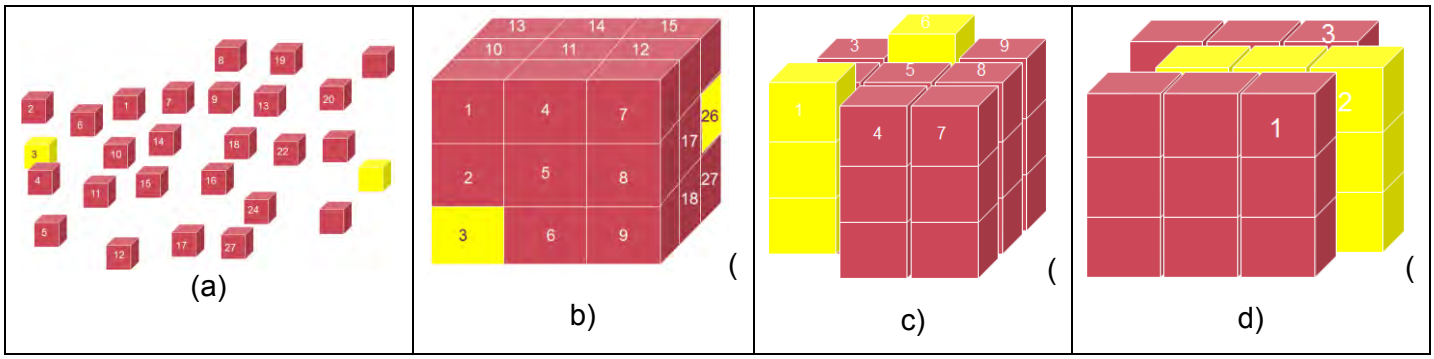


Figure 3.2: a) Zero dimensions, b) 3_Dimensions, c) 2_Dimensions, and d) 1_Dimensional lots

3.8.2 Three-dimensional sampling

Consider 27 blocks arranged in a 3 x 3 x 3 array (Figure 3.2b). If our smallest sampling unit is a single block that we could extract from the array. There are two blocks that are completely inaccessible to any sampling activity, one in the centre of this array and the block immediately beneath it. The principle of correct sampling, which requires that all blocks be equally accessible, cannot be followed in three-dimensional situations as shown in Figure 3.3, and should be avoided where possible. The only sure way of obtaining a representative sample is to reclaim the stockpile and sample across a stream of the conveyor belt as the pile is being rebuilt.

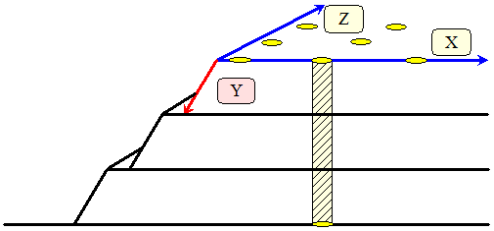
Figure 3.3: Three dimensional sampling on a stockpile means that the principle of correct sampling is violated (Source: (a) http:// yosemite.epa.gov/R10/CLEANUP.NSF, (b) http://www.labradorironmines.ca/images/124m.jpg&imgrefurl (c) http://epg.modot.org/files/2/26/1001.jpg)

A three-dimensional ore deposit can be thought of as an integrated stack of two-dimensional slices, each slice being one bench height thick as shown in Figure 3.2c. In such cases the ideal shape of observation is a cylinder which must penetrate the entire thickness of the bench. Provided the mineralisation is horizontal and parallel to the bench surface drill holes can be positioned so that they will intersect the deposit optimally. Rarely are mineral deposits perfectly horizontal so that the effectiveness of delimitation in two-

dimensions is reduced. Where the mineralisation is vertically dipping, effective channel samples can be collected across the floor of the pit; samples from vertical RC blast holes are meaningless.

3.8.3 Two-dimensional sampling

Consider 27 blocks in a 3 x 3 array of nine stacks, each stack consisting of three blocks each (Figure 3.2c). By numbering the stacks 1 to 9 in two dimensions we could take a random sample by choosing two stacks at random. By defining the sample in the vertical dimension we have artificially eliminated this dimension of the lot. So now we only have two dimensions and a cylinder, or a drill bit as shown in Figure 3.4, is the correct geometry for two-dimensional sampling. By reducing the number of sampling dimensions from three to two we can improve the chances of taking a good sample.



Source: Pitard, FF, 2009

Figure 3.4: Vertical drilling rods a) and b) mean the sample position is defined in two dimensions (x and y co-ordinates) and sample is collected across the third dimension, c) a three-dimensional deposit is an integrated stack of two-dimensional slices

3.8.4 One-dimensional sampling

Consider the 27 blocks in three slices of 9 blocks each and we choose one of the nine block slices at random for the sample (Figure 11.2d). By grouping the blocks in slices of nine-blocks each in two dimensions and numbering them in the other dimension we have one-dimensional sampling. By defining the sample as a slice across the line of nine-block slices we have eliminated two dimensions. Thus the correct geometry for one-dimensional sampling is a slice across the other two dimensions of the material as illustrated in the conveyor belts shown in Figure 3.5. So a sample or cut across a stream moving at constant speed is the most practical way to ensure the best sample.

Figure 3.5: Conveyor belts carrying particulate materials reduce the sampling dimensions of the lot to one dimension that can be easily cut by cross-stream or cross-belt samplers.

In summary the principle of correct sampling requires that

- Every part of the lot has the same chance as every other part of the lot of being sampled, and
- The integrity of the sample is preserved during and after sampling.

There are several things that can be done to reduce the chances of not taking a correct sample.

- Reduce the number of sampling dimensions if possible. Correct sampling of three-dimensional lots is not practical so try to sample across one or two dimensions where possible.
- Define the correct sample by using the correct geometry for the sampling dimension; a cylinder for two-dimensional sampling and a slice for one-dimensional sampling.
- Choose the right sampling tool. The tool must be capable of taking the sample as defined and the tool must be correctly used in collecting the sample
- Preserve the integrity of the sample.

3.9 Heterogeneity

The importance of a clear definition of heterogeneity is paramount and this has already been covered to some extent in the section dealing with large and small scale variability. **Homogeneity** can be defined as: the condition of a lot under which all elements of the lot are exactly (strictly) identical. At the level of a sample a well-mixed liquid or gas might be thought of as approaching homogeneity. **Heterogeneity** is defined as: the condition of a lot under which these elements are not strictly identical. It therefore follows that homogeneity is the zero of heterogeneity and is an inaccessible limit.

3.9.1 Understanding heterogeneity

There are several types of heterogeneities and care must always be taken to define exactly which type one is referring to.

3.9.2 Heterogeneity of a statistical population

In a first analysis it is possible to identify two types of heterogeneity when a lot is considered as a statistical population. The first definition is that of Constitution Heterogeneity (CH) which is responsible for FSE. The second definition is that of Distribution Heterogeneity (DH) which is responsible for GSE, as shown in ***Figure 4.1.***

3.9.3 Constitutional Heterogeneity

(CH) is the difference in grade internally between one point in a fragment and another. Compositional and constitutional heterogeneity are the same thing and it gives rise to the fundamental sampling error (σ^2_{FE}) and can be quantified. CH is due to differing particle size, shape, density chemical composition, grade and other physical properties; it is unchanged by mixing, but ***increased*** by crushing. Any action that increases the CH will also increase the variance. The FSE is directly proportional to the cubed size of the fragments and inversely proportional the mass of the sample, so **reducing the size of the particles** by crushing, and **increasing the mass of the sample** will reduce the variance. Reducing the fragment size, once you are below the liberation size of the mineral of interest, does not decrease the CH. Total CH is the sum of all compositional heterogeneities of individual fragments. It is the main challenge when trying to collect representative and consistent samples. The FSE is the difference between what we measure in the sample and what the true value of the lot is.

3.9.4 Distribution Heterogeneity

(DH) is best understood by considering the lot to comprise a composite of increments that are all strictly identical in composition, i.e. distributionally homogeneous. DH arises when all the increments are not strictly identical in composition. DH is gives rise to the segregation and grouping errors (σ^2_{GSE}) and can be quantified. How particles are distributed makes a difference to the sample we take and the result we get from assaying the sample. Ore fragments in the lot to be sampled segregate principally due to density, but shape and size characteristics of the material being sampled may also contribute. Once material is segregated the lot is no longer homogenous and particles no longer have the same probability of being selected during the sampling process. Mixing and blending the lot before sampling, i.e. break up the tendency for dense minerals to group together, helps to reduce DH, as does incremental sampling. Mixing and segregation are highly transient phenomena and this type of variability introduces a bias that varies in time and space. Particles that differ in size, density, and shape are susceptible to poor mixing and agitation may in fact increase segregation. The best way to overcome the problem is to take as many small increments from different parts of the lot as is feasible, and composite them as a single more representative sample. The smaller the individual increments we use to composite the larger sample, the closer we approach the theoretical best which is random sampling particle by particle. DH is markedly increased beyond the liberation size due to the fact that segregation becomes so much easier because the differences in density are maximised beyond liberation.

Together these forms of material variability determine how consistent and representative our sampling is. Even if we increase the mass of sample and reduce the particle size by crushing, residual heterogeneity will persist if fragments are segregated in the pile to be sampled. The sampling error is reduced to the

fundamental error if the bulk is homogenized by crushing and mixing before sampling (assuming that equipment operation errors have been eliminated). The relationship between CH_L and DH_L is as follows:

$$DH_L = CH_L \frac{1+\xi\gamma}{1+\gamma}$$

where $\xi =$ Segregation factor : Characterises the type of distribution of the constituents in the lot

$\gamma =$ Grouping factor : Characterises the size of the groups that become increments

3.9.5 Heterogeneity of a sequential population

In a second analysis other types of heterogeneity can be identified if the lot is considered a sequential population.

The total heterogeneity contribution **(h)** of a given fraction towards the corresponding lot is the sum of only three major components:

Short-range, *random* heterogeneity **(h_1)**
Long-range, *non-random* heterogeneity **(h_2)**
Periodic heterogeneity **(h_3).**

Each type of heterogeneity occurs at different points on a conveyor belt.

- **h_1** occurs between close-range samples on a belt, separated by a distance of 50 cm.
- **h_2** is due to long-range changes in material composition between samples taken every 60 minutes.
- **h_3** is a very long-range or large-scale cyclic phenomenon relating to the changes that occur in a plant every 48 hrs.

Therefore, the total heterogeneity contribution **$h = h_1 + h_2 + h_3$**. The variance of the total variability is called the variance of the continuous sampling selection error HE, $S^2_{HE} = S^2_{HE1} + S^2_{HE2} + S^2_{HE3}$

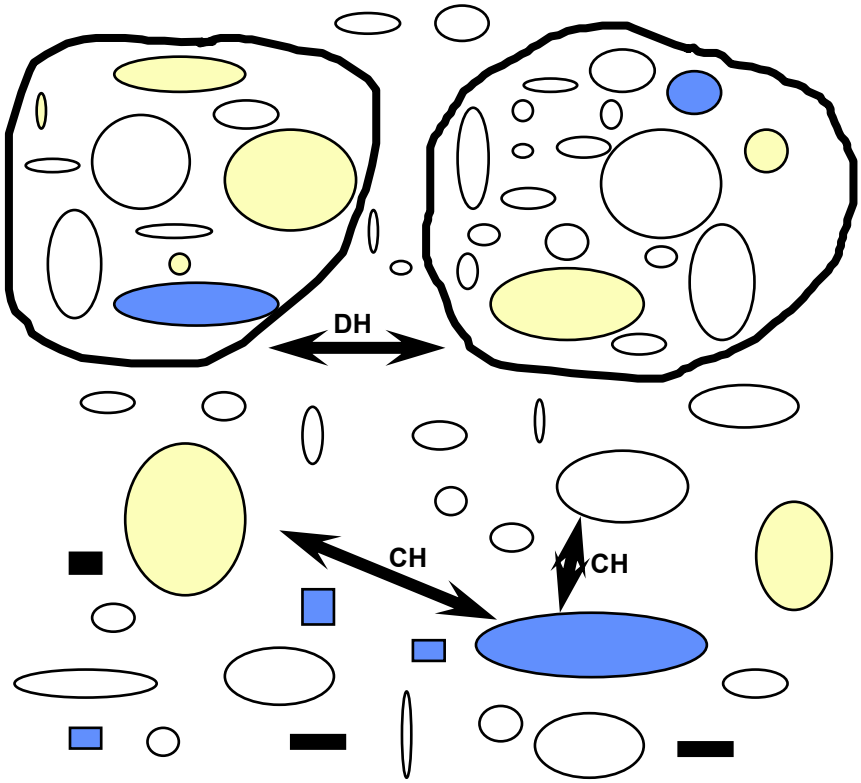


Figure34.2: Constitution heterogeneity (CH) is the heterogeneity between individual particles comprising the sample whereas distribution heterogeneity (DH) is the heterogeneity between groups of fragments in the sample. (Used by permission of FF Pitard, Sampling Theory and Methods, Short course presented at the University of the Witwatersrand 2006)

3.9.6 Heterogeneity Fluctuation Error (CE)

Process Integration Error (PIE): new terminology Each kind of heterogeneity is the source of a different type of error in the sample.

$\mathbf{h_1}$ is a fixed property of the stream that leads to the short-range heterogeneity fluctuation error, or Process Integration Error PIE_1**.** It is random and cannot be changed but can be mitigated by increasing the sample mass.

$\mathbf{h_2}$ leads to the long-range heterogeneity fluctuation error, or Process Integration Error PIE_2 **.** It is non-random but can be mitigated by increasing the sampling frequency.

$\mathbf{h_3}$ leads to the periodic heterogeneity fluctuation error, or Process Integration Error PIE_3**.** It is cyclic and can be hidden if the sampling interval is in phase with the cycle.

Although it is not possible to change $h_{1\text{-}3}$, choices can be made about the size of the fluctuation error in order to optimise the sampling protocol. The Fluctuation Error is cumulative such that:

$$\mathbf{PIE = PIE_1 + PIE_2 + PIE_3}$$

PART 1: BACKGROUND AND BASICS

4 BROKEN ORE SAMPLING AND THE THEORY OF SAMPLING

"There is no such thing as reliable feasibility studies, unbiased ore grade control, accurate environmental assessments or effective process control, if you cannot identify and minimize……sampling variability or errors". F. F. Pitard.

This book began in 2003 as a set of notes for students in the School of Mining Engineering doing the third year course in Technical Valuation of Mineral Deposits at the University of the Witwatersrand. The importance and interest in sampling expanded beyond the point of being a simple addendum to the existing notes on Statistical Evaluation and Geostatistical Methods of Mineral Evaluation. The tutorial exercises demonstrate the principles taught and allow students to explore the range of problems faced in this little appreciated and highly important subject. The notes were compiled principally from short-course material on the Theory of Sampling and Practice made available by Francis Pitard author of a shortened, digested, English version of the of 1988 volume by Pierre Gy[7] and Dominique François-Bongarçon. These experts, as well as Kim Esbensen and Geoff Lyman, have presented postgraduate short-courses through the Geostatistical Association of South Africa (GASA) in the GDE programme in the School of Mining Engineering on the Theory of and Practice of Sampling. The work of Patricia Smith (2001) has also been used at various places in these notes. Other sources are listed in the references. This book is by no means a complete statement of the issues and problems associated with the sampling of broken ores; instead it tries to provide a workable tool for practitioners and students as well as giving a broad introduction and overview of the Theory of Sampling.

4.1 The Purpose of Sampling

The purpose of sampling is to collect and analyse a small representative portion of a larger mass of material referred to as the lot. The true unknown value of each sample is an unbiased estimator of the true unknown value of the lot and the average of several analytical results is the estimated value ascribed to the lot. The ultimate aim of any sampling procedure is that the sample submitted for analysis is representative of the lot. In mathematical terms representativeness is the linear combination of the accuracy and the precision. Accuracy is the mean (squared) of the sampling errors, while precision is a function of the variance of the sampling errors. The important point is that any definition of representativeness must take both the accuracy and the precision into account. In order to reach for truly 'representative sampling' the multi-stage process from lot to analytical aliquot, involving one or more steps of grain size reduction and mass reduction, must be done in accordance with the principles specified in the Theory of Sampling (TOS). In fact the ***sampling process*** is the only way that representivity can be controlled and maintained.

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[7] Gy, P.M. 1988. *Heterogeneite, Echantillonnage, Homogeneisation*, Editions Masson, Paris, 1988.

However because we have analysed a sample and not the lot, an error is associated with the estimated value that we accept. The error is equivalent to a deviation from the true, but unknown value of the lot. Because we know neither the size nor the source of the error on any individual sample, neither do we know the true value of the lot. Values obtained from sampled material are the basis for nearly all decisions made about a mining project, from exploration to mine closure. Identifying and minimizing sampling variability is the most important issue in a sampling procedure. If sampling is improper, the assay value returned by the laboratory bears little resemblance to the actual value in the ground and the outline of the 'ore' contact on the plan will not have the slightest resemblance with reality (Figure 1.1). Geological modelling and geostatistical computation will be fruitless and financial losses arising from sampling errors may be larger than is generally realized. In practice, most mining operations have small budgets and under-equipped sample preparation laboratories.

Figure: 4.1: Defining the orebody on the basis of sample results will give a totally incorrect picture if the sampling is not correct

As has been shown by Robinson[8] [9] and De Korvin and Shipley [10] decisions based on sampling information have far-reaching financial implications involving millions of rand or dollars. The myths that sampling errors are self-compensating, that sampling precision is not that important an issue, that sampling issues are well known and can be easily solved using common sense, that analytical laboratories are accurate and precise, and that current industry practice is perfectly acceptable, should be exposed since nothing could be further from the truth! Actually, all these unfounded beliefs are a direct departure from basic professional due diligence; it is a very serious matter. Simply taking one sample means there is no method of identifying any associated error. The so-called duplicate analysis on samples undertaken by laboratories is a means of comparing values as a form of internal audit on the analytical procedures. Verifying an analytical result by duplicate or triplicate analysis is restricted to the last of the sampling procedures, namely the analytical and does not interrogate any of the preceding steps in the sampling event. This raises the important point that what is usually termed Quality Assurance and Quality Control (QA/QC) is a vitally important aspect of audits

[8] Robinson G.K. 2004 How much would blending a stockpile reduce variation? Chemometrics and Intelligent Laboratory Systems, 74: 121-133.
[9] Robinson G.K. 2005. Economic justifications for capital expenditure on sampling and blending. Second World Conference on Sampling and Blending. Australian Institute of Mining and Metallurgy Publication Series No 4/2005. 111-117.
[10] De Korvin, A. and Shipley, M.F. 2005. Sampling rules to achieve quality maximize benefit and minimize financial loss: Managerial Finance, Vol. 31, No 3: 1-18.

of laboratory practice and procedures. Unfortunately it is usually restricted to the analytical laboratory, but QA/QC should extend to all practices and procedures that precede this final step, namely sampling and sample preparation.

Pierre Gy's TOS is mathematically sound, conceptually correct and provides a practical template of the methods and processes for minimising the seven main sources of error, in any sampling result. It also provides the structural framework, a unified view on sampling processes and procedures. Whether using a dragline or a spatula, the same principles apply at different scales. Until the turn of the millennium however it had attracted a relatively few specialist adherents, but this number is growing as the concepts are understood and the importance of sampling is appreciated.

4.2 What is a Sample?

Sampling in the context of this manual deals with "the selection of individual observations (samples) intended to yield some knowledge about a population" (the lot) using methods of statistical inference. "Each observation measures one or more properties (mass, grade, metal content, etc.) of the observable entity" (i.e. the sample). Sampling is a selection process that may be probabilistic or non-probabilistic. Probabilistic or simple random sampling means each fragment in the lot (population) has the same probability as every other fragment of being included in the sample (increment). The sampling ratio n/N is the size of the sample (n) relative to the size of the lot (N). If for example a random sample consists of five fragments and there are 1000 fragments in the lot, the sampling ratio is 5/1000 = 0.005 or 0.5%. This means that each fragment in the population has a 0.5% chance of being selected in the sample. If any fragment has less than a 0.5% chance of being selected then the sample is not perfectly random and the selection process is biased. Results from probability theory and statistical theory are combined in the Theory of Sampling which is the only verified guide to practice'[11]. The word "sample" is defined[12] as 'a small part or quantity intended to show what the whole is like', with a strong emphasis on the idea of representativeness i.e. equiprobabilistic sampling. Many understand the process to be purely mechanical, without an appreciation that the sampling process is the selection of an increment or subsample that should carry all the essential characteristics of the lot. The difference between the sample and the lot is referred to as the sampling error; the art being to identify and minimize all such errors in the process. Ingamells and Pitard (1986) highlight the importance of the variability of results saying that it reflects characteristics of the parent lot including the degree of segregation, heterogeneity, effective size of the ore mineral, and nugget content. The best samples are representative of the whole, they should be unbiased meaning that the average grade of aliquots is the grade of the lot, and precise, meaning that the differences between estimates is less than a minimum acceptable threshold. A good example of representivity is the DNA signature of living organisms. Irrespective of the cell's position in the body, head or foot, its contained DNA molecule carries a signature for the whole organism in its strands. Thus the value of the sample depends on how well it represents the lot. Half a strand of DNA would not be representative, it could only be considered an incomplete specimen,

[11] Sampling (statistics). From Wikipedia, the free encyclopaedia. http://en.wikipedia.org/wiki/Sampling_%28statistics%29
[12] Complete Wordfinder. 1993. The Readers Digest Oxford Complete Wordfinder. Readers Digest. 1892p.

but because the DNA molecule is so small relative to any body part that might be sampled, the molecules are almost always intact.

The total sample error is a linear combination of all the errors that accumulate at each step of the sampling procedure. In his elegant approach Pierre Gy identified seven sampling errors, not all of which may be present in any sampling event. With time the number of errors has been expanded from Gy's original seven to nine or ten depending on one's interpretation of errors and the setting in which the samples are taken. However, whether you sample a sip of juice through a straw or collect a half tonne sample of coal from a conveyor belt, the nature of the sampling errors remain the same. Every step of the sampling procedure generates errors specific to a particular characteristic of the lot or specific to a sampling method and/or device. Appropriate action to eliminate or minimise such errors occurs after their identification.

4.3 Difference between samples and measurements

Usage of the word 'sample' is deeply embedded in the literature and has come to mean a small and representative fraction of a bigger lot for which we want to know the content of a material of interest. Practitioners use the word sample to mean a 50cm length of split core or a bag of broken fragments collected from conveyor belt or a pile of cuttings around a reverse circulation drill hole. To many the condition of the material prior to the time of extraction has no relevance, so there is little difference between a pile of broken ore and unbroken, in-situ material in the mining face. According to Francois-Bongarcon (2013), the distinction between a 'sample' as used in the Theory of Sampling, is not to be confused with a 'measurement' which may be classified as nominal (categorical), ordinal (rank) or scale (continuous) variables. He is of the opinion that is different to in-situ heterogeneity associated with a measurement and that the two are not to be confused. A measurement has an in-situ heterogeneity which is different to that of the sampling heterogeneity and hence it also has a different probability space. The differences between a measurement and a sample shown in Table 4.1 for comparison have been considered by Francois-Bongarcon (2013).

Table 4.1: Differences between measurements and samples

CHARACTERISTIC	MEASURING	SAMPLING
The support	Support in the X, Y, and Z dimensions with correlations and auto-correlations. Material is compact and in-situ. If you can define the geometry in space then you are talking about a measurement. The channel sample represents itself.	No support. Material is crushed or broken to various degrees of comminution and is not in-situ
Correlations and autocorrelations	They exist and form the basis of geostatistics	All correlations between fragments is broken because of comminution of the ore.
Applied discipline	Interpolations between measurements – involves the application of the variogram in	Statistics for random independent variables. No interpolation between samples. All correlations and hence ability to interpolate

	geostatistics to describe and quantify macro-correlations	between samples is broken. Theory of sampling – representativeness.
Separation in space	Measurements are separated by distance or time from one another. Taking a sample from a conveyor belt is the same as a measurement at a coordinated position in space	Samples are separated by time, as a proxy for distance from one another
Statistics	We define a group of random variables (RV) characterised by the mean and the standard deviation of the variable. Random function (RF) is defined by the variogram.	
Physical aspects	There is a particular geometric space associated with the volume of material extracted as a measurement. The sample cannot be re-taken or re-sampled.	Material is sampled by division. The lot can be homogenised and resampled several times.
Are data collected at non-random points in space and time?	Yes = Geostatistics	No = Theory of Sampling

Descriptive statistics

The objective in sampling a lot of broken ore is that by taking a representative physically manageable portion of the lot (sample) we might determine the metal grade in the entire lot. The lot could be seen as the population and the sample as the portion drawn from the population which is analysed to derive a measurement (Figure 4.2). A number of measurements are required to calculate statistics, the mean and the variance.

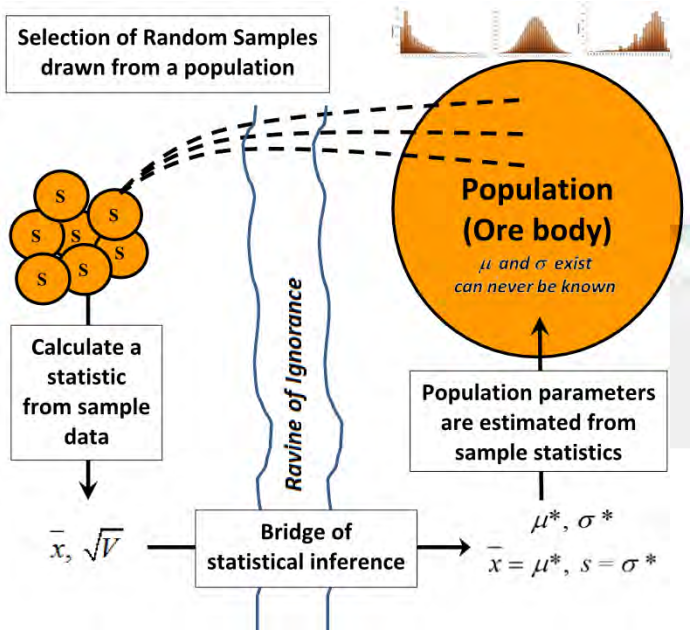

Figure 4.2: Relationship between population, samples, statistics and parameters

The process of statistical inference is used to convert descriptive statistics into inferential statistics or estimates of the population parameters. So a parameter is a number relating to the population (Greek letters) and the statistic is a numerical property relating to the sample (Roman letters) which is used to estimate the corresponding population parameter. Population parameters μ (mean) and σ (standard deviation) really exist, but can never be fully known even when the deposit is completely depleted. While grade of the lot cannot change, individual samples taken from the lot will have a range of values, and constitute a distribution generally lognormal, normal or negatively skewed depending on the commodity, which is fully defined by the mean and the variance (Figure 4.2).

4.4 The Role of Sampling

Sampling is the basis for all decision making in the mineral production process. The content, usually of metals, but of any substance or material of interest, in exploration drill core, channel samples cut from a mining face, and large scale commercial sales of mineral commodities, e.g. coal, iron ore, and manganese is determined through a process of analysing a sample. The analysis provides a mean and a variance of the metal content, the necessary information for a reasonable decision to be made. Errors, measured in terms of relative variance, precision, bias, or representativeness, are statistical values with associated confidence intervals or associated risks. Graphical representations, such as variograms and statistical process control scattergrams provide a method for measuring the identity and magnitude of sampling errors. Control of product quality of large tonnages of relatively cheap commodities by sampling, means even small biases can have a huge economic impact, leading to incorrect technical decisions or dissemination of misinformation.

4.5 The Nature and Size of Sampling Errors

The word “sample” is defined[13] as ‘a small part or quantity intended to show what the whole is like’, with a strong emphasis on the idea of representivity. Hence the best samples carry all the essential characteristics of the lot, something like the DNA signature of biological organisms. Irrespective of which cell is sampled, it’s contained DNA molecule carries in its strands the genome for the whole organism. The unique characteristics of DNA means that half a strand would not be representative, it is only an incomplete specimen, but because the DNA molecule is so small relative to any body part that might be sampled, the molecules are almost always intact. In the same way the representativeness of a sample is a function of the way it is extracted from the lot. The process of sampling is the delimitation, extraction, preparation and analysis of a single or multiple aliquots that are representative of a much larger lot, and the analytical result obtained for the sample is then used as a proxy for the value of the lot (Figure 2). However, because we have analysed a sample and not the lot, there is an error inherent in the value we accept.

[13] Complete Wordfinder. 1993. The Readers Digest Oxford Complete Wordfinder. Readers Digest. 1892p.

The error is in the form of a deviation from the true, but unknown value of the lot. Sampling is never a purely mechanical event; the process leading to the selection of an aliquot must be appreciated. The difference in grade between the sample and the lot is referred to as the sampling error, the art being to identify and minimize all such errors in the process. Measuring the size and source of this error on any individual sample is difficult because we do not know the true value of the lot, nor do we know the size or the source of the error.

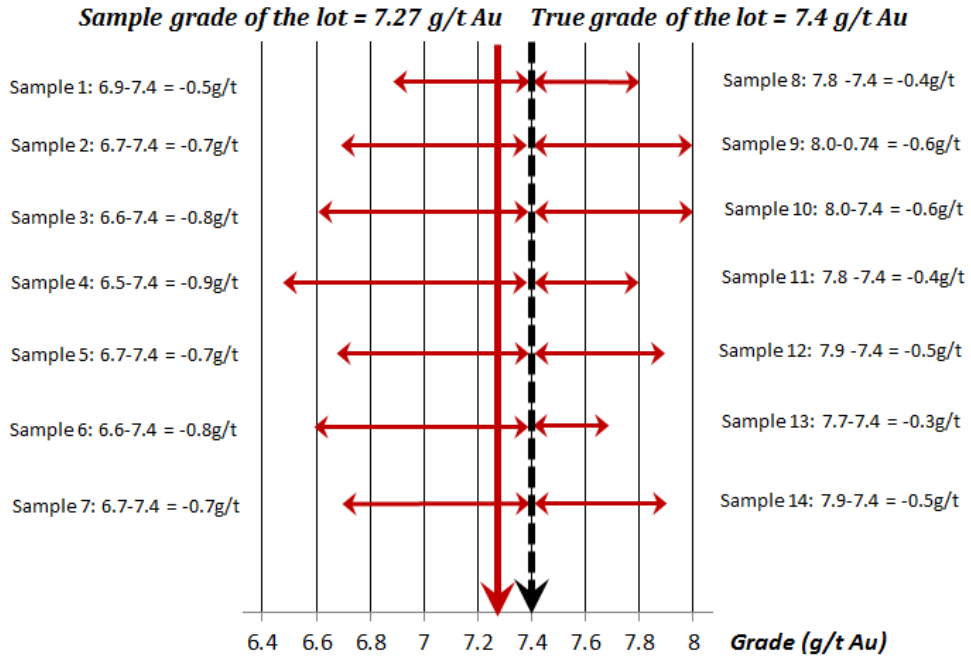

Figure 4.2: Distribution of random errors of 14 samples around a true mean of 7.4 g/t Au, with a systematic bias of 0.13 g/t Au on the low side; the estimated value returned by the assays is 7.27 g/t Au, with a standard deviation of 0.63 g/t Au

Samples of broken ore, either in the form of drill cuttings or fragmented ore recovered from the blast pile in open pit or underground operations are truly representative of the lot only if the principles embodied in the Theory of Sampling have been adhered to. From the several hundreds of tons of broken ore, we extract an aliquot weighing 1 - 50 g to be submitted for analysis. The starting material is probably 10's of centimetres in diameter while the end product will be a few grams at -75µ microns. Errors associated with the sample value are ubiquitous and are the difference between the true grade of the lot and the estimate. Generally the errors include a random component which results in scatter about the mean, and a systematic component that will bias the analytical result and the estimate. Sample values constitute a distribution, an important point being that the sampling errors are likewise from a distribution with the same standard deviation, but a different mean. Lognormal sample values have sample errors with an identical distribution shape as that for the sample data as shown in Figure 3. The only way to ensure there is no error is to analyse the lot – a logistically and financially impossible task, which defeats the point of sampling in the first place.

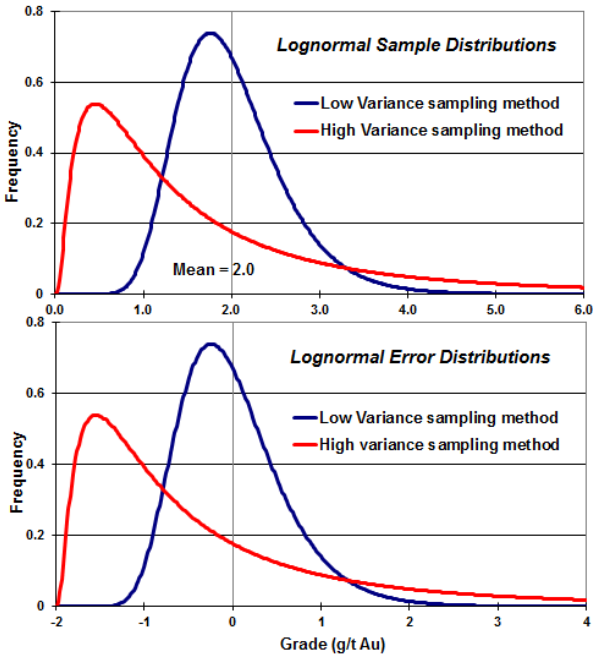

Figure 4.3: Lognormal sample distribution is matched by and identically shaped distribution for the sampling errors; the zero point of the sampling errors lies along the mean at 2 g/t Au

Representative sampling of particulate material is a stepwise reduction of the mass and the fragment size from the lot to the aliquot, schematically shown in Figure 4. The number of steps involved is a function of material characteristics and the requirements of the analytical procedure. Historically sampling is seen as a two stage process, the extraction of the sample from the lot, and the analysis of the sample in the analytical laboratory. Suspicions about aberrant sample results usually mean that mining companies spend money on upgrading their analytical facilities. However, Pierre Gy indicates that analytical biases range from 0.1 to 1%, whereas primary sampling biases can range for 50% to 100%[14]. Thus primary sampling is the main focus of the so-called Theory of Sampling, first established by Gy in 1950[15], and provides the principles for correct, i.e. representative sampling.

The total sample error is a linear combination of all the errors that accumulate at each step of the sampling procedure. Errors specific to a particular characteristic of the lot or specific to a sampling method and/or device, are generated at every step of the sampling procedure. These errors must be identified as those which can be either eliminated, or only minimised, and the appropriate action taken. Nearly all mining project-related decisions from exploration to mine closure are based on sample values, and sometimes with enormous financial implications (Robinson, 2004[16] and 2005[17], De Korvin and Shipley, 2005[18]). Assay results from incorrect sampling provide unrealistic geological ore body models, incorrect in-situ ore grades, and flawed mineral resource and reserve estimates that could lead to financial losses. Sampling as a

[14] Gy, P. 2004. Sampling of discrete materials – a new introduction to the theory of sampling. I Qualitative approach. Chemometrics and Intelligent Laboratory Systems 74. 7-24.
[15] Esbensen, K.H. 2004. 50 Years of Pierre Gy's "Theory of Sampling" – WCSB1: a tribute. Chemometrics and Intelligent Laboratory Systems 74. 3-6.
[16] Robinson G.K. 2004 How much would blending a stockpile reduce variation? Chemometrics and Intelligent Laboratory Systems, 74: 121-133.
[17] Robinson G.K. 2005. Economic justifications for capital expenditure on sampling and blending. Second World Conference on Sampling and Blending (WCSB2). Australian Institute of Mining and Metallurgy Publication Series No 4/2005. 111-117.
[18] De Korvin, A. and Shipley, M.F. 2005. Sampling rules to achieve quality, maximize benefit, and minimize financial loss: Managerial Finance, Vol. 31, No 3: 1-18.

discipline is mired by false beliefs that errors are self-compensating, that precision is not that important, that analytical laboratories are accurate and precise, that current industry practice and a common sense approach are acceptable (Pitard, 2008). Therefore identifying and minimizing sampling errors is the most important issue in a sampling procedure.

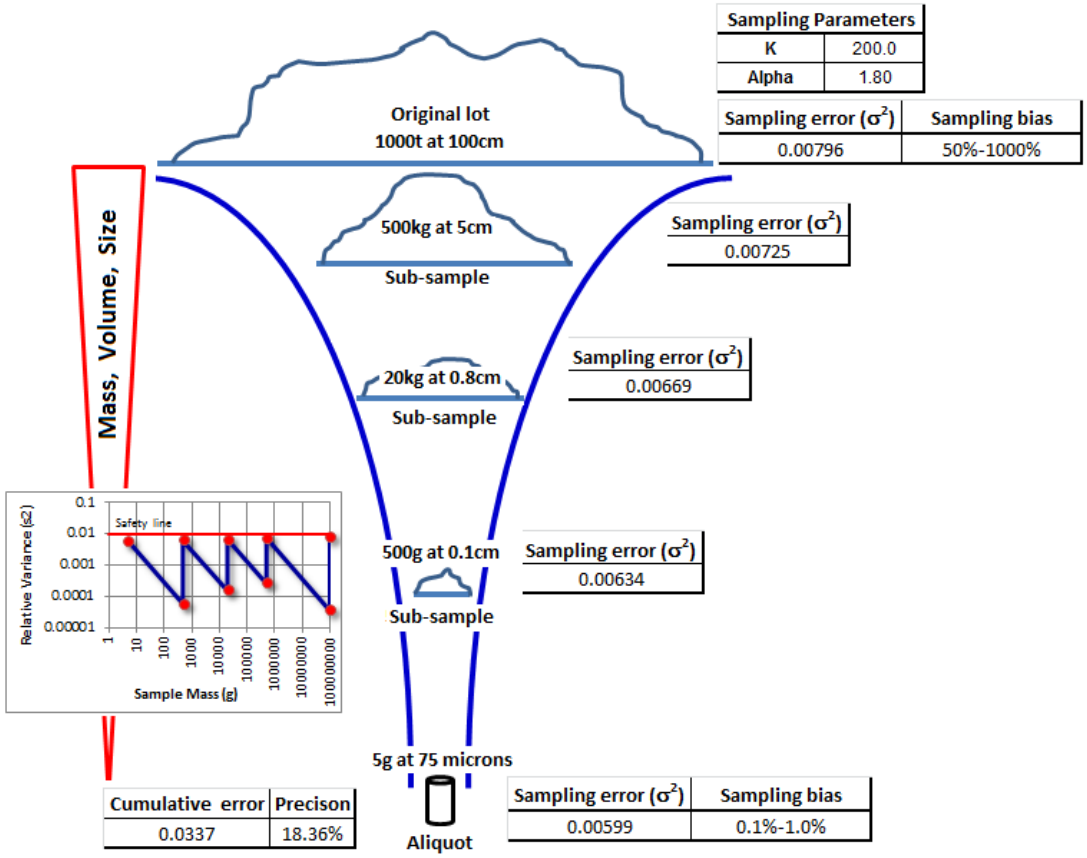

Figure 4.4: Schematic representation of the stepwise reduction in mass, volume and fragment size that occurs during the sampling process

Unlike the cells of organisms, most materials do not provide us with a DNA-type molecule that carries an inherent representativeness; it is left to the sampling champion to define, identify and extract this smallest representative unit, ultimately to present it to the analyst who defines for us what the characteristics or parameters of the lot are. Thus the scale of observation is paramount in understanding optimal sampling protocol. The condition and nature of the material to be sampled will determine the procedure adopted by the sampler in the process of defining the characteristics or parameters of the lot. The parameters of the lot are fixed, but the sampler is again responsible for defining and identifying and minimizing the differences in the parameters of the lot and the sample, i.e., the sampling error. Furthermore we may only be interested in one or two parameters of the lot and the sampling procedure will have to be tailored to ensure that this characteristic is preserved in the sample.

Many companies, particularly those in the chemical and mining industries take huge numbers of samples from their plants every month believing that samples provide them with the necessary control on systems. Many samples are simply used as a monitoring mechanism without well-defined objectives as to what is being monitored and why differences in sampling results arise. Changes in the composition of solid, liquid

or gaseous streams are dealt with reactively by making changes in the plant, but the factors responsible for the changes are never clearly identified. A further problem is that without clear objectives of what is to be achieved through the sampling programme it is very difficult to optimize a sampling protocol.

Generally the control of variability in a plant is monitored through the analysis of samples, which for practical and economic reasons are a small amount of material taken in such a way that it is considered to be representative. Usually when two samples are taken from a stream at the same locality the results will be quite different, but the differences mask variability at a large scale and at a small scale.

4.6 Sampling Errors and Sampling Variability

The difference between sampling errors and sampling variability must be emphasised. No variability would be evident between one sample and the next in a perfectly homogenous population. Sampling variability is always a function of sampling errors. The spread of sampling results is due to the variability arising from the fact that the parent lot is not homogeneous, and generally the variability decreases as the sample size increases. Even if a perfectly optimised and implemented protocol existed during sampling, so that reduction, sub-sampling and assaying were error-free, random samples taken in order to estimate the mean of a lot would all give quite different results. Sampling errors arise because of a wide range of sampling problems associated principally with human and mechanical interventions in the sampling procedure of one form or another, i.e. the interaction of the sample cutter with the material stream and its strong dependency on equipment design. In addition errors may be inherent in the sampled material irrespective of how careful the sampler may have been in eliminating bias. Understanding how these errors arise and the methods by which to eliminate or mitigate them, is the principal subject in the Theory of Sampling and of this text.

The variability and the associated confidence interval are calculated statistically. A confidence interval, defined at a certain level, is the range of sample values between upper and lower bounds that contain the true mean. For example, when sampling the entire lot, a 95 per cent confidence interval implies that 19 out of 20 samples contain the true mean. A change in the estimate of the mean is not statistically significant if the value lies within the upper and lower limits of the confidence interval, i.e. is less than the sampling variability. An alternative to the confidence interval is the Coefficient of Variation (or per cent precision) which expresses the sampling variance as a percentage of the mean squared[19]. The Coefficient of Variation has the advantage of being a relative, rather than an absolute measure and indicates whether estimates have a high or low level of precision or reproducibility. It is also possible to calculate the percentage uncertainty that exists in an estimate. If the relative uncertainty measured in terms of standard deviations sigma (σ) associated with a sample mass of 20kg is ±3% then the mass on repeat measurements could vary between 19.4kg and 20.6kg with 68% confidence. If the 2σ uncertainty is ±6% the mass could vary between 18.8 and 21.2kg with 95% confidence. A sample of mass 500g, would have an associated

[19] Sampling variability. 2008. http://www.statistics.gov.uk/about/data/guides/LabourMarket/methods/quality/sampling.asp

uncertainty of R = 19% with 68% confidence, or 57% with 98% confidence. That is repeat assays on other 500g samples would vary from 0.2% to 0.8% even if reduction, sub-sampling and assaying were error free.

4.7 Face Sampling in the South African Underground Mining Context

The extensive amount of work that has been done in regard to sampling of the mineral deposits of South Africa, and the gold and platinum mines in particular must be considered. The history of sampling techniques for narrow Witwatersrand-type gold reefs has been documented in an excellent article by Fred Cawood[20] who examined the effects of sampling practice and its impact on the Mine Call Factor in Witwatersrand gold mines. Rather than changing the sampling protocol his intention was to stimulate debate on the importance and method of mine sampling on narrow reef mines. In Krige's[21] opinion, both experience and best available procedures are essential if sound judgement is to be applied to decisions based on mine sampling results.

In regard to metal accounting, numerous authors[22] emphasize the distinction between real and apparent gold losses arising from poor sampling protocols and actual metal losses in the movement of mineralized rock from the face to its final location in the gold ingot. The first, and often only indication of metal content in the ores between the face and the mill, is the exposed and sampled reef face which provides the principal control point at which metal balance is established. Discrepancies between gold output at the smelt shop and the initial estimates of gold content by evaluators are captured in a single ratio of these two values referred to as the Mine Call Factor (MCF). While low MCF's are most often ascribed to the real losses of fine-grained rock particles and very fine-grained liberated gold metal enroute to the mill, the apparent losses are ascribed to poor sampling protocols at the reef face. This is particularly true in cases where the reef is highly carboniferous and reef widths are narrow, such as the Carbon Leader Reef and the Vaal Reef for which MCF's as low as 50 per cent have been recorded at some mines. This implies an over-estimation of 50 per cent if all losses are attributed to poor sampling procedures.

In his overview of the historical development of sampling techniques on the Witwatersrand Cawood[20] notes that the important aspects to be considered in sampling are the geology, the mineralogical characteristics of the reef and the channel widths. The earliest recorded sampling protocol for the tabular Witwatersrand–type reefs is that provided by Watermeyer and Hoffenberg[23] in 1932 who suggested sampling intervals of about 15ft (1.5m) in development and 15 to 20ft (4.6 – 6.1m) in stopes. They suggested sample sections could be 6in (15cm) for narrow reefs and 15in (38cm) for wide reefs, the important point being that they recognised that different reefs required different sampling protocols. They also accepted that in narrow reefs the width

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[20] Cawood, F. C. 2004. Underground face sampling on narrow gold reefs: Sampling practice and its impact on the Mine Call Factor on Witwatersrand gold mines: Lessons learnt. Journal of the Institute of Mine Surveyors of South Africa, Johannesburg, Volume xxxi No. ?? Date. Pp //-??.

[21] Krige, D.G. (1952). A statistical approach to some basic mine valuation problems in the Witwatersrand. *Journal of the Chemical, Metallurgical and Mining Society of South Africa*, Johannesburg, March 1952. pp. 201 - 215.

[22] De Jager K. and Anderson, D. G. etc

[23] Watermeyer, G. A. and Hoffenberg, S. N. (1932). Witwatersrand Mining Practice. *The Transvaal Chamber of Mines, Gold Producer's Committee*, Johannesburg, 1932. pp. 895.

of the sample be increased until an acceptable mass (not exceeding 5pds, about 2.3kg) had been collected. According to Cawood[20] *"Great emphasis was placed on the importance of measuring reef, channel and stope widths accurately, always true (normal) to the reef plane."* pg 6.

Working in North American mines in 1932 Hoover[24] also provided a sampling protocol for narrow reefs and veins saying that 'the width of the sample groove should be two or three times the usual width' implying that sample mass is a critical feature.

In his significant second edition volume entitled "Underground practice in mining", published in 1938, Beringer[25] describes the method of chip sampling on the South African Witwatersrand as well as the role and qualifications of a good sampler, saying he should be technically trained and honest. In his attempts to ensure that samples across the mining industry were comparable Beringer[25] emphasised a single standard protocol for all mines. He was the first to insist that half an inch (1.5cm) of waste rock on either side of the reef be included in the sample because of gold enrichment that occurs along the reef-waste contacts. Neither the mass nor the width of the sample is specified, the sample width in particular could vary from 3 to 12in. (8-30cm) depending on the reef width, provided a calico sample bag 9 by 14 inches is filled three quarters.

In 1946 mine sampling practice was comprehensively described by Jackson[26] who opposed the inclusion of external contact waste because it led to overvaluation when using accumulation values (cmg/t). He clearly understood that reefs were variable from place to place and across reef types and made allowances for this in the way samples were taken. Although he did not suggest it directly the implication from his descriptions of reef variability and sample interval is that samples should be in the region of four pounds (1.8kg) in mass. He also suggested that each mine establish a ratio for sample mass to the mass it represents. This is equivalent to the so-called *sampling fraction* defined as the proportion of sample size to population size. Further refinements and consistent updates of the sampling protocol for Wits-type reefs were provided in 1947 by Truscott[27] who again described sampling intervals in development ends and stopes and emphasised the importance of accuracy in the measurements of distances from survey pegs, measurements of reef widths and channel widths, and general due diligence using diagrams and sketches in the delineation and extraction of reef samples. This author suggests sample masses should range between two and five pounds (0.90-2.3kg).

In 1947 Herbert Sichel[28] contributed an insight on the sampling interval based on the channel width, variability of the grade and the type of reef. He also provided a view on the size of the ideal sample and

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[24] Hoover, T. J. 1933. The Economics of Mining. *Stanford University Press*, California. pp. 547.

[25] Beringer, B. 1938. Underground practice in mining: Second edition. *Mining Publications Ltd. (Proprietors of the mining magazine)*, Salisbury House, London, 1938. pp. 277.

[26] Jackson, J. O. 1946. Mine Valuation: Rand Practice. Louis Gordon Booksellers, Springs (South Africa), May 1946. pp. 155.

[27] Truscott, S.J. 1947. Mine Economics: Second Edition. *Mining Publications Ltd. (Proprietors of the mining magazine)*, Salisbury House, London, 1947. pp. 366.

[28] Sichel, H. S. 1947. An experimental and theoretical investigation of bias error in mine sampling with special reference to narrow gold reefs. Transactions of the Institute of Mining and Metallurgy (London) 56 (Bulletin No. 485) (1947), p. 403. *Discussion and comment on Sichel's paper by:*

warned about the dangers of over-sampling the softer conglomerate portions of a reef relative to the harder adjacent waste rock, stating that if this is allowed to occur, overvaluation could be as much as 100 percent or more. In work published in 1952, Harrison[29] also notes the propensity to over-sample and consequently over-value especially in narrow reefs. It appears that he is the first to suggest that the bias introduced by chip sampling could be overcome by diamond drill sample methods. Sichels[28] most significant contribution came in 1947 when he identified the lognormal distribution for gold assay values and described the "*bias error in mine sampling theory*" that can be introduced due to both poor sample delimitation and incomplete or incorrect extraction of the sample so delimited. These are identical to what Gy identified as the Increment Delimitation Error, arising from the incorrect marking off of samples before their extraction e.g. when the amount of waste included in a sample varies from one sample to the next, and the Increment Extraction Error, arising when the geometry of the sample extracted fails to match that of the sample that was delimited. The fact that the average value is calculated using a weighted average calculation '*weighting the assay value by the reef width*' also means that unless the waste and reef portions are very carefully measured a bias error (A. R. O. Williams in Sichel[28]) ???.

Sichel[28] emphasised the importance of equivalent 'support' among samples in order for them to be comparable and recognised that the bias introduced by sampling the waste adjacent to the reef is directly proportional to the amount of waste included in the sample width. It appears that he was also the first to understand the inverse relationship between bias and sample mass "Heavier samples have smaller errors"[20]. In addition Sichel applied a set of correction factors to sample values and later (1966[30]) published a method for estimation of means and associated confidence limits for small samples from lognormal populations. Cawood[20] notes that the work of Sichel[28] led to a review of both the principles of sampling and the calculation of the '*best estimate'* from assay results. The relevant points for this text are that Richardson noted that Sichel had turned attention to the way in which samples were taken from the reef and suggested ways in which sampling might be mechanised. As mentioned by Cawood[20] in the past century no such economical or efficient sampling tool has yet been devised. The best tool is still conceptual and should ideally be a '*rectangular borehole'* that can take perfectly symmetrical samples to an even depth every time.

The effect of clustering of gold grains and the occurrence of large grains (nuggets) on sample values was probably the reason that duplicate check samples (in the same groove or immediately adjacent to it) had become standard practice by 1960[20]. In a study on the variability of gold reefs and the reproducibility of such 'duplicate results' Rowland and Sichel[31] (1960) found that the standard deviations of the logarithms of the ratio between the original value and the duplicate assay is constant, a fact they based their sampling control charts on. The fact is that these are not duplicate samples, nor indeed can they be and that the large variations they encountered are probably related to the nugget effect.

SJ Truscott (pp. 27 - 33), JB Richardson (pp. 33 - 35, 40 - 41), EH Lloyd (pp. 35 - 36), D Gill (p. 36), ARO Williams (pp. 36 - 37), T Pryor (pp. 37 - 38), CM Richardson and WH Wilson (pp. 38 - 40).

[29] Harrison, R. N. 1952. Comment on the Mine Call Factor. *Journal of the Institute of Mine Surveyors of South Africa*, Johannesburg, December 1952. pp. 102 - 104.

[30] Sichel, H. S. 1966. The estimation of means and associated confidence limits for small samples from lognormal populations. Journal of the South African Institute of Mining Metallurgy. In "Symposium on Mathematical Statistics and Computer Applications in Ore Valuation", Johannesburg 1966. pp. 106-123.

[31] Rowland, R. St. J. and Sichel, H. S. 1960. Statistical quality control of routine underground sampling. *Journal of the South African Institute of Mining and Metallurgy*, Johannesburg, January 1960. pp. 251 - 284.

According to Krige (1964) the sample spacing in stopes (about 3-5m) depends largely on the rate of face advance. Although it is not stated a possible reason for this is so that there should be evenly distributed sampling coverage across the reef plane. Differences in the stope sampling interval were mentioned as early as 1932 by Watermeyer and Hoffenberg. They did not specifically state that this was due to their recognition that different reef types or different grade variability required different sampling interval, but the concept is implied in their statements. The more important issue is that implicit in their thinking was that samples had a range or area of influence and that this could be adjusted by changing the sampling interval. Krige[32] also recognised that increasing the sample mass would lead to a reduction in variability of the samples, but that no benefits would arise from trying to determine the optimum size that should be taken. Although he does not state it specifically, the overriding principle to be invoked is that 'bigger is better', but it should be guided by the caveat that the same support size for all samples is just as important.

The most recent and comprehensive coverage of sampling practice in South African gold mines is provided by Storrar[33] (1987) in his classic documentation of South African mine valuation. He covers every aspect of mine sampling including management of sampling crews, tools and equipment, the sampling procedure in general, and sample spacing. The emphasis in his work is that Delimitation of the sample and Extraction of the sample should match one another, and that all measurements should be made with due diligence and accuracy. What Gy refers to as Increment Preparation Errors are described by Storrar[33] under a section entitled "Avoidance of False Values" (p. 46) and includes warnings against contamination, spillage, and fraud. Storrar also has a brief section that deals with Broken Ore Sampling, details of which are described more fully in a later section.

Storrar[33] and Cawood[20] undertook a number of simple experiments to demonstrate the effects of mis-measurement of channel width and reef width on the estimated grade. Both Investigators found substantial over-valuation on narrow reefs if measured widths are not equivalent to the actual sample widths, a combination of the Increment Delimitation and Increment Extraction Errors. He also identified that carboniferous reefs are subject to a 17 percent over-valuation because the softer, richer carbon-rich layer at the bottom of the reefs is more easily sampled (Increment Extraction Error). If the external waste above and below the reef is correctly marked off, but not correctly chipped out a 27 percent over-valuation on grade is possible (Increment Extraction Error).

4.7.1 Lessons from Wits-type Sampling Practice

In the light of the Theory of Sampling developed by Pierre Gy it is informative to review historical sampling practice on South African mines and identify how sampling errors were appreciated and accounted for in the principles underlying sampling practice. Although the sampling errors were not named in the same way as Gy had done, it is clear that all the errors and their influence on valuation practices were understood.

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[32] Krige, D.G. (1966). Two-dimensional weighted moving average trend surfaces for ore valuation. *Journal of the South African Institute of Mining and Metallurgy*, Symposium on mathematical statistics and computer applications in ore valuation, Johannesburg, March 1966. pp. 373 - 380.
[33] Storrar, C.D. 1987 South African Mine Valuation. Chamber of Mines of South Africa. Johannesburg. 1987. Pp. 470.

Prior to the opening of the Far West Rand Goldfields sampling specifications on the Central Rand addressed sample support in terms of size, shape and mass, and the sample representativeness in terms of the sampling interval. The concept of a samples area of influence also probably lay at the root of the early samplers need to specify what the sample intervals ought to be; this became more evident at a later stage[27]. Even as early as 1932 there was an understanding that wide reefs and narrow reefs required different sampling protocols and that if the principle of representivity was to be upheld then narrow reefs would not provide sufficient sample mass unless the samples were made wider. Discovery of the Carletonville Free State goldfields meant that some further highlighted the need for different protocols for the wide Main, Main Reef Leader, and Kimberley reefs found in the Central Rand, and the narrow carboniferous reefs of the Carbon Leader and later the Vaal Reefs. In particular it was clear that the standard Central Rand sampling protocol in the carboniferous reefs led to consistent overvaluation, usually by orders of magnitude. The underlying concept in all of the sampling protocols was that sufficient sample mass should be recovered, but either no samples masses were specified or if so they were linked to the volumes of specific sample bags[25] rather than a specific mass. Prior to the work of Sichel and Krige it seems that no one actually linked a reduction in grade variability to increases in sample mass.

Warnings against over-sampling of the softer conglomerates[24] or the softer carboniferous portions of reefs were given, while others recognised the importance of employing technically skilled samplers[25] for this important task. It is clear that early on[25] an appreciation for what is currently termed gold deportment was evident in that the enrichment of gold along waste-reef contacts was accommodated by including a portion of waste from the hanging- and foot-wall contacts. The use of weighted averages for grades was also beset by problems of mis-measurement[26] of the included waste and reef waste ratios.

Sichel's analysis of bias error in mine sampling theory which emphasised the link between increased sample mass and reduction in grade variability was more akin to the approach taken by Gy in his analysis of sampling errors. Increasing the sample mass to reduce grade variability was also suggested by Whitten[34] (1966) who considered sample mass as a matter of convenience, tradition or history. However Sichel's method for overcoming these problems was to apply mathematically sound correction factors.

4.8 Benefits of the Theory of Sampling

Any sampling process aims to ensure that the sample bears all the characteristics of the lot from which it was taken. The characteristics of the material being sampled, the equipment used to extract the sample, sample handling after it is collected, plant processes and procedures, and the analytical process itself may contribute to the Total Sampling Error (TSE). The TSE is shown in a disaggregated form in Table 4.1, the identification and taxonomy of these errors being amongst the most important of Gy's contributions to the TOS.

[34] Whitten, T. E. H. 1966. The general linear equation in prediction of gold content in Witwatersrand rocks, South Africa. *Journal of the South African Institute of Mining and Metallurgy*, In "Symposium on Mathematical Statistics and Computer Applications in Ore Valuation". Johannesburg, March 1966. pp. 124 - 141.

Table 4.1: Taxonomy of sampling errors in regard to material characteristics, plant processes, sampling equipment, and analytical procedures

	Sources of Error	Nature of Error
Total Sampling Error	***Material characterisation***	***Fundamental Sampling Error (FSE)***, Related to Compositional Heterogeneity due to the material properties of the lot
	(Sampling uncertainties)	***Grouping and Segregation Error (GSE)*** Related to Distributional Heterogeneity due to grouping and segregation in the lot
	Sampling equipment and Materials handling ***(Sampling errors)***	***Increment Delimitation Error (IDE)***, Geometry of outlined increment is not completely recovered; can be completely eliminated
		Increment Extraction Error (IEE), Material extracted does not coincide with the delineated increment; can be completely eliminated
		Increment Preparation Error (IPE), All sources of non-stochastic variation after extraction of the material; error should always be nil
		Increment Weighing Error (IWE), May be relevant or not, depending on specific context. Sample increment mass not proportional to the mass based interval or material throughput
	Plant Process and Procedures ***(Combined sampling uncertainties and errors)***	***Short-range Process Integration Error (PIE_1)***
		Long-range Process Integration Error (PIE_2)
		Continuous Selection Error (CSE), Random, Time, and Cyclical fluctuations, usually sub-divided into Random and Non-random effects
	Analytical processes (Mainly sampling errors)	***Analytical Error (AE)***, All sources of error associated with materials handling and analytical processes in the laboratory

4.9 The Sampling Protocol

The process of sampling refers to the actions of identifying what increment must be extracted (IDE), the action of increment extraction (IEE), the sample handling and preparation process (IPE) to produce a final aliquot for presentation to the analytical technology, and the geochemical or fire assay-type analysis of the final aliquot. Generally the actions and procedures along the route from lot-to-aliquot are well understood, at least in each stage, by the operators involved in the processes, and comprise what is known as the Sampling Protocol. However it is rare to see the total lot-to-aliquot process carefully documented and displayed in a convenient place where irrespective of the person involved, the so-called protocol to produce an acceptable result can be followed without ambiguity.

The defining framework within which the protocol is constructed is referred to as the sampling nomogram. This is a staggered series of comminution and mass reduction processes applied to the broken ore and designed so that changes in the variance of the Fundamental Sampling Error with changes in fragment size and increment mass can be followed. The nomogram is constructed so that the FSE should not exceed 10% precision at any point along it. The two constants K and Alpha (α) are determined through an

experimental process of calibration for each specific ore type. These values are then substituted into a modified version of Gy's formula and form the basis for compiling a nomogram.

Francois-Bongarçon (2011) has summarised a number of essential concepts which Pierre Gy contributed to our understanding in the Theory of Sampling. The seven essential concepts are presented in Table 4.2 together with a brief explanation of each.

Table 4.2: Seven essential concepts of Francois-Bongarçon (2012) in TOS with a brief explanation

	Essential concepts	Explanation
1	Once the sample is taken there is no way to tell if it is a good or bad sample	It is the sampling process from lot to aliquot that must be understood and managed. The economic impact of sampling errors and bias is substantial
2	A sample is one value taken from a distribution of all possible values	In nature metals are distributed according to a given mean and variance
3	Errors or bias arise from problems that may be Circumstantial or Structural	Circumstantial: Always random, transient, uncontrollable and unpredictable Structural: Manageable and consistent It is essential to distinguish between the two
4	There are two types of heterogeneity: Constitutional and Distributional	Constitutional: relating to the internal composition of fragments (within fragment variability) Distributional: relating to heterogeneity between groups of fragments (between fragment variability)
5	Correct Sampling	Each fragment must have the same statistical chance as every other fragment of being in the sample
6	The effects of segregation must be mitigated	This can only be done with a large number of increments with a small mass
7	Sampling of broken ore is different to in-situ measurements	Sampling material from a moving conveyor belt is different to underground chip sampling or core drilling, the latter being simply in-situ measurements

Esbensen (2008) has outlined what he refers to as the seven unit operations, actions which can be taken to overcome or mitigate the sampling errors listed in Table 6.1.

Companies often seek advice from those qualified in the area of sampling theories and methods; yet fail to implement the recommended changes. This usually arises because there is either a lack of effective management at senior level or a lack of understanding regarding the need for correct sampling protocols and the importance of variability. Rectifying these three components is vital to ensure strong support and long-term benefits to a company.

The key to ***effective management*** is that structural problems in the sampling systems are identified. This means that where reconciliations and material balances are not in sync management should be aware of any underlying problems and the likely contributions to thuis. Bad sampling of blasthole cuttings is a structural problem, as is a poor protocol and a poorly designed mechanical cross- stream sampler. Unless

the structural problem is identified and dealt with it could last indefinitely with enormous consequences at times.

If the sampling protocol is sub-standard reconciliations will always be poor regardless of how well the sample is prepared or how careful laboratories are. Having identified problems there must also be a willingness to invest in solutions. Circumstantial problems must be identified as these are usually manifested in areas where substantial financial savings can be made. Once all the sources of variability have been understood it is essential that management respond in a proactive, rather than reactive, manner.

Correct sampling requires that management and those involved in sampling processes and who use the results are aware of the errors, identified by Pierre Gy, which occur in the sampling event. It is particularly important that management appreciates the nature, number and types of variability and its attendant problems and opportunities. Furthermore, management must also be aware that all sampling errors enhance variability and therefore substantial visible and hidden costs can be generated. Analysis of variability requires a strong understanding of statistics, geostatistics and chronostatistics. While geostatistics provides a means of analysing variability in the ore body, chronostatistics provides a means of analysing variability in the plant.

The solution to understanding variability in a plant or process is to “divide and conquer”. When a problem is separated into its component parts and the contribution of each is quantified and understood then meaningful solutions can be implemented. Additionally, once management is capable of examining and analysing variability it should implement a constant improvement strategy whereby aspects of plant variability are constantly monitored.

5 ESTABLISHING DATA QUALITY OBJECTIVES

5.1 Data Quality Objectives (DQO)

Linking sampling methods and procedures to the purpose and reason for undertaking the exercise, is vital. Samples collected for one purpose may be worthless for another. Sampler and analyst alike must identifying the need for the sample, understanding the why's and wherefore's of the exercise. Individual samples may not be exorbitantly expensive, but the cumulative cost can be staggering, especially if there is no one (except an accountant) to say when an appropriate limit has been reached. Sampling costs make it imperative that products of the sampling exercise are used in as many applications as possible. The stages of the sampling process include:

a. Defining the lot;
b. Specifying the variables;
c. Specifying a sampling method;
d. Selecting a sample size;
e. Implementing the sampling process;
f. Analysing the sample, and;
g. Compiling the data and reviewing the sampling process.

Such processes are carried out daily in an innumerable number of settings, perhaps without much consideration of the stages involved. Data Quality Objectives (DQO) address the question of why and with what purpose the samples are collected. The USA Department of Energy provides a Data Quality Objectives home page[6] explaining that the DQO Process "*is a strategic planning approach based on the Scientific Method to prepare for a data collection activity. It provides a systematic procedure for defining the criteria that a data collection design should satisfy, including when to collect samples, where to collect samples, the tolerable level of decision error for the study, and how many samples to collect, balancing risk and cost in an acceptable manner. Using the DQO Process will assure that the type, quantity, and quality of environmental data used in decision making will be appropriate for the intended application, resulting in environmental decisions that are technically and scientifically sound and legally defensible. In addition, the DQO Process will guard against committing resources to data collection efforts that do not support a defensible decision*[35]." The intended scale of the sampling operations undertaken by the EPA is generally much larger than that for a mining or exploration company, but the *modus operandi* is relevant irrespective of scale.

DQO provides a template for managing data collection activities in, exploration, ore reserve evaluations, feasibility studies, ore grade control, processing, and trade of commodities. It is a strategic planning tool that provides the interface to regulatory and legal structures such as the reporting codes and listing rules of securities exchanges (SAMREC, JSE Listing Rules) within which sampling theory and practice is

[35] USA Department of Energy. 2008. Data Quality Objectives Home Page
http://www.epa.gov/osw/hazard/correctiveaction/resources/guidance/qa/epaqag4.pdf Accessed on 7th November 2012

conducted and controlled to provide answers to relevant questions at any phase of a sampling study[36]. Before samples are collected for any project, DQO must be established. It provides sequential planning guidelines that bring efficiency to a project allowing effective and defensible decisions to be made. Only data of the appropriate quantity and quality can support correct decisions. DQO integrates the sampling needs of a project with site-specific concerns about planning, collection, analysis and interpretation of sample data. The appropriate sample types, location, number, quantity, quality, sites, and acceptable levels of error needed for decision making are ensured through DQO. It prevents allocation of resources to non-cost effective sampling efforts. DQO comprises the seven steps, shown in Figure 1. Application of the first six steps of the DQO process produce four qualitative and quantitative statements (EPA (1994)[37]):

a) Clarify the study objective
b) Define the most appropriate data to collect
c) Determine the most appropriate sites from which to collect the data
d) Specify tolerable limits on sample errors.

5.2 DQO Process

The EPA (1993, 2000)[38] has identified seven sequential steps (Figure 1); the first six being the generation of decision performance criteria to be used in the design of the data collection procedure. The final step is to optimize the design within the constraints of time, money and manpower. As a result, it is important to carefully integrate DQO into all sampling activities performed in the field, at the plant, at the laboratory, and for trade of commodities. It is equally important to integrate DQO into statistical studies.

5.2.1 State the problem

Define the elements of the problem in detail so that the aim of the study is unambiguous. A planning team of technical experts develop the DQO with input from all stakeholders. The problem to be solved through sampling data must be described and existing information compiled. A chain of command and accountability for each of the following seven points must be defined.

5.2.2 Identify the decision

The decision statement that the study will resolve, namely the projects economic feasibility using the selected cut-off grades, crushing, grinding, and reduction process, must be defined. Sample data should bring resolution to the main study question which then allows the decision statement to be formulated and defined. The decision statement is the basis for any actions to be taken. Iterative and multiple decisions, such as the need for better control sampling, sub-sampling, and assaying accuracy and precision, for the

[36] Meyers, J. C. 1997.Geostatistical error management. Quantifying uncertainty for environmental sampling and mapping. Van Nostrand Reinhold an International Thomson Publishing Company. Pp.571.
[37] Grumbly, T. P. 1994. Institutionalizing the Data Quality Objectives Process for EM's Environmental Data Collection Activities Memorandum, Department of Energy, United States Government.
[38] EPA 2000. Data Quality Objectives Process for Hazardous Waste Site Investigations EPA QA/G-4HW. *U.S. Environmental Protection Agency Quality System Series* documents. Office of Environmental Information Washington, DC 20460. Pp 143.

quantification of ore reserves, and for effective definition of comminution processes, are resolved on a flow chart.

5.2.3 Identify the inputs to the decision

The type and source of information required to answer the decision statement is identified. Variables to be measured must be defined. The number and types of samples required to answer the questions are stated. Sample data provides information to trigger actions at levels that may be risk or threshold based.

5.2.4 Define the study boundaries

Spatial and temporal boundaries of the study are described in detail. Specifications for accuracy and precision for each constituent of interest and for exploration, feasibility, processing, and trade purposes are established. The appropriate support size, the scale of the decision making, and the smallest unit for which a decision can be made, must be defined. Optimise the strategy for obtaining the necessary data, including the human resources requirements, necessary equipment, and necessary working environment.

5.2.5 Develop a decision rule

A decision rule must state and define the variable of interest that characterizes the population, the support size and scale of decision making, and the action levels. Use of appropriate software for variograms, relative difference plots, and control charts is mandatory. A decision rule in the form of an “if….then” statement is developed.

5.2.6 Specify tolerable decision-error limits

Tolerable limits on decision errors define the performance goals for the data collection design. They are based on the consequences of an incorrect decision. Identify the decision errors and set up the null hypothesis. Specify the region where consequences of decision errors are minimal and assign probability limits to the boundaries of the region. Action levels are defined where the variable exceeds or is less than the boundaries of the region.

5.2.7 Optimise the design for obtaining the data

An effective data collection design for input data to the DQO is established. Activities to optimise the design include reviewing existing data, assessing its internal consistency, and identifying gaps or deficiencies. Data collection designs and optimal sample sizes that satisfy the DQO must be selected. Operational details and assumptions must be documented. The number of samples or use of a larger support volume should be considered to improve sampling outputs.

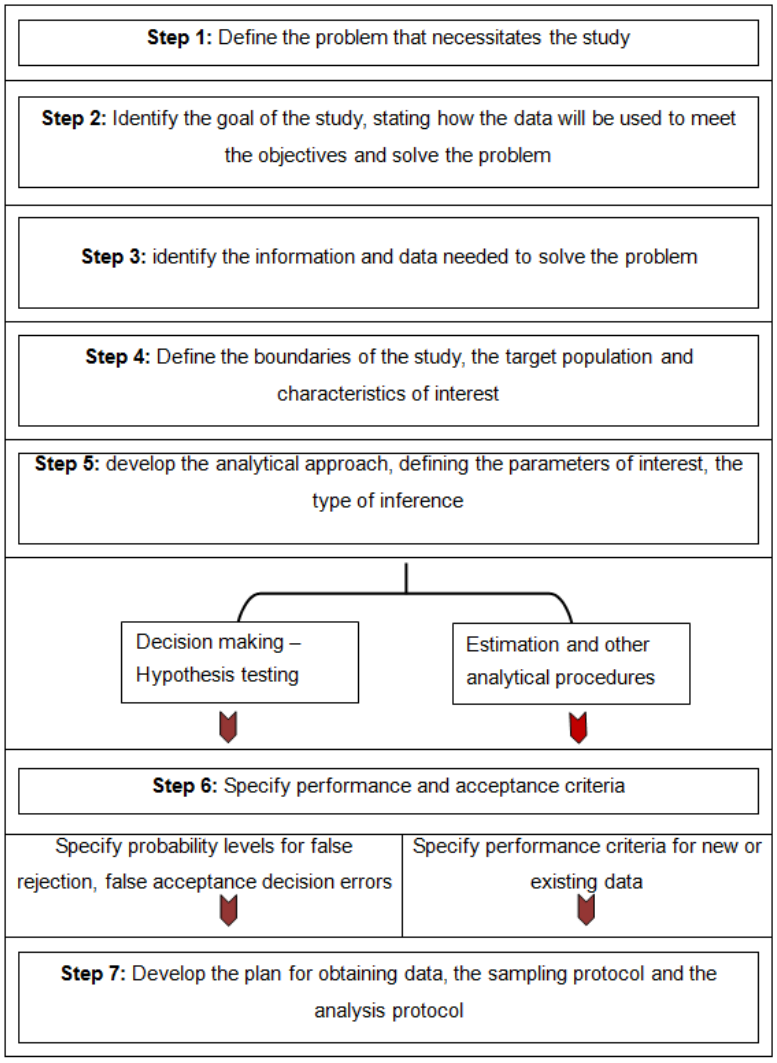

Figure 5.1: Seven steps in the Data Quality Objectives Process[39]

Sample collection may be for commercial, technical, or administrative purposes (Gy[7]), but the reasons may be wider in scope. Maintaining a robust data base and the use of good laboratories with acceptable QA/QC procedures is essential. Cost saving targeted at sampling equipment and procedures introduces considerably higher invisible costs. Pitard [61] suggests that effective management understands the nature of material variability and appreciates the role of sampling in maintaining and promoting organizational goals. Whatever the reason application of DQO is essential as it engages the reason for sampling with an optimal approach to the process. It avoids the pitfall of taking samples on a regular basis which are redundant or not fit-for-purpose.

[39] Guidance for the Data Quality Objectives Process. EPA QA/G4, USEPA, 1993.

6 THE ECONOMICS OF SAMPLING

The economics of sampling is a subset of the economics of information which simply examines the cost of obtaining additional information and the value, positive or negative, associated with that information. Several investigators[40] have tried to quantify the losses (or gains) that can be attributed to poor (or good) sampling practice. More specifically they ask about the purpose of collecting additional samples, the cost of the exercise, who will pay for the exercise, the economic impact sampling might make on the value of the project, the effect of new sampling on project cash flows, and the benefit to the mining operation. Answers to these questions can be obtained through a quantitative analysis of the risk associated with grade control sampling.

Figure 6.1: Sampling ore and host lithologies at Ernest Henry, (**Iron Oxide Copper-Gold Deposits in Australia** 2005.)
Photographs used with permission by Mike Porte: http://www.portergeo.com.au/tours/iocg05/album/iocg05photo14.asp

In his analysis of the problem Bucknam (1997) showed that an improvement of $70 in the value for a block of ore compared to the $20-$30 cost associated with an additional sample. The value of the several thousand blocks comprising the deposit means an increased value in the order of several millions of dollars.

6.1 SUPPORT SIZE

The term support is used in the mining industry to refer to the shape, size, mass and volume of a sample or block of ore. Typically a sample from a Witwatersrand type gold mine stope face is cut across the reef at right angles to its dip and will weigh 2 - 3kg. Such samples are commonly said to have **point support**[41]. A

[40] Bucknam, C.H. 1997. Simplified Quantitative Risk Analysis (SQRA) for Ore Control Sampling @ http://www.bucknam.com/sqra.html. 6p.

[41] Morgan, C J. 2005. Analysing spatial data via geostatistical methods. An MSc dissertation, Faculty of Science, University of the Witwatersrand, Johannesburg. pp 279.

series of 15 or 20 face samples, at two metre intervals will usually mean a block of ground 30m by 30m is evaluated on the basis of these samples; the average gold grade of all face samples being attributed to the block. A 30 x 30 m block of gold-bearing ore, commonly referred to as **block support**, will weigh up to 2500t. Because the mean of the face samples is attributed to the blocks the variability in grade between the face samples will be much larger than that between blocks as shown in Figure 2. Larger support implies smaller variability and vice versa. Using point samples above the cut-off to value blocks will result in overvaluation while the reverse is true; using point samples below the mean will result in an under valuation.

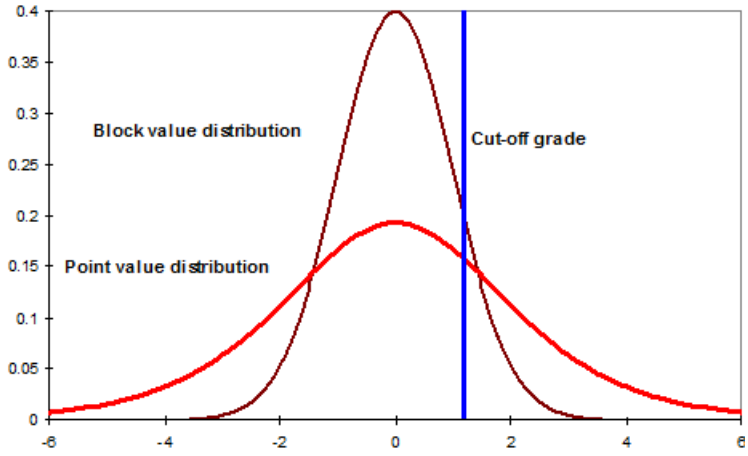

Figure 6.2: Although the means are identical the point value distribution has greater variability than the block value distribution. A smaller proportion of blocks lie above the cut-off than point values

Morgan (2005)[42] has shown that the mineral grade of a block of ore is mathematically equivalent to the average grade of all points within that block.

6.2 MISCLASSIFICATION OF ORE

Most mining operators are largely unaware of how much ore ends up on the waste dump? Misclassification of ore as waste is a hidden cost in any selective mining operation. Standard grade estimation methods have the drawback that they producing good global estimates for the entire deposit, but poor local estimates for selective block scale mining. The degree of confidence in the estimated values is also difficult to quantify. If a cut-off grade is introduced, portions of production that report to the mill should be on the waste dump and portions that should be on the waste dump end up in the mill (Figure3). In both cases the grade falls and we end up with less grade and more tonnes in the mill than expected. Waste blocks, misclassified as ore, dilute the head grade to the mill and is reflected in metal accounting reconciliations as a lowering of the head grade. Even if the mill reconciliation appears to be correct, it fails to account for ore blocks misclassified as waste. There should at least be evidence of this in the reconciliation; if there is no such evidence then sampling is not being done correctly.

[42] Op cit p.23.

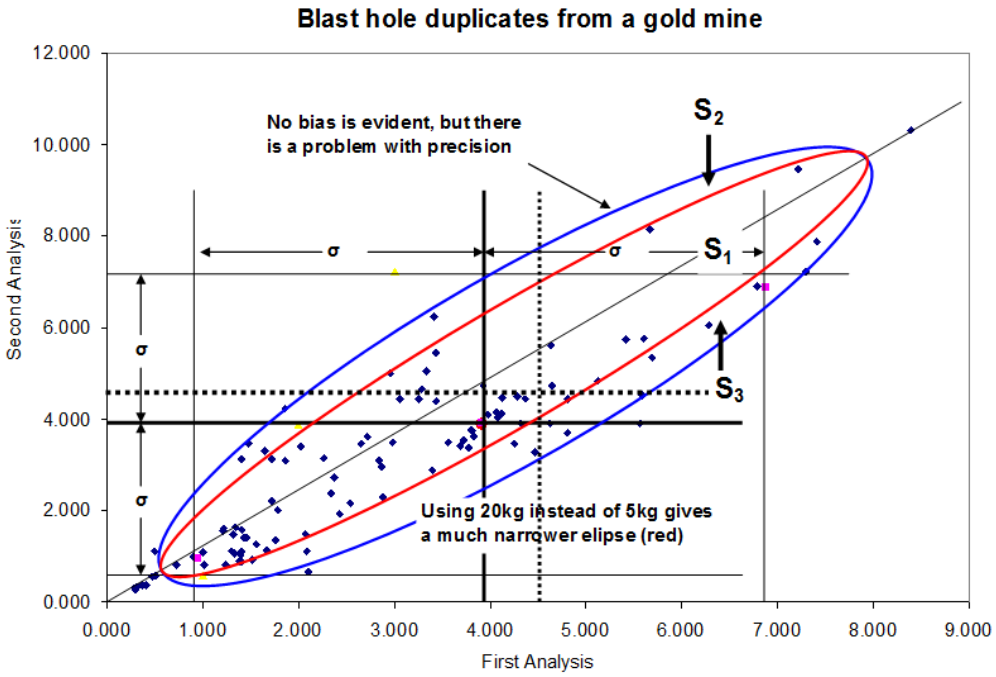

Figure 6.3: The size of the precision ellipse affects misclassification; better precision leads to less misclassification of ore

This misclassification results in a hidden loss of revenue that is never unearthed, unless you treat your waste dump – mill operators are often pleasantly surprised at the grade of "low grade" stockpiles.[43]. There are occasions when a mine will try to improve productivity through increasing the cut-off grade, the underlying logic being driven by the economic incentive that an increase in cut-off grade will increase the average grade above cut-off. The problem in the case of gold and other lognormally distributed mineralisation is that sampling is pushed into an area of the distribution where precision is a problem. Loss of precision can lead to significant misclassification of the ore because of the nugget effect.

An example is a management decision to feed a mill with ore at an average grade established during a feasibility study. Random variability of the ore grade affects the precision, which in turn affects the selection procedure leading to misclassification of the ore. If the expected grade is not achieved, management may be tempted to increase the cut-off grade thereby reducing the ore reserve, but not really solving the problem.

S_1 is equivalent to the amount sold in one year, namely the material that goes to the plant; (S_2 + S_3) is lost to the waste dump. In order to improve precision we must move to a narrower ellipse (Figure 4). By increasing the sample mass you can do a better job of grade control and produce more copper. One reason that improvements are not sustainable is the rate at which mining takes place. Before the data can be analysed and the information sent to the pit crew the bench has been mined. The level of precision at which the cut-off grade is established is critical to the long term well-being of the operation.

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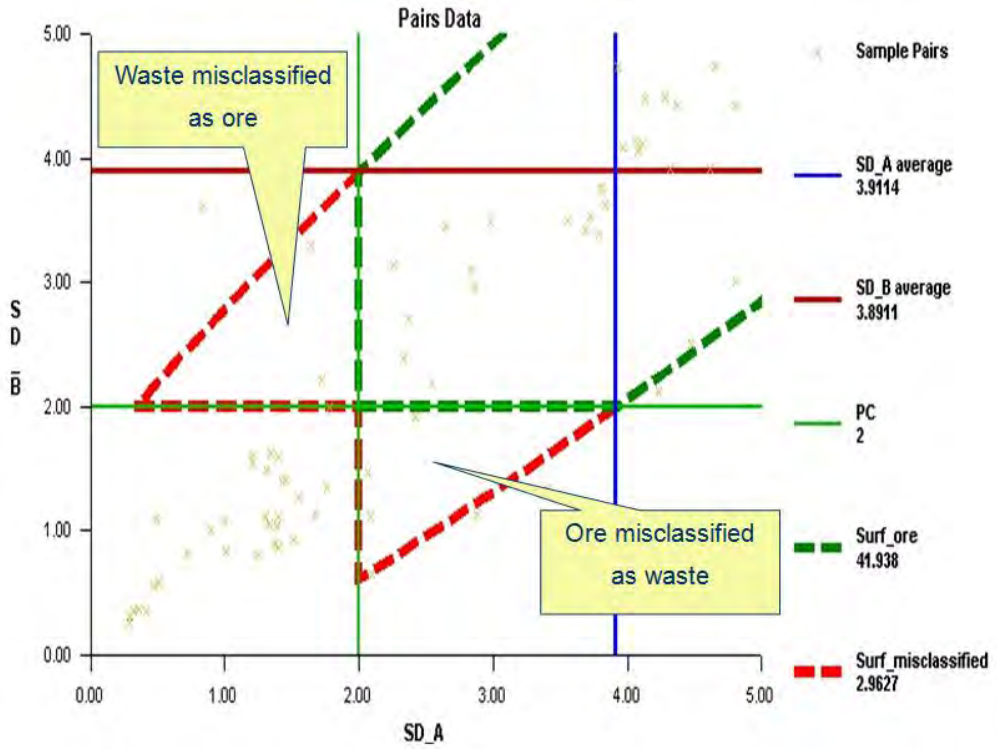
[43] www.vulcan3d.com/pdf/grade_control.pdf. Knowing the odds of the grade control game www.vulcan3d.com

6.3 DIFFERENCES BETWEEN ERROR AND UNCERTAINTY

No measurement is exact because of poor calibration, repeated measurements are different, or external variables change the measurement and for these reasons a measured value must be accompanied by some statement of accuracy[44]. In any exercise involving measurement all we ever produce is an estimate of the true value. It may be a good estimate or a bad estimate depending on the numerous factors affecting the way the measurements are made, but the result, an average of all the measurements, is only ever an estimate of the true value. It is also impossible to define the true value because measurements deviate from the true value randomly or systematically and such deviations are called errors. An analysis of the errors allows us to calculate the uncertainty, but at the end of the day “a statement of uncertainty tells *what we think* about our measurement more than it tells about the measurement itself (thanks to Ron Wittmann)”

6.3.1 Definition of uncertainty

According to the International Organization for Standardization (ISO) uncertainty (of measurement and error are defined respectively as[45]): “a parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurement.', and for error: “the result of a measurement minus a true value of the measurand."



[44]. Young, Matt. 2000. Uncertainty Analysis: Based on a talk given to the Laser Measurements Short Course, Boulder, Colorado, August, 2000. www.mines.edu/Academic/courses/ physics/phgn471/**uncertainty**.htm

[45] International Organization for Standardization (ISO), ``*Guide to the expression of uncertainty in measurement*'', Geneva, Switzerland, 1993.

Figure 6.4: The precision ellipse and the misclassification of ore. (Cost of sampling precision $70643909. Pairs relative standard deviation = 0.2439. Multiple =2). Source: Pitard (2005)

Uncertainty is "a feature arising from the process of measurement that characterises the dispersion of the values around a typical value."[46] This definition of uncertainty focuses on the range of values that the analyst believes could reasonably be attributed to the measurand. Uncertainty about a result may arise from many possible sources, such as incomplete definition, sampling, uncertainties of weights and volumetric equipment, reference values, approximations and assumptions incorporated in the measurement method and procedure, and random variation. Generally *uncertainty* relates to the concept of *doubt* and refers to the limited knowledge about a particular value. *Uncertainty of measurement* however does not imply doubt about the validity of a measurement.

6.3.2 Components of uncertainty

Uncertainty of measurement comprises many components. Components of Type A estimations are evaluated from the standard deviation of a statistical distribution of results. Components of Type B estimations are also evaluated using the standard deviation of an assumed probability distribution based on experience or other information. Overall uncertainty is a composite of contributions from a wide source of uncertainty and it is often possible to evaluate the combined effect of several components. Each component can be expressed as a standard deviation known as a standard uncertainty. For a measurement of any variable y, the standard uncertainty [$u_c(y)$], is an estimated standard deviation.

The **expanded uncertainty** *U*, is the interval within which the value of the measurand is believed to lie. *U* is obtained by multiplying $u_c(y)$, the standard uncertainty, by a **coverage factor** *k,* based on the level of confidence desired. For an approximate level of confidence of 95%, *k* is 2.

6.3.3 Error and uncertainty

An important distinction separates error and uncertainty. Error is the difference between an individual result and the unknown **true value**; thus the value of a known error can be applied as a correction to the result.

By contrast uncertainty takes the form of a range, and, if estimated for an analytical procedure and defined sample type, may apply to all determinations so described. In general, the value of the uncertainty cannot be used to correct a measurement result. For example a result may have a negligible error after correction, but the uncertainty may still be very large, simply because the analyst is very unsure of how close the result is to the true value. The uncertainty of the result of a measurement should never be interpreted as representing the error itself, nor the error remaining after correction.

6.3.4 Components of error

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[46] http://www.measurementuncertainty.org/mu/guide/index.html?content_frame=/mu/guide/uncertainty.html

An error has two components, namely, a random component and a systematic component. **Random errors** also called indeterminate errors or noise arise from statistical fluctuations, unpredictable variations in repeated observations of the measurand due to the precision limitations of the measuring device. Random error can be minimized but never be eliminated. The random error of an analytical result cannot be compensated by correction but it can usually be reduced by increasing the number of observations.

Systematic errors also called determinate errors are reproducible inaccuracies that occur consistently in a definite direction, with a consistent magnitude and have an assignable cause[47]; a constant component of error. They are difficult to identify and cannot be analysed statistically. They may be detected and eliminated when calibrating against a standard. It is independent of the number of measurements made and cannot therefore be reduced by increasing the number of analyses under constant measurement conditions.

So we might conclude that for any measurement there is a combination of both uncertainty and error that could be summarised as follows:

$$\text{Measurement} = \text{Best estimate} \pm \text{Error} \pm \text{Uncertainty}$$

A further type of error is a spurious error or blunder. Errors of this type invalidate a measurement and typically arise through human failure or instrument malfunction. Measurements for which spurious errors have been detected should be rejected.

6.4 SIGNIFICANT FIGURES[48]

A significant figure is any digit 1 to9 and any zero which is not a place holder[49]. The number of significant figures in a value can be defined as all the digits between, and including, the first non-zero digit from the left, to the last digit. For example, 0.44 has two significant figures, and the number 66.770 has 5 significant figures. Zeroes are significant except when used to locate the decimal point, as in the number 0.00030 which has 2 significant figures. Zeroes may or may not be significant for numbers like 1200, where it is not clear whether two, three, or four significant figures are indicated. To avoid this ambiguity, such numbers should be expressed in scientific notation so that 1.2×10^3 clearly indicates only two significant figures or 1.20×10^3, indicating three significant figures.

Depending on the precision of the measuring instrument and our ability to estimate where a measurement lies on a graticule, you may be uncertain about what number to write after the decimal point. For example

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[47] http://chemlabs.uoregon.edu/Classes/Exton/Misc/determinate.html

[48] http://www.physics.unc.edu/~deardorf/uncertainty/UNCguide.html

[49] http://www.rit.edu/~physics/uncertainties/Uncertaintiespart2.html

an accurate measurement of the diameter of a new tennis ball with a straight ruler is constraint by the problem of parallax. The diameter you read off may be 8.3 or it may be 8.5; we think it is 8.4, but we could be out by 0.1cm in either direction, so we record the measurement as 8.4±0.1cm. It is cumbersome to report our uncertainty about length in this way for every measurement we make so we use significant figures to imply the precision of a measurement, without having to be explicit about the uncertainty. We assume an uncertainty of ±0.1cm in the last digit without having to state it explicitly (you must include units). When taking a series of measurements the unavoidable uncertainty will cause the measurements to scatter about the mean. It should be noted that there is a true value which is unknown and essentially unknowable, but for most work we simply assume that the mean equals the true value.

Calculators often display many digits, only some of which are meaningful (significant in a different sense). Estimating the area of a circular playing field, by pacing off a radius of 9 meters and using the formula; area = πr^2. A calculator might return a value of 254.4690049m^2, an extremely misleading value for the area of the field because it suggests that you know the area to within a fraction of a square millimetre! – an absurd result. You only know the radius to one significant figure; the final answer should also contain only one significant figure. A more truthful answer is to report the area as 300 m^2; but such a format is also misleading, since it could be interpreted to have three significant figures because of the zeroes. The better way to report the number would be to use scientific notation: 3□ x 10^2 m^2.

Thus the number of significant figures reported for a value implies a certain degree of precision. In fact, **the number of significant figures implies an approximate relative uncertainty:**

1 significant figure suggests a relative uncertainty of about 10% to 100%
2 significant figures suggest a relative uncertainty of about 1% to 10%
3 significant figures suggest a relative uncertainty of about 0.1% to 1%

For example a value with 2 significant figures, like 99, suggests an uncertainty of ± 1, or a relative uncertainty of ± 1%. Precision is sometimes reported as relative or fractional uncertainty:

$$\text{Relative uncertainty} = \left|\frac{\text{Uncertainty}}{\text{Measured quantity}}\right|$$

so if x = 75.5 ± 0.5g, the fractional uncertainty is

$$\text{Relative uncertainty} = \left|\frac{0.5}{75.5}\right|$$
$$= 0.0066$$
$$= 0.66\%$$

The smallest 2-significant figure number, 10, also suggests an uncertainty of ± 1, which in this case is a relative uncertainty of ± 10%. The ranges for other numbers of significant figures can be reasoned in a similar manner.

6.4.1 Use of Significant Figures for Simple Propagation of Uncertainty

By following a few simple rules, significant figures can be used to find the appropriate precision for a calculated result for the four most basic math functions, all without the use of complicated formulas for propagating uncertainties. For multiplication and division, the number of significant figures that are reliably known in a product or quotient is the same as the smallest number of significant figures in any of the original numbers. Example:

$$
\begin{aligned}
&\quad 6.6 \text{ (2 significant figures)} \\
&\times 7328.7 \text{ (5 significant figures)} \\
&= 48369.42 \\
&= 48 \times 10^3 \text{ (2 significant figures)}
\end{aligned}
$$

For addition and subtraction, the result should be rounded off to the last decimal place reported for the least precise number, in the following examples:

$$
\begin{array}{ll}
223.64 & 5560.5 \\
+54 & +0.008 \\
\hline
278 & 5560.5
\end{array}
$$

If a calculated number is to be used in further calculations, it is good practice to keep one extra digit to reduce rounding errors that may accumulate. Then the final answer should be rounded according to the above guidelines.

6.4.2 Uncertainty and Significant Figures

Reporting a result to more significant figures than are reliably known is untruthful; likewise the uncertainty value should not be reported too much precision. The density of copper for example should not be reported as: Measured density = 8.93 ± 0.4753 g/cm^3

Uncertainty in the measurement is never much better than about $\pm$ 50% because of the various sources of error. Therefore, to be consistent with this large uncertainty in the uncertainty (!) the uncertainty value should be stated to only one significant figure (or perhaps 2 significant figures if the first digit is a 1). **Experimental uncertainties should be rounded to one (at most two) significant figures.** Uncertainty in the density measurement is about 0.5 g/cm^3, telling us that the digit in the tenths place is uncertain, and should be the last one reported. Other digits, in the hundredths place and beyond, are insignificant, and should not be reported: Measured density = 8.9 ± 0.5 g/cm^3

To help give a sense of the amount of confidence that can be placed in the standard deviation, the following table indicates the relative uncertainty associated with the standard deviation for various sample sizes. Note that in order for an uncertainty value to be reported to 3 significant figures; more than 10,000 readings would be required to justify this degree of precision!

N	Relative Uncertainty.*	Significant Figures Valid	Implied Uncertainty
2	71%	1	± 10% to 100%
3	50%	1	± 10% to 100%
4	41%	1	± 10% to 100%
5	35%	1	± 10% to 100%
10	24%	1	± 10% to 100%
20	16%	1	± 10% to 100%
30	13%	1	± 10% to 100%
50	10%	2	± 1% to 10%
100	7%	2	± 1% to 10%
10000	0.7%	3	± 0.1% to 1%

*The relative uncertainty is given by the approximate formula:

$$\frac{\sigma_{ru}}{\sigma} = \frac{1}{\sqrt{2(n-1)}}$$

Experimental values should be rounded to an appropriate number of significant figures consistent with its uncertainty. Thus the last significant figure in any reported measurement should be in the same decimal place as the uncertainty. The practice of rounding experimental results to be consistent with the uncertainty estimate gives the same number of significant figures as for simple propagation of uncertainties for adding, subtracting, multiplying, and dividing.

Caution: When conducting an experiment, it is important to keep in mind that precision is expensive (both in terms of time and material resources). Do not waste your time trying to obtain a precise result when only a rough estimate is require. The cost increases exponentially with the amount of precision required, so the potential benefit of this precision must be weighed against the extra cost.

7 UNDERSTANDING SMALL-SCALE AND LARGE-SCALE VARIABILITY

7.1 Introduction

An appreciation of the nature of variability in all its forms lies at the heart of understanding the Theory of Sampling and its application in the minerals industry. The tools necessary to adequately analyse and evaluate variability include conventional statistics, geostatistics, and chronostatistics. These three approaches to the analysis of variability allow the complex components of the sampling problem to be disaggregated into basic components and for each to be analysed and evaluated independently. Esbensen[50] (2008) has proposed seven sampling unit operations consisting of three principles and four practical procedures by which TOS is guided, shown in Table 6.1.

Table 6.1: Seven Sampling Unit Operations comprising three principles and four practical procedures

Three Principles	1. Heterogeneity and Materials Characterization	Involves Heterogeneity test, Mineral Liberation Analysis, grain size distribution analysis etc.
	1. Lot Dimensionality Reduction	Reclaiming any 3_D lot on a conveyor belt will reduce the lot to 1_D
	2. Variography	Requires the analysis of time series data from a plant or process
Four Practical Procedures	3. Mixing / blending	Normally used once in planning / optimization of a new sampling process
	4. Particle Size Reduction	Used as active steps in the sampling process (often used several times, in combination
	5. Representative Mass Reduction	Mass must be reduced using a rotary splitter or a riffle splitter
	6. Incremental Sampling	The sample should be made up of as many small increments as possible

The three principles are steps preceding any sampling event that ensure it is optimal when implemented. The first principle is that heterogeneity is both a ‘within-fragment’ and ‘between-fragment’ characteristic of the lot. The distribution of the component of interest within-fragments, (the constitutional heterogeneity), and between-fragments, (the distributional heterogeneity), in the host rock must be characterised by means of Heterogeneity Tests. The second principle is that the dimensionality of the lot be reduced as far as possible depending on the nature and the mobility of the lot. Any stockpile that essentially represents a 3-D lot should be reclaimed and moved along a conveyor and be incrementally sampled by a cross- belt sampler, or off a conveyor where the falling stream is incrementally sampled by a cross-stream sampler before the stockpile is rebuilt. This principle also requires that the increments be as small as possible and as numerous as possible for the composite sample to be truly representative of the lot. The third principle requires an understanding of sample variability on a moving conveyor belt, as in one-dimensional lots through application of variography and chronostatistics. Understanding the behaviour of variability in a particular material over time provides a clear indication of the frequency of sampling events so that processes variability is accommodated in the sampling procedure. These three principles are usually only

[50] Esbensen, K., Friis-Petersen, H.H., Petersen, L., Holm-Nielsen, J.B., and Mortensen, P.P. 2007. Representative process sampling in practice: Variographic analysis and estimation of total sampling errors (TSE) Chemometrics and Intelligent Laboratory Systems 88 (2007). 41-59.

applied once during the initial stages of planning and optimising the sampling protocol. By contrast the four practical procedures described below may be used many times during a sampling event and are usually used together with one another.

The four practical procedures[50] include firstly mixing and blending the lot in an attempt to homogenise the material as far as possible before the sampling event. Mixing and blending also aims to minimise both the distributional heterogeneity and the Grouping and Segregation Error that arises when the component of interest is significantly denser than the host gangue material. The second procedure involves particle size reduction through the process of crushing (comminution). The Fundamental Sampling Error is a cubed function of the particle size of the fragments being sampled so even the slightest reduction in particle size reduces the FSE considerably. Representative mass reduction is the third procedure critical to the outcome of a representative sample. Because the Fundamental Sampling Error is inversely proportional to the sample mass bigger samples are usually better, but because most analytical procedures required 30g or less of material the process that reduces the mass from the sample to the aliquot must be done representatively. For this reason the step of mass reduction of the lot is usually preceded by a step of comminution. The final procedure is incremental sampling in which the largest number of increments with the smallest possible size should be composited to form the sample. By following these three principles and four procedures there is strong assurance that the TOS is being properly implemented.

Depending on the type of sampling processes and depending on the reasons for taking the samples, e.g. metallurgical accounting or process control, variability occurs at different scales, including the small scale (PIE1) and the large-scale. Large-scale variability is further divided into large-scale, non-periodic sampling variability (PIE2), and large-scale, periodic sampling variability (PIE3).

7.2 Small-scale Sampling Variability: Process Integration Error (PIE1)

Small-scale variability between one sample and the next arises from two distinct sources, namely sampling error and sampling bias (Table 2). Sampling errors are due to the inherent ‘within-fragment’ variability also known as constitutional heterogeneity occurring inside fragments of broken ore. Sampling errors may also arise from the distributional heterogeneity ‘between groups of fragments’ that occur due to segregation of high-density constituents of interest from the host-rock gangue. Because it is inherent in the composition of the ore, this kind of error can never be eliminated, although the application of the correct protocol can mean that it is minimised.

Small-scale variability may also be a function of sampling bias that is induced during the delimitation or extraction of the sample from the process plant, an exploration borehole, an open pit production bench, or a conveyor belt. The bias induced by the interaction of the sampling tool with the broken ore can be eliminated if the sampling process and the sampling equipment are matched to the characteristics of the material being sampled such as the fragment size and the mass of material extracted.

Table 6.2: Types, sources, size, and mitigation of small-scale variability

Type of variability	Source of variability	Size of variability	Methods to minimise the variability
Sampling error: Fundamental Sampling Error (FSE) Grouping and Segregation Error (GSE)	Arises from constitutional and distributional heterogeneity	50-100%	Can never be eliminated, but can be measured and optimised to a minimum
Sampling bias: Increment Delimitation Error (IDE) Increment Extraction Error (IEE) Increment Preparation Error (IPE) Increment Weighting Error (IWE)	Arises from the interaction of sampling equipment with broken ore fragments	10-50%	Can be totally eliminated by optimising the sampling equipment to extract a correct sample
Sampling Error and Bias: Analytical Error (AE)	Arises from errors and bias in the extraction and submission of the aliquot to the analytical technology	1-5%	Application of TOS at laboratory scale

Once identified the types and sources of small-scale variability are minimised by implementing a "Saville Row"-type solution affected through the sampling protocol. The protocol is designed to accommodate the heterogeneity of the broken ore being sampled, and to minimise the bias associated with each sampling event during sub-sampling or the collection of increments. It must be clearly articulated and adhered to by all involved in order that sampling events, one to the next, are consistent in every respect. Once the protocol has been established it must then be implemented. If small-scale variability becomes too large it may mask the larger-scale variability one is trying to identify and control in a process plant.

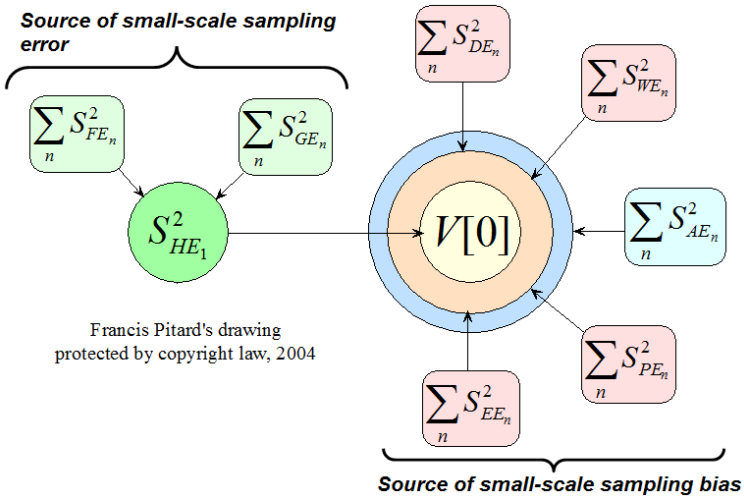

Figure 7.1: Sources of random small-scale sampling error and bias V[0] ***(Source: Pitard [61])***

7.3 Optimising the Sampling Protocol

Prescribing exact procedures for delimiting and extracting a sample is the only way to optimise the sampling protocol. Elimination of small-scale variability due to sampling error requires that an optimal sampling protocol be established through the use of Heterogeneity tests[51] and material characterisation[52].

[51] Pitard (2005).

The sampling parameters, in the case the K and alpha factors in Gy's formula, of the material being sampled must be quantified and understood. The heterogeneity carried by a given constituent to be sampled must be characterised prior to the selection of a sampling protocol in compliance with well-defined DQO. Thorough microscopic observations, mineralogical studies, and mineral processing studies can greatly help to customise heterogeneity tests. Gy (1979[53], 1983, 1992[54]), François-Bongarçon (2001[55], 2003[56]), Pitard (1993[57], 2002[58], 2003[59], 2009[60]), and Magri (2011) suggested many ways to perform such critically important tests, the final objective of which is to calculate a sampling nomograph specific to the constituent of interest, and ultimately lead to the selection of an appropriate sampling protocol in compliance with DQO.

FSE only affects the sampling process once an increment of broken ore is extracted. Mineralised rock is broken into fragments ~0.2 - 2.5 cm in diameter during blasting or drilling and the FSE decreases as the sample mass increases. For a combination of a given mass and a given particle size FSE is a minimum, assuming that everything else is optimised. FSE is usually the most substantial error and can be resolved with diligent and careful experimentation.

GSE arises because of the one-dimensional force of gravity. Segregation is induced in a lot of material because of differences in density, shape of a pile, size and shape of particles etc. Most equipment designed to homogenize material actually induces segregation and the only way in which this error can be minimised is by compositing many small-volume, small-mass correctly extracted increments from a lot.

Determining a correct sampling mode such as systematic, systematic random, stratified random, (link with PIE3) is also an essential first step in ensuring the sampling protocol is optimised.

7.4 Implementing a Sampling Protocol

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[52] Esbensen, K. 2008. Sampling Theory and Methods. A Short Course presented by Prof K. Esbensen through the Geostatsitical Association of south Africa, in the School of Mining Engineering at the University of the Witwatersrand. August, 2008.
[53] Gy, P, 1979 and 1983. Sampling of particulate materials, theory and practice, in *Developments in Geomathematics 4* (Elsevier Scientific Publishing Company).
[54] Gy, P, 1992. Sampling of Heterogeneous and Dynamic Material Systems: Theories of Heterogeneity, Sampling and Homogenising (Elsevier, Amsterdam).
[55] François-Bongarçon, D, 2003. Theory of sampling and geostatistics: an intimate link, in *Proceedings Elsevier: 50 years of Pierre Gy's Theory of Sampling: First World Conference on Sampling and Blending,* pp 143-148 (Chemometrics and Intelligent Laboratory Systems).
[56] François-Bongarçon, D and Gy, P, 2001. The most common error in applying 'Gy's Formula' in the theory of mineral sampling and the history of the Liberation factor, in *Mineral Resource and Ore Reserve Estimation – The AusIMM Guide to Good Practice*, pp 67-72 (The Australasian Institute of Mining and Metallurgy: Melbourne).
[57] Pitard, F, 1993. *Pierre Gy's Sampling Theory and Sampling Practice*, Second Edition (CRC Press: Boca Raton).
[58] Pitard, F, 2002. Practical and theoretical difficulties when sampling gold, in Proceedings *Mineral Processing Plant Design, Practice, and Control*, Volume 1 (The Society for Mining, Metallurgy and Exploration, Inc).
[59] Pitard, F, 2003. Effects of residual variances on the estimation of the variance of the fundamental error, in *Proceedings Elsevier: 50 years of Pierre Gy's Theory of Sampling: First World Conference on Sampling and Blending,* pp 149-164 (Chemometrics and Intelligent Laboratory Systems).
[40] Pitard, F, 2009. Sampling Theory and Practice. A postgraduate course presented in the School of Mining Engineering at the University of the Witwatersrand through the Geostatistical Association of Southern Africa.

An optimised protocol is implemented by defining the procedure to extract a correct aliquot from a broken ore lot. Biases arise during the sampling process because most sampling equipment is flawed by design to the extent that neither the manufacturers, nor those taking the samples, are aware of the identity or nature of these errors. Pitard[61] defined the Increment Materialisation Error (IME) as the sum of IDE, IEE, IWE and IPE, the four independent errors generated by the practical implementation of a sampling protocol.

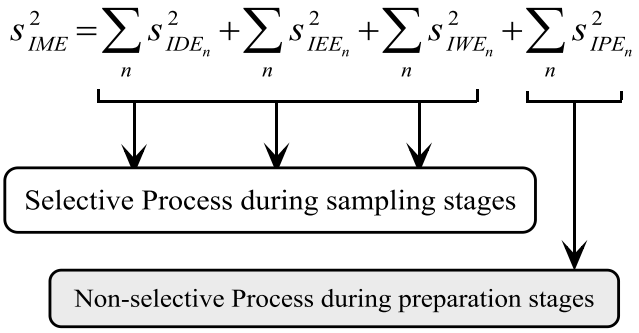

Figure7.2: Errors introduced during implementation of the sampling protocol (Source: Pitard[61])

It is not possible to calculate s^2_{IME} or provide a meaningful value to it. For companies undertaking feasibility and valuation studies a fair market estimation of the share value, the sampling standard is correct, equi-probabilistic sampling systems for optimising all operations[61].

The variance s^2_{IME} of the IME shown schematically in ***Figure 6.2*** as the sum of the IDE, IEE, IWE, and IPE which arise from selective processes during the sampling events. IPE is a non-selective error arising during the preparation stage. It should be emphasised that σ^2 is used for infinite populations, but in practice we use s^2 because we are dealing with finite numbers of samples, or limited amounts of material.

The most important principle to ensure sampling correctness during implementation of the protocol is that *all fragments in the lot must have an equal chance of being collected in the sample*. Failure to observe this principle means that the rule of sampling correctness is not upheld and it is certain that a bias will be introduced. The result is that the mean of the errors is not zero, and the variability around the bias (variance of each error) is not constant, since the sampling bias is never constant as it heavily depends on transient segregation.

Implementation of the sampling protocol requires that both labour and machinery be matched for the task. Identification, coupled with diligent mitigation, of these sources of error is essential in minimising sampling biases. Samplers who have no appreciation for the problems associated with the sampling process may be using the best equipment, but still end up taking poor samples. In the same way samplers who are extremely diligent and understand the TOS cannot be expected to take correct samples with inferior equipment. Samplers must have an appreciation of the TOS and must be provided with the correct equipment if samples taken are to be correct.

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[61] Pitard, F. F. 2005. Sampling Correctness - A Comprehensive Guideline. Australian Institute of Mining and Metallurgy, 2nd World Conference on Sampling and Blending, Sunshine Coast, QLD, 9 - 12 May 2005. 55-66.

Implementing the sampling protocol requires that firstly the sample be correctly defined or delimited and, secondly, that it be correctly extracted. The first problem is one of correct geometry and must obey the principle that *'all fragments in a lot must have an equal chance of being included in the sample'*. This leads to a consideration of the sampling dimensions of the lot and the pre-condition that sampling dimensions must be reduced as far as possible. IDE's arise because in a geometric sense, that is the isotropic volume of observation, the sample does not come from where it is expected to come from, while IEE arises because the sampling tool is selective. IDE and IEE are both severe generators of sampling bias and if such errors are introduced there is no way of backtracking to remedy the situation.

IEE is related to sample recovery. If coarse cuttings, because of their weight, stay at the bottom of an RC drill hole the machine used is responsible for introducing an IEE. Similarly, if the sampling tool preferentially collects fine material (because it cannot collect the large fragments) an IEE is introduced.

7.5 Preserving Sample Integrity

When a physical characteristic of material is altered through any preparation processes such as drying, screening, crushing or pulverizing the integrity of that sample is destroyed and a IPE introduced. As part of protocol implementation the integrity of any sample must be preserved. This requires that fines are not lost during the sample preparation stage, nor that the physical characteristics or composition of the material is changed when drying in an oven that may be too hot. IPE includes contamination, loss, alteration and human error as well as errors due to fraud and sabotage. IPE's are incorrectly considered to occur mainly in the analytical laboratory, but they may occur at any non-selective stage of the protocol

7.6 Analytical Error (AE)

Finally, the AE associated with the last stage of a sampling protocol must be minimised. Not surprisingly companies tend to focus on the analytical laboratory once the existence of sampling errors is identified. New and more rigid procedures are introduced to match the new and improved analytical equipment. Laboratory staff are urged to perform their tasks with renewed determination to improve precision and reduce errors. However, laboratories are usually staffed with highly qualified, diligent people possessing an analytical bent who are responsible for introducing the least of errors into the sampling process.

Issues to be addressed with regard to AE include the contrast between scope and principle. It may be asked if the analytical procedure is compatible with what is being done. "*Is the sulphur in copper cathodes correctly analysed if the method used is close to the detection limit?*" Above 80 microns gold is coarse and submitting such materials to fire assay is totally incompatible with the objective of determining gold content. In such cases a cyanide bottle-roll or screen analysis for gold would be a more appropriate method. Other issues include the additive and overlapping interferences in spectral emissions on specific types of equipment or the proportional interferences due to the so-called "matrix effect". Drying temperature baselines, dissolution techniques, the composition of dissolution residues and contamination or losses may all accumulate and contribute to AE's.

7.7 Summary of Small-scale Variability

Pitard refers to the sum of all small-scale variability as V[0], with the primary aim of an optimised protocol being to minimize the size of V[0]. The concept of V[0] is schematically summarised in ***Figure 3.1*** which provides a means of identifying and classifying the components of the sampling error at a time and distance of zero. At the core of V[0] is a combination of FSE and GSE. This is the absolute minimum for V[0] which becomes inflated by four main bias generators, namely IDE, IEE, IWE and IPE. Finally, the value of V[0] can also be inflated by AE although this is generally the smallest of the combined errors. An understanding as to how V[0] is constituted is vital to a correct understanding of small-scale variability.

7.8 Large-Scale, Non-Periodic Sampling Variability: Process Integration Error PIE2

Large-scale variability occurs within large scale processing or production plants. It is a variability that is often represented on a process control chart and that can be characterised using a variogram and chronostatistics. A plant superintendent wanting to measure and control large-scale variability in a plant process relies on samples taken over a period of time – typically time series data. Many companies take huge numbers of samples on a daily basis with no well-defined objectives about what is to be done with the results, how to analyse the results, or what to do with them. Neither do they know how to correctly interpret them. More often than not they have an unspoken or poorly articulated sampling protocol that has been used for years with no sound scientific reason for the manner or purpose in which samples are taken. On the basis of the results from the many samples taken the plant superintendent attempts to interpret the results, identify the causes of variation, and subsequently introduces control measures to reduce or eliminate what can be termed the large-scale variability of the various product streams in the plant.

Disaggregating sampling problems into their large-scale and small-scale component parts, followed by identifying and disaggregating the sources of variation into their component categories is a first step in resolving large-scale variability. Methodical resolution of the components of variability identifies the elusive sources of error associated with process control problems. Data gathered during statistical process control aims to quantify large-scale variability, but this may be masked or compounded by small-scale variability that arises from other sources. An example of the Manganese metal content in a process plant is shown in Figure 3.3 with the mean value, the 99% confidence limits, the target value and the Lower Specification Limit (LSL).

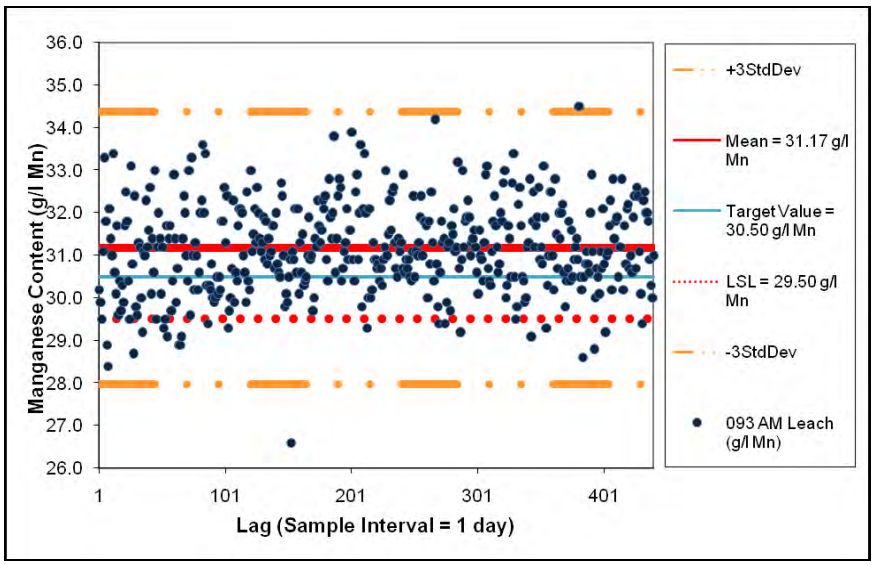


Figure 7.3: A variability plot of manganese content over a 400-day interval showing the mean value, the 99% confidence limits, the target value and the Lower Specification Limit (LSL)

Large-scale variability can only be quantified and analysed once small-scale variability has been addressed and controlled. Companies that are not aware of the consequences of bad sampling, or that fail to address the optimisation of small-scale variability are in no position to resolve the problems associated with large-scale variability. Evaluation of sampling '*correctness*' in regard to small scale variability, for example IDE and IEE, should be complemented with bias testing. Bias testing has limitations because there is no such thing as a *consistent sampling bias*. Bias is a transient phenomenon, changing on a daily, hourly, or even minute-by-minute basis. Negative bias tests do not mean that there is no bias. Attempts to rectify bias by introducing a correction factor simply lead to more damage in the database.

These problems aside, the main aim of process control sampling is to identify process cycles or variability on a large-scale in order to optimise sampling intervals. Strongly developed cyclicity is shown in the Mn circuit leach process represented by the moving average and by the variogram of the circuit leach in Figure 3.4a and 3.4b. In analysing large-scale variability one should be aware of the Interpolation Error (IE) that can occur in both time and space. A variogram is a powerful analytical tool for analysing large-scale variability in process plants.

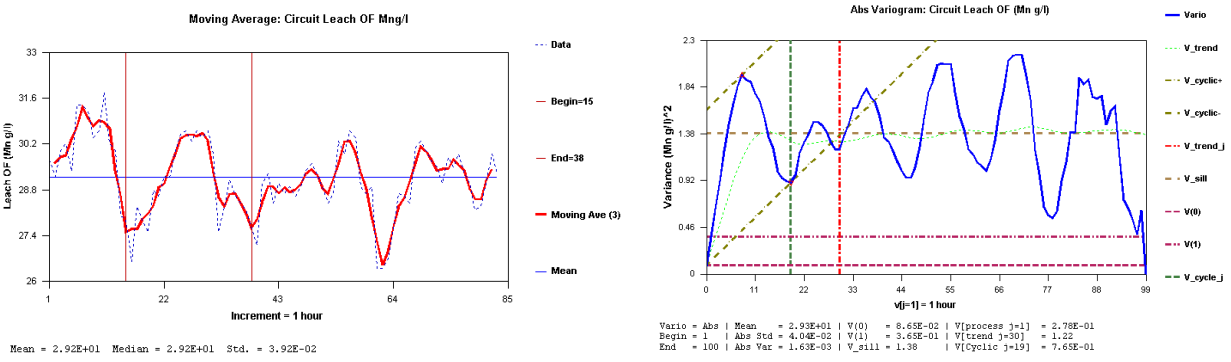


Figure 6.4: a) Five times moving average analysis of Mn in a circuit leach and b) the same data shown as a variogram reflecting the extreme cyclicity of the process

Identifying cycles and their associated causes within plant processes provides an opportunity to improve one's control of the process and ensure that the product is within customer specifications. Cross-belt or cross-stream sampling at regular time intervals may induce a bias if the flow-rates vary between the sampling intervals. Systems extracting samples that are proportional to the flow-rate will minimise the Interpolation Error.

The sampling interval should be optimised in process streams for commercial purposes. Variography is the only means by which to characterise short-term process behaviour, and the only way to optimise the sampling interval at which processes should be monitored and controlled. Variographic experiments[62], involve sampling at short intervals (e.g. every few minutes), and longer intervals (e.g. every hour), to quantifying the random, non-random, and cyclic variability (Gy, 1979, 1983, 1992; Pitard, 2003). Methods for disaggregating the components of large-scale, non-periodic variability are dealt with in detail in later sections.

[62] Op cit

8 MATERIAL CHARACTERISATION

8.1 Metal Distribution in Nature

The metal content of an ore body is a complex function of geology, structure, deformation, the intensity of hydrothermal alteration and other geological parameters. The average value of 100 samples may approach the true value of the metal content, but the range of values is a reflection of the inherent or constitutional heterogeneity of the ore body. The distribution of calorific value (MJ/kg), % sulphur, % ash, fixed carbon, and moisture content in coal deposits is Normal. In such cases the mode, median, and mean are more or less equal and the Coefficient of Variation (CoV) is generally less than 0.33. Samples from deposits with Normal distributions are likely to yield values closer to the mean than elements with negatively skewed distributions, such as iron, manganese, chromite, and alumina. Strongly positively skewed lognormal distributions are even less likely to return sample values close to the mean, with CoV's well above 0.33.

Differences in metal distribution from one deposit to the next are due to the host minerals or host rock. Ferrous metal ores carry 45 to 70 per cent of the metal in the principal mineral, e.g. haematite with 66% Fe in iron ore deposits, or varying proportions of pyrolusite (MnO_2), haussmanite (Mn_3O_4) and braunnite (Mn_6SiO_{12}) in dark brown to black manganiferous oxides. Sampling presupposes that the assay will return a value close to the average, approximately 63 %Fe, in the case of the 1511 iron analyses shown in Figure 1.1. Skewness in the distribution means that any sample selected at random from the lot has a slim chance of returning a value of 63 %Fe; it has a 57% chance of being greater than the average, and a 43% chance of being less than the average. Even with our best efforts to eliminate all the potential sources of error, the likelihood that the sample result will return a value we anticipated, is small.

Incremental sampling is the only way to overcome the naturally skewed distribution of metals in nature. Sampling of a one-dimensional lot of particulate ore using a cross-stream sampler captures the full range of possible grade variability - in effect we are sampling the whole lot in an incremental way to get an average. The greater the number of increments the smaller must be the incremental sample mass, but the more evenly spread will be the selection from the potential range of grade variations.

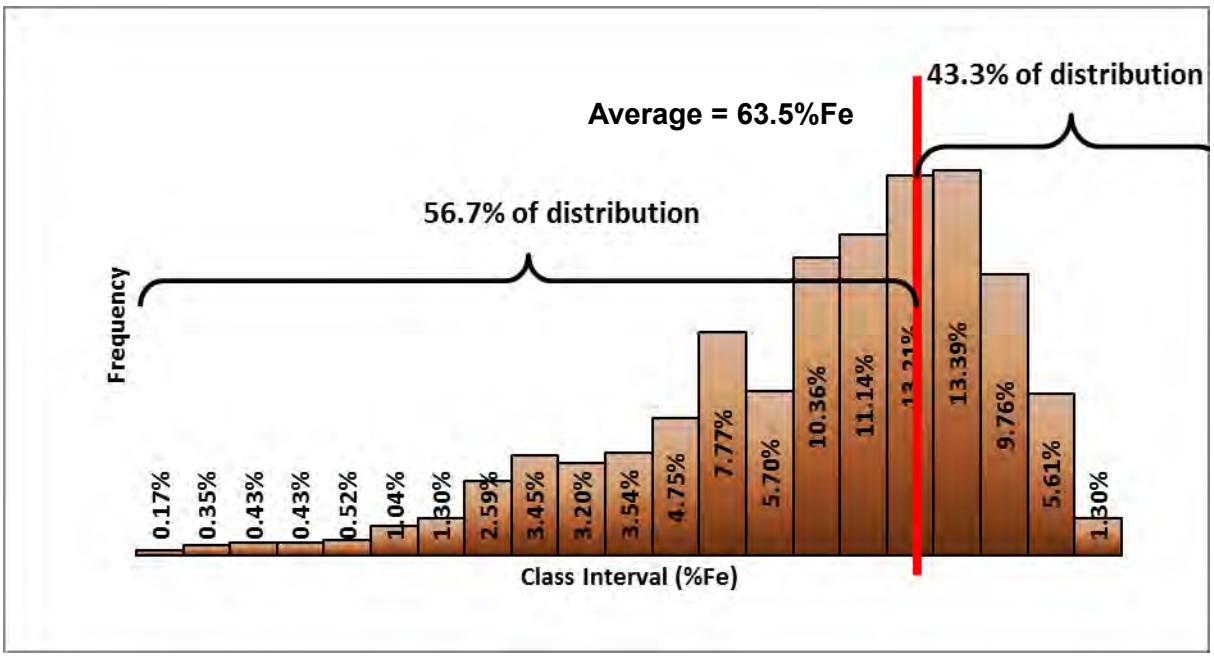


Figure 8.1: Distribution of 1511 iron ore analyses with an average of 66.1%Fe

Furthermore elements in the same ores may have different distributions. The 860 manganese grades have a more-or-less normal distribution, but potassium (K) in the same ores, shown in Figure 2.2 is lognormally distributed.

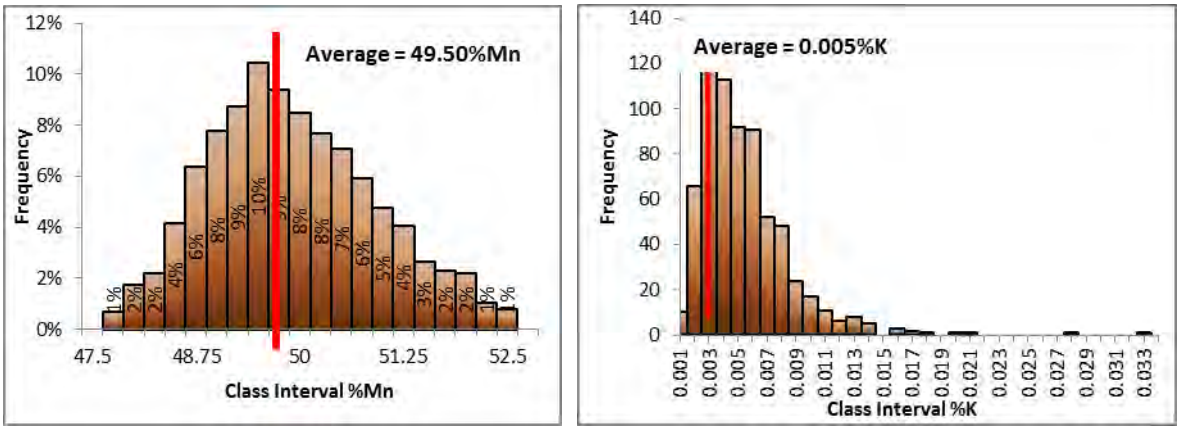
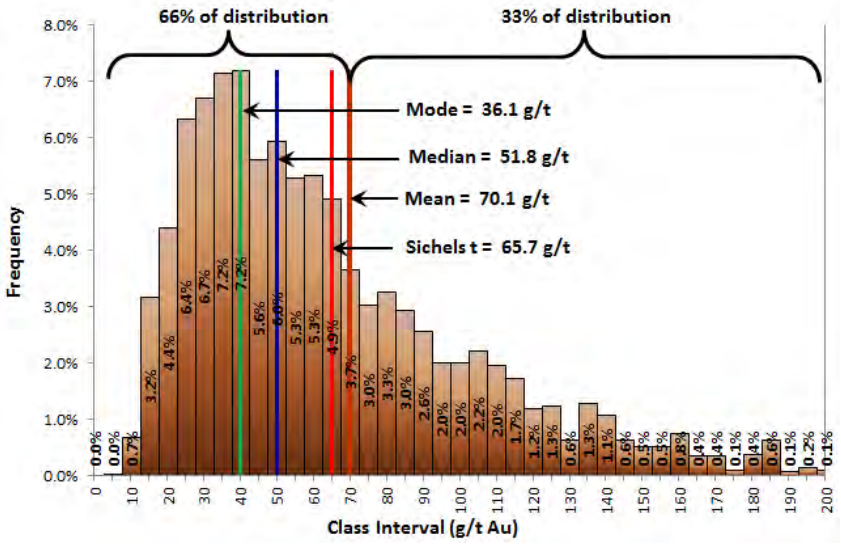


Figure 8.2: Distribution of %Mn and potassium (%K) in 860 assays of manganese ores

Precious metals often display a lognormal distribution as shown in Figure 2.3 for 21578 gold assays (Geostokos Limited, http://software.kriging.com/) from the a South African gold mine. In this case 66% of the distribution lies below the mean of 65.7g/t and 33% lies above it, again making it unlikely that any one sample will return a mean grade of 65.7g/t gold.



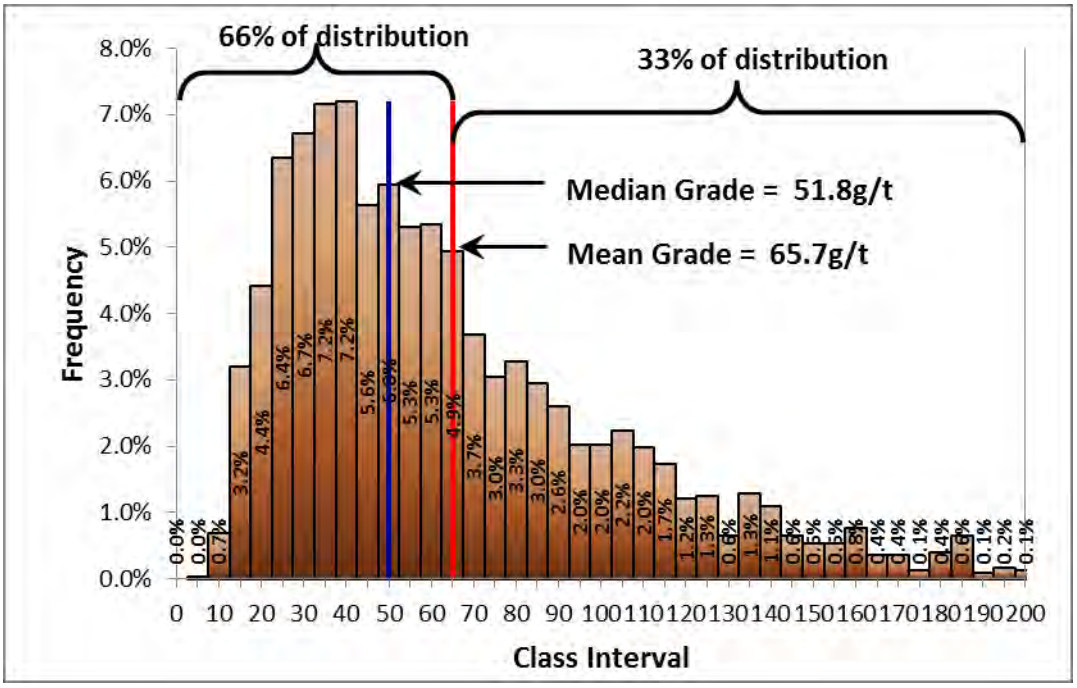


Figure 8.3: Distribution of 21578 gold assays from a South African gold mine

The arithmetic mean of lognormally distributed gold grades has been shown to be a quite inaccurate estimate of the mean (Krige, 1962; Sichel, 1966), if there are less than 40 samples. When the average is to be estimated from more than 40 samples the formula in Equation 1 can be applied:

$$\tau^* = e^{\mu^* + 0.5(\sigma^*)^2} \qquad (1)$$

Where μ^* and σ^* are the logarithmic mean and standard deviations, respectively. A more accurate estimate for the mean of a lognormal distribution using less than 40 samples is the Sichel's-t estimate.

The definition of a lognormal distribution arises from the fact that transformation of the distribution to ln(g/t Au) values produces a Normal distribution, as shown for data from a South African gold mine in Figure 2.4.

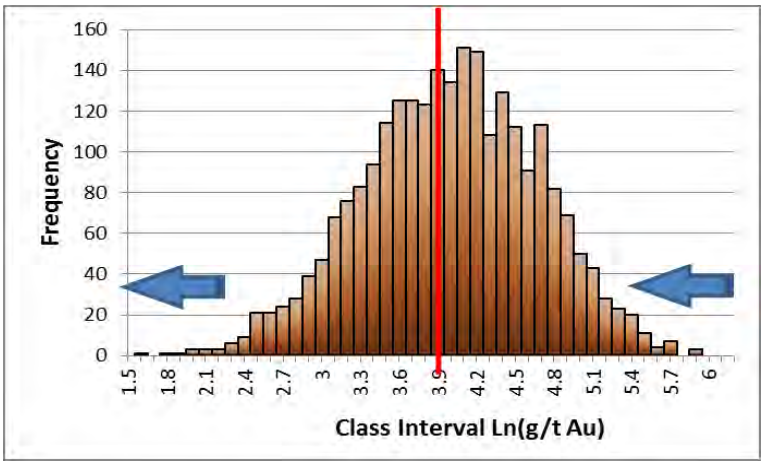


Figure 8.4: Distribution of Ln(g/t Au) for 21578 gold assays from a South African gold mine

Clearly the shape of the lognormal distribution means that extracting a single sample from a *lognormally* distributed parent population is unlikely to yield a good estimate of the mean. In fact there is only a 5% chance that the sample value will be more-or-less equal to the mean (Figure 2.3), and a much greater chance, in fact 66% that the sample chosen will underestimate the mean, probably being closer to the mode than any other value; there is a statistically greater chance of underestimating the grade in lognormal distributions. If the cut-off is below the mean there is a much greater chance of overestimating the block grade and vice versa.

8.2 Central Limit Theorem (CLT)

The CLT describes the characteristics of the means of a sufficiently large number of independent random variables for any distribution, normal, lognormal, or negatively skewed, with a mean μ and variance σ^2. In such cases the sampling distribution of the means approaches a normal distribution with a mean (μ) and a variance σ^2/N as N, the sample size increases. The counter-intuitive outcome of the CLT is that no matter what the shape of the parent distribution, the sampling distribution of the mean approaches a normal distribution. Furthermore, a normal distribution is approached very quickly where N, the number of values in the computation of each mean, increases (Davidmlane, 2011). Regardless of the distribution of the parent population, the mean of the population of means is always equal to the mean of the parent population from which the population samples were drawn. In addition the standard deviation of the population of means is always equal to the standard deviation of the parent population divided by the square root of the sample size (N), and the distribution of means will increasingly approximate a normal distribution as the size N of samples increases. A consequence of CLT is that if the average for any measurement is calculated, the distribution of our average tends toward a normal one. However one should not forget that the iron, potassium and gold distributions shown in Figures 2.1, .2.3, and 2.4, are the products of sampling, sub-sampling, and analytical processes and the effects of the CLT, and yet the skewness of the distributions is

maintained. A consequence of CLT is that if the average for any measurement is calculated, the distribution of our average tends toward a normal one.

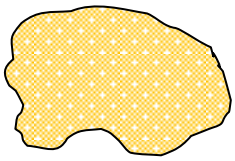
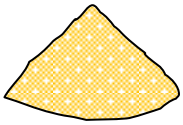
Type of ore deposits		Unbroken in-situ ore		Comminuted particulate ores		Milled analytical powders - at or below liberation size
In-situ unbroken rock 	Blasting and fragmentation	Crushed ore 	Comminution	Comminuted ores 	Liberation Size	Milled ore - powders 
Iron ore, manganese ores, chromite ores, vanadium ores, bauxite ores		Negatively skewed distributions		Approaches normality due to CLT		Normal distribution Aliquot size = 1-2g Analytical method: Typically XRD or wet chemical
Coal deposits		Normal distributions		Approaches normality due to CLT		Normal distribution Aliquot size = 1-2g Analytical methods: Thermal decomposition, XRD, or wet chemical
Gold, platinum, base metals (copper lead, zinc, nickel)		Positively skewed distributions (Lognormal)		Approaches normality due to CLT		Poisson distribution Aliquot size = 30-50g Analytical methods: Fire assay

Figure 2.6: Changes in metal distribution as the degree of ore comminution increases

So there are three domains in which the response to sampling activity is quite distinctive, that of the unbroken in-situ ore, that of broken ore at different stages of comminution, and finely milled ore in which 85% of the constituent of interest has been liberated. A hundred samples taken from different ores will produce different distributions depending entirely on the nature of the material and the type of mineralisation. Samples collected from an unbroken coal seam would have calorific values, ash, and sulphur contents that are all normally distributed, while sampling of in-situ gold ores would reveal a high nugget effect and a strong lognormal distribution. The distribution of constituents of interest in a stream of broken ore subjected to incremental sampling, irrespective of the stage of comminution, will progressively tend towards normality because of the averaging influence of the CLT and the large number of increments.

Finally ores that have been very finely milled, at or below the liberation size, usually comprise the materials from which an aliquot is selected for assay or analysis. Aliquot selection is a sampling stage in its own right and such materials have their own distribution depending on the nature of the metals and their habit. At each stage of particle size reduction there is a corresponding reduction in the mass of the sample. The relationship between the sample mass and fragment size is captured in the nomogram which aims to maintain the balance between the rate at which both of the processes, particle size reduction and mass reduction proceed. Once the milling stage is reached i.e. the point at which the constituent of interest is liberated, the nature and the characteristics of the native metal or the metal host will determine the nature of the sampling behaviour. Ingamells and Pitard (19??), Pitard (2009), and others have shown that for gold bearing ores in these regions, and particularly for very fine-grained, milled powders (95% passing 75 microns), it is typically the Poisson distribution that begins to dominate the sampling processes. Gold, because of the extreme malleability does not break during comminution or milling and may deform, take on a different shape such as a platelet or rounded grain, or even roll into cigar shapes, but it will not break into smaller fragments. At this stage the gold in the very finely milled powder will take on a Poisson distribution in terms of its sampling behaviour.

The characteristics of the original distribution of the metal in nature also have some serious implications with regard to the way in which stockpiles are managed. Blending stockpiles of ferrous metal ores can greatly enhance the overall resource extraction and the export revenues that are generated form these ores. Typically a million tons of low grade iron ore for example at 60% Fe can be blended with 500000t ore at a grade of 63%Fe to produce a lot of 1.5Mt with a n overall grade of 61%Fe which, because of the way in which sales contracts are set up, will sell at a premium. This is especially true in South Africa where blending of iron, manganese, vanadium, and chromite ores is crucial to improving overall profitability, and managing resource allocations.

On the other hand the blending of precious metal ores and those with lognormal distributions is a sure way of destroying value, even though there may appear to be better allocation of the resource and improved resource exploitation.

8.3 Materials Related Uncertainties and Errors

The subtleties between the definitions of errors and uncertainty in regard to sampling have been addressed by Pitard (2009) who quotes a statement by Gy (1967) saying “....:tradition has established the word *error* as common practice, though it implies a mistake that could have been prevented, while statisticians prefer the word *uncertainty* which implies no responsibility”. This distinction is important because INE, FSE and GSE relate to the inherent heterogeneity and material characteristics the material being sampled, and are *uncertainties* rather than deliberate errors. He further clarifies the issue by identifying the major sampling *errors,* namely IDE, IEE, IPE, IWE, as biases. Differences between the true grade of the lot and that returned by the sample assay is a combination of uncertainties and errors arising from both heterogeneity of the material being sampled, poor sampling equipment design, materials mishandling, and derelict duty on part of operators to follow the rules of sampling correctness. The differences in grade between the lot and

the sample arising at the analytical stage include a combination of operator error (minimal at the analytical stage), and uncertainties due to materials heterogeneities. Errors at the analytical stage arise from failure to understand the Poisson nature of mineral, and metal distributions at low concentration, e.g., highly malleable materials such as gold and other precious metals, as well as very hard and brittle minerals which do comminute at the same rate as the gangue in which they are hosted.

8.4 Constitutional, Distributional, and Process Heterogeneity

The parts or elements of a perfectly homogeneous material are all exactly (strictly) identical, and is both constitutionally and distributionally homogenous. Homogeneity is the condition of a material, at the level of a sample, under which all elements might be thought of as identical, and it follows that homogeneity being the zero of heterogeneity, is an inaccessible limit. Constitution Heterogeneity (CH) which is responsible for FSE, and Distribution Heterogeneity (DH) which is responsible for GSE, are considered here. Together these forms of material variability determine how consistent and representative our sampling is. Even if we increase the mass of sample and reduce the particle size by crushing, residual heterogeneity will persist if fragments are segregated in the pile to be sampled. The sampling error is reduced to the fundamental error if the bulk is homogenized by crushing and mixing before sampling (assuming that equipment operation errors have been eliminated).

8.4.1 Constitutional heterogeneity (CH)

CH refers to heterogeneity within the lot arising from the internal differences in composition of the fragments, specifically the ***between-fragment*** variability. The fundamental sampling error (σ^2_{FSE}) is due to constitutional heterogeneity because of differing particle size, shape, density chemical composition, grade and other physical properties; it is unchanged by mixing, but increases significantly as the material is crushed or milled, especially as the average grain size of the constituent minerals is approached. Any action that increases the CH will also increase the variance of the FSE. Reducing the size of the particles by crushing, and increasing the mass of the sample reduces the FSE variance. Reducing the fragment size, once the mineral of interest is liberated, does not decrease the CH. Total CH is the sum of all compositional heterogeneities of individual fragments. The FSE is the difference between what we measure in the sample and what the true value of the lot is. Probabilistic sampling is the only way in which a representative sample can be extracted form a constitutionally heterogeneous lot, i.e. a lot in which all the fragments are not compositionally identical. Gy (1979, 1982) and Pitard (1993) proposed the following formula for the constitutional heterogeneity:

$$\sigma^2_{SE} = \frac{1}{n-1}\sum\left(\frac{a_s - a_L}{a_L}\right)^2$$

8.4.2 Distributional heterogeneity (DH)

If a lot is considered to be composed of individual increments, the distributional heterogeneity is the ***between-increment*** variability within the lot. Distributional Heterogeneity arises from segregation within the

lot due to the tendency for fragments with the same density or size to group together. Even visually in a completely unsorted concentrate of heavy minerals the grouping and segregation effects are evidenced by the grouping of different density and size materials as shown in Figure 2.5.

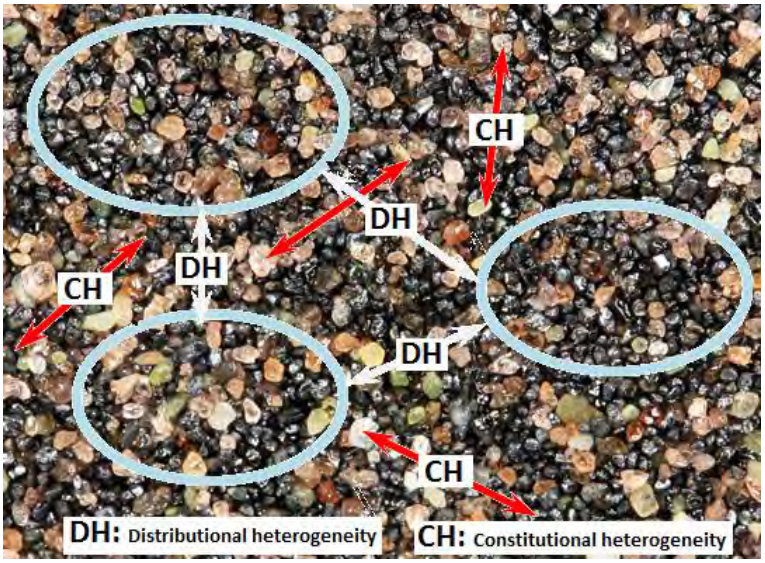


Figure 8.5: Comparison of constitution and distribution heterogeneity in a lot; local CH within an increment arises between individual particles in the sample (red arrows), while DH between arises between groups of more-or-less similar sized increments (white arrows and blue ovals)

The tendency for dense material components to group and segregate under gravity, gives rise to the segregation and grouping errors (σ^2_{GSE}) which can be quantified. This tendency is used to good effect in heavy mineral separation and gold gravity concentration circuits. Examples are seen in iron ore stockpiles where the finer-grained material segregates as long streaks on the sides of the stockpiles (Figure 2.6), and coal stockpiles where larger fragments accumulate around the base perimeter of the stockpile.

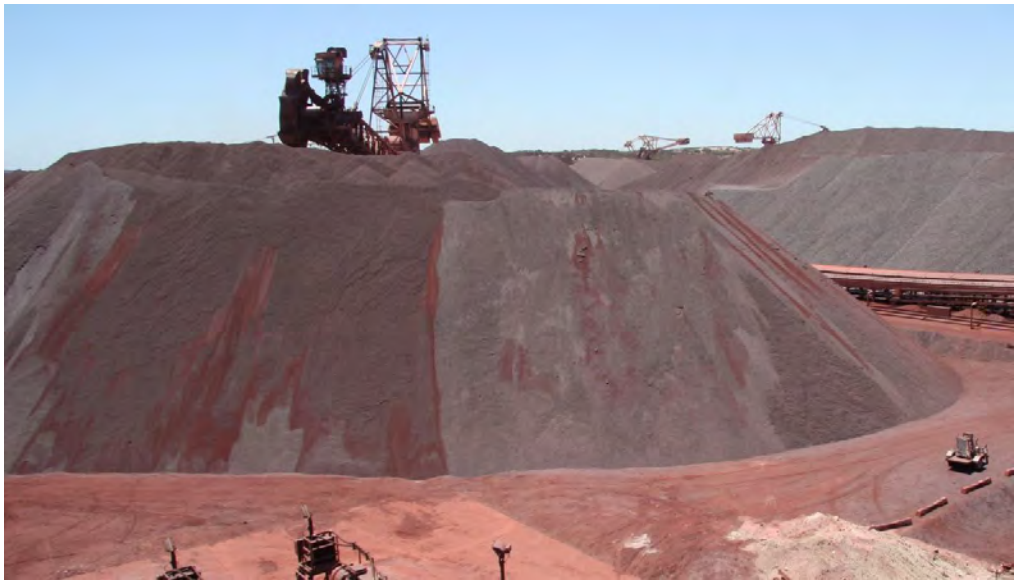


Figure 8.6: Segregated streaks of finer material (light red) across the sides of an iron ore stockpile, Saldanha Bay (used by permission Kumba Resources, 2010).

A distributionally homogeneous lot would yield a perfectly representative increment (sample) even if it were collected under non-probabilistic conditions. Ore fragments in the lot to be sampled segregate principally due to density, but shape and size of the fragments being sampled also contribute. Once material is segregated the lot is no longer homogenous and particles no longer have the same probability of being selected during the sampling process. Mixing and blending the lot before sampling helps to reduce DH, as does incremental sampling. Mixing and segregation are highly transient phenomena and this type of variability introduces a bias that varies in time and space. Particles that differ in size, density, and shape are susceptible to poor mixing and agitation may in fact increase segregation. The best way to overcome the problem is to take as many small increments from different parts of the lot as is feasible, and composite them as a single more representative sample. The smaller the individual increments we use to composite the larger sample, the closer we approach the theoretical best which is random sampling particle by particle. DH is markedly increased beyond the liberation size due to the fact that segregation becomes so much easier because the differences in density are maximised beyond liberation. The relationship between CH_L and DH_L is as follows:

$$DH_L = CH_L \frac{1+\xi\gamma}{1+\gamma}$$

$$where\ \xi = Segregation\ \ factor : Characterises\ the\ type\ of\ \ distribution\ of\ \ the\ constituents\ in\ the\ lot$$

$$\gamma = Grouping\ \ factor : Characterises\ the\ size\ of\ \ the\ groups\ that\ become\ increments$$

Details of Process Heterogeneity are provided in Section 9 dealing with aspects of variography.

9 STRUCTURAL AND CIRCUMSTATNTIAL PROPERTIES

9.1 Logical Process Control Strategy

Correct protocols for sampling, grade control, process control, material balancing, commercial settlements and environmental monitoring is the only means by which to optimise mineral extraction. Attempts to correct the negative outcomes or consequences in a system, without identifying structural (causes) or circumstantial properties (effects) may result in incorrect and ineffective protocols. A property is said to be *primary* when it depends solely on the structure; it is therefore independent of circumstances. It is said to be *secondary* when it depends on both the structure and circumstance.

9.2 Structural Property

A structural property in a system is the cause that gives rise to one or more consistent, permanent, guaranteed negative effects that cannot be controlled or changed. A structural property reliably delivers consistent sampling error or bias as an outcome that arises from the application of procedures or equipment that is thought to be correct, but is not. A poor sampling protocol, a faulty sampling device, an inappropriate analytical procedure, the faulty design of a storage silo, or stacker-reclaimer etc., are all structural properties that can be fixed once they are identified. Identifying structural properties that induce error and bias takes time and effort.

9.3 Circumstantial Property

A circumstantial property is not a function of procedures or equipment, it depends solely on chance. There is not much one can do about these undesirable effects. A circumstantial property is a function of conditions which we are not able to control. Strong segregation induced in a stockpile of ore, by a stacker/reclaimer system may change due to a circumstantial property, a change in moisture content, a change in flow-rate, a change in particle size distribution, etc. Time and money can be lost because of too much emphasis on mitigating the effects of a cause. Changing the cause, the stacking/reclaiming system, is the only thing one could do.

9.3.1 Logical relationship between a primary structural property and a secondary circumstantial property

Answer yes or no, in chronological order, to the following two questions:

		Does the primary Structural Property exist?	
		Yes	No
Is the secondary Circumstantial property observed?	Yes	Certain	Possible
	No	Impossible	Probable

9.3.2 Logical relationship between sampling correctness and sampling accuracy ([40]Pitard, F, 2009.)

Answer, in chronological order, the following two questions.

		Is the sampling device correct?	
		Correct	Incorrect
Is the sample accurate or biased?	Accurate	Certain	Possible
	Biased	Impossible	Probable

9.3.3 Logical relationship between the correctness of a stacking/reclaiming facility and the constancy of the feed it delivers to the process ([40]Pitard, F, 2009.)

Answer, in chronological order, the following two questions:

		Is the stacker reclaimer correct or incorrect?	
		Correct	Incorrect
Is the grade from the reclaimer constant?	Constant	Certain	Possible
	Variable	Impossible	Probable

10 A CRITICAL REVIEW OF SAMPLING AND SAMPLING SELECTION MODES

10.1 NON-PROBABILISTIC SELECTION

. Non-probabilistic sampling is deterministic, purposive or authoritative. Sampling errors for non-probabilistic selection processes cannot be logically connected to the mode of selection. The largest portion of the lot has zero probability of being selected. Non-probabilistic sampling is always biased to the extent that the samples are of little practical value. Non-probabilistic sampling practices are found in commercial sampling, mining evaluations, process control, ore grade control, pilot plants, and environmental assessments. Non-probabilistic sampling should never be used when making important financial decisions.

10.1.1 Deterministic sampling

Also known as “grab sampling” it usually occurs in a single event, the sampler collecting material from easily accessible portions of a 3_D lot with a scoop or shovel. The invisible cost generated using deterministic sampling is substantial. Even if numerous increments are taken, a large portion of the lot at the bottom of a truck or drum, at the center of a conveyor belt or stockpile, which is inaccessible to the sampling tool. Because the probability of selection for most units in the lot is zero grab sampling can never produce accurate results.

10.1.2 Purposive sampling

Purposive Sampling is at the discretion of the operator and is neither accurate nor equitable. It is effectively specimen collecting since units of the lot which are not accessible to the sampler are simply ignored.

10.1.3 Authoritative sampling

The validity of authoritative sampling rests on an individual, who has personal knowledge and acquaintance with the lot to be sampled. The sample is selected with no regard to randomisation. While the method is not recommended authoritative sampling is often necessary for environmental assessments in order to separate hot sectors from others in a contaminated area.

10.2 PROBABILISTIC SELECTION

Probabilistic selection implies that all the components of a lot are submitted to the selection process and have an equal chance of being selected.

10.2.1 Probabilistic sampling of movable lots

A “movable” lot is small enough to be handled in totality for the sole purpose of its sampling. All zero-dimensional aliquots that are sub-sampled in the laboratory are in this category. The two probabilistic processes which can be used are the:

10.2.2 Increment process

The lot is transformed into a one-dimensional stream from which increments are collected with a cross-stream sampler.

10.2.3 Splitting process

The lot is partitioned into several fractions, one of which is selected at random as a sample.

10.2.4 Probabilistic sampling of immovable lots

An immovable lot may be arranged as a one-, two- or three-dimensional lot. There is no probabilistic sampling of immovable lots, simply because the principle of correct sampling cannot be upheld, but the best approximation is a form of incremental sampling. For example three- dimensional lots can be flattened to a two-dimensional lot and drilled (augured) on a regular grid.

10.3 SAMPLING SELECTION MODES

Three sampling selection modes that incorporate an element of randomness in their protocols are of importance, namely Random Systematic, Simple Stratified Random, and Authoritative Stratified Random sampling. Application of these modes to one-dimensional lots, i.e. streams of solids on a conveyor belt, slurries, or liquids, and two-dimensional lots spread out over areas, is considered.

10.3.1 Sampling Selection Modes - Two-Dimensional Lots (Areas)

Two-dimensional lots could be represented by a flat copper cathode about 70 x 80 cm, or an undulating area of land of several hectares. A sector is a fraction of the area, whose size may be determined statistically in the case of a cathode or by the rules of local regulatory commissions in the case of land.

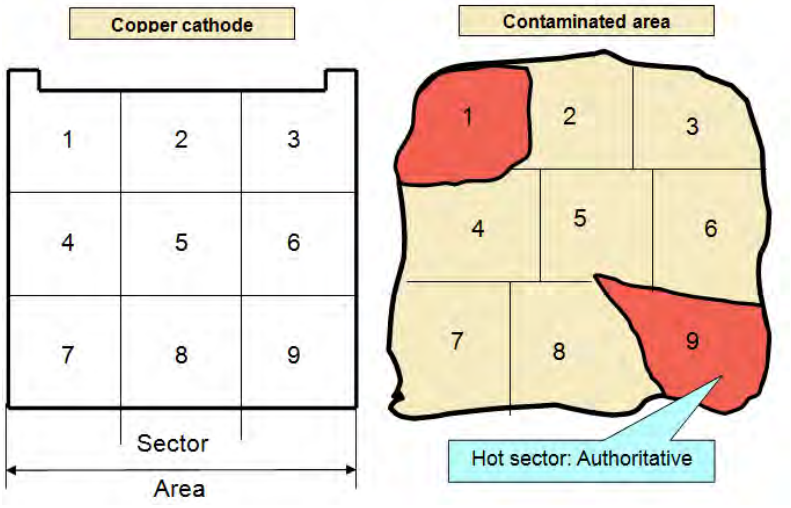


Figure 10.1: Two-dimensional lots a) copper cathode and b) contaminated land areas for sampling by random systematic or random stratified sampling methods ([40]Pitard, F, 2009.)

The categories of stratum and sub-stratum within the two-dimensional lot are defined in **Figure 9.2**.

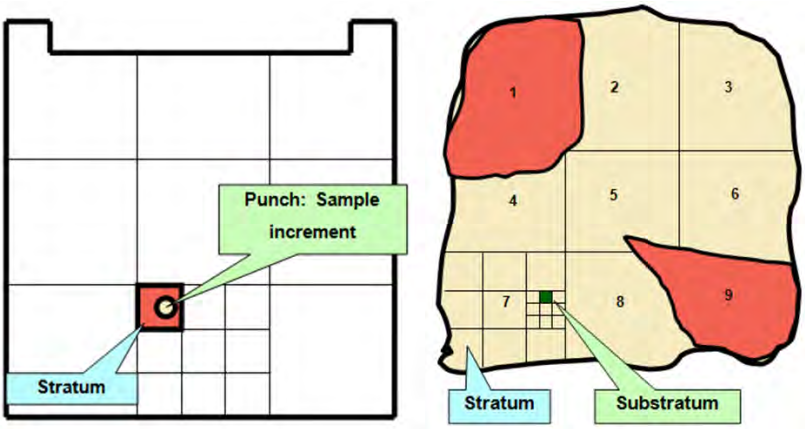


Figure 10.2: Position and hierarchy of stratum and sub-stratum in a two-dimensional lot, a) copper cathode and b) contaminated land area ([40]Pitard, F, 2009.)

Having defined the stratum and sub-stratum (**Figure 9.2**) it is possible to select either random systematic or stratified random sampling modes. The Random Systematic sampling mode is shown in **Figure 9.3a** while the Stratified Random sampling mode is shown in **Figure 9.3b**.

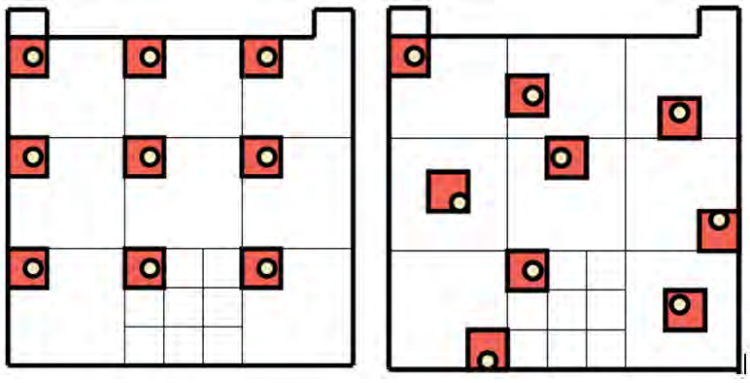


Figure 10.3: Position and hierarchy of a) stratified random sampling and b) random systematic sampling in a two-dimensional copper cathode ([40]Pitard, F, 2009.)

Both sampling modes are defensible, but if one has numerous samples it is probably better to use random systematic sampling. If only a minimum number of samples are required stratified random sampling may suffice.

10.3.2 Sampling Selection Modes - One-Dimensional Lots (Streams)

i. Random systematic mode for a stream

Random systematic is the most popular and most reproducible selection mode. In the presence of regular cycles this mode is to be avoided as sampling in phase means it is unlikely that you will ever identify the cycles in the stream. An example of the random systematic mode is shown in **Figure 5.4a.**

ii. Stratified random mode

The stratified random selection mode is used in material balance exercises and is the most accurate selection mode in the presence of cycles. An example of a stratified random mode is shown in **Figure 5.4b.**

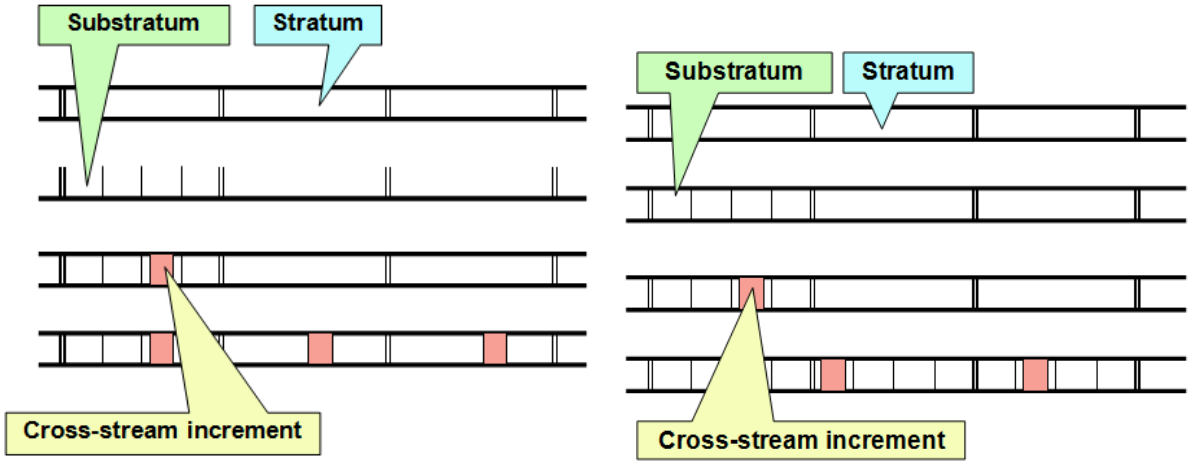


Figure 10.4: Random systematic selection mode for one-dimensional streams; the starting point must be positioned at random within the first stratum, and b) Stratified random selection mode for one-dimensional streams ([40]Pitard, F, 2009.)

11 PROBABILITIES AND SAMPLING DISTRIBUTIONS

Statistical concepts are relevant to the Theory of Sampling and provide insights to sampling distributions. For diamond deposits the statistics of the 'rare event' can be modelled by application of the Poisson's distribution, as can very low-grade particulate materials. The Poisson distribution is the limiting case of the binomial distribution, and linkages between the Poisson and lognormal distributions will be discussed.

11.1 Probability

TOSsing coins and dice or selecting red balls from a bag containing red and white balls are the common examples used to describe probabilities. If an event may or may not happen, the probability P(E) that it will happen is evenly distributed between zero and one. A probability of zero is assigned to events that could never happen; the probability that the sun will not rise in the morning is zero, while a value of one is assigned the probability that the sun will rise. A coin TOSsed in the air will come down on its head or tail, so there are only two ways in which it could land, but only one way in which it will land, hence the probability of a head or a tail is a half or 0.5. The total probability for these two TOSses of the coin is 0.5 + 0.5 = 1. Each time a die is thrown there are six probable outcomes; there are six ways it could land but only one way it will land, so there is a probability of 1/6 that any number between one and six will appear. For a one or a six to appear the probability is 2/6 or 1/3. For a bag containing two red and six white balls there is a probability of 2/8 or 0.25 of selecting a red ball and 6/8 or 0.75 of selecting a white ball. These outcomes are the result of *one trail* of a *statistical experiment*, while all the outcomes are referred to as the *sample space*[63]. All the events are based on prior knowledge of potential outcomes within the constraints of the sample space, and are referred to as *a priori* probabilities. In cases where there is no way of knowing the possible limits of the sample space, historical data of similar events is analysed and provides an estimate of the probability, a so-called *empirical probability*[63].

Probabilities are governed by addition and multiplication rules[63]. The ***addition rule*** says that if the probability that a coin will come up heads is ½ and the probability that it will come up tails is ½, the total probability that it will be heads or tails is 1. This rule only applies where the events are mutually exclusive, if one happens the other can't happen. For a bag containing four red balls, six orange balls and eight white balls the probability of choosing a red or an orange ball is:

$$\frac{1}{18}+\frac{1}{18}+\frac{1}{18}+\frac{1}{18}=\frac{4}{18} \text{ for red balls}$$

$$\frac{1}{18}+\frac{1}{18}+\frac{1}{18}+\frac{1}{18}+\frac{1}{18}+\frac{1}{18}=\frac{6}{18} \text{ for orange balls}$$

$$\text{Together it is } \frac{4}{18}+\frac{6}{18}=\frac{10}{18} \text{ or } 0.556$$

[63] Caswell, F.1991. Success in STATISTICS (Second Edition) Coventry Polytechnic, John Murray (Publishers) Ltd. pp. 356.

The ***general addition rule*** applies when two events, A and B can occur independently or simultaneously[63]. A common example is that of drill holes in an oil field. We want to find the probability of either event A or event B occurring when A and B are not mutually exclusive, as well as the probability that both occur simultaneously. The general rule for the addition of probabilities is:

$$P(\text{A or B}) = P(\text{A}) + P(\text{B}) - P(\text{A and B})$$

As an example say there are 30 boreholes drilled. Ten have oil and 20 are dry, but 4 of the dry holes have gas and 9 of the wet holes have gas. We want to know what the chances are of intersecting a dome that carries both oil and gas. So the probability that the hole will be both wet and gas is:

$$\begin{aligned} P(\text{Wet or Gas}) &= P(\text{Wet}) + P(\text{Gas}) - P(\text{Wet and Gas}) \\ &= \frac{10}{30} + \frac{13}{30} - \frac{4}{30} \\ &= 0.63 \end{aligned}$$

The ***multiplication rule*** applies when the final outcome is a compound result of other simpler outcomes that are not independent. An obvious example is the chance of throwing a six, three times in a row. A dice can end up six different ways (1/6) in one throw and the rule states that the probability of two or more independent events occurring is the product of their individual probabilities. In general the form of the multiplication rule is:

$$P(\text{A and B}) = P(\text{A}) \times P(\text{A}/\text{B})$$

You have a slim chance of throwing three sixes in a row:

$$\frac{1}{6} \times \frac{1}{6} \times \frac{1}{6} = \frac{1}{216} = 0.0046$$

In metal reduction plant a cross-stream sampler collects 20 increments before sending them to the lab for analysis. In the first sample, five of the increments are not representative because of bridging of the material across the cutters of the sampler. What is the probability that the sample is representative? In a second round of increment collection 3 of the 20 increments are not representative because of a mechanical problem. If increments are collected and examined before they are combined into a single sample, what are the chances that both increments will not be representative? For the first batch:

$$\begin{aligned} P(\text{Unrepresentative}) &= \frac{5}{20} = 0.25 \\ P(\text{Representative}) &= 1 - 0.25 = 0.75 \end{aligned}$$

For the second batch:

$$P(\text{Unrepresentative}) = \frac{3}{20} = 0.15$$
$$P(\text{Representative}) = 1 - 0.15 = 0.85$$

Since the events are independent, the chance of selecting two increments that are unrepresentative is:

$$P(\text{Unrepresentative}) = 0.25 \times 0.15 = 0.0375$$

11.2 Binomial Distribution

A binomial distribution is applicable in a case where a mixture of minerals is made of two different components that occur as discrete grains, particles or clusters in roughly equivalent proportions. If the number of particles or clusters is very large the distribution may appear continuous. If the outcome of a trial in an experiment is mutually exclusive such that it is either heads or tails, a dry hole or wet hole, etc., the binomial distribution tells us the probability of obtaining 5 heads if a coin is TOSsed a dozen times or the probability of three holes being dry if we drill ten oil wells in a field of known prospectivity. According to Caswell[63] the binomial distribution is useful in situations where there is a fixed number of trials and the number of successes or failures is counted The binomial distribution of oil wells has only two mutually exclusive outcomes, the well is either wet, usually termed success, or dry, termed failure, and if the probability of success of one trail is p then the probability of failure is q, so that p+q=1. If n boreholes are drilled, the probabilities of success are given by the successive terms in the binomial expansion of the term $(p+q)^n$, and the probability of x successes in n trials is given by:

$$P(x) = \binom{n}{x} q^{n-x} p^x \text{ for } x = 0,1,2,.....n$$ with p and q remaining constant from one trial to the next.

In an oil field that is traditionally known to have a success rate of 33.3% we want to know the chances of drilling a wet hole if we budget to drill 12 holes. A hole is either wet or dry:

$$P(\text{dry}) = 33.33\% = 0.3333 = q$$
$$P(\text{wet}) = 1 - 0.3333 = 0.6666 = p$$

We don't want more than 4 dry holes in 12:

$$P(\text{zero or 3 dry}) = P(X = 0 \text{ or } 3)$$
$$= P(X = 0) + P(X = 1) + P(X = 2) + P(X = 3)$$
$$P(X) = \binom{n}{x} q^{n-x} p^x$$
$$= \binom{12}{x} (0.666)^{12-x} (0.333)^x$$

What is the probability that these wells will be dry?

$$P(X=0)=\binom{12}{0}(0.666)^{12}(0.333)^{0}$$
$$=\frac{12!}{0!(12-0)!}\times(0.666)^{12}\times 1$$
$$=\frac{12!}{1\times 12!}\times(0.666)^{12}$$
$$=1\times(0.666)^{12}$$
$$=0.0076$$

What is the probability of exactly one successful hole?

$$P(X=1)=\binom{12}{1}(0.666)^{11}(0.333)^{1}$$
$$=\frac{12!}{1!(12-1)!}\times(0.666)^{11}\times(0.333)$$
$$=\frac{12!}{1\times 11!}\times(0.666)^{11}\times(0.333)$$
$$=12\times(0.666)^{11}\times(0.333)$$
$$=0.0304$$

What is the probability of exactly two successful holes?

$$P(X=2)=\binom{12}{2}(0.666)^{10}(0.333)^{2}$$
$$=\frac{12!}{2!(12-2)!}\times(0.666)^{10}\times(0.333)^{2}$$
$$=\frac{12!}{1\times 10!}\times(0.666)^{10}\times(0.333)^{2}$$
$$=12\times 11\times(0.666)^{10}\times(0.333)^{2}$$
$$=0.2513$$

So the cumulative probability for exactly three holes is:

$$P(0)+P(1)+P(2)+P(3)=0.289$$

We conclude that the probability that there will be three dry holes in this field is 28.9% if 12 holes are drilled. Furthermore the probability for more than three successes is:

$$P(>3)=(1-0.289)$$
$$=0.711$$

11.3 Poisson distribution: The limiting case of the Binomial Distribution

When dealing with components in a mixture and if the number of particles is very large the distribution may appear continuous. The shape of the distribution of assay grades will depend on the relative proportions of

the two components in the original material. Let's call the proportion of clusters (or nuggets) of a constituent of interest p, then the proportion of the other constituent q, is given by q = 1 – p. N is number of samples taken from the lot, with replacement the probability in this case is:

$$P(x) = \left[\frac{n!}{x!(n-x)!} \right] \times p \times q^{(n-x)}$$

Where the variance = npq and the mean value = np. For our purposes this may not appear very interesting, but according to Pitard (1993)[64] the Poisson distribution is a limiting case of the binomial distribution for binomial probabilities when p becomes very small i.e. $P \leq 0.5$, and occurs where the component of interest occurs in trace amounts or very low grade in the lot relative to the other constituents.

Where one of the constituents occurs as discrete grains relative to the other and the proportion of one constituent is very small in relation to the other there is a transition from the binomial distribution to the Poisson distribution. Gold ore for example, where the gold occurs as a trace element constituent in relation to the gangue, is a limiting case and is best represented by the Poisson distribution.

11.4 Poisson's Distribution

The Poisson distribution can be used to describe the occurrence of rare events such as natural disasters and extreme value statistics, but for this distribution to be applicable the mean should be a constant. Samples yielding discrete, but generally low values of a random variable (i.e. alluvial gravels along ancient watercourses containing nuggets of gold; diamonds in perched river terrace gravels; coastal marine gravels containing diamonds; rare indicator minerals in river gravels, or trace minerals in thin sections) are distributed according to Poisson's distribution. Experiments that yield numerical values for a random variable *r*, the number of events occurring either during a given time interval or within a specified region, are Poisson experiments. The time interval could be any unit of time, seconds, minutes, hours, days, months, years, while the region could be a line segment, an area, or a volume. In the first case *r* might be the number of vehicles crossing an intersection per hour, the number of tourists visiting a game park per year, while in the second *r* may represent the number of breaks in an oil/gas pipeline, the number of typing errors per page, or the number of defects in a block of granite. There is no upper limit to the value for r, but as it becomes very large the Poisson probability becomes negligible. The Poisson distribution is strongly positively skewed at a mean of 0.1 and changes shape rapidly for mean values between 0.1 and 2, becoming more and more symmetrical as the mean rises beyond 2. This distribution is generally thought of as a discrete distribution, applicable when the critical component of interest is present in trace amounts, very low concentrations or as isolated or low-frequency grains randomly distributed in a very much larger

[64] Pitard, F, 1993. *Pierre Gy's Sampling Theory and Sampling Practice*, Second Edition (CRC Press: Boca Raton).

volume of gangue[65], an attribute which Stanley[66] (1998) refers to as "the rare grain effect". According to Pitard[64], Poisson's distribution is a limiting case of the binomial distribution for probabilities $P \leq 0.5$ when $n \sim$ infinity, is such that np is constant. Sinclair and Blackwell[67] (2002) stated that Poisson's distribution can be used to approximate binomial distribution where p is very small and n is very large. The Poisson distribution has the following form:

$$P(X = r) = \frac{z^r \, e^{-z}}{r!} \text{ where } r = 0, 1, 2, 3....$$

And where z is the hypothetical average number of nuggets per sample, r is any specific number of samples (r = 1, 2, 3,…n), and P(X=r) is the probability that r nuggets will occur in the sample, and the value of e is 2.7183. Worded another way: the Poisson distribution gives the probability P_r of finding r grains in ***a single sample*** when the average number of grains per sample is z[65]. For a typically low grade diamond deposit in which the diamonds are discrete stones, the Poisson's distribution gives the probability of finding n diamonds in a single sample when the average number of diamonds per sample is z. Typical examples of low-frequency type materials in the mineral industry are alluvial gravels along ancient watercourses containing nuggets of gold, or costal marine gravels containing diamonds. In fact Gilfillan[68] has noted that alluvial deposit pose special problems, usually being overvaluation, because they are evaluated using selected and unrepresentative samples that rely on visual estimation of specific heavy minerals rather than scientific determination. As the sample size decreases the probability of capturing a gold nugget or a diamond in the sample decreases dramatically. However if a diamond or nugget is found in the sample the unknown average grade is so high that the result is considered unrealistic. Typical Poisson distributions for different values of z (the hypothetical number of nuggets in a sample) are shown in Figure 5.1. It is interesting to note that by the time z = 7 the distribution is indistinguishable from a normal distribution.

[65] Ingamells, C.O. and Pitard, F.F. 1986. Applied Geochemical Analysis. A series of Monographs on Analytical Chemistry and its Applications, Volume 88. A Wiley-Interscience Publication, John Wiley and Sons, Inc., New York. pp. 733.
[66] Stanley, C.R., 1998. NUGGET: PC-software to calculate parameters for samples and elements affected by the nugget effect: In: M. Vallee and A.J. Sinclair (Eds.), Quality assurance continuous quality improvement and standards in mineral resource estimation: Exploration and Mining Geology, Vol.7, No. 1 and 2: p139-148.
[67] Sinclair, A.J. and Blackwell, G. H. 2002.Applied Mineral Inventory Estimation, Cambridge University Press. 381p.
[68] Gilfillan, J. F. 2001. The resource database audit. In (Ed: A. C. Edwards) *Mineral Resource and Ore Reserve Estimation – The AusIMM Guide to Best Practice*. Australian Institute of Mining and Metallurgy, Melbourne. Pp91-96.

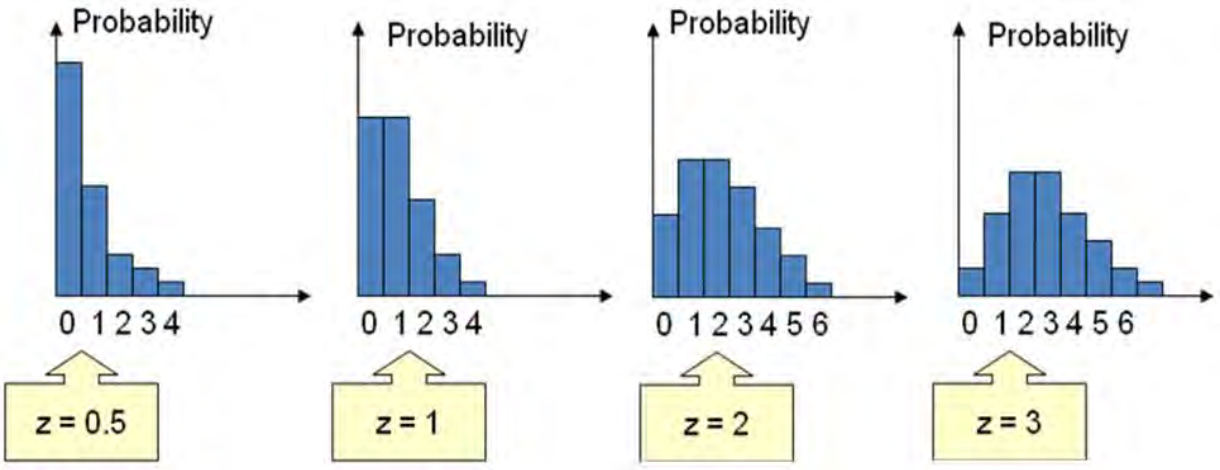


Figure 11.1: Typical Poisson histograms for different values of z. Source: Pitard 2008

The histograms in Figure 10.1 indicate that if the average number of nuggets is 0.5 the chances of getting no nuggets per sample is high while the probability of getting 4 nuggets is relatively small. When z = 1 the probability of getting 0 or 1 nuggets per sample is exactly the same, as is the probability of getting 2 or 3 nuggets per sample when z = 3. The shape of the Poisson and lognormal distributions is almost identical when z is small, and more often than not a lognormal distribution is assumed when it is in fact a Poisson distribution (Pitard, 2007). Characteristics of Poisson's distribution worth noting are firstly that the number of grains per sample is independent of that found in any other sample, secondly that the probability of a single grain occurring in a sample is proportional to the size of the sample, and finally that the probability of more than one grain in a very small sample is negligible.

Poisson's distribution is a very simple but convenient distribution and has the remarkable feature that the ***mean is equal to the variance***, both having the value *z*, which is equal to the hypothetical average number of grains per sample. Equality between the mean and the variance implies a Poisson distribution, but Miller and Kahn[69] (1962, p.378) noted that a mean/variance ratio greater than 1 suggests a clustering effect amongst the grains, whilst a ratio less than unity suggests regular dispersion of, or a repelling amongst, the components of interest.

Poisson's distribution also has the characteristic that the ***standard deviation of the number of grains per average sample n equals the square root of that number***, so that: $\sigma_z = \sqrt{z}$, and the variance is then given by $\sigma^2_z = z$ and according to Stanley[66] the precision is given by what he calls "the two standard deviation coefficient of variation" (pg 4):

$$\text{Precision} = \text{CoV} = \frac{\sigma}{z} = \frac{\sqrt{z}}{z} = \frac{200}{\sqrt{z}}$$

[69] Miller, R.L., and Kahn, J.S. 1962. Statistical analysis in the geological sciences: John Wiley and Sons Inc., New York: 483p.

If we multiply both sides of the standard deviation equation by c, the contribution of a single grain to an assay value, the standard deviation of the assay values is given by:

$$c\sigma_z = \sigma = c\sqrt{z}$$

And the variance is given by:

$$(\sigma^*)^2 = s^2 = c^2 z$$

Thus if the number of grains per sample decreases the estimation of σ^* becomes very unstable, so that for the estimated standard deviation s, to have meaning the number of samples analysed must be very large as n decreases.

11.5 Normal distribution

It is not the intention here to deal exhaustively with the normal distribution as it is well known and details are available in almost any text book. In this text the main aspects of the Normal distribution as they relate to sampling problems in the minerals industry will be covered. In particular there are two aspects, the Central Limit Theorem (CLT) which states that the distribution of the means from repeated sampling of any distribution will be Normally distributed, and the Standard Normal Distribution (SND) which together provide a basis for the determination of optimal sample sizes.

Provided the two questions, “What level of confidence is required?” and “What is the largest allowable difference (D) between the estimated mean and the true value of the mean?” can be answered, the necessary sample size can be calculated. The minimum sample size required to estimate the mean at a specified level of confidence is given by the equation:

$$n = \frac{z_\alpha^2 \times \sigma^2}{D^2}$$

A slightly modified example for the ferrochrome industry is taken from Meyers (1997) and assumes that the average value for vanadium in ferrochrome is found to be 480ppm with a standard deviation of 35ppm, using 6 samples. Customer specifications insist that a load of ferrochrome shall not exceed 500ppm V_2O_5. Management want to know how confident the mill is about the average vanadium content at the current rate of sampling, and how many samples are required if the error on the mean must be less than 35ppm, with 95% confidence that the true mean is less than 500ppm.

A 95% confidence interval equates to a z-value of 1.645 from the SND tables and making appropriate substitutions in the above equation gives:

$$n = \frac{z_\alpha^2 \times \sigma^2}{D^2}$$
$$= \frac{(1.645)^2 \times (35)^2}{(500-480)^2}$$
$$= \frac{3314.8}{400}$$
$$= 8.3 \text{ say } 9 \text{ samples.}$$

In such a case there needs to be an increase in the number of samples taken because there is insufficient confidence in the mean value.

11.6 Lognormal Distribution

11.6.1 Testing for lognormal distributions

It is essential that any data from a mineral deposit be examined to determine the parent population distribution from which the samples are drawn. Samples from a parent distribution, suspected to be lognormal, can be checked using one of three methods.

i. Simply inspect the data. If there are many small values and a few high values these could indicate a lognormal distribution.
ii. Calculate the mean and the standard deviation and then calculate the coefficient of variation, $\text{CoV} = \sqrt{V}/\bar{g}$. If CoV < 0.333 the samples probably come from a normal distribution. If CoV > 0.333, then any suspicions would be reinforced as to the values coming from a lognormal distribution.
iii. Plotting a probability curve that results in a straight line is a clear indication that the data is drawn from a lognormal population.

It is now possible to determine all the necessary population parameters for the GASA reef (Table 10.1) grade data in logarithmic terms

$$\text{Logrithmic mean} = 1.6537 \log_e \text{g/t}$$
$$\text{Logrithmic Std Dev} = 1.5708 \log_e \text{g/t}$$

For large numbers of samples, n>40, calculate τ^* the mean of a lognormal distribution

$$\tau^* = e^{\mu^* + 0.5(\sigma^*)^2}$$

$$\mu^* = \bar{y} = \text{estimate of logarithmic mean, and}$$
$$(\sigma^*)^2 = \text{estimate of logarithmic population variance}$$

The GASA set contains only 27 pieces of information (n<40), but simply for purposes of illustration the mean for the GASA data is:

$\tau^* = e^{1.6537+0.5(1.5708)^2} = 17.95\ g/t\ Au$. Compare this result with the arithmetic mean, 17.89 g/t Au.

Table 11.1: GASA Gold Data*

Data	Reef Grade	log e Grade
1	0.13	-2.04
2	0.53	-0.63
3	0.63	-0.46
4	0.94	-0.06
5	1.52	0.42
6	1.97	0.68
7	2.04	0.71
8	2.56	0.94
9	2.65	0.97
10	3.60	1.28
11	3.73	1.32
12	3.80	1.34
13	5.11	1.63
14	5.45	1.70
15	5.98	1.79
16	6.28	1.84
17	6.44	1.86
18	7.52	2.02
19	7.92	2.07
20	8.82	2.18
21	9.51	2.25
22	13.58	2.61
23	28.59	3.35
24	32.50	3.48
25	47.90	3.87
26	68.66	4.23
27	204.56	5.32
Mean	**17.89**	**1.65**
Var		**2.38**
Std Dev		**1.54**
Pop Var		**2.47**
Log Pop SD		**1.57**

** This is part of a set of data that was compiled by the Geostatistical Society of South Africa for use among geostatisticians as a standard test set for comparing statistical concepts.*

For small numbers of samples, n<40, use the so-called "Sichel's t estimator of the mean". By this method we calculate the median and then add an amount that depends on the number of samples being used, to bring it to the mean. Sichel's t estimator is given by:

$$t = e^{\mu^*} \times \gamma_n(V)\ g/t\ Au$$

The term γn(V) is the equivalent part that is added to the median to get to the mean. It is necessary to interpolate values of γn (V) from Sichel's tables. It is also important to remember to use logarithmic sample variance. Sichel calculated his tables in terms of sample variance, not population variance. Although he developed a set of tables it is necessary to interpolate between values to get the factor γn (V) for estimating the mean. Using a value of 3.1326 for γn(V):

$$t = e^{\mu^*} \times \gamma_n(V)\ g/t\ Au$$
$$= e^{1.6537} \times 3.1326$$
$$= 16.37\ g/t\,Au$$

Typically the frequency histogram of precious metal values is positively skewed giving rise to lognormal distribution, such as is shown in Figure 10.2. Although the parameters of the frequency distribution may differ from mine to mine, the basic lognormal characteristics are inherent. According to Krige (1994) the occurrence of a definite shape in the distribution of sample values in gold deposits was first discovered by Watermeyer[70] in 1919, but it was only in the late 1940's that Herbert Sichel identified it as lognormal distribution. This distribution is positively skewed, the tail being dragged out to the higher values.

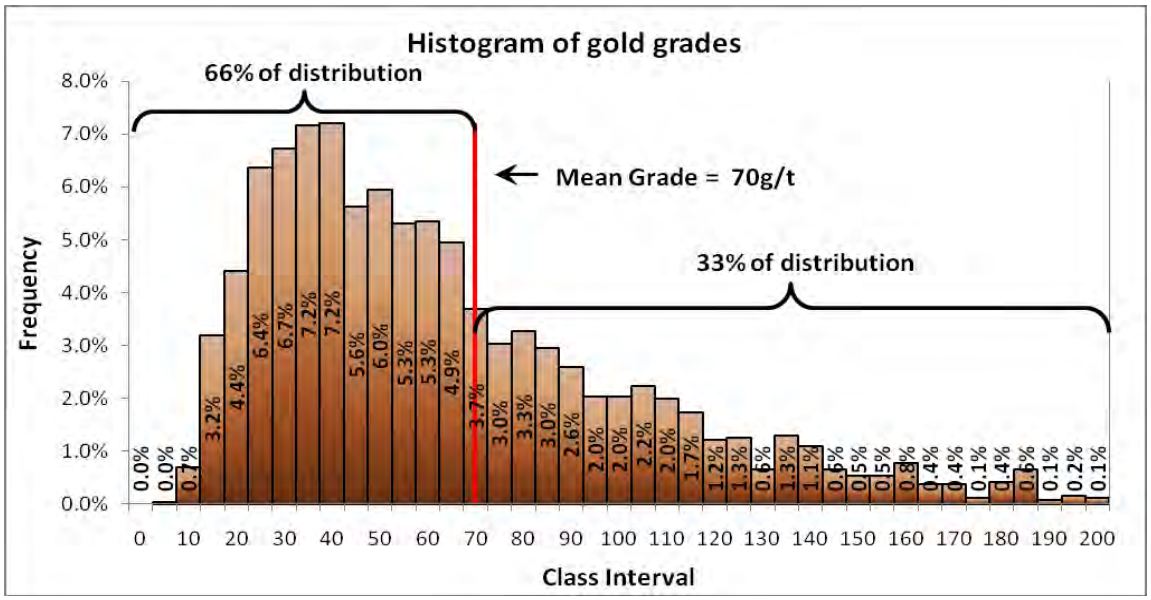


Figure 11.2: Lognormal frequency distribution for 1000 values showing that 2/3 of the distribution lies below the mean

Lognormal distribution can be completely described by the logarithmic mean and the logarithmic standard deviation, the mean lying at the centre of gravity of the distribution. A significant feature of lognormal distribution is that the shape of the curve for samples (point support) is the same as that for the grade distribution of larger blocks of ore, except that the variability of the point samples is much greater than that for the blocks. However, Krige (1994) also showed that the relative variations between blocks of ore of equal size (areal extent) are fairly stable and possibly constant. Thus, the size of the sample does not affect the overall lognormal distribution pattern of the gold values.

Lognormal distribution has specific problems with regard to sampling in that the selection of one sample from the distribution (shown in **Figure 10.2**) has a 66% chance of being less than the mean. There is, therefore, a 33% chance that when sampling is above the mean the value will be so high that re-sampling will be necessary. The probability of collecting a sample from a particular grade category is shown in **Figure 10.2.** Thus there is a 7.2% chance that the grade will be in the range 30 to 35 g/t Au.

[70] Watermeyer, G.A. 1919. Application of the theory of probability in the determination of ore reserves. Journal of the Chemical, Metallurgical and Mining Society of South Africa, Vol. 19. January, 1919.

In low-grade blocks there will inevitably be some high-grade values reflected on the assay sheet. There will also be some low grades, even when sampling a high-grade block of ore. Because any sampling event draws only a limited number of samples from a population with an infinite number of samples, the likelihood of finding a set of duplicate samples with identical values, even if re-sampled in exactly the same channel, is highly unlikely.

The greater the number of samples used to calculate the average, the closer will be the estimated average to the true average of the population. Again, because the distribution is lognormal there will be occasions when the sample suite contains some values so extreme that they could seriously affect the arithmetic mean of the values. The simplest way of dealing with such "outliers" is to cut them, reducing their value to a more acceptable level (despite the absence of hard and fast rules as to how this should be done). Geostatistical purists abhor such practices, claiming they are interference with the data and as serious an offence as increasing values thought to be too low. Another way of mitigating the influence of very high values is to use the geometric rather than the arithmetic mean.

11.6.2 Sampling in lognormal distributions

The arithmetic mean of n underground samples is an unbiased estimator of the population mean. It is almost invariable that the physical process of sampling introduces a bias into the population of actual values and is a major contributor to a low Mine Call Factor71 (MCF). Assume that a sample is to be taken at random in a block of ore for which the cut-off grade is 1.7g/t gold with a frequency of distribution of gold grades as shown in Table 6.2. If the grade to be sampled is close to the cut-off, the chances that the grade will be fall on either side of the mean are not the same. There is 4.33% chance that the grade will lie in the range1.5 to 1.7g/t and a 3.42% chance that it will lie in the range 1.7 to 1.9g/t. If the range is expanded to 0.8g/t, there is a 13% chance that the grade lies between 1.7 and 2.5g/t, and a 26% chance that it lies between 0.9 and 1.7g/t. There is exactly twice the chance that the block of ground will be sent to the waste dump rather than to the mill. In the range of grade categories 40% above and below the cut-off grade, the distribution frequency is about 40% (26.18 plus 13.09). The chances of not exceeding a 40% error are about four in ten. In other words, six samples out of ten will be in error by more than 40% of the true, mean value of the block of ore. Thus according to Krige72 (1994) the practice of assigning a block of ground in an open pit to the waste dumps or the mill on the basis of one sample alone is unfounded.

Table11.2: Per cent frequency distribution for different gold grade categories

Grade (g/t Au) as a % of mean block value	% Frequency
0.00%	0.00%
11.85%	4.93%
23.69%	12.89%

[71] Krige, D. G. 1962. Statistical applications in mine valuation. Journal South African Institute of Mining and Metallurgy, January 1962.

[72] Krige, D.G. 1994. A statistical approach to some basic mine valuation problems on the Witwatersrand. Journal South African Institute of Mining and Metallurgy, March 1994. pp. 95-111. First Published Dec. 1951. Vol. 52, No.6. pp 119-139.

35.54%	12.59%		
47.38%	11.18%		
59.23%	8.16%		
71.07%	7.25%		
82.92%	6.45%	**26.18%**	
94.76%	4.33%		**CUT-OFF GRADE = 1.7 g/t**
106.61%	3.42%		
118.46%	4.63%		
130.30%	3.02%	**13.09%**	
142.15%	2.01%		
153.99%	1.81%		
165.84%	2.01%		
177.68%	1.21%		
189.53%	1.41%		
201.37%	1.41%		
213.22%	1.21%		

11.7 The Sample Grade Distribution

According to Pitard (1993)[73] the theory of sampling is a preventive tool which is like an "insurance policy" against major misunderstandings. Ambiguity is avoided if sampling is undertaken in such a way that the grade distribution can be assumed to be Normal. This is not always possible, especially for trace and precious metals, where the grade is likely to follow a Poisson distribution. If the grade follows a Poisson distribution, the average will be an unbiased estimator of the real mean a_L of the lot, assuming that the sampling operation was handled correctly. If the constitution heterogeneity is large, and few data are available, the apparent average drifts toward the mode of the distribution. If the sample grade as is normally distributed in the absence of systematic sampling error, the average grade of the samples can be taken as the average grade of the initial lot. A systematic sampling error is prevented if the following conditions are fulfilled:

- The entire lot is freely accessible to the sampling tool, so that an equal chance for each constituent of the lot to be part of the sample exists.
- The sampling scheme is impartial, so that an equal chance for each constituent to be part of the sample exists.
- The distribution of the grade as of the sample obeys a normal distribution, which is an optimistic assumption in the case of trace elements.

[73] Pitard, F.F. 1993. Pierre Gy's Sampling Theory and Sampling Practice. Heterogeneity, Sampling Correctness and Statistical Process Control. CRC Press, , New York. 488p.

12 SAMPLING EXPERIMENTS IN THE POISSON DISTRIBUTION[74]

12.1 Poisson Distribution Sampling Experiment using a Silica-Chromite mixture

This demonstration uses a mixture of silica sand and chromite to illustrate the value of sampling theory to develop optimised sampling protocols and evaluate uncertainties. For example a series of assays may be used to estimate grade, particle size, distribution, and the degree of liberation. The mixture of grains has the characteristics listed in Table 7.1.

Table 11.1: Characteristics of the Silica Sand- Chromite mixture

Characteristic	Silica Sand	Chromite
Density	2.65	4.6
Colour	White	Black
Grain size	-500, +250 microns	-710, +500 microns
Assume density	9.0g/cc (~Base metal density)	18.0g/cc (~Gold density)
Assumed gold content	0.1%Au	70%Au
Average gold grade for mixture	0.45%Au	

The mixture is thoroughly mixed, but there is evidence of grouping and segregation in the experiment. There is sufficient silica sand and chromite to provide 100 spoonful's of the mixture, the mass of each spoonful being about 13.25g.

Table 12.2: Data from the Silica Sand – Chromite experiment

In Table 7.1 the actual density is about 2.65g/cc, so the actual volume of the spoon is about 4.9cc, say 5cc. Each participant collects two spoonful's of the mixture without trying to select black grains, there being sufficient material for 100 spoonful's. Each person should count the number of chromite grains in each spoonful and the data for the experiment are listed in Table 7.2.

[74] Ingamells, C.O. Sampling Demonstration, AMAX Research and Development Laboratory, 5950 McIntyre Street, Golden, Colorado 80401. USA. This work was presented by Oliver Ingamells at the IPMI Symposium, San Francisco, March 18-19, 1980.

We now draw two Poisson histograms; one for the Observed Distribution from our experimental data and one for the theoretically Expected Distribution with a mean of 1.27. The hypothetical average number of chromite grains in the sample is given by **z**, and **P(x = r)** is the probability of **r** nuggets or particles appearing in a sample. We then compare them to see if there is statistical evidence to prove that the sampling of this mixture does in fact follow a Poisson distribution.

Table 12.3: Poisson statistics for nuggety gold ores

r	$P(x=r)=\frac{z^r}{r!}e^{-z}$	Percentage
0	$P(x=r)=\frac{z^r}{r!}e^{-z}=-e^{-}=$	% should have 0 gold particles
1	$P(x=r)=\frac{z^r}{r!}e^{-z}=-e^{-}=$	% should have 1 gold particles
2	$P(x=r)=\frac{z^r}{r!}e^{-z}=-e^{-}=$	% should have 2 gold particles
3	$P(x=r)=\frac{z^r}{r!}e^{-z}=-e^{-}=$	% should have 3 gold particles
4	$P(x=r)=\frac{z^r}{r!}e^{-z}=-e^{-}=$	% should have 4 gold particles
5	$P(x=r)=\frac{z^r}{r!}e^{-z}=-e^{-}=$	% should have 5 gold particles
6	$P(x=r)=\frac{z^r}{r!}e^{-z}=-e^{-}=$	% should have 6 gold particles

Having compiled this data we can now draw this Poisson distribution as a histogram of Observed outcomes. Leave enough space to draw the histogram of Expected outcomes for comparison sake.

Table 12.4: Results of sampling experiment using 100 samples of one spoonful each

No of Samples	Expected No of grains/spoonful	Observed No of grains/spoonful	Total No of Grains
0	28.08	27	0
1	35.67	35	34
2	22.65	24	48
3	9.59	11	30
4	3.04	1	4
5	0.77	1	5
6	0.16	1	6
Total			127

In order to make a meaningful comparison between the two histograms we calculate a χ^2 **goodness of fit statistic.** The χ^2 goodness of fit statistic is essentially a comparison of the two histograms shown in Figure 12.1.

$$\chi^2=\frac{(28.08-27.0)^2}{28.08}+\frac{(35.67-35.0)^2}{35.67}+\frac{(22.65-24.0)^2}{22.65}+\frac{(9.58-11.0)^2}{9.58}+\frac{(3.04-1)^2}{3.04}+\frac{(0.937-2)^2}{0.937}=1.77$$

The test statistic has 2 degrees of freedom ν = n - 2 degrees of freedom so ν = 4, where ν is the number of categories (one degree of freedom is lost because expected frequencies are constrained to sum to 12, and a second degree of freedom is required for estimation of the parameter λ. For c = 5 categories there are three 3 degrees of freedom. The critical value for χ^2 for ν = 4 and P = 0.05 is 9.488. The test statics at 1.77 is much less than this value so we accept the hypothesis of equality between the expected and observed distributions and conclude that the Poisson distribution is an appropriate model for this distribution. The selection of chromite nuggets has been randomly made within the lot from which aliquots are selected.

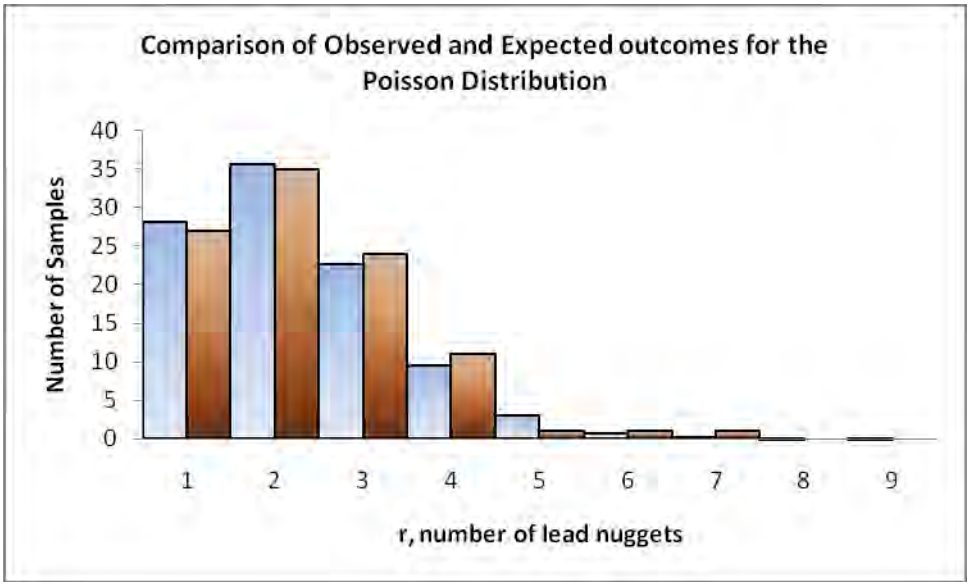


Figure 12.1: Comparison of Observed and Expected outcomes for the Poisson Distrbution

Average grade for the 100 samples is = 127/100 = 1.27 grains/sample and the average overall grade of one spoonful (and the total mixture) is calculated as 0.127%Au. So in each sample (one spoonful) the contribution to the average grade of 0.127%Au is split in the following way:

- 0.001% Au is due to the background grade in the rice;
- 0.126% Au is due to 1.27 chromite grains per sample.

Since 1.27 chromite grains contribute 0.126%Au, one chromite grain contributes 0.126/1.27 = 0.09921 %Au to the overall grade of the mixture in each sample. This is C = 0.09921%Au, the “contribution of a single chromite grain”. As an approximation, the number of rice grains is so large that replacing one of them by a single chromite grain will not appreciably reduce their number or alter the mass of the rice grains. The overall gold content is X, and the gold content in the gangue is L = 0.001%Au. So the overall gold content can be written as:

$$X = L + CZ \quad (1)$$

If an individual assay is X_i and the number of grains in the individual samples is Z_i then:

$$X_i = L + CZ_i \quad (2)$$

It is thus possible to change grain-counts (Z_i) to average grades (X_i) for the one-spoonful samples.

12.1.1 Example 1: Convert Numbers of Grains to Sample Grade

Let's take our data for the one spoonful samples and calculate X_i from what we know about Z_i. The results are listed in Table 12.3.

Table 12.5: Conversion of numbers of grains to sample grades for the 72 single spoonful samples

Z_i	No of Samples	No of Black grains (Z_i)	Contribution From Gangue	Total grade X_i (%Au)
0	27	0 = 0	0.001	0.001
1	35	1 x 0.09921 = 0.09921	0.001	0.10021
2	24	2 x 0.09921 =0.19842	0.001	0.19942
3	11	3 x 0.09921 = 0.29763	0.001	0.29863
4	1	4x 0.09921 = 0.39684	0.001	0.39784
5	1	5x 0.09921 = 0.49605	0.001	0.49705
6	1	4x 0.09921 = 0.59526	0.001	0.59626

If we are to sub-divide the 100 samples into 25 composites consisting of 4 spoonful's each we get the distribution of grains/ sample shown in Table 12.4.

Table 12.6: Sampling results of experiment using 25 composites of 4 spoonfuls's each

No of Samples	No of grains/spoonful	Total No of Grains
0	0	0
1	1	1
2	3	6
3	5	15
4	2	8
5	3	15
6	2	12
7	3	21
8	4	32
9	0	0
10	1	10
11	1	11
	Total	**131**

Average grade for the 25 samples is = 127/25 = 5.08 grains/sample and the average overall grade of one spoonful (and the total mixture) is calculated as 0.127%Au. So in one spoonful the contribution to the average grade of 0.127%Au is split in the following way:

- 0.001% Au is due to the background grade in the rice;
- 0.126% Au is due to 5 chromite grains.

Since 5 chromite grains contribute 0.126 %Au, one chromite grain contributes 0.126/5 = 0.0252 %Au.

12.1.2 Example 2: Convert Numbers of grains to Sample Grade for Composite Samples

Now let's calculate X_i the sample grade from what we know about Z_i the number of grains, for the 18 composite samples as listed in Table 5.

Table 12.7: Conversion of numbers of grains to sample grades for the 18 composite samples

	No of	No of	Contribution	Total grade
Z_i	Samples	Black grains (Z_i)	from Gangue	X_i (%Au)
0	2	0 x 0.07 = 0.00	+ 0.1	0.100
1	0	0 x 0.07 = 0.00	+ 0.1	= 0.170
2	1	2 x 0.07 = 0.14	+ 0.1	= 0.240
3	1	3 x 0.07 = 0.21	+ 0.1	= 0.310
4	5	4 x 0.07 = 0.28	+ 0.1	= 0.380
5	2	5 x 0.07 = 0.35	+ 0.1	= 4.450
6	2	6 x 0.07 = 0.42	+ 0.1	= 0.520
7	1	7 x 0.07 = 0.49	+ 0.1	= 0.590
8	3	8 x 0.07 = 0.56	+ 0.1	= 0.660
9	0	0	0	= 0.00
10	1	10 x 0.07 = 0.70	+ 0.1	= 0.800

Now suppose the only information we have is the list of assay values given in Table 6 which are divided into two groups; 100 single analyses and 25 composite analyses (average of 4 values).

Table 11.8: 100 single analyses also rearranged as averages for 25 composite samples

0.1002	0.1002	0.1002	0.1994	0.1002	0.1002	0.1994	0.1994	0.2986	0.0010	0.2986	0.1994	0.0010
0.1002	0.0010	0.2986	0.0010	0.1002	0.1994	0.0010	0.0010	0.1002	0.1002	0.1002	0.1994	0.0010
0.1002	0.1994	0.2986	0.1994	0.1002	0.4971	0.1002	0.1002	0.1994	0.0010	0.0010	0.0010	0.1002
0.1002	0.1994	0.2986	0.1994	0.0010	0.0010	0.0010	0.0010	0.1002	0.1002	0.1002	0.0010	0.0010
0.1002	0.1250	0.2490	0.1498	0.0754	0.1994	0.0754	0.0754	0.1746	0.0506	0.1250	0.1002	0.0258

0.0010	0.1002	0.2986	0.1002	0.1002	0.0010	0.1002	0.1002	0.1002	0.0010	0.0010	0.2986
0.1002	0.0010	0.1994	0.1994	0.1994	0.1994	0.1994	0.1994	0.1002	0.1994	0.0010	0.0010
0.0010	0.1002	0.1002	0.1994	0.1994	0.0010	0.1994	0.1002	0.2986	0.2986	0.1002	0.1994
0.1002	0.1002	0.1002	0.1994	0.5963	0.0010	0.0010	0.3978	0.2986	0.2986	0.1994	0.1002
0.0506	0.0754	0.1746	0.1746	0.2738	0.0506	0.1250	0.1994	0.1994	0.1994	0.0754	0.1498

What information can be derived from this data? We might want answers to the following questions:

a. How reliable is a single assay when a 110g sample is taken?
b. What weight of sample should be taken?
c. What proportion of the gold residues in isolated high-Au grains?
d. Is there a problem in terms of segregation?
e. Should one take one large sample for assay or would several smaller samples be better?
f. If the material is a sample of a shipment, how representative of it is the sample?
g. Should the shipment be re-sampled?
h. If so how many samples and of what size should be taken?

These calculations require estimates of parameters to be calculated; if estimates are poor the calculated values may be misleading.

12.2 Calculate the Visman[Error! Bookmark not defined.] Constants

The standard deviations and the average content are calculated from the data in Table 7.6.

The standard deviation S_1 for 100 values = 1.1694 (variance = 1.3676). Mass of samples W_1 = 27g

The standard deviation S_4 for 25 values = 0.0669 (variance = 0.0045). Mass of samples W_4 = 110g

The average gold content for the 100 values is X = 0.127%Au.

12.2.1 Visman Heterogeneity Constant A,

Constant A measures the small-scale non-uniformity of a material, i.e. the nugget effect.

$$\begin{aligned} A &= \frac{W_1 W_4 (S_1^2 - S_4^2)}{(W_4 - W_1)} \\ &= \frac{27 \times 110 \times (1.1694^2 - 0.0669^2)}{(110 - 27)} \\ &= \frac{2970 \times (1.3676 - 0.0045)}{(83)} \\ &= 48.77 \end{aligned}$$

12.2.2 Visman Segregation Constant B,

Constant B measures unmixing or large-scale non-uniformity. A perfectly blended mixture would have B equal to zero.

$$\begin{aligned} B &= S_1^2 - \frac{A}{W_1} = 1.3676 - \frac{48.77}{27} = -0.4463 \\ &= S_4^2 - \frac{A}{W_4} = 0.0045 - \frac{48.77}{110} = -0.4389 \end{aligned}$$

12.3 Calculate Z_i the average number of nuggets per sample

From the list of assays we want to find L, the background Au content of the samples (i.e. the grade of the white grains). We make a best guess that the lowest grades, probably also the most numerous samples with this grade is 0.001 %Au.

If we assume that L = 0.001 %Au, it is possible to calculate the average number, Z, gold-alloy (chromite) grains per 27g sample.

$$Z = \frac{(X-L)^2}{S_1^2}$$
$$= \frac{(0.127-0.001)^2}{1.3676}$$
$$= 0.0116$$

12.4 Calculate the probability that Z is correct

We can now check the assumption that L = 0.100 %Au. The probability that the lowest assay value L = 0.100 %Au represents the gold content of the gangue is:

$$P = 1-(1-e^{-Z})^N$$
$$= 1-(1-e^{-1.27})^{72}$$
$$= 1.0000$$

This probability, equivalent to 100% provides strong support for our assumption that the lowest assay represents the back ground grade L=0.100% and that our estimate for Z = 1.27 is correct.

12.5 Calculate the Poisson probabilities

Poisson probability for samples that contain on average Z nuggets of gold is:

$P = e^{-Z}Z^n / n!$ and for Z = 1.27 the possibilities are listed in Table 7.7.

Table 12.9: Expected Poisson numbers of grains per sample if Z = 1.27

Probability of Grains	P(r)	Expected
0	0.281	20
1	0.357	26
2	0.227	16
3	0.096	7
4	0.030	2
5	0.008	1
6	0.002	
TOTAL	**0.999**	**72**

The total probability adds up to 1.0 so the expected distribution is found by multiplying each probability by 72, the number of samples taken. The expected and observed distributions are compared in Table 7.8. The observed distribution is what we found experimentally, the expected distribution is what was calculated above.

Table 12.10: Comparison of Expected and Observed black grain distribution

No of Samples with Black grains	Expected	Observed
0	20	20
1	26	27

2	16	16
3	7	5
4	2	4
5	1	0
TOTAL	**72**	**72**

From this we can calculate a χ^2 test of goodness of fit which is a comparison of the two these histograms shown in Figure 7.1

12.6 Calculating a χ^2 goodness of fit statistic

The χ^2 goodness of fit statistic is essentially a comparison of the two histograms shown in Figure 7.1.

$$\chi^2 = \frac{(20.0-20.0)^2}{20.0} + \frac{(26.0-27.0)^2}{27.0} + \frac{(16.0-17.0)^2}{16.0} + \frac{(7.0-5.0)^2}{5.0} + \frac{(2.0-4.0)^2}{4.0} + \frac{(1.0-0.0)^2}{0.0}$$
$$= 0.4114$$

The test statistic has v = n -2 degrees of freedom giving 6 -4 = 2 degrees of freedom, where v is the number of categories (one degree of freedom is lost because expected frequencies are constrained to sum to 36, and a second degree of freedom is required for estimation of the parameter λ. For c = 5 categories there are three 3 degrees of freedom. The critical value for χ^2 for v = 4 and x = 0.05 is 9.488. The test statics at 0.4114 is much less than this value so we accept the hypothesis of equality between the expected and observed distributions and conclude that the Poisson distribution is an appropriate model for this distribution. The selection of gold nuggets has been randomly made within the lot from which aliquots are selected.

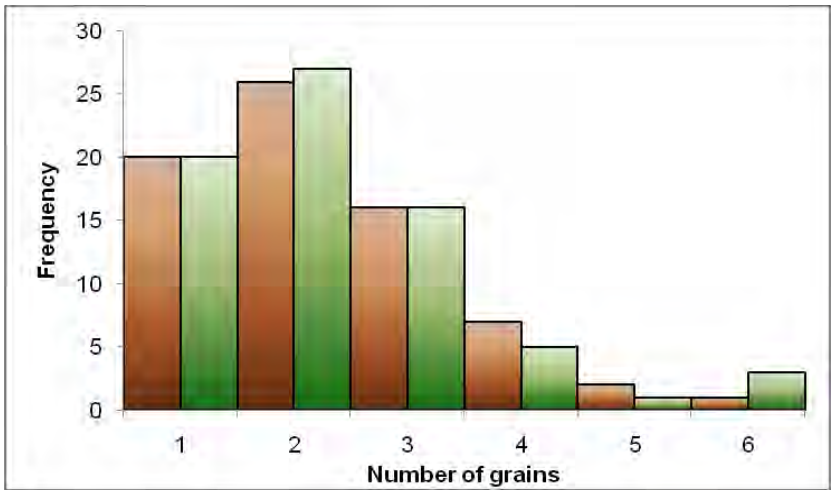


Figure 12.2: Comparison of the Expected and Observed Poisson distributions for the given experiment

12.7 Calculate the contribution c of a single nugget to a single assay

The outstanding characteristic of the Poisson distribution is that the variance is equal to the mean so:

$$S_1^2 = C^2 Z \quad so$$
$$C = \frac{S_1}{\sqrt{Z}}$$

So the contribution of a single nugget there is:

$$C = \frac{S_1}{\sqrt{Z}} = \frac{0.3108}{\sqrt{1.27}} = 0.276$$

This compares well with our earlier estimated value of 0.280.

12.8 Calculate the mesh size d_N of the nuggets

$$A = \rho \times d^3 \times (X - L)(H - L) \quad so$$
$$d_N = \sqrt[3]{\frac{A}{(X - L)(H - L) \times \rho}}$$
$$= \sqrt[3]{\frac{3.4055}{(0.45 - 0.10)(70 - 0.10) \times 18}}$$
$$= 0.198$$

Where A is the Visman constant of heterogeneity, H is the gold content of the black grains and d is their density.

12.9 Calculate the Sampling Constant K_S

12.9.1 From the relative variance of the data (use S_1 and W_1)

$$K_S = R^2 W_1 = \frac{S_1^2}{X^2} W_1 \times 10^4$$
$$= \frac{0.3108^2}{0.45^2} \times 39 \times 10^4$$
$$= 186083$$

Where R(%) is the Relative Variance

12.9.2 From the physical characteristics of the material; the constituent of interest is less than a few per cent of the whole:

$$K_S = \frac{10^4 (X - L)(H - L) \times \rho \times d^3}{X^2}$$
$$= \frac{10^4 (0.45 - 0.10)(70 - 0.10) \times 18 \times 0.198^3}{0.45^2}$$
$$= 168172$$

Note this calculation is very sensitive to the value of u since it is cubed. The sampling constant Ks is defined as the weight in grams which must be taken from a well-mixed mixture to ensure a sampling uncertainty of no more that 1% of the amount present with 68% confidence.

12.10 Calculate the Sample Weight for a Specific Uncertainty

Once the sampling constant K_S has been estimated the sample weight required for a specific uncertainty can be calculated:

$$W = \frac{K_S}{R^2}$$

It is also possible to calculate the uncertainty that exists in a given weight of sample:

$$R = \sqrt{\frac{K_S}{W}}$$

If the sample mass is 20kg the uncertainty associated with the sample is

$$R = \sqrt{\frac{K_S}{W}} = \sqrt{\frac{180000}{20000}} = \pm 3\%$$

That is ±3% of the amount present with 68% confidence or ±9% with 97.5% confidence.

A sample of mass 500g, would have an associated uncertainty of R = √(180000/500) = 19% with 68% confidence, or 57% with 98% confidence. That is repeat assays on other 500g samples would vary from 0.2% to 0.8% even if reduction, sub sampling and assaying were error free.

12.11 Calculate the Increment Mass in order to give an Optimal Total Sample.

The optimal individual sample size is given by $W_{Optimal}$ = A/B = 3.4055/0.0093 = 367g.

12.12 Calculate the Number of Increments for the Optional Sample (using the Optimal Mass)

If n samples of 367g are properly reduced and analysed, the uncertainty (at 68% confidence) in the average of all assays is:

$$S = \pm\sqrt{\frac{A}{NW} + \frac{B}{N}}$$

$$and \quad if \quad W = W_{Optimum} = \frac{A}{B} \quad then$$

$$S = \sqrt{\frac{2B}{N}}$$

If we take a total sample mass of 1000g, composited by means of different numbers of samples, the sampling uncertainty when N increments are composited for the mixture used here, can be calculated.

$$S = \pm\sqrt{\frac{A}{NW} + \frac{B}{N}}$$
$$= \pm\sqrt{\frac{3.406}{1000} + \frac{0.0093}{N}}$$

And a table of values can be calculated as shown in Table 7.8

Table 12.12: Comparison of sampling uncertainty relative to the number of increments collected

No of increments	Sample mass	Sampling uncertainty
1	1000g	0.11
5	200g	0.073
10	100g	0.066
100	10g	0.059
1000	1g	0.058

The change in the sampling uncertainty with increased numbers of increments is shown in Figure 7.2, there being very little reduction beyond 30 samples.

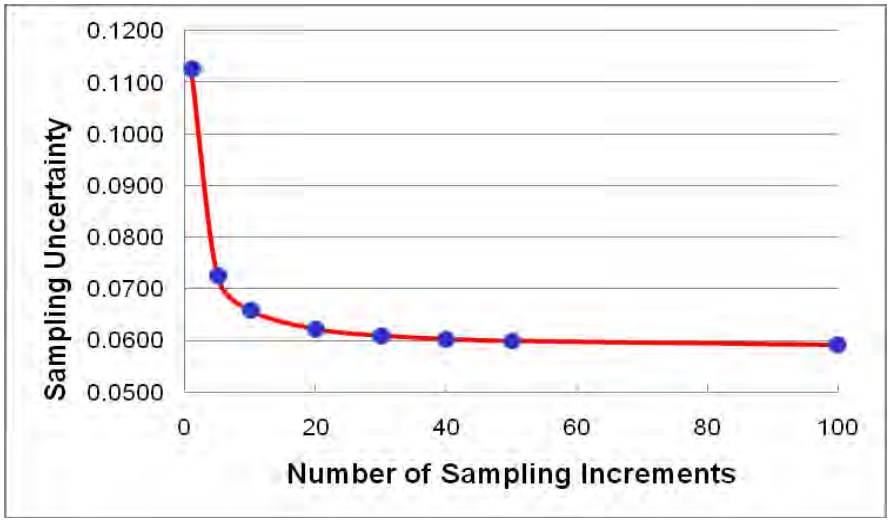


Figure 12.3: Changes in sampling uncertainty with increasing numbers of increments

There is very little additional reduction in sampling uncertainty beyond 30 increments. Using 10 increments of 100g each would be just as good.

12.13 Minimizing Sampling Costs

If 10 samples have to be collected, how much should each sample weigh if we are to minimize sampling costs? If the variable is now the mass (W) with N being a constant at 10 increments, the equation becomes:

$$S = \pm\sqrt{\frac{A}{NW} + \frac{B}{N}}$$
$$= \pm\sqrt{\frac{3.406}{10W} + \frac{0.0093}{10}}$$

If the number of increments is fixed at 10, but the mass of the increments changes, the corresponding change in sampling uncertainty is shown in Table 7.9 and is shown in Figure 7.3.

Table 11.13: Comparison of sampling uncertainty relative to the mass of increments collected

Mass (g)	No of Increments	Sampling Uncertainty
1	10 x 1 g samples	0.58
5	10 x 5 g samples	0.26
10	10 x 10 g samples	0.19
20	10 x 20 g samples	0.13
30	10 x 30 g samples	0.11
40	10 x 40 g samples	0.10
50	10 x 50 g samples	0.09
100	10 x 100 g samples	0.07
200	10 x 200 g samples	0.05

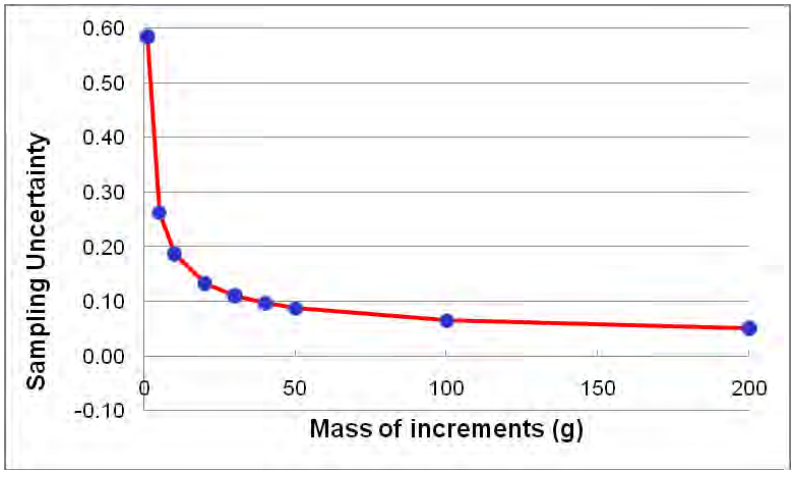


Figure 12.4: Decline in the sampling uncertainty as the mass of the sample increases; the number of increments is fixed at 10

From Figure 12.4 it is evident that there is very little decrease in the sampling uncertainty for sample mass greater than 50g.

12.14 Review of Calculations

H = 70% in nuggets

X = 0.45% Au (Average grade mixture)

S_1 = Std Dev of 39g samples (0.01084%Au)

S_4 = Std Dev of 156 samples (0.0093%Au)

L = Background gold value in gangue (0.100%Au)

From which it is possible to calculate

A = Visman's Homogeneity Constant = 3.406

B = Visman's Segregation Constant = 0.0093

Z = No of nuggets per average sample = 1.27 (actual 1.25)

u = Mesh size of nuggets = 0.198cm (actual 0.168 to 0.238)

K_S =Sampling constant = 180000g

$W_{Optimal}$ = Optimal sample weight = 367g

From this experiment it has also been demonstrated that:

C = Contribution of one black grain to a 39g sample

$$C = \frac{S_1^2}{(X-L)} = \frac{0.3108^2}{(0.45-0.10)} = 0.276$$

W_{min} = Sample weight for Z = 1

$$W_{\min} = \frac{A}{(X-L)^2} = \frac{3.406}{(0.45-0.10)^2} = 27.80$$

Z = Number of black grains per 39g sample

$$Z = \frac{W(X-L)}{(Hu^3 d)} = \frac{39\times(0.45-0.10)}{(70\times 0.198^3 \times 18)} = \frac{13.65}{9.7806} = 1.396$$

Compared to an actual value of 1.25

$$Z = \frac{S_1^2}{C^2} = \frac{0.3108^2}{0.276^2} = 1.268$$

Compared to an actual value of 1.25

N = Total number of grains per 39g sample

$$N = \frac{H\rho_H}{C\rho_L} = \frac{70 \times 18}{0.276 \times 9} = 507 \ \ grains$$

d = Mesh size in cm

$$d = \frac{W_1}{\rho_L N} = \frac{39}{9 \times 507}$$

q_W = Weight proportion of black grains

$$q_W = \frac{(X - L)}{(H - L)} = \frac{(0.45 - 0.10)}{(70 - 0.10)} = 0.005$$

Z_i = Number of black grains in a specific sample

$$Z_i = \frac{(X_i - L)(X - L)}{S_1^2} = \frac{(0.94 - 0.10)(0.45 - 0.10)}{0.3108^2} = \frac{0.294}{0.0966} = 3.04$$

The number of black grains in a specific samples, with given grade are listed in Table 6.10.

Table 12.14: Actual and calculated numbers of black grains in samples of given grade

Xi	Zi (calculated)	Zi (Actual)
0.1	0.000	0
0.38	1.015	1
0.66	2.029	2
0.94	3.044	3
1.22	4.058	4

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PART 2: ESTABLISHING AN OPTIMISED SAMPLING PROTOCOL

13 DETERMINATION OF THE FUNDAMENTAL SAMPLING ERROR (FSE)

13.1 Constitution Heterogeneity (CH)

The Constitutional Heterogeneity, the average variance between fragments within small movable lots of fragmented ore, must be quantified. CH arises due to the difference in composition between individual fragments, the internal variance of the individual fragments being of no consequence here. The between fragment variability in a lot L is schematically shown in ***Figure 13.1***.

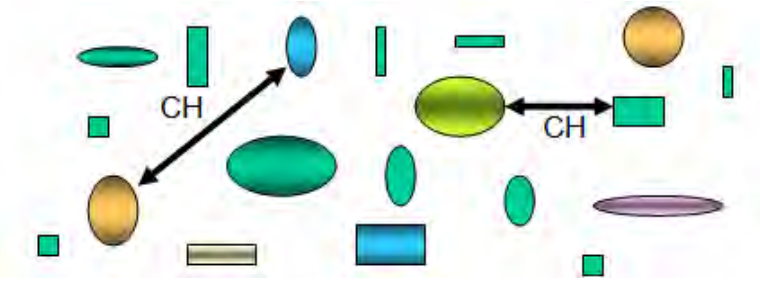

Figure 13.1: The Constitutional Heterogeneity between fragments in the lot must be quantified. **([40]Pitard, F, 2009.)**

13.2 Heterogeneity h_i carried by one fragment

The limiting case of perfect homogeneity, where the grade of any sample is exactly equal to the grade of the lot, can be expressed mathematically as $a_i = a_L$. Heterogeneity h_i is expressed as $a_i \neq a_L$. The greater the difference, the greater is the heterogeneity. The heterogeneity for any individual fragment is the difference between the grade of the fragment and the grade of the lot, $a_i - a_L$, and because it is easier to work with dimensionless numbers we divide by the term a_L to give:

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

The average mass of one fragment is the mass of the lot divided by the number of fragments:

$$\overline{M_i} = \frac{M_L}{N_F}$$

Where:

a_i **-** the content of a given fragment *i*

a_L **.** the average content of the lot *L*

h_i - the heterogeneity carried by a given fragment *i*

M_i - the weight of a given fragment *i*

$\overline{M_i}$ - the average weight of any fragment

M_L - the weight of the lot *L*

N_F - the number of fragments in the lot *L*

On average the mass of the fragment under consideration M_i will not be too different from the average mass of the fragments comprising the lot. The heterogeneity h_i must be adjusted by the mass of the fragment M_i, relative to the mass of the average fragment as follows:

$$h_i = \frac{(a_i - a_L)}{a_L} \times \frac{M_i}{\overline{M_i}}$$

The average h_i carried by a single fragment is therefore:

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

CH_L is the relative, intrinsic dimensionless variance of the heterogeneities of all N_F fragments in the lot.

The ***mean of heterogeneities*** will be zero since some h_i will be positive and some negative so:

$$\overline{CH_L} = \frac{1}{N_F} \sum_{i=1}^{n} h_i = 0$$

The ***variance of heterogeneities*** is the FSE of the material under consideration. Therefore:

$$Var(CH_L) = s^2(h_i) = \frac{1}{N_F} \sum_{i=1}^{n} h_i^2$$

Substitute for h_i to give the CH_L carried by the fragments

$$\begin{aligned} CH_L &= \frac{1}{N_F} \sum_{i=1}^{n} \left(\frac{(a_i - a_L)}{a_L} \times \frac{N_F M_i}{M_L} \right)^2 \\ &= \frac{1}{N_F} \sum_{i=1}^{n} \frac{(a_i - a_L)^2}{a_L{}^2} \times \left(\frac{M_i}{M_L} \right)^2 \times N_F^2 \\ &= N_F \sum_{i=1}^{n} \frac{(a_i - a_L)^2}{a_L{}^2} \times \left(\frac{M_i}{M_L} \right)^2 \end{aligned}$$

13.3 An Estimation of IH_L

Although there is no specific use for this equation it is the basic starting point for the theory of sampling as developed by Pierre Gy. For practical reasons eliminate N_F from the CH_L equation, by multiplying CH_L by a constant factor, M_L/N_F.

$$CH_L \times \frac{M_L}{N_F} = CH_L \overline{M_i} = IH_L$$

where

$$IH_L = \sum_{i=1}^{n} \frac{(a_i - a_L)^2}{a_L^{\,2}} \times \frac{M_i^2}{M_L}$$

Thus CH times the average mass of all fragments is a new identity termed the Intrinsic Heterogeneity IH_L. It is also known as the intrinsic or constant factor of heterogeneity having units of mass, a useful feature when using the FSE for optimisation of the protocol. Later it will be seen that it is equivalent to the terms:

$$Kd_N^{\alpha} \quad and \quad fgc\ell d_N^3$$

13.4 Classifying the lot in terms of Size and Density

According to Neufeld[39] the Intrinsic Heterogeneity IH_L can be determined accurately for any material by separating the material into a two-dimensional grid of volume and density fractions, followed by assaying and weighing each of the fractions. However the use of such an equation is impractical and so a simplified version is used.

IH_L can be modelled in a more user-friendly way by classifying the original lot into different sizes and different density fractions. Imagine that a lot of ore is classified in terms of different size fractions using a large selection of screens along the top row in Figure 7.2. Once this has been done take each size fraction and classify it in terms of density along each of the columns under each of the size fractions in Figure 7.2.

Sizes Densities	α_1 Gangue	α_2	α_3	α_4	α_5	α_6	α_7 Liberated metal	....	Masses β
β_1 Coarse	$\alpha_1\beta_1$	...	...	...	...	...	$\alpha_7\beta_1$	....	...
β_2	...	...	...	...	...	...	...	....	...
β_3	...	...	...	...	...	...	...	....	...
β_4 Medium	$\alpha_4\beta_4$			Fragments: $a_{\alpha\beta}$ ρ_β v_α			$\alpha_7\beta_4$		M_β
β_5	...	...	...	...	...	...	...	....	...
β_6	...	...	...	...	...	...	...	....	...
β_7	...	...	...	...	...	...	...	....	...
β_8 Fine	$\alpha_8\beta_8$	...	...	...	...	...	$\alpha_7\beta_8$	....	...
.....	...	...	...	...	...	...	...	....	...
Masses α	...	...	...	M_α	...	...	...	....	

Figure 13.2: Schematic representation of a lot of crushed ore in different size fractions along the rows and different density fractions along the columns of the matrix. Each square in the matrix has a reference in terms of a combination of grade ($a_{\alpha\beta}$), size (v_α) and density (ρ_β) **([40] Francois-Bongarçon 2012)**

So if a lot is separated into fractions based on size and density, the fragments in any size/density class can be grouped and the average fragment $F_{\alpha\beta}$ in the lot $L_{\alpha\beta}$ can be represented by:

Its volume: $v_\alpha = f_\alpha d_\alpha^3$

Its density: ρ_β

Its average grade: $a_{\alpha\beta}$

Its mass: $M_{F\alpha\beta} = \rho_\beta v_\alpha = \rho_\beta f_\alpha d_\alpha^3$

The average mass of any group ($M_{F\alpha\beta}$) is equal to the mass of the lot ($M_{L\alpha\beta}$) divided by the number of groups ($N_{\alpha\beta}$), and of course the mass is the volume times the density.In each square of the matrix there is a relevant size/density combination or reference in terms of combination of size and density, e.g. $\alpha_2\beta_1$, as shown in ***Figure 13.2***.

13.5 A Simplification of IH_L

Generally the histograms of size along mass and grade distribution in rows ($\alpha_1, \ldots \alpha_n$) are more or less the same, but the histograms of densities along the mass and grade distribution in columns are very different. Experience shows that the grade of the fraction $a_{\alpha\beta}$ varies more from one density fraction to another than from one size fraction to the next. Consequently all the values of the grade $a_{\alpha\beta}$ may be replaced by the average grade a_β of the density fraction L_β and hence: $a_{\alpha\beta} \approx a_\beta$

Also, the ratio of $M_{L\alpha\beta}/M_{L\beta}$ varies little from one density fraction to the next, so replacing the ratio $M_{L\alpha\beta}/M_{L\beta}$ with an average value, $M_{L\alpha}/M_L$ is a good approximation. Because of this we make two simplifications, firstly, the grade depends more on the density $\rho_{\alpha\beta} = \rho_\beta$ than on size $v_{\alpha\beta} = v_\alpha$ so we write $a_{\alpha\beta} \sim a_\beta$, and secondly, the size distribution does not vary much between density classes. In effect this says that the proportion of the size fraction α in the density fraction β is equal to the proportion of the size fraction α in the lot. Therefore we can write:

$$\frac{M_{L\alpha\beta}}{M_{L\beta}} \approx \frac{M_{L\alpha}}{M_L} \;\; so\ we\ write \;\; M_{L\alpha\beta} \approx \frac{M_{L\alpha} M_{L\beta}}{M_L}$$

Using these equalities we can easily make the substitutions resulting in the following the formula:

$$M_{F\alpha\beta} = \frac{M_{L\alpha\beta}}{N_{\alpha\beta}} = v_\alpha \rho_\beta$$

$$IH_L = \sum_{i=1}^{n} \frac{(a_i - a_L)^2}{a_L^{\,2}} \times \frac{M_i^2}{M_L}$$

$$= \sum_{\alpha=1}^{n} \sum_{\beta=1}^{n} (v_{\alpha\beta}\rho_{\alpha\beta}) \left(\frac{(a_{\alpha\beta} - a_L)^2}{a_L^{\,2}} \right) \times \left(\frac{M_{L\alpha\beta}}{M_L} \right)$$

If we use groups of similar fragments, based on their size and their density, rather than individual fragments to approximate the IH_L we can determined IH_L for each of the different size fractions. These results can be summed to give a total value for IH_L:

$$Total\ IH_L = (IH_L)_{\alpha 1} + (IH_L)_{\alpha 2} + (IH_L)_{\alpha 3} + ... + (IH_L)_{\alpha k}$$

where

$$(IH_L)_\alpha = \sum_{\alpha=1}^{n} \sum_{\beta=1}^{n} (v_{\alpha\beta}\rho_{\alpha\beta}) \left(\frac{(a_{\alpha\beta} - a_L)^2}{a_L^{\,2}} \right) \times \left(\frac{M_{L\alpha\beta}}{M_L} \right)$$

This equation is still too complex to be used meaningfully, and the two terms expressed as $\beta\alpha$ must be eliminated, but based on laboratory results and field experience it can be simplified as follows:

$$IH_L = \sum_{\alpha=1}^{n}\sum_{\beta=1}^{n} v_{\alpha\beta}\rho_{\alpha\beta}\left(\frac{(a_{\alpha\beta}-a_L)^2}{a_L^2}\right)\cdot\left(\frac{M_{\alpha\beta}}{M_L}\right)$$

$$= \sum_{\alpha=1}^{n}\sum_{\beta=1}^{n} v_{\alpha}\rho_{\beta}\left(\frac{(a_{\beta}-a_L)^2}{a_L^2}\right)\cdot\left(\frac{M_{\alpha}M_{\beta}}{M_L^2}\right)$$

$$= \sum_{\alpha=1}^{n}\sum_{\beta=1}^{n} v_{\alpha}\left(\frac{M_{\alpha}}{M_L}\right)\cdot\rho_{\beta}\left(\frac{(a_{\beta}-a_L)^2}{a_L^2}\right)\cdot\left(\frac{M_{\beta}}{M_L}\right)$$

$$= \left[\sum_{\alpha=1}^{n} v_{\alpha}\left(\frac{M_{\alpha}}{M_L}\right)\right]\times\left[\sum_{\beta=1}^{n}\rho_{\beta}\left(\frac{(a_{\beta}-a_L)^2}{a_L^2}\right)\cdot\left(\frac{M_{\beta}}{M_L}\right)\right]$$

$\Downarrow$ $\Downarrow$

X : Size fraction Y : Density fraction

This equation is separated into terms in α shown in part *X* and terms in β shown in part *Y,* the first being related to the ***size fraction*** and the second being related to the ***density fraction***. It is now possible to estimate *X* and *Y* separately and later combine them.

13.6 Calculation of the Size Fraction, *X*

13.6.1 Part One: The Fragment Shape Factor, f

f_α is defined as a fragment shape factor, also called the coefficient of cubicity, is a measure of fragment deviation from the cubic shape[39]. It is a correcting factor taking into account the fact that fragments are not perfect cubes. This factor has no dimension. If fragments are perfect unit spheres with r = 1, the volume of the sphere is $4/3\pi r^3$ = 0.523 and the shape factor f = 0.523. Most minerals have a shape factor close to 0.5 and for our purposes this is the standard value that will be used.

Table 13.1: Fragment shape and corresponding F value

Shape	F value
Cube	1
Sphere	0.523
Coal	0.45
Iron ore	0.50
Pyrite	0.47
Quartz	0.47
Ordinary rock	0.50
Biotite, mica, scheelite	0.1
Liberated gold	0.2
Acicular minerals	>1

The value for v is $v_\alpha = f_\alpha d_\alpha^3$. This can be substituted to give:

$Size\ \ fraction$:

$$X \approx \sum_{i=\alpha}^{n}\frac{v_\alpha M_{L\alpha}}{M_L} \approx \sum_{i=\alpha}^{n}\frac{f_\alpha d_\alpha^3 M_{L\alpha}}{M_L}$$

And because the Shape Factor is relatively constant for different size fractions we write:

$$Size \quad fraction: X \approx f \sum_{i=\alpha}^{n} \frac{d_{\alpha}^{3} M_{L\alpha}}{M_L}$$

13.6.2 Nominal size of fragments, d_N

Fragments in a lot have a distribution, the nominal size being the maximum particle size in the lot to be sampled. In practice, d_N is taken to be the mesh size that retains 5% of the lot being sampled. So for example if you sieve a sample using a 0.17-cm screen opening and 5% of the sample is retained on top of the sieve then d_N = 0.17 cm.

13.6.3 Part Two: The Fragment Size Distribution Factor or Granulometric Factor g

Our definition of the Size Fraction X requires that we fully understand the distribution of size fractions in a sample. While it may be theoretically possible to determine all the different size fractions contained in a sample, it is not easy in practice. However the fragment size distribution can be accounted for by using both the granulometric factor g, and the nominal fragment size, d.

g is a correcting factor taking into account the fact that all fragments are not the same size as **d**. In this approach one must clearly define what *d* is. If *d i*s defined as the square opening of a sieve that would retain exactly 5% of the investigating material by weight, then:

Degree of Calibration	Granulometric factor
For non-calibrated material:	g = 0.25
For material calibrated between two screens	g = 0.50
For naturally calibrated materials, corn or beans	g = 0.75

g is a dimensionless number. According to Long [75]one can sufficiently describe types of duplicates by an appropriate measurement of the particle size at which the material is divided (split), and the mass of the split. Gy (1992) has made an extensive investigation, utilizing many different kinds of crushing and grinding equipment, to ascertain what measurement of particle size best characterizes a sample. Gy found that the screen size that allows 95 percent of the sample to pass is the measure which is most independent of the method by which the material was crushed or ground, and most independent of the range of particles sizes (i.e., it applies well to both fine and coarse materials). More recently, François-Bongarçon (AMEC, personal communication) has provided a theoretical explanation of why this is so.

The sum term can easily be estimated by performing a size distribution analysis of the material under investigation. This will be done in a later Heterogeneity Test. The nominal size and the granulometric factors can now be substituted in the Size Fraction, X as follows:

---

[75] Long, Scott D. 2007. Assay quality assurance-quality control program for drilling projects at the pre-feasibility to feasibility report level. (5th edition). *AMEC* Mining Consulting Group, Suite 300, 2001 W Camelback Rd, Phoenix, Az, 85015 USA

$$Size\ \ fraction: X \approx f\sum_{i=\alpha}^{n}\frac{d_{\alpha}^{3}M_{L\alpha}}{M_L} \approx fgd_N^3$$

13.7 Calculation of the Density Fraction, Y

13.7.1 Part One: The Mineralogical Factor, c

$$Density\ \ fraction: Y \approx \sum_{\beta=1}^{n}\rho_{\beta}\left(\frac{(a_{\beta}-a_L)^2}{a_L^2}\right)\cdot\left(\frac{M_{\beta}}{M_L}\right)$$

As shown above the size fraction is Y is proportional to the sum of all variances $(a_{\beta}\text{-}a_L)^2$ for all density fractions. Y reaches a minimum Y_{min} when the material is completely homogenous and a maximum Y_{max} when the constituent of interest is completely liberated, at which point the material is a mixture of gangue only and mineral only. Thus there are two density fractions, one L_M containing pure mineral with a density of ρ_M, and another the density fraction of gangue L_g, with a density of ρ_g. In addition the grade of the critical mineral is 1 ($a_M = 1$), and the grade of the gangue is zero ($a_g = 0$). Also the grade of the lot a_L is the ratio of the mass of critical mineral to the mass of the lot ($a_L = M_M/M_L$), and the mass of the lot M_L, is the sum of the mass of critical minerals and the mass of the gangue ($M_L = M_g + M_M$).

The mineralogical factor is often given without any explanation of its derivation in many textbooks. The full description of the derivation is provided by Gy, 1967, Pitard, 1993, and Neufeld[39]. If the descriptions of a_M and a_L are substituted in to the equation Y for the Density fraction the equation for the Mineralogical Factor c at Y_{max} can be derived.

$$Density\ \ fraction: Y \approx \sum_{\beta=1}^{n}\rho_{\beta}\left(\frac{(a_{\beta}-a_L)^2}{a_L^2}\right)\cdot\left(\frac{M_{\beta}}{M_L}\right)$$

$$\begin{aligned}
Y\max &= \rho_M\left(\frac{(a_M-a_L)^2}{a_L^2}\right)\cdot\left(\frac{M_M}{M_L}\right)+\rho_g\left(\frac{(a_g-a_L)^2}{a_L^2}\right)\cdot\left(\frac{M_g}{M_L}\right)\\
&= \rho_M\frac{(1-a_L)^2}{a_L^2}a_L+\rho_g\frac{(0-a_L)^2}{a_L^2}\cdot\left(\frac{M_L-M_M}{M_L}\right)\\
&= \rho_M\frac{(1-a_L)^2}{a_L}a_L+\left(\rho_g\frac{(a_L)^2}{a_L^2}\right)\cdot(1-a_L)\\
&= \rho_M\frac{(1-a_L)^2}{a_L}a_L+\rho_g(1-a_L)
\end{aligned}$$

Where c is defined as Y_{max} and has units expressed in (g/cc), λ_M is the density of the constituent of interest (Column β_1 in ***Figure 13.2***), and ρ_g the density of the gangue (Column β_2 in ***Figure 13.2***). Solving the above equation for the constituent of interest gives Y_1.

$$Y_1 = \rho_M\frac{(1-a_L)^2}{a_L}$$

Solving the general equation for the gangue gives Y_2.

$$Y_2 = \rho_g(1 - a_L)$$

If the grade of the material comprising the lot (a_L) is very low, less than 10% (0.1), then the mineralogical factor reduces to:

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

If the grade of the lot is very high, in excess of 90% (0.9) the equation reduces to:

$$c = \rho_g(1 - a_L)$$

If the grade is above 10% and below 90%, c must be calculated using the complete formula[39]. The value used for a_L may be the average grade or the cut-off grade although it is better to use the average grade for grade control purposes.

The fact that the value of the lot a_L occurs in the calculation for the Mineralogical Factor may seem odd[39]. The critical content or grade of the lot a_L is expressed as the proportion of an element, rather than as a percentage or grams per ton number. If for example an ore contains 5% zinc and 0.35% copper the decimal proportion or fractional concentration of the metal of interest that is calculated in the following way:

- For zinc giving an assay of 5% and occurring as sphalerite (ZnS) would have a decimal proportion of sphalerite of: $a = \frac{64 + 32}{64} x \frac{5}{100} = 0.075.$
- For copper giving an assay of 0.35% and occurring as chalcopyrite would have a decimal proportion of chalcopyrite of: $a = \frac{63.55 + 55.85 + 2(32.06)}{63.55} x \frac{0.35}{100} = 0.0101$

This also means through this factor the sample variance depends on the grade of the lot being sampled, and 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 (François-Bongarçon, 2002, pg. 477).

13.7.2 Part Two: The liberation factor, ℓ

13.7.2.1 Definition of ℓ (After Dominique Francois-Bongarçon, 2013)

Francois-Bongarçon (2013) has established a clear mathematical definition for the liberation Factor which is reproduced with some additional explanation here. In the case of a sample of material S_L from an infinite liberated lot the relative variance is given by:

$$Rel.Var.[S_L] = \frac{cfgd_N^3}{M_S}$$

When the mineral of interest is not fully liberated the sampling variance for a sample *S* is somewhat lower and we write:

$$Rel.Var.[S] = \frac{cfg\ell d_N^3}{M_S} \quad where \;\; 0 < \ell < 1 \qquad \text{Equation 1}$$

Revert back to the liberated lot, letting a sample S_L be taken so that the average number of fragments *N* in sample S_L is the same as that in sample *S.* This means that the mass of S_L will be such that:

$$M_{S_L} = N \times m_L \;\; and \;\; N = \frac{M_S}{m}$$

In this case m_L and *m* are the average fragment masses in the liberated and non-liberated lots, respectively. So the relative variance for the liberated lot becomes:

$$Rel.Var.[S_L] = \frac{cfgd_N^3}{M_{SL}} \qquad \text{Equation 2}$$

If this is the case then the ratios in the model are:

$$\frac{m_L}{m} = \frac{fgd_\ell^3}{fgd_N^3} = \left(\frac{d_\ell}{d_N}\right)^3 = \frac{M_{S_L}}{M_S}$$

We can replace:

$$M_{S_L} \;\; by \;\; M_S\left(\frac{d_\ell}{d_N}\right)^3$$

In Equation 2, to give:

$$Rel.Var.[S_L] = \frac{cfgd_\ell^3}{M_{SL}} = \frac{cfgd_\ell^3}{M_S\left(\frac{d_\ell}{d_N}\right)^3} = \frac{cfgd_N^3}{M_S} \qquad \text{Equation 3}$$

Now divide Equation 1 by Equation 3 to give:

$$\ell = \frac{Rel.\ Grade\ Var.[S]}{Rel.\ Grade\ Var.[S_L]}$$

Thus the ratio of sample variance (S) to the liberated sample variance (S_L) with the same number of fragments is a precise, rigorous and objective definition of the liberation factor, ℓ. Thus the size of the initial sample S is irrelevant, and the ratio of M_{SL} to M_S remains constant:

$$\frac{M_{S_L}}{M_S} = \left(\frac{d_\ell}{d_N}\right)^3 = constant$$

The variance in Equation 4 are in the inverse ratio of the sample mass and therefore in the same ratio to each other and thus unaffected by changes in M_S.

13.7.2.2 Methods for estimating the Liberation Factor

Method 1: This is also known as the mineralogical method and requires knowledge of the critical content of the lot aL and the critical content of the largest fragments, a_{max}. Following microscopic investigation of the coarsest fragments to be sampled, a few of the largest fragments, where the content of the constituent of interest is high, are isolated. These fragments are assayed to find the maximum content which is defined as a_{max}.

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

This formula is used when looking at the coarsest fraction. Take about 300kg of material and crush it all to the same size, about 1.2cm. Separate this into 50 samples of 6kg each. Screen out the 1cm sized fragments and select those fragments with the highest sulphide content or ash content depending on what one is analysing for. This can be very difficult for gold ores unless there is a clear association between sulphide and gold content. Analyse the 1cm fragments for the mineral of interest. Assume the following results are obtained: Average content of ore = 1.5%Cu, a_L = 0.015, and the average content of 1cm fragments = 15%Cu, a_L = 0.15:

$$\ell = \frac{a_{\max} - a_L}{1 - a_L}$$
$$= \frac{0.15 - 0.015}{1 - 0.015}$$
$$= 0.15$$

Method 2: In mineral processing when one knows the liberation size dl of the constituent of interest, there is an approximate model to quickly calculate the value of ℓ as a function of the nominal fragment size d.

$$\ell \approx \sqrt{\frac{d_\ell}{d}}$$

This equation is only a rough model and should not be taken for granted. Many people have vastly misused this equation. Under no circumstance should it be used unless there is mineral processing information at hand, or if one already knows the sampling constant K introduced later in the Heterogeneity Test. Assuming d_ℓ ~100 microns it is then possible to calculate the liberation factor when fragments are at 1.5cm in diameter.

$$\ell \approx \sqrt{\frac{d_\ell}{d}}$$

$$\ell \approx \sqrt{\frac{0.001}{1.5}}$$

$$\ell \approx 0.0258 \; or \; 26 \; microns$$

The liberation size, $d\ell$ of a constituent of interest is defined as the size below to which 95% of the material must be ground in order to completely liberate at least 85% of the constituent of interest. Convention dictates that one never liberates all the minerals. The best crush only liberates 85%-90% of the mineral. Such relationships are shown in the liberation curves of Figures 2.14 to 2.16.

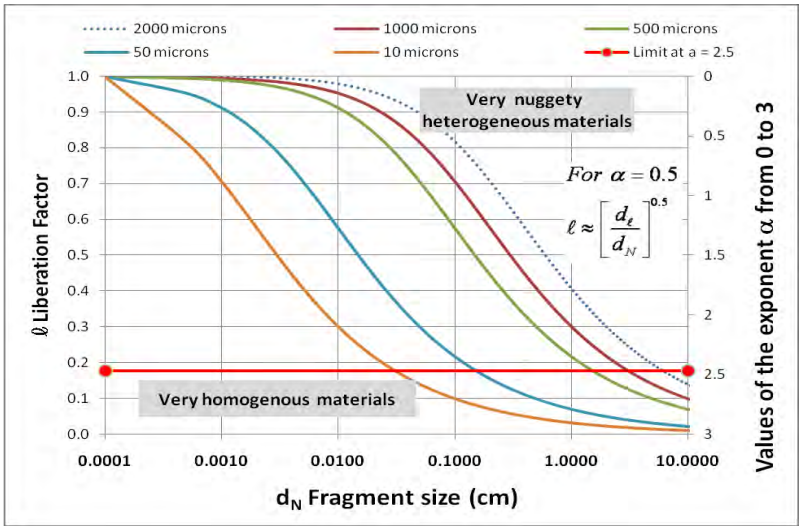

Figure 13.3: Curve for changes in the liberation factor from 0 to 1 with changing fragment size for α = 0.5

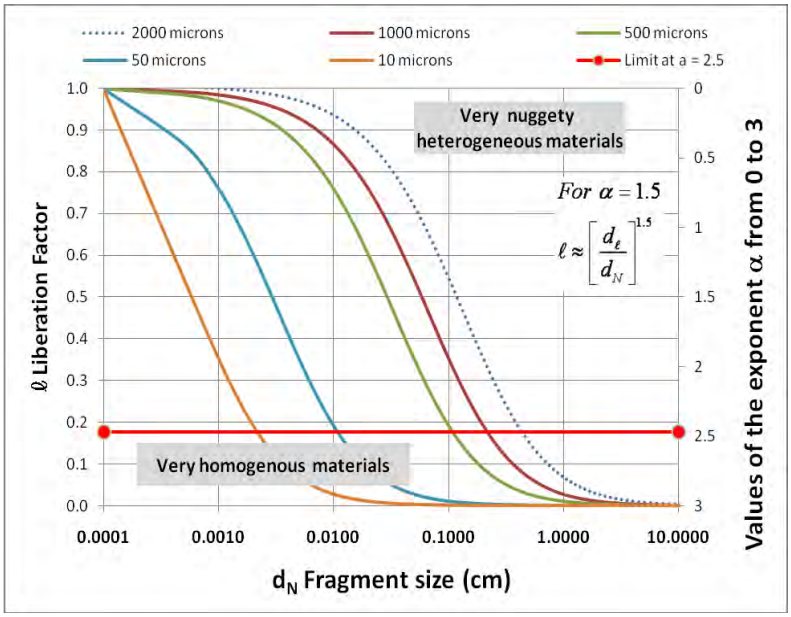

Figure 13.4: Curve for changes in the liberation factor from 0 to 1 with changing fragment size for α = 1.5

For the liberation of a single mineral from its host gangue material the equation given above only produces good results in a range between about 0.01cm and 1cm as shown in Figure 13.4. Beyond these limits the equation does not produce stable results. Once there is more than one mineral of interest the assistance of a mineralogist is required in order to resolve the problems of liberation.

Method 3: In recent experimental work Dominique François-Bongarçon investigated the liberation factor and suggested that the factor should be calibrated according to the nature and characteristics of the material being sampled. This so-called calibration method for the liberation factor has the form

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

Where the exponent b, is a value related to the slope of the calibration line above the liberation size dl. The value for b can vary between 0 and 3 depending on the nature of the ore and requires calibration of the particular ore type. Exponent b takes values close to 1.5 in most gold ores as well as in cases where it has not been possible to calibrate the exponent and so we can write:

$$\ell = \left(\frac{d_\ell}{d_N}\right)^{1.5}$$

There is a change in the form of the relative variance of the FSE when the ores become fully liberated.

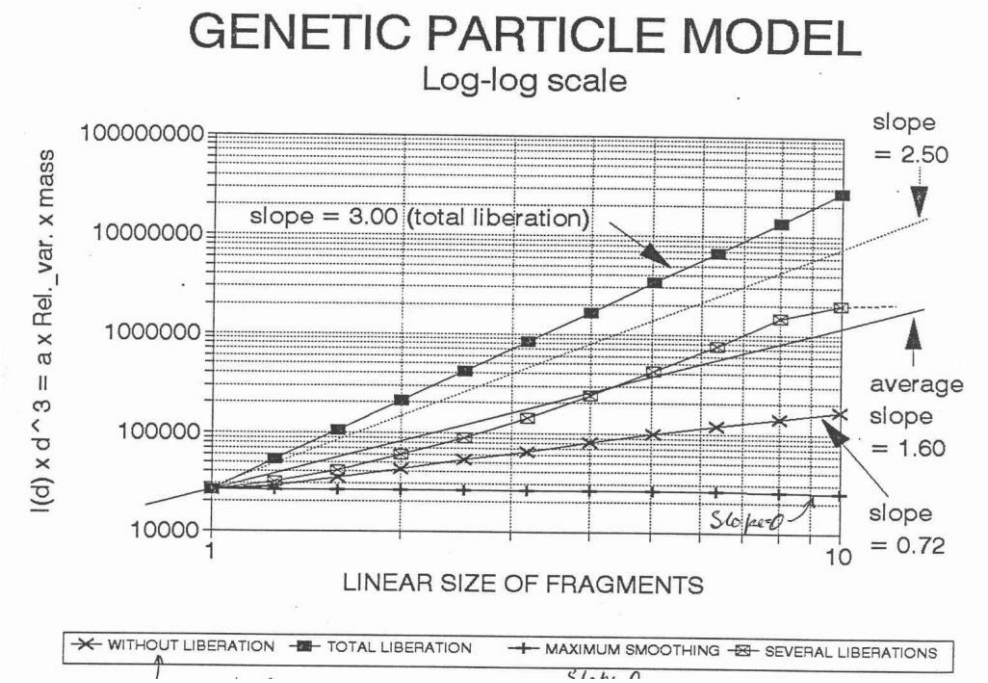

Figure 13.5: Comparison of linear fragment size with Relative variance x mass. Source: François-Bongarçon, D. 2002. Course notes from a course entitled “Sampling in the mining industry: Theory and Practice”.

Beyond liberation size further comminution does not change the variability of the individual rock fragments (François-Bongarçon 1998). From the log-log graph of I(dl) x d3 vs. fragment size (Figure 13.5) we know that the slope of the line below the liberation size (dl) is equal to 3. Beyond the liberation size the slope of the line is less than 3 and is given by α = 3-b and the effective range for b is actually 1 to 2 (Figure 13.5). The value of b can never be less than 1 otherwise the value for Y is overestimated. We also know according to the work by François-Bongarçon that:

$$\ell = \left(\frac{d_\ell}{d_N}\right)^b = \left(\frac{d_\ell}{d_N}\right)^{3-\alpha}$$

where b is a factor related to the slope of the line and varies between 0 and 3. The underlying insight to this factor is shown in Figure 13.6.

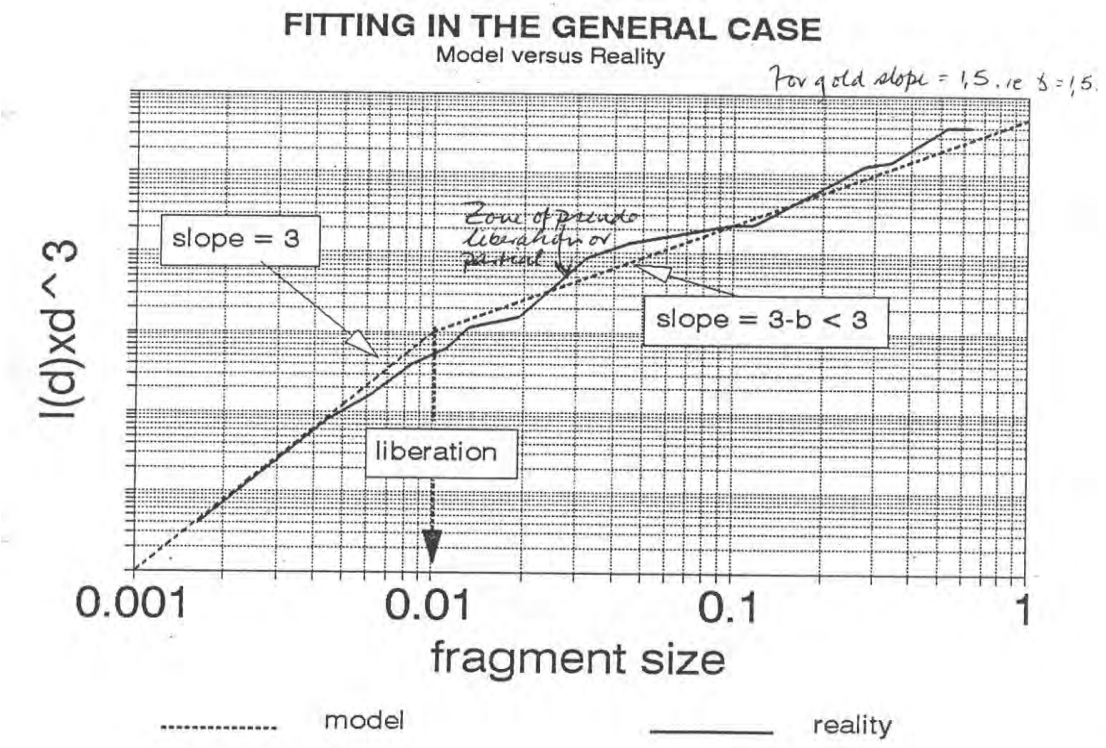

Figure 13.6: Fitting a general case to fragment sizes above and below the liberation size (0.01); Above the liberation size slope α = 3-b; below the liberation size slope α = 3 Source: François-Bongarçon, D. 2002. Course notes from a course entitled “Sampling in the mining industry: Theory and Practice”.

Thus two distinct cases arise, one for materials below the liberation size, in which case ℓ=1 and Gy’s formula can be written:

$$\sigma_R^2 = \frac{K' \times d_N^3}{M_s}$$

where K’ = c x f x g, and one for materials above the liberation size, in which case Gy’s formula takes the form:

$$\sigma_R^2 = \frac{c \times f \times g \times \ell \times d_N^\alpha}{M_s}$$

where α = 3-b.

Since we know:

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

we can substitute for ℓ in Gy’s equation and further write:

$$\sigma_R^2 = \frac{cfgd_N^3}{M_s} \times \left(\frac{d_\ell}{d_N}\right)^b = \frac{cfgd_N^3}{M_s} \times \frac{d_\ell^b}{d_N^b} = \frac{cfgd_\ell^b}{M_s} \times \frac{d_N^3}{d_N^b}$$

$$= \frac{cfgd_\ell^b \times d_N^{3-b}}{M_s}$$

and because $\alpha = 3 - b$ *we can write*

$$\sigma_R^2 = \frac{cfgd_\ell^{3-\alpha} d_N^\alpha}{M_s}$$

Using the calibration method the general case for Gy's formula can be written as

$$\sigma_{FSE}^2 = \frac{c \times f \times g \times d_\ell^{3-\alpha} \times d_N^\alpha}{M_S}$$

Where α is a parameter for specific deposits which can be 'calibrated' to a particular ore type or the mineralization associated with a particular mineral deposit François-Bongarçon1 (1993) reported values of α at about 1.5 and Assibey-Bonsu[76] listed values between 0.76 and 1.15. Afewu and Lewis[77] (1998) reported values of 1.01 for low grade ores (5g/t) and 1.13 for high grade ores (60g/t). Current research has indicated that α = 1.5 for most low grade gold ores (see1). Equation (4) can be rearranged so that

$$M_S = \frac{c \times f \times g \times \ell \times d_N^3}{\sigma_{\mathrm{FSE}}^2}$$

We now have all the tools necessary to answer the three questions stated at the beginning of this section.

Again it is important to emphasise that σ^2 is used for infinite populations, but in practice we use S2 because we are dealing with finite, limited amounts of material.

13.8 The Form of IH_L

It is now possible to combine all the elements of the estimation equation in a simplified form that combines:

IH_L is composed of the following components each of which can be calculated or estimated

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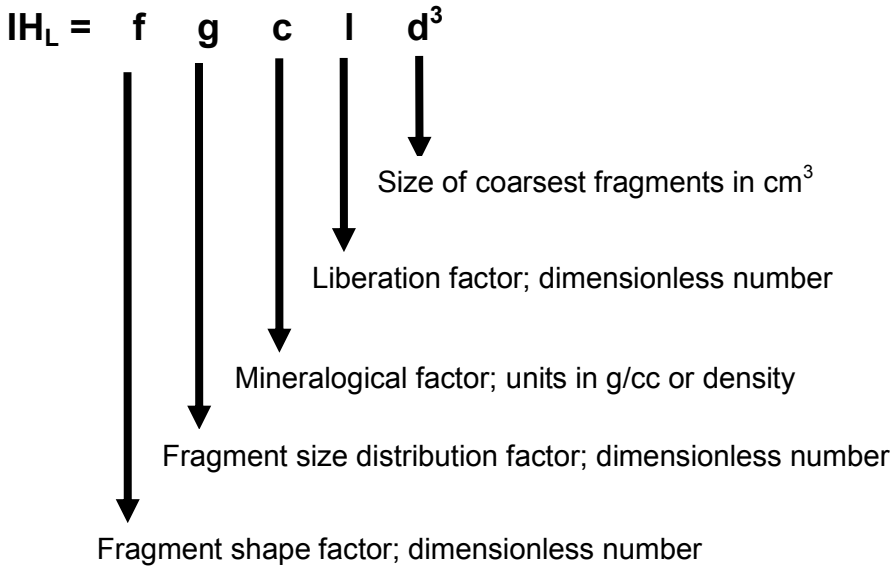
[76] Assiby-Bonsu, W. (1996). A summary of present knowledge on the representative sampling of ores in the mining industry. Journal of SAIMM, vol. 96, no. 6. 1996. pp. 289-293.

[77] Afewu, K.I. and Lewis, G.O. (1998) Metallurgical laboratory Techniques Colloquium. SAIMM, Johannesburg. 1998.



The estimation of the invariant heterogeneity IH_L has units of mass in grams and has the form:

$$IH_L = cfg\ell d_N^3$$

13.9 Variance of the Fundamental Sampling Error (FSE)

In Theory of Sampling, Dr. Pierre Gy demonstrated that the:

Mean of the Fundamental Sampling Error (FSE): When sampling is correctly implemented, the mean m(FSE) is negligible.

Variance of the Fundamental Sampling Error (FSE): is given by the first order approximation:

$$\sigma_{FSE}^2 = \frac{1-P}{PM_L} IH_L$$

Where *P* is a constant probability of selection applied to all fragments in the material to be sampled. By definition:

$$P = \frac{M_S}{M_L}$$

Where M_S is the sample weight and M_L the lot weight. After substitution the following is obtained:

$$\sigma_{FSE}^2 = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) IH_L$$

FSE is defined as an error taking place when the increment selection is correct and when the increments making up a sample are made up of only one fragment selected at random. Therefore it is a limited case. FSE is the minimum sampling error possible in each sampling stage of a sampling protocol.

$$\sigma_{FSE}^2 = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) f\,g\,c\,\ell\,d^3$$

Provided that the mass of the lot is 10 times larger than the mass of the sample it is possible to drop the term $1/M_L$ so that the equation for FSE becomes:

$$\sigma_{FSE}^2 = \frac{f\,g\,c\,\ell\,d^3}{M_S}$$

Sampling Constant K is then defined as: $K = c\,f\,g\,\ell$

13.10 Compiling the Sampling Nomogram

We now have all the calibrated constants for a particular ore type that was analysed in the sampling tree experiment with Three Series of analyses. These give us the necessary data in order to compile a sampling nomogram for the particular ore type. As shown previously;

$$\sigma_R^2 = \frac{K\,d^3}{M_S}$$

Taking logarithm of both sides gives:

$$\ln\left(\sigma_R^2\right) = \ln\left(\frac{1}{M_S}\right) + \ln\left(Kd^3\right)$$

$$\ln\left(\sigma_R^2\right) = (-1)\ln\left(M_S\right) + \left[3\ln(d) + \ln(K)\right]$$

This last equation can be used to build a chart which shows that for a given stage of comminution (i.e. a fixed value of particle size d_l), the term $\ln(K) + 3\ln(d_l)$ is a constant say c(d) and

$\ln\left(\sigma_R^2\right) = -\ln(M_s) + ¢(d)$. So for a given fragment size d, this is a line with a slope of -1.

Input for calculating σ_R^2: We should note here that Gy's formula changes when we are below the liberation size i.e. for $d < d_0$, that is for fragments below the liberation size. Using an estimate of K = 15.00 we can compile a table of values (Table 7.1) for the nomogram by substituting the following values in the equation: $\sigma_R^2 = \frac{K \times d_N^{2.5}}{M_{sample}}$

Table 13.2: Compilation of data for use in a Sampling Nomogram

Mass (gm)	Fragment size (cm)	Standard Relative variance ($S^{2)}$	Point	Precision
400000	20	0.067082039	A	25.9%
400000	1	0.0000375	B	0.6%
5000	1	0.003	C	5.5%
5000	0.5	0.00053033	D	2.3%
1000	0.5	0.00265165	E	5.1%
1000	0.15	0.000130713	F	1.1%
200	0.15	0.000653566	G	2.6%
200	0.05	4.19263E-05	H	0.6%
30	0.05	0.000279508	I	1.7%
30	0.005	8.83883E-07	J	0.1%
5	0.005	5.3033E-06	K	0.2%

Using these data we can now compile the nomogram shown in Figure 7.6. The 10% relative error line is shown in blue on Figure 7.6 and the precision of points falling below this line are generally acceptable. The region below 16% precision in the nomogram is set according to Data Quality Objectives. Above the 16% line the sampling operation involves a domain where the error distribution is non-normal and may be unacceptably skewed, which means the precision may get out of control, and possibly out of the validity of our model. Any point along the nomogram should not exceed 16% precision.

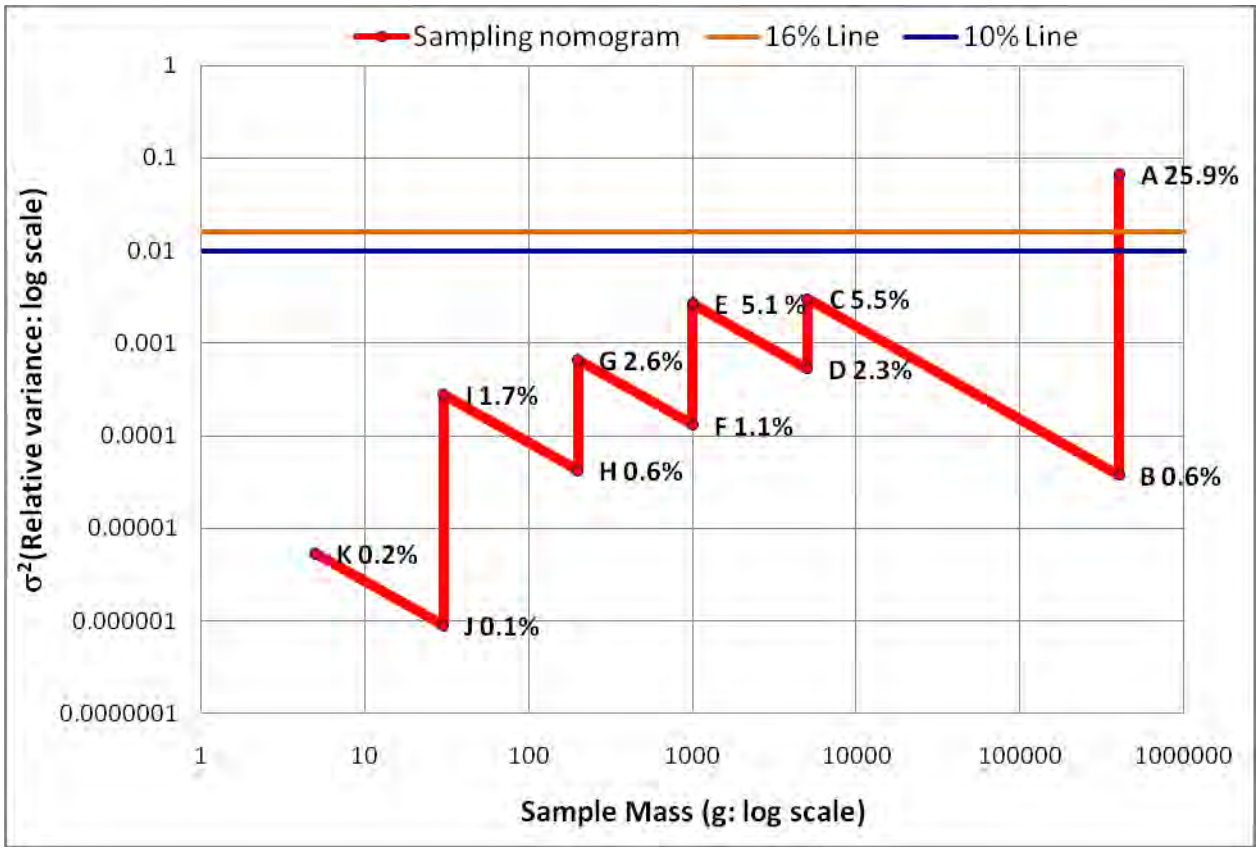


Figure 13.4: Sampling nomogram that plots the Relative Variance against the sample mass (g)

13.11 Importance of Sampling Nomograms

For each sub-sampling stage any sampling operation (mass reduction at constant rock particle size d_N) can be plotted on the chart as a path along a straight line of slope -1 (there is one such sampling per comminution size). On ***Figure 7.6***, (A) – (B) is such a path. The mass corresponding to Point A is the weight of crushed material. The mass corresponding to Point B is the weight of material split out from the next stage of comminution. The difference between σ_R^2 for B and σ_R^2 for A represents the segregation-free relative sampling error variance introduced during the step of mass reduction. Crushing and grinding stages, which do not introduce errors, and thus do not contribute to the variance, are represented by vertical segments. Thus the entire sampling preparation protocol can be visualized, with the contribution of each stage of the overall precision variance appearing clearly (***Figure 7.6***). Such charts constitute valuable tools for the design, assessment and control/improvement of sampling processes.

Comment on the proposed sampling procedure based on the Sampling Nomogram: Any sampling operation (mass reduction at constant rock particle size d) can be interrogated at each sampling stage. The difference between σ_R^2 for B and σ_R^2 for A represents the segregation-free relative sampling variance for the stage. Crushing and grinding stages, which do not contribute to the variance, are represented by vertical segments. The entire sampling preparation protocol can thus be visualized, with the contribution of each stage of the overall precision variance appearing clearly.

It is important to note that a nomogram, because it is derived from the Heterogeneity Test, describes only the behaviour of the sampling variance due to the heterogeneity of the ores i.e.,. The Heterogeneity Test is designed in such a way that the nomogram that is compiled from these data eliminates as far as possible the grouping and segregation effect and the variability due to the In-Situ Nugget Effect, (INE). Hence the nomogram only takes the variance s^2_{FSE} into account, it does *not* represent the total sampling error (TSE) or the combined influence of the In-situ Nugget Effect and the problems associated with segregation. The total small-scale error, which is the sampling variance, can be calculated and represents only that proportion of the overall error which arises due to the compounded effects of FSE, which together should not exceed the Safety Line. There is no strict rule with regard to what the value for the Safety Line should be. It depends on what the purpose of the nomogram is in order to determine what is acceptable. ***Table 7.2*** provides some guidelines.

Table 13.3: Guidelines for the size of the safety margin in a typical nomogram

Purpose of Nomogram	Base Metals	Precious Metals
Exploration Ore Grade control Routine sampling Environmental sampling	±10%	±15%
Material Balance Process Control	±5%	±10%
Commercial concentrates Trade of commodities	±2%	±1%
Laboratory standards	Very stringent protocols	Very stringent protocols

14 HETEROGENEITY TESTING

14.1 Introduction

The aim of the Heterogeneity Test is to determine the sampling constants for different styles or types of mineralisation as a basis for designing and optimizing sampling and sample preparation protocols for exploration drillhole samples, blasthole samples, conveyor belt samples, etc[78]. Heterogeneity testing is a critically important step in material characterisation that focuses on determining the sampling constant K in the formula Gy proposed for determining the Fundamental Sampling Error.

$$s^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L} \right) \times K d^3_N$$

The test is performed by controlling d_N to a size of one centimeter so that the exponent of d_N is irrelevant and the value of d^3_N is one. The mass of each sample is controlled to an exact weight so that M_S is exactly known. The variance of the selected samples is also known which leaves K as the only unknown value that can be solved for. The first step should also include a mineral deportment study allowing the nature of the mineral distribution, the mineral associations and mode of occurrence, the size distribution of the mineral grains (especially the possibility of nugget formation or occurrence), and the sampling characteristics of the ores to be determined. Heterogeneity testing is not done on a regular basis, in fact it probably only needs to be done once for a given type of mineralisation over the lifetime of a project, but it is essential that the process followed is done correctly from the beginning. Results from the Heterogeneity Test should be congruent and support the results of the deportment study.

The following section describes a recommended test that can be used in order to achieve an accurate determination of IH_L if it is difficult to estimate the liberation factor of a constituent of interest. This is particularly true for minor constituents such as gold, molybdenum, copper, arsenic etc. The test provides a very good definition of the value of K and is recommended for optimising sampling protocols during exploration, at the mine and at the mill. The idea is to experimentally estimate the error variance S^2_{FE} and on this basis compile a nomogram that describes the practical sampling unit operations that allows one to optimise the sampling protocol.

14.2 The Heterogeneity Test

Usually one hundred samples are collected, depending on the material being characterised, and these are assayed for each constituent of interest. The relative variance S^2_{ai} of the assays or analyses is calculated and under the specific conditions set by the experiment, the following equation for the relative variance of FSE can be written:

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[78] Magri, E. J. M. 2004. Some experiences in heterogeneity tests and mine sampling. Mining Engineering Department, University of Chile, Av. Tupper 2069, Santiago, Chile

$$s^2_{FSE} = \frac{s^2_{a_i}}{\bar{a}_i^2}$$

Where s^2_{ai} = the variance of the 100 assay values and $\bar{a}_i^2$ is the mean of the 100 assay values squared, so s^2_{FSE} is a dimensionless number.

14.3 Precautions to be taken

a. Performing the Heterogeneity Test on 1-cm fragments is ideal. The average size of the fragments between the 1.25-cm screen and 0.65-cm screen is 1.05 cm. 1.05-cm is very close to the ideal 1 cm, so the exponent of d_N does not play any role in the calculation of the sampling constant.

b. Samples collected to perform the Heterogeneity Test should consist of relatively few fragments in order to make the Analytical Error negligible. Usually, the fragments are collected one by one until a weight nearest to 50 grams is obtained; it simplifies the calculations if using sample mass instead of number of fragments.

c. Fragments collected to make each sample must be selected one by one at random, in order to destroy any Distributional Heterogeneity (DH) and minimize the contribution from the Grouping and Segregation Error.

d. It is essential that no outlier values, high or low (***Figure 11.1***) are cut from the set of analyses. What we call outliers constitute an extremely important component of the data set and carry information that can be obtained no other way. To eliminate the so-called outliers distorts the outcomes of any analytical procedure and gives the wrong answers for any grade related studies.

14.4 Description of the Heterogeneity Test

a. Collect 300kg of ore in as large fragments as can be handled from a mine.

b. Dry the 300kg overnight at 110^0C.

c. Crush the material to about 95% passing minus 1.9-cm with a Jaw Crusher with an opening adjusted accordingly. This means that if you pass 300kg through a 1.9-cm screen, that 15kg should remain on top of the screen because it is too large to pass through the screen. If there is 25kg remaining on top of the screen, all the rejects must be passed through the jaw crusher and through the screens until only 15kg remains. The lot is then correctly classified as [95% -1.9-cm].

d. Split the 300kg into two lots; **Sub-lot A** weighing 260kg and **Sub-lot B** weighing 40kg.

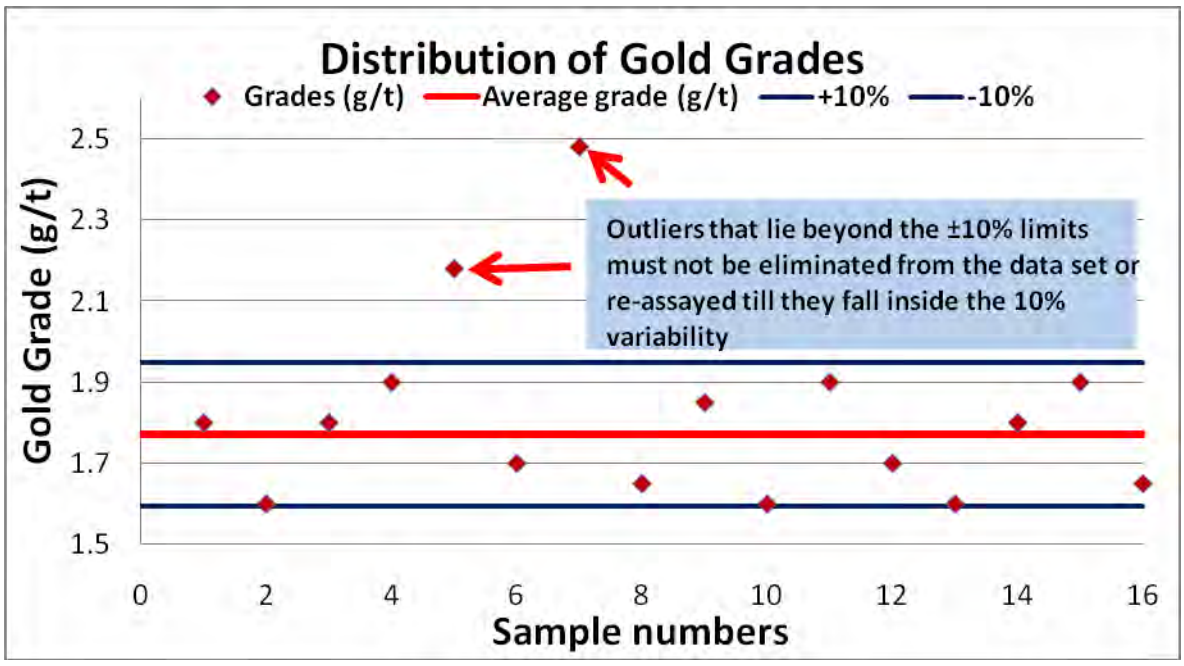


Figure 14.1: Distribution of gold grades between the ±10% limits set by the analytical labaoratory showing the so-called outliers in the data set

14.4.1 Treatment of Sub-lot A

a. Screen the entire **Sub-lot A** 260kg through the 1.25cm and 0.63cm screens.

b. Weigh this size fraction.

c. Spread the –1.25cm+0.63cm fraction on a clean surface: The Heterogeneity Test will be performed on this fraction.

Top Screen (cm)	Bottom Screen (cm)
1.266	0.630

The average d_N between these two screens is 1.05cm on average as shown in the following calculation.

$$d_N = \sqrt[3]{\frac{d^3_{\text{upper screen size}} + d^3_{\text{lower screen size}}}{2}}$$

$$= \sqrt[3]{\frac{1.266^3 + 0.63^3}{2}} = \sqrt[3]{1.1396} = 1.0445\text{cm}$$

d. From this fraction (about 50kg), collect 100 samples. Each sample must be made of p fragments selected randomly one at a time, up to 50g. (Use 50-gram as a target, collecting whatever number of fragments it takes to do so. So later on you have M_S = 50 grams in the formula). Number these samples from 1 to 100, weigh each of them, and record values for p. Equivalent mass is 5000g.

e. Pulverize each sample directly in an enclosed ring and puck pulverizes (LM5 mill) to about 95% minus 106 micron.

f. Assay samples 1 to 100 for gold ***using 50g aliquots***.

g. **NBB:** Recombine any remaining material from the –1.25cm+0.63cm fraction with the balance of **Sub-lot A**.

14.5 Sample treatment for balance of material *after* the Heterogeneity Test

The following description has been recently used to examine the behaviour of variances in different size fractions of a lot selected for a Heterogeneity Test. The process is not recommended for every Heterogeneity Test, but should be considered as a means of complete material characterisation at the start of new mineral extraction or mining operations.

a. The remaining re-combined material in **Sub-lot A** amounts to about 250kg. Starting with the -1.9cm screen, set up and screen all the material (about 250kg) through the 8 screens listed in Table 8.1 and the relative size of the screen mesh is shown in ***Figure 8.2***. Record the weight of each size fraction. [Number of samples = 8 at about 32kg each].

Table 14.1: List of screens to be used in analytical work

	Top Screen (cm)	Bottom Screen (cm)	Approximate mass	No of fractions
1	1.900	1.277	32.5kg	32 x 1000g
2	1.266	0.630	32.5kg	32 x 1000g
3	0.630	0.335	32.5kg	32 x 1000g
4	0.335	0.168	32.5kg	32 x 1000g
5	0.168	0.084	32.5kg	32 x 1000g
6	0.084	0.042	32.5kg	32 x 1000g
7	0.042	0.0212	32.5kg	32 x 1000g
8	0.0212	Collecting pan	32.5kg	32 x 1000g
		Totals	**250kg**	

*It is highly unlikely that the mass of the crushed material will be distributed in this even manner. A normal distribution is much more likely.

b. Using a riffle splitter, split each one of the 8 size fractions into 32 samples according to the scheme illustrated in Figure 8.3. Record the weight of each of the 32 samples in each size fraction. [Number of samples for each size fraction = 32 at about 1000g each. Total number of samples = 8 x 32 = 256. 256 samples at about 1000g each = about 256kg].

c. From the 32 samples for each size fraction select any two samples at random, and set them aside for polished section and/or petrography.

d. Crush the remaining 30 samples in each size fraction to 95%-106µ and assay for gold using a 30g aliquot.

e. Each of the 8 size fractions will have 30 assays [Number of assays 8 x 30 = 240 assays] using a 30g aliquot.

f. Weigh the remaining 95% -106µ fraction for each of the original size fractions and using a pan or a Nelson concentrator separate the gold from the pulverized powder. Mount the gold recovered in this way in a polished section.

g. The final treatment of the gold grains separated from the pulverized material is questionable, but it is essential that we get an idea of the amount of gold in the pulverized material and the size distribution of these gold grains. It is essential to inspect the grains using a binocular microscope immediately after recovery from the powders (either by panning or using a Nelson concentrator). Following that, it is best to mount the gold grains in polished sections as a permanent record.

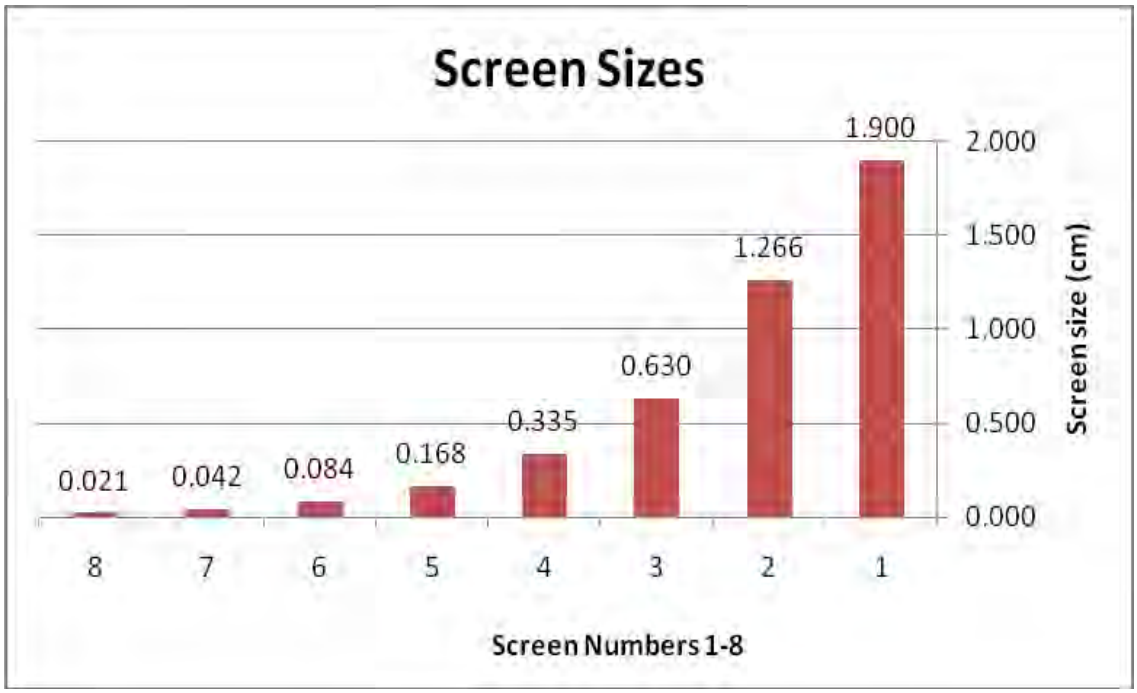


Figure 14.2: Relative mesh sizes for the eight screens used in this test work following a Heterogeneity Test

14.6 Description of the Sampling Tree Experiment or Duplicate Series Test

This is a well-known test, but it needs careful supervision in order to get correct samples for analysis. It requires approximately 40kg of material that is split from the total lot in Step 3 of Part A of the procedure described above.

a. The 40kg of material (split from the original 300kg) is classified as 95%-1.9cm after jaw crushing.
b. Split the 40kg into 128 equal sized samples weighing approximately 312g each. Select 32 samples at random from the 128 samples.
c. Recombine the remaining 96 samples to give a single lot with a mass of about 30kg and crush it to 95%-0.63cm.
d. Split the 30kg into 96 equal sized samples weighing approximately 312g each. Select 32 samples at random from the 96 samples.
e. Recombine the remaining 64 samples to give a single lot with a mass of about 20kg and crush it to 95%-0.2cm.

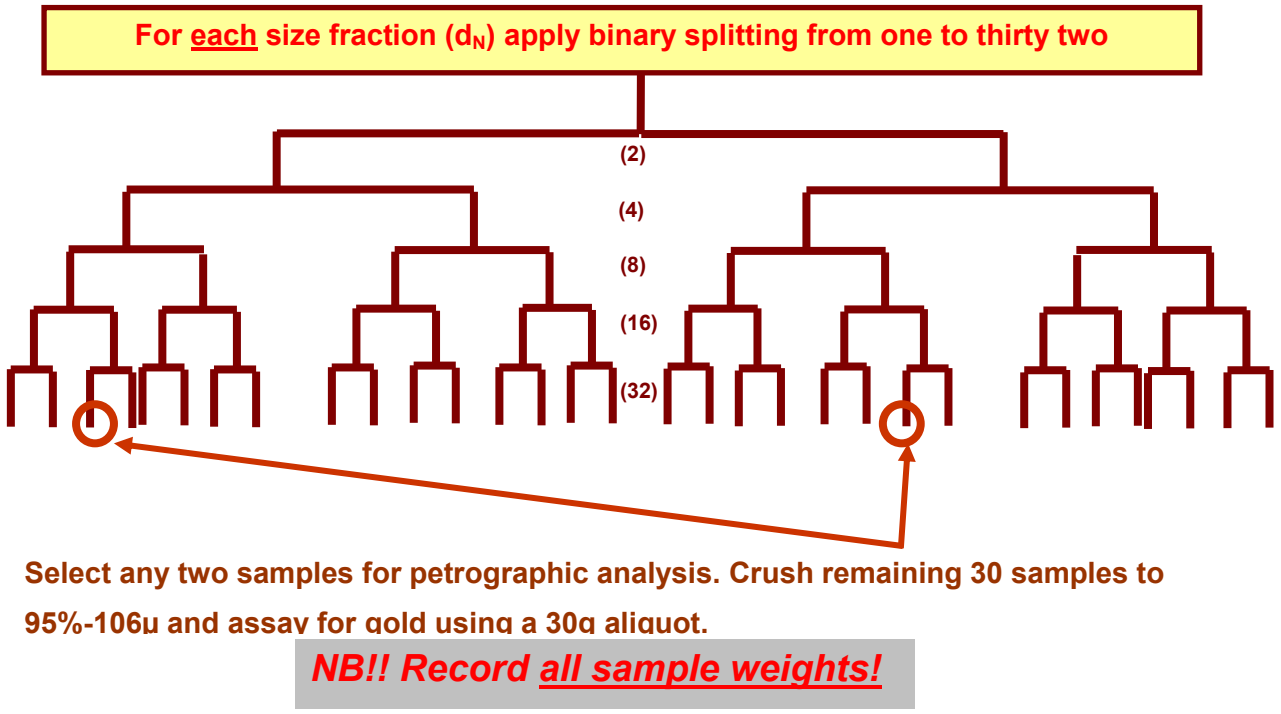


Figure 14.3: Illustration of binary splitting using a riffle splitter to produce 32 samples for each size fraction

f. Split the 20kg into 64 equal sized samples weighing approximately 312g each. Select 32 samples at random from the 64 samples.

g. Recombine the remaining 32 samples to give a single lot with a mass of about 10kg and crush it to 95%-0.05cm.

h. We are now left with four series of samples each comprising about 10kg of material at the following sizes:

Series	**4**	**3**	**2**	**1**
Size	95%-1.90 cm	95%-0.63 cm	95%-0.2 cm	95%-0.05 cm
Mass	~10kg	~10kg	~10kg	~10kg
Samples	32 x 312g	32 x 312g	32 x 312g	32 312g

i From each of the four series two samples are selected for confirmation of granulometry and polished sections (8 samples altogether).

j. The total number of samples for fire assay using 30g per aliquot is 120.

k. Methodology for reducing the assay data and plotting the derived results has been explained by Assibey-Bonsu (1996) and Minnitt et al (2007). These data can provide the basis for calculating the sampling parameters.

14.7 Analysis of Size Fractions

Some of the material can be used to analyse the different size fractions. Crush all the material in each size fraction to 1mm and take a 1kg sample from each screen size. Pulverise the sample and submit an aliquot

for assay. Generally it will be found that the fines have very different grades to the coarse materials. These results should sound warning bells for problems that could arise. A summary of assay outputs that will be used in various tests is shown in Table 14.4.

Table 14.4: Summary of assay outputs used in various tests

Sub-lot	Description	No. of Assays
Sub-lot A Heterogeneity Test	100 assay results from 50g aliquots	100
Sub-lot A	18 assays, 3 for each of 6 size fractions at 95% -106	18
Sub-lot B	48 assays, 3 for each of 16 splits at 95% -106 μm	48
Sub-lot C1	48 assays, 3 for each of 16 splits at 95% -106 μm	48
Sub-lot C2	48 assays, 3 for each of 16 splits at 95% -106 μm	48
Sub-lot D	48 assays, 3 for each of 16 splits at 95% -106 μm	48
Sub-lot E	Screen analysis and polished micro-sections	5
	Total	315 assays

14.8 The Difference between C and K

Whereas we have previously defined the sampling constant K as: $K = f\,g\,c\,d_{\ell}^{b}$, for the assay data from Heterogeneity Test we define C_{α} as the sampling constant for a specific size fraction α. The assay data for a copper deposit are listed in Table 8.3 and the relative variance (σ^2) of the 100 assays of material specifically classified as d_{α} = 1cm is given by the total variance of the assays divided by the mean squared:

$$\sigma^2 = \frac{s_{ai}^2}{\bar{a}_i^2}$$

$\bar{M}_{si}$ The average weight of the 100 samples (which must be calculated), then:

$$C_{\alpha} = \frac{s_{ai}^2}{\bar{a}_i^2}\frac{\bar{M}_{Si}}{d_{\alpha}^3} = f_{\alpha}g_{\alpha}c_{\alpha}\ell_{\alpha}$$

Assuming that the variability of the liberation factor obeys the model:

$$\ell = \frac{\sqrt{d\ell}}{\sqrt{d}}$$

The fundamental sampling error at any particular stage of a sampling protocol is given by Pierre Gy's well-known formula:

$$s_{FSE}^2 = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] \times Kd^3$$

Where: C is the sampling constant composed of the following factors f * g * c * l. K is the sampling constant composed of the following factors f*g*c* d_{ℓ}^{b} . d_{ℓ} is the liberation size. Again it is important to emphasise that σ^2

Again it is important to emphasise that σ^2 is used for infinite populations, but in practice we use s^2 because

we are dealing with finite, limited amounts of material. Because of the difficulties of estimating l at each stage of the protocol some investigators[79] have proposed the following empirical formula:

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

Where d_l is the liberation size of the minerals of interest, d_N is the size of the fragments, and b is an exponent usually given as 0.5. So:

$$C_\alpha = f_\alpha \times g_\alpha \times c_\alpha \times \sqrt{\frac{d\ell}{d_\alpha}}$$

The first three factors are independent of the maximum diameter of the fragments (d) so that from one size fraction to another the term $f_\alpha g_\alpha c_\alpha \sqrt{d\ell}$ remains reasonably constant. Therefore, call this term factor **K** - it does not change from one stage of comminution to another. Therefore sampling constant C can be written:

$$C_\alpha = \frac{K}{\sqrt{d_\alpha}} \text{ or } K = C_\alpha \sqrt{d_\alpha}$$

Thereafter, for each of the different sampling stages, the constant C_α can be derived from the above equation using the different values of **d**.***Table 8.3: Results of a heterogeneity test from a copper project (total copper)***

Sample	No of Fragments	Weight/g	%T Cu	Sample	No of Fragments	Weight/g	%T Cu
1	50	65.60	0.68	51	50	92.41	0.67
2	50	73.50	0.74	52	50	97.21	0.66
3	50	80.63	0.72	53	50	73.53	0.59
4	50	72.22	0.61	54	50	76.22	0.76
5	50	89.35	0.73	55	50	85.86	0.45
6	50	87.58	0.74	56	50	64.96	1.69
7	50	58.38	0.45	57	50	67.58	0.76
8	50	66.51	0.64	58	50	78.03	0.56
9	50	88.82	0.71	59	50	72.57	0.46
10	50	79.68	0.71	60	50	79.79	0.94
11	50	100.58	0.62	61	50	86.82	0.75
12	50	78.81	0.97	62	50	75.32	0.74
13	50	85.88	0.73	63	50	74.79	0.69
14	50	97.86	0.71	64	50	78.29	0.76
15	50	89.83	0.66	65	50	83.85	0.79
16	50	96.09	0.64	66	50	75.64	0.86
17	50	74.48	0.65	67	50	92.32	0.77
18	50	82.11	0.76	68	50	67.75	0.67
19	50	84.10	0.72	69	50	73.22	0.61
20	50	86.30	0.34	70	50	87.96	0.71
21	50	66.98	0.62	71	50	91.06	0.65
22	50	91.53	0.65	72	50	73.68	0.53
23	50	75.00	0.97	73	50	78.57	0.67
24	50	52.06	0.77	74	50	82.06	0.62
25	50	88.63	0.73	75	50	73.34	0.75

[79] Francois-Bongarçon, D. 1998. Extensions to the Demonstration of Gy's Formula. January, 1998.

26	50	58.17	0.69	76	50	75.96	0.62
27	50	84.32	0.75	77	50	79.33	0.83
28	50	57.39	0.55	78	50	82.06	0.84
29	50	78.56	0.73	79	50	79.38	0.71
30	50	74.34	0.73	80	50	78.06	0.62
31	50	86.26	0.63	81	50	88.52	0.71
32	50	85.28	0.71	82	50	70.72	0.72
33	50	66.69	0.83	83	50	72.24	0.78
34	50	80.27	0.65	84	50	70.59	0.45
35	50	89.65	0.69	85	50	79.85	0.54
36	50	67.17	0.89	86	50	76.98	0.64
37	50	82.97	0.62	87	50	70.69	0.97
38	50	78.52	0.61	88	50	74.02	0.77
39	50	88.26	0.57	89	50	67.57	0.76
40	50	69.67	0.58	90	50	103.15	0.74
41	50	78.07	0.74	91	50	84.39	0.89
42	50	93.68	0.68	92	50	87.18	0.69
43	50	63.58	0.88	93	50	70.28	0.71
44	50	94.13	0.69	94	50	66.5	0.62
45	50	74.82	0.37	95	50	88.19	0.58
46	50	78.87	1.56	96	50	75.29	0.62
47	50	93.56	0.87	97	50	81.74	0.88
48	50	71.97	0.84	98	50	65.74	0.74
49	50	60.38	0.77	99	50	77.53	0.68
50	50	70.81	0.61	100	50	84.82	0.34
				Sum		**7873.51**	**71.07**
				Average		**78.7351**	**0.7107**
				Variance		**102.314**	**0.0324**
				Rel Var		**0.016504**	**0.0642**

14.8.1 Analysis of Results from the Heterogeneity Test for Copper Ores

The Heterogeneity Test, as earlier described, was performed for total copper in a copper project. Results from the 100 collected samples made of 50 fragments each are shown in ***Table 8.3***. A simplified protocol for sampling blast holes is as follows:

A 4000g sample is collected at the mine. The maximum fragment size *d* is 1.9cm. At the laboratory the sample is dried and sent to a sample preparation robot. There it is split to 2000g and crushed to 95% -0.3cm using a jaw crusher. It is further split into 100g then pulverized to 95% -106 micron. Finally, a 0.25g sample is used for the analytical procedure. The following statistics can be derived from the data listed in ***Table 13.3.***

Statistic	Weight/g	%T Cu
Mean	78.7351	0.7107
Variance	102.314	0.0324
Rel Var	0.016504	0.0642

The following exercises should be done.

a. Calculate *K*
b. Calculate a sampling nomograph
c. Is this protocol optimum?
d. What can one do to improve it?

The value for *C* is derived as follows:

$$C_\alpha = \frac{s_{ai}^2}{\bar{a}_i^{-2}} \frac{\bar{M}_{Si}}{d_\alpha^3}$$
$$= 0.064146 \times \frac{78.7351}{1.05^3}$$
$$= 4.363$$

So the value for *K* is:

$$K = C_\alpha \sqrt{d_\alpha}$$
$$= 4.363 \times \sqrt{1.05}$$
$$= 4.71$$

The value for K used in the compilation of the nomogram shown in ***Figure 8.4*** was 4.48. In order to compile the nomogram you need values for:

$$\sigma_{FSE}^2 = \frac{K d_N^{3-\alpha}}{M_S} \quad (1)$$

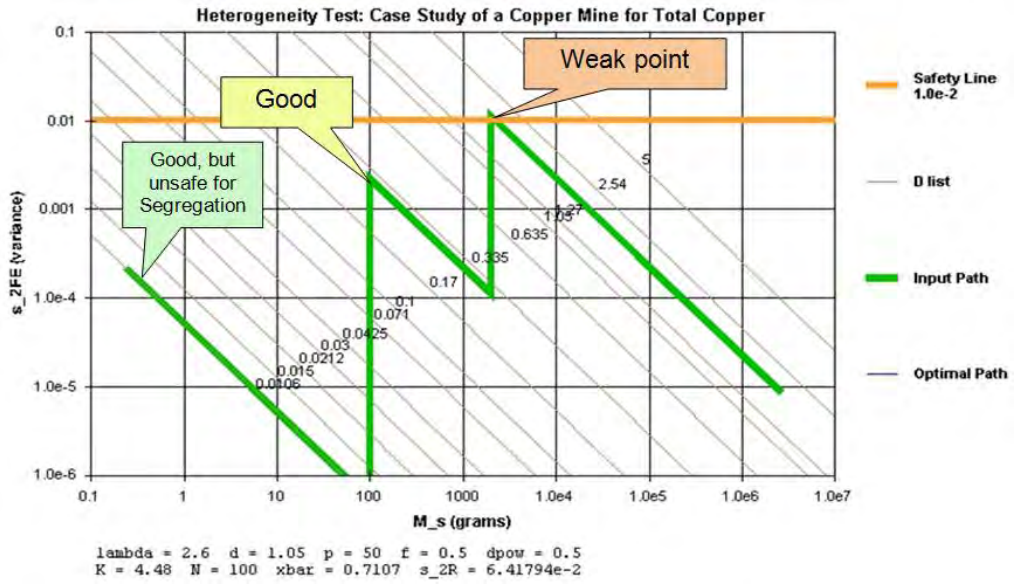


Figure 14.4: Nomogram compiled from data from Heterogeneity test for total copper **([40]Pitard, F, 2009.)**

Table 14.5: Sample mass and fragment size used for calculating the relative variance

Size [cm]	Mass [g]	Stand.Relat.Variance	Rel Std Dev	Position
20.0000	1,000,000,000	0.000008426	0.29%	A
20.0000	1,000,000	0.008425504	0.29%	B
2.5000	1,000,000	0.000046545	0.68%	C
2.5000	10,000	0.004654477	6.82%	D
0.2000	10,000	0.000008426	0.29%	E
0.2000	1,000	0.000084255	0.92%	F
0.0500	1,000	0.000002633	0.16%	G

0.0500	50	0.000052659	0.73%	H
0.0050	50	0.000000167	0.04%	I
0.0050	5	0.000001665	0.13%	J
	Incremental variance	0.013	11.53%	

The nomogram was compiled using OSP software provided by Francis Pitard Sampling Consultants. The small-scale error in this case is under control but this is where segregation can cause a major error because it is transient and can change all the time. Having completed the Heterogeneity Test it is now possible to continue the study and examine other issues such as the distribution of metal in different size fractions and liberation curves.

The relative variance shown in column three of ***Table 14.4*** was calculated using Equation (1), substituting values for K = 4.71 and α = 0.5 and applying the sample mass and fragment sizes shown in ***Table 14.4***; these data are plotted in ***Figure14.4*** to give a nomogram for copper ores.

It must be noted that fine material must not be lost during these exercises. It is also evident from ***Table 14.6*** that copper grade doubles when going from the coarsest to finest fractions. This is a clear warning that IEE and IPE may be occurring during the implementation of the protocol.

Table 14.6: Grade – Size analysis for copper ores

Size Fraction	Weight after drying and crushing	% Total Copper
+1.25 cm	32kg	0.65
-1.25 +0.63 cm	59kg	0.72
-0.63 +0.335 cm	17kg	0.75
-0.335 +0.17 cm	13kg	0.75
-0.17 +0.085 cm	9kg	0.79
-0.085 +0.0425 cm	7kg	0.92
-0.0425 +0.0212 cm	7kg	1.12
-0.0212 cm	6kg	1.27

The results of the heterogeneity test can be carried further and the values of the constant *C* can be plotted against the fragment size to produce the points shown in ***Figure 8.5.*** These points have not been joined yet it is feasible that they could form a continuous curve asymptotic to the axes Thus we are able to compare theory with reality.

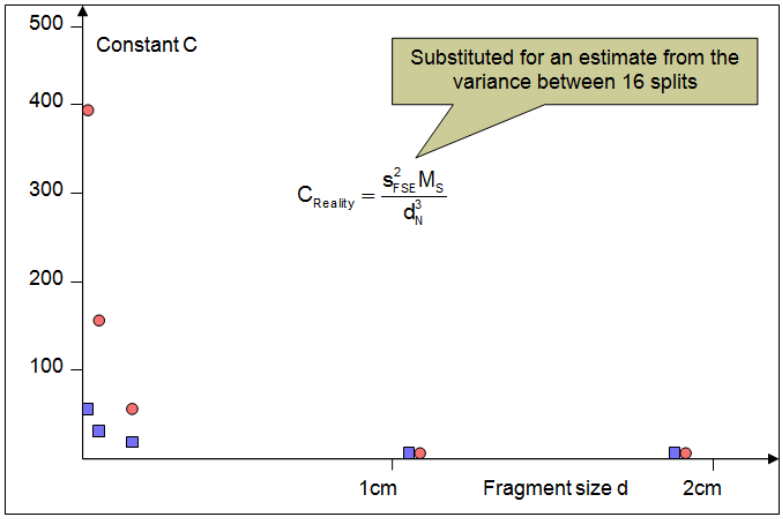


Figure 14.5: Variation in value of C for different fragment sizes; usually duplicate samples lead to a much higher value of C, especially for small size fragments, because other dominant errors are taking control of the overall variability. (e.g., GSE, AE, etc…) **([40]Pitard, F, 2009.)**

15 DETERMINATION OF FUNDAMENTAL SAMPLING ERROR FROM PARTICLE SIZE DISTRIBUTION

Determination of particle size distribution in a lot is a very important criterion because much depends on it: the quality of the product, efficiency of a process, compliance clauses in a commercial contract, assessment of the accuracy of a sampling system, and assessment of the representivity of a sample. Some initial definitions are required:

- **L_c:** Particle size class of interest
- **a_{Lc}:** Proportion of L**c** in the lot L
- **M_{Lc}:** Weight of the class of interest
- **N_{Lc}:** Number of fragments in L**c**
- **F_j:** A fragment of L**c**
- **M_{Fj}:** Weight of the fragment F**j**
- **a_{Fj}:** For each fragment of L**c** $a_{Fj} = 1$
- **F_{Lc}:** Average fragment of the class L**c**
- **M_{FLc}:** Weight of F_{Lc}

15.1 Calculation of IH_L

The following calculation of IH_L is given:

$$IH_L = \sum \frac{\left(a_i - a_L\right)^2}{a_L^2} * \frac{M_i^2}{M_L}$$

in the class of interest $L_c \rightarrow a_i = a_{Fj} = 1$

out of the class of interest $L_c \rightarrow a_i = 0$

$$IH_L = \sum_j \frac{(1-a_{Lc})^2}{a_{Lc}^2} \times \frac{M_j^2}{M_L} + \sum_i \frac{M_i^2}{M_L}$$

$$IH_L = \sum_j \frac{(1-a_{Lc})^2}{a_{Lc}^2} \times \frac{M_j^2 \times a_{Lc}}{M_{Lc}} + \sum_i \frac{M_i^2}{M_L}$$

$\Downarrow$ $\Downarrow$

constants X_{Lc} X_L

$$IH_L = \frac{(1-a_{Lc})^2}{a_{Lc}} X_{Lc} + X_L$$

And because the term a_{Lc}^2 is very small it is discarded and the result is:

$$IH_L = \frac{(1-2a_{Lc})}{a_{Lc}} X_{Lc} + X_L$$

X_{Lc} and X_L should be expressed in practical terms because M_i and M_j are not known. This is relatively easy when one has a rough idea of the particle size distribution. It is sufficient to substitute F_i or F_j of a given particle class by the ***average fragment*** of this class. Again we need some further definitions:

N_{Lx}: number of fragments in the class Lx
F_{Lx}: average fragment of the class Lx
M_{FLx}: weight of the average fragment F_{Lx}
a_{Lx}: proportion of Lx in the lot L

By definition

$$M_{FLx} = \frac{M_{Lx}}{N_{Lx}}$$

Then changing Mi by the average number of fragments:

$$X_L = \sum_{+} \frac{M_i^2}{M_L} = \sum_{x} \frac{N_{Lx} M_{FLx} M_{FLx}}{M_L} = \sum_{x} \frac{M_{FLx} M_{Lx}}{M_L}$$

However:

$$M_L = \frac{M_{Lx}}{a_{Lx}}$$

Thus:

$$X_L = \sum_{x} (M_{FLx} \times a_{Lx})$$

Similarly:

$$X_{Lc} = \sum_{+} \frac{M_j^2}{M_{Lc}} = \frac{N_{Lc} M_{FLc} M_{FLc}}{M_{Lc}} = \frac{M_{Lc} M_{FLc}}{M_{Lc}}$$

$X_{Lc} = M_{FLc}$

Therefore:

$$IH_L = \frac{(1-2a_{Lc})}{a_{Lc}} M_{FLc} + \sum_{x} M_{FLx}\, a_{Lx}$$

A rough screen analysis can provide all the numbers required for this calculation. All terms in this formula can either be calculated or estimated when one has a ***rough idea of the particle size distribution,*** and a rough screen analysis gives you all the information required. The average weight of the fragments of a particle size class can be estimated in two different ways; either by direct measurement of the weight of a given number of fragments selected at random within the class, or by calculation using the following two formulas:

$$M_{FLc} = v_{FLc}\lambda = f\lambda d_{FLc}^3$$

$$M_{FLx} = v_{FLx}\lambda = f\lambda d_{FLx}^3$$

The average dimensions of the fragments can be calculated using:

$$d_{FLc} = \sqrt[3]{\frac{(\text{upper opening})^3 + (\text{lower opening})^3}{2}}$$

$$d_{FLx} = \sqrt[3]{\frac{(\text{upper opening})^3 + (\text{lower opening})^3}{2}}$$

Hence:

$$IH_L = f\lambda\left[\left(\frac{1}{a_{Lc}} - 2\right)d_{FLc}^3 + \sum_x d_{FLx}^3 a_{Lx}\right]$$

So from a size distribution analysis this can be estimated quite accurately. Primary interest is in the proportions of size fractions that do not exceed 25%. The second term in the above equation represents other size fractions of interest. Again if a_{Lc} is small then the term $\sum_x a_{Lx} \approx 1$ and the last term on the right can be approximated using the particle size distribution factor g, so we write:

$$IH_L = f\lambda\left[\left(\frac{1}{a_{Lc}} - 2\right)d_{FLc}^3 + gd^3\right]$$

Then the variance of FSE will be:

$$s_{FE}^2 = \left(\frac{1}{M_s} - \frac{1}{M_L}\right)f\lambda\left[\left(\frac{1}{a_{Lc}} - 2\right)d_{FLc}^3 + gd^3\right]$$

This formula can often be simplified if:

- $M_L > 10M_S$
- d_{FLc} is not much different from d

- a_{Lc} is small

Then:

$$s_{FSE}^2 = \frac{f\lambda}{M_s}\left(\frac{1}{a_{Lc}} - 2\right)d_{FLc}^3$$

NB*:* If the coarse fragments represent a large proportion of the lot, approximately more than 20%, the term gd^3 ***should be kept.***

Assessment of the representivity of a sample: If a sample is *to be representative of all the fragment size fractions,* then it should be representative of the largest fragments which is the most difficult condition to fulfill. Thus, by definition it can be written: $d_{FLc} = d$ and $a_{Lc} = 5\% = 0.05$, then:

$$s_{FSE}^2 = 18f\lambda\frac{d^3}{M_s}$$

Assuming, of course, that the investigated material is not calibrated so g ~ 0.25. This equation is easy to use and provides a value for the FSE for relatively little effort.

15.2 Calculation Examples (Examples a-d)

Application of these equations is shown in the following sections. Worked answers for many of these examples are provided at the back of this book.

a. What mass of soil should be sampled to evaluate heavy metals assuming that the precision of FSE is given as ±15% and interest is in the coarse fraction?

From a regulatory standpoint *d* is about 1cm so that $a_{Lc} = 5\% = 0.05$ and the standard values for f = 0.5 and a density λ ~2.6 are used:

$$\begin{aligned} M_s &= \frac{f\lambda}{s_{FSE}^2}\left(\frac{1}{a_{Lc}} - 2\right)d_{FLc}^3 \\ &= \frac{0.5\times 2.6}{(0.15)^2}\left(\frac{1}{0.05} - 2\right)\times 1^3 \\ &= 57.78\times 18 \\ &\approx 1040\text{g} \end{aligned}$$

The generally accepted procedure would be to assay 100g of soil with a weak acid (TCLP test). The result shows that 100g is too small in magnitude and so it would be better to use 1 040g of material.

b. What mass is required if one wants to sample broken ore at a size of 20cm and a density of 2.5?

$$M_s = \frac{f\,\lambda}{s_{FE}^2}\left(\frac{1}{a_{Lc}} - 2\right) d_{FLc}^3$$
$$= \frac{0.5 \times 2.6}{(0.15)^2}\left(\frac{1}{0.05} - 2\right) \times 20^3$$
$$= 55.55 \times 18 \times 8000 \approx 8000000$$
$$\approx 8\text{tons}$$

The above indicates that there is way of properly representing the coarse fraction with the types of samples. The only point at which one could properly sample this material is at a draw point.

c. Given the same conditions, in order to sample a kimberlite dump with a top size of 12cm and a density of 2.5, about 1.8 tons of material would be required. This does not mean the sample would be representative for estimating the diamond contents; it simply means that if the lot is very large and if the mass of the sample is less than 1.8 tons the sample cannot be representative of anything.

d. Similarly, a stream of manganese ore with a density of about 3.8 and a top size of 1.9cm would require about 10.4kg of material.

$$M_s = \frac{f\lambda}{s_{FSE}^2}\left(\frac{1}{a_{Lc}} - 2\right) d_{FLc}^3$$
$$= \frac{0.5 \times 3.8}{(0.15)^2}\left(\frac{1}{0.05} - 2\right) \times 1.9^3$$
$$= 84.44 \times 18 \times 6.859 \approx 10420$$
$$\approx 10.4\text{kgs}$$

Caution should be exercised when using this formula; if it's not representative for the size fraction, it is not representative for anything including the metal. For gold mineralisation it is essential to find out how and where the gold is distributed and to screen out the particular size fraction carrying the gold and analyse this fraction. Again, this does not mean the sample would be representative for estimating the manganese content; it simply means that if the lot if very large and if the mass of the sample is less than 10 Kg the sample cannot be representative of anything.

15.3 Calculation of the Mineralogical Factor, c

The mineralogical factor is the maximum heterogeneity the sample can attain if liberation of the constituent of interest is complete. At this point there is a direct correlation between *c* and the grade of the material. The lower the grade the bigger the problem that will be encountered. Large values for *c* mean that one is faced with large sampling problems, something most people generally fail to take account of.

$$s_{FSE}^2 = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \times fgc\ell d_N^3$$

Samples of gold ores have been found to contain around 2ppm, in which the gold occurs as small nuggets with a density of about 16.0g/cc (the average density of the surrounding porphyry is about 2.8g/cc). Calculate the mineralogical factor.

Another example: The gold ore contains 32g/t gold with the density of the gold particles at about 16.0 and the density of the in-situ host rock at 2.7t/m^3

15.4 Calculation of the Mineralogical Factor and the Liberation Factor

i. In a galena concentrate gold content is around 100ppm and occurs as native metal at a density of about 16.0 with the content at about 2 500ppm and has as a complex sulfide of formula AgPbSbS3 of approximate density 5.0. The density of the galena is 7.5, with atomic weights: Ag = 108, Pb = 207, Sb = 122, S = 32. Calculate the mineralogical factor for silver and gold in a galena concentrate ($AgPbSbS_3$)

ii. For the same ore as above calculate the liberation factors of gold and silver in this concentrate.

15.5 Calculate the Mineralogical Factor for Gold in Stream Sediment

The gold content of a reliable anomaly is arbitrarily fixed at 10ppb. The density of gold is assumed to be approximately 16.0. The background gold content is assumed to be undissolved by the cyanide leach procedure. Calculate the mineralogical factor for these sediments.

15.6 Calculate the Liberation Factor at Various Stages of Comminution

Gold ore is crushed to minus 4 inch (10cm) prior to reaching a cross-stream sampler, then to minus 1 inch (2.5cm) prior to reaching the secondary sampler, to minus 10 mesh (0.17cm) prior to reaching the tertiary sampler and finally to minus 100 # (0.015cm) prior to fire assay. Assume a gold grade of 32g/t. Calculate the liberation factor and FSE for each stage of the process. The liberation factor ℓ can be estimated using a form more familiar to process engineers, namely the liberation size, for situations where liberation is not complete when $d_N > d_\ell$, and the following model is acceptable for this purpose:

$$\ell = \sqrt{\frac{d_\ell}{d_N}} = \left(\frac{d_\ell}{d_N}\right)^{0.5}$$

The necessary data is provided in the following ***Table 9.5.***

Table 15.1: Factors necessary for calculating FSE

Fragment size (cm)	Liberation factor	K'	Sample mass, M_S
10	0.02	625000	40000
2.5	0.04	625000	20000

0.17	0.15	625000	5000
0.15	0.52	625000	500

15.7 Calculate the Liberation Factor at Various Stages of Comminution

Gold ore is crushed to minus 10cm (4 inch) prior to reaching a cross-stream sampler, then to minus 2.5cm (1 inch) prior to reaching the secondary sampler, to minus 0.17cm (10 mesh) prior to reaching the tertiary sampler and finally to minus 0.015cm (100 mesh) prior to fire assay. The average gold content of this ore is around 2ppm (0.058oz/t). A mineralogical investigation of the heavy fraction obtained from a gravity concentration shows that 85% (i.e. definition of the liberation size d_l) of the gold is liberated when the material is pulverized to minus 40 microns. Calculate the liberation factor of gold as the ore reaches:

a. The primary sampler:

b. The secondary sampler:

c. The tertiary sampler:

d. The balance room prior to fire assay:

15.8 Calculate the Liberation Factor at Various Stages of Comminution

The need is to determine the gold content of an underground deposit. A face sample is taken routinely for ore grade control. The average gold grade is expected to be around 32g/t. Samples are crushed to 95% minus 1.05cm, using a jaw crusher, before further splitting. A quick x-ray investigation shows that some of the fragments between 1.25cm (1/2 inch) and 0.635cm (1/4 inch) occasionally contain up to 7750g/t. Calculate the liberation factor of the gold in the average face sample as delivered by the jaw crusher.

15.9 Calculate the comminution size

From the tailings of an old mine a 50kg composite sample was collected. The maximum particle size *d* of the material is about 1/4 inch (0.67cm). The requirement is to send a sub-sample not heavier than 2kg to a laboratory. The 2kg sub-sample will be used to determine the average gold content of the tailings. The gold content is expected to be around 1ppm. It is known from a mineralogical study and former analyses that the gold is finely disseminated in the rock and that the maximum grade locally encountered in ¼ inch particles is no more than 100ppm. Use 16.0 for the density of gold. The allotted standard deviation of FSE is 2S = ±15% so S_{FE} = 7.5% which is the limit required by a feasibility study. To what size *d'* should the 50kg sample be crushed before splitting? Is the problem solvable with a 50kg composite? Is a 50kg sample of material with a liberation factor of l = 0.0001 and a total range of standard deviation of FSE equal to 15% adequate as a sample? Use the formula:

$$s^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \times cfg\ell d^3_N$$

15.10 Calculation of Sampling Parameters and FSE

A routine reverse circulation sample weighs about 60 000g. The material is 95% minus 1.05cm. The approximate gold content is expected to be around 1.5g/t. A Heterogeneity Test shows that fragments between 1.25cm (1/2 inch) and 0.635cm (1/4 inch) occasionally contain up to 350g/t gold. The sample is submitted to the following protocol:

Step 1: The material is split as received at the drilling site until a 4 000g sample is obtained.

Step 2: At the laboratory the 4 000g sample is crushed to 95% minus 0.335cm (6 mesh) using a roll crusher.

Step 3: The minus 0.335cm (6 mesh) sample is split to about 250g.

Step 4: The 250g sample is pulverized to 95% minus 0.015cm (100 mesh) using a plate pulverizer.

Step 5: A 30g sub-sample is finally collected for fire assay.

1. Calculate the sampling constant C for each sampling stage. (Use 16.0 as the density of gold particles.)
2. Calculate the constant factor *K* valid for all stages of comminution.
3. Calculate the standard deviation of FSE for each sampling stage (expressed in relative %).
4. Calculate the standard deviation of the total FSE (expressed in relative %).
5. What are the weak points of this protocol?
6. What can we do about them?

16 THE IN-SITU NUGGET EFFECT (INE)

16.1 Characteristics of the INE

The well-known geostatistical term “nugget effect”, refers to the inherent random variability of ores at time or distance zero V(j=0), on the experimental semi-variogram. The so-called “In situ Nugget Effect” was identified and named by Dr FF Pitard (1993), as a specific sampling error, and he believes that the term Nugget Effect by itself is misleading as a large part of it, V(j=0) has nothing to do with Nugget Effect. He emphasises that the true In Situ Nugget Effect, is grade variability in the rock before it is fragmented. Pitard (2009) considers the INE to be inherent in and specific to the internal constitution of the ores being investigated and arises because of the clustering of numerous small gold grains or the occurrence of larger individual grains referred to as nuggets. An illustration of the two forms of INE clusters and large individual grains are shown in ***Figures 16.1a*** and ***16.1b.***

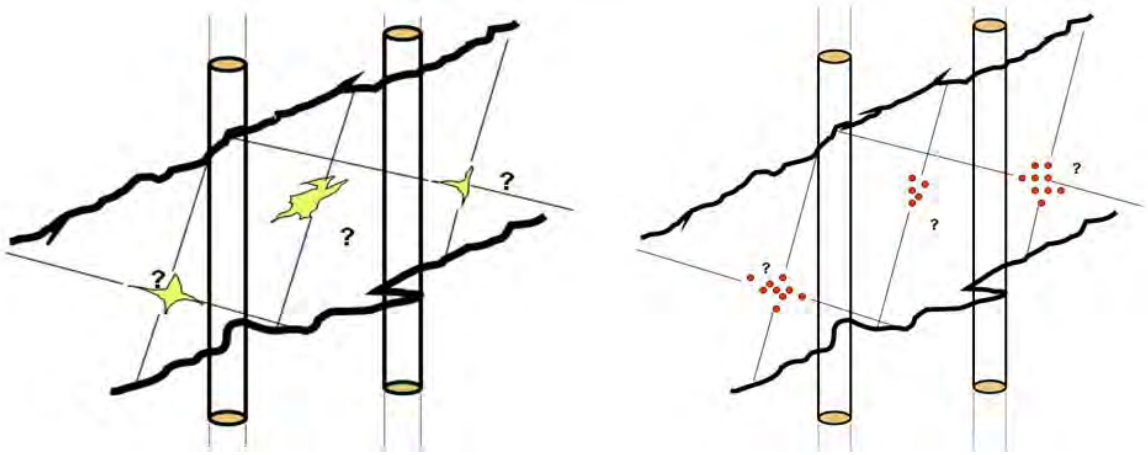


Figure 16.1a: Gold occurring as solid nuggets, and b) Gold occurring as clusters of very small grains (Pitard, F, 2009)

The random variability is a combination of errors arising from primary sampling, sub-sampling and analytical variance, including FSE, GSE, the aggregated IDE, IEE, IPE, and IWE errors associated with n steps of comminution and splitting, and the AE. According to Pitard (2009) the original Theory of Sampling did not include “the uncertainty created by the selection of a certain mass for an unbroken module of observation during diamond core, reverse circulation, or blasthole drilling” (p183). Pitard (1993), is of the opinion that the random variability [V_0], also includes a component of the true In Situ Nugget Effect, an error inherent in the ore to be sampled before the material has been fragmented by blasting. Lyman (2011) on the other hand considers the relationship between the variance associated with the sampling of an in-situ mineral material, and the intrinsic (constitutional) heterogeneity once it is broken, to be a somewhat grey area of sampling theory.

Lyman (2011) examined in situ heterogeneity of ore bodies by modelling the behaviour of variograms of discrete mineral grains by calculating variogram functions for model mineral phases as they might appear in thin sections. In this way he demonstrated that a mineral texture has an associated variogram which

characterises its spatial covariance. This was done by modelling point-counts and finite increment volumes of discrete mineral grains representing the phase of interest as they might appear in an ore texture under a mineralogical microscope. In this way covariance functions for model, both nuggety and non-nuggety, consisting of cubic grains for each of 2 phases, at concentrations as low as 1.62%, were calculated. Variograms were then calculated from 10000 isotropically and randomly orientated points for a series of different sample sizes. The process is in fact geostatistical modeling at a very small scale, and reflects the typical change of support effects of an increase in the range and a decrease of the sill as the sample size increased (Lyman, 2011). In all of his analyses Lyman was able to demonstrate that the variograms all pass through the origin irrespective of sample size. From these results he deduced that any real nugget effect observed in a variogram arises only from the sampling, preparation, and analytical variance of in situ sample.

The results of Lyman's research indicate that generally there is no direct link between in situ heterogeneity and particulate heterogeneity of an ore. There is however a special case that refers to diamonds, but is also true for nuggety gold where, for a given state of comminution, not much of the gold is liberated and the average grade does not vary much with spatial position. In such cases the sampling constant does not depend on the state of comminution, and the comminuted sample will have precisely the same grade variance as that of the particulate sample, provided the in situ and particulate samples have the same mass (Lyman, 2011).

In conclusion Lyman (2011) proposes that the number of samples per cubic metre required to reduce the relative estimation error to a fixed target value is the appropriate measure of the in situ heterogeneity of an ore body. The support of such samples must of course be standardised. He mentions that this measure of in situ heterogeneity, which will vary depending on the analyte of interest, is parallel to the use of the sampling constant K_S as a measure of heterogeneity in particulate material.

In regard to the INE Pitard (2009) states, "Unless this uncertainty is properly understood it can result in major sampling errors, especially on the selection of diamond drill diameters". Generally the INE is referred to as an error rather than an uncertainty, and it arises because precious and base metal mineralisation may occur as larger individual nuggets, as numerous small grains of sulphides, or as clusters of small grains. Typically gold, silver, platinum, copper, arsenic, molybdenite, and tin mineralisation (cassiterite) occur in this form, as do the deleterious elements in iron, alumina, manganese, and chromite ores. The important point is that the primary natural occurrence of these metals is as nuggets or clusters of grains rather than being evenly disseminated throughout the host rock giving rise to a lognormal distribution, with many low values, and a few very high grade values. It is the very few high grade values that lie in the tail-end of the lognormal distribution that give rise to the INE. An illustration of the two forms of INE clusters and large individual grains are shown in Figures 16.1a and 16.1b.

Statistically these two types of gold deportment generate exactly the same kinds of sampling problems. Molybdenite mineralisation tends to do exactly the same thing in terms of its distribution in the porphyry host rocks. The molybdenite occurs as clusters of grains rather than being evenly disseminated throughout

the rock. Generally the geologist has to log many meters of core before he gets a good feel for the size of the molybdenite aggregates. Different types of gold mineralisation in borehole core and channel samples across Witwatersrand-type strata-bound reefs, should be observed, noted and emphasised, especially since the size of the gold grain or the equivalent size of a small-grain aggregate will affect the sampling behaviour of the ores ***Figure 16.2***.

Both basic[80] and elementary[81] instruction in sampling of Witwatersrand-type reefs is provided for gold and platinum mine employees through the South African Chamber of Mines and the Gold Fields Academy. These courses provide a well-rounded and specific approach to sampling and metal accounting issues as they relate to narrow, gold-bearing placer reefs of the Witwatersrand Supergroup. They emphasise, determination of ore value, compilation of the ore reserve, efficient control of grade, potential life of mine, and identification of unpayable ores, as reasons for regular sampling. In terms of support the so-called narrow-reef channel sample is identical to a borehole intersection except that the method and position of sample recovery is different. Consequently errors arising from the insitu nugget effect (INE) in regard to borehole sampling are also directly applicable to channel sampling. The principle difference is that samples collected during channel sampling are broken and there may be some liberation of gold particles.

The common practice of splitting borehole core with a diamond saw in order to sample half and preserve half of the core as a record, is a practice which immediately doubles the variance of the In Situ Nugget Effect associated with the selected support mass. Very rarely do we consider the damage that is inflicted on the results because of sampling split core.

Figure 16.2: Typical gold nugget and the cluster equivalent; the upper line for the local agglomerate is a little lower, so the equivalent agglomerate size takes voids between mineral grains into account **([40]Pitard, F, 2009.)**

In the general case of borehole intersections the ore is not broken, but it is still necessary to quantify the INE arising from the cluster equivalent. For the in-situ case where there is no liberation, the liberation factor plays no role in calculations using Gy's formula. In the case of a cluster, the size of the largest cluster or agglomerate is equivalent to the size of the mineral of interest, d_m. A number of examples illustrate the method.

16.2 Exercises in Calculating INE

[80] Chamber of Mines. 2008. Basic mine sampling. Gold Fields Business and Leadership Academy, GFBLA/MRM-EVA-BASAMP.118p.
[81] Anderson, C. J. 2000. Elementary mine sampling. Gold Fields Trust, GFA/MIN/SER/M008. 95p.

16.2.1 Arsenic impurity

A porphyry copper deposit in Chile is drilled for its arsenic content. For the floatation process to deliver copper concentrates within arsenic specifications for the smelters the mine must either eliminate or carefully blend any ore with an arsenic content higher than 100ppm. The exploration program uses 2m-lengths of half-NQ diamond core drilling samples. It is common to observe a cluster of arsenic minerals or neighbouring veinlets within a tiny volume (approximately 1lt), more or less equivalent to a single 5cm particle.

Determine what the sample mass should be to ensure the relative standard deviation s_{INE} is no larger than ±15%. Also calculate how many 2m half-NQ core samples, full NQ core samples, half-HQ core samples, full HQ core samples and RC 50kg samples should be averaged before obtaining a relevant database for ore grade control purposes. Do the same exercise if the cluster agglomerate equivalent was 1cm and if the expected grade was 500 ppm, or 20 ppm. Use the equation for the in-situ nugget effect and rearrange for mass:

$$s^2_{INE} = \frac{c \times f \times g \times d^3}{M_S}, \text{ where } c = \frac{\lambda_M}{a_L}$$

$$M_S = \frac{f \times g \times \lambda_m \times d^3}{s^2_{INE} \times a_L}$$
$$= \frac{0.5 \times 0.25 \times 4.5 \times 5^3}{(0.15)^2 \times 0.0001}$$
$$= 31.2 \text{ tons}$$

A very useful means of representing this problem is through the nomogram of fragment size, in this case the size of aggregates or nuggets, against sample mass as shown in ***Figure 16.3***. This representation of the mass-cluster or nugget size relationship indicates how sample mass will change as the size of the clusters or nuggets in the borehole core changes. A rapid change in sample mass with a changing size of nuggets is evident so that going from 5cm to 1cm means that the required mass reduces from 31t to 250kg – a remarkable result!

16.2.2 Molybdenum by-Product

A porphyry copper deposit in Chile is drilled for its molybdenite content. The expected average molybdenite content is 0.040%. The exploration program uses half 2mt NQ diamond core drilling samples. For molybdenite it is common to observe a cluster or neighbouring veinlets within a tiny volume (approximately 1lt) more or less equivalent to a 1cm single particle.

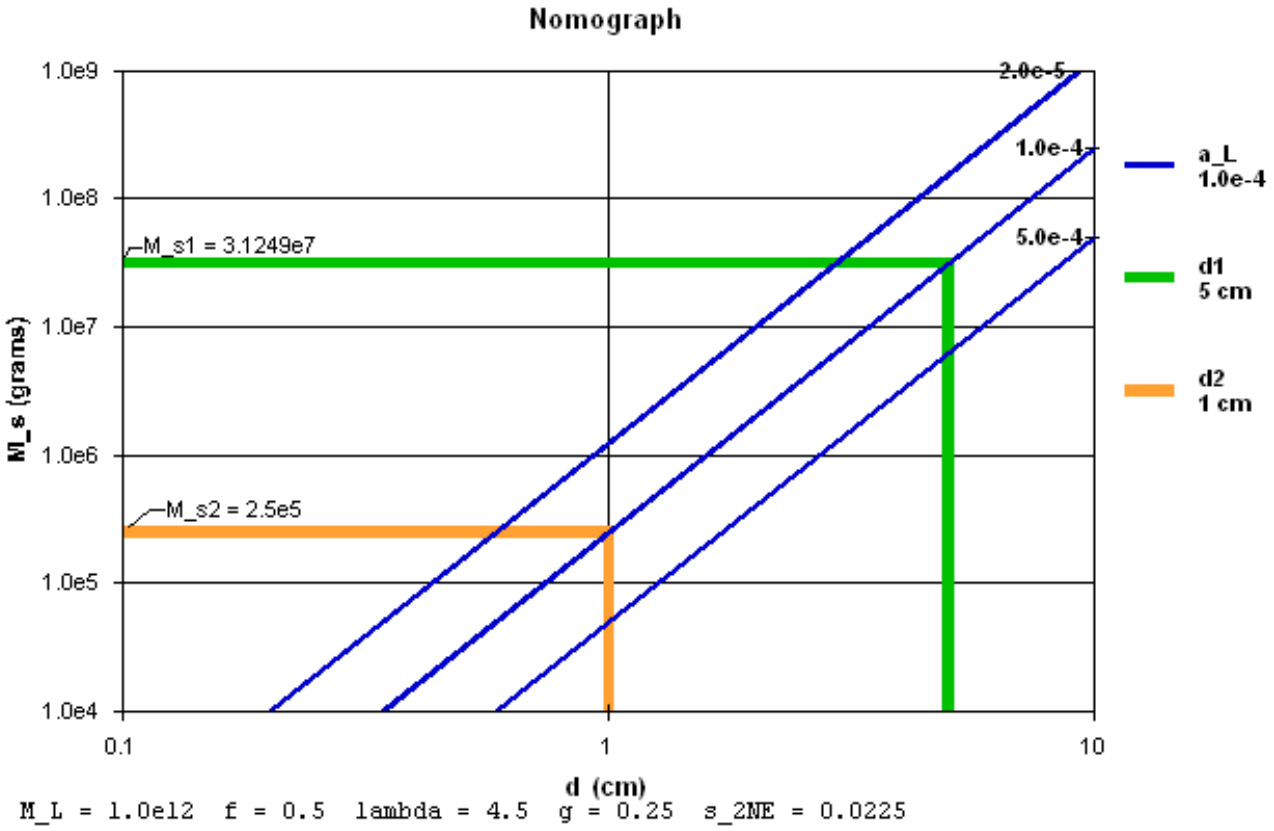


Figure 16.3: Nomogram of sample mass vs. nugget (cluster) size in coarse-grained gold ores **([40]Pitard, F, 2009.)**

Establish what the sample mass should be to ensure the relative standard deviation S_{INE} is no larger than ±15%. Also determine how many 2m lengths of half-NQ core samples, full NQ core samples, half-HQ core samples, full HQ core samples and RC 50kg samples should be averaged before obtaining a relevant database for ore grade control purpose. Do the same exercise if the cluster agglomerate equivalent was 0.3cm and if the expected grade was 0.100% or 0.020%.

Using the modified version of Gy's formula the required mass of sample material as shown in the nomogram of ***Figure 16.4*** is about 63 kg. If the size of the cluster equivalent is 0.3 cm the mass required reduces to about 1.7 kg.

Again, as with the previous example, the effect of reducing the top size of the clusters or nuggets is substantial. If the nugget size changes from 1 cm to 0.3 cm the required sample mass is reduced from 62 kg to around 1.7 kg.

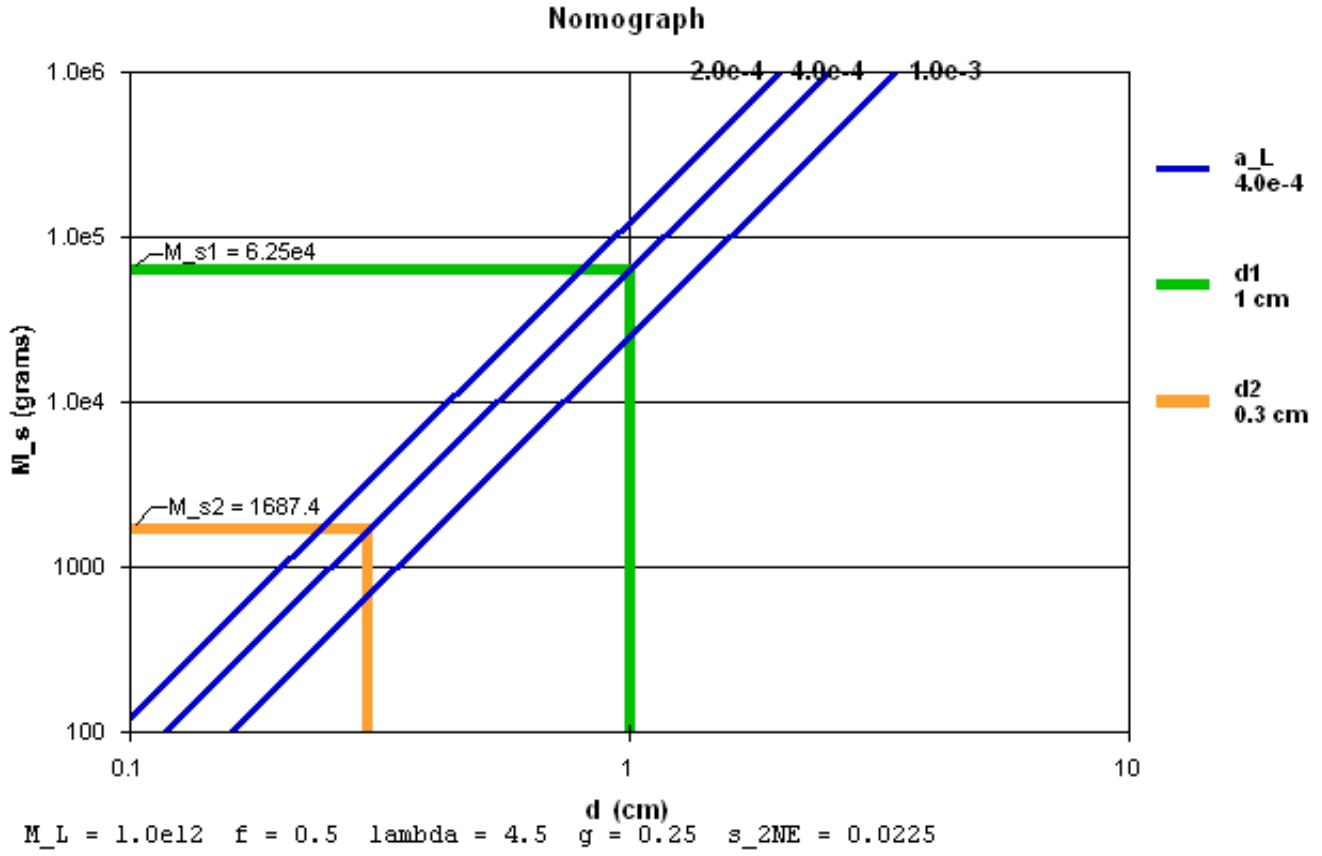


Figure 16.4: Nomogram for molybdenite cluster size vs. sample mass in a porphyry deposit **([40]Pitard, F, 2009.)**

16.2.3 Coarse gold and clustering of fine gold

Exploration of a deposit, with coarse, commonly visible gold, in Brazil using HQ core is expected to give an average grade of 1.50g/t. The exploration program uses full 3mt HQ borehole core samples. Clusters of several gold particles amounting to a single particle with an equivalent size of 0.25cm can be observed in within a tiny volume, approximately one liter.

Calculate what the sample mass should be to ensure the relative standard deviation S_{INE} is no larger than ±15%. Also determine how many 3mt full HQ core samples and RC 60kg samples should be averaged before obtaining a relevant database for ore grade control purpose. Do the same exercise if the cluster agglomerate equivalent was 0.05cm and if the expected grade was 0.75g/t, or 3.00g/t.

Using a density of 16 for gold (because it is probably an amalgam of gold and silver) suggests sample masses of 370kg for material at a top size of 0.25cm and a mass of 3kg if the nugget size is reduced to 0.05cm. The nomogram solution for this example is shown in ***Figure 16.5.***

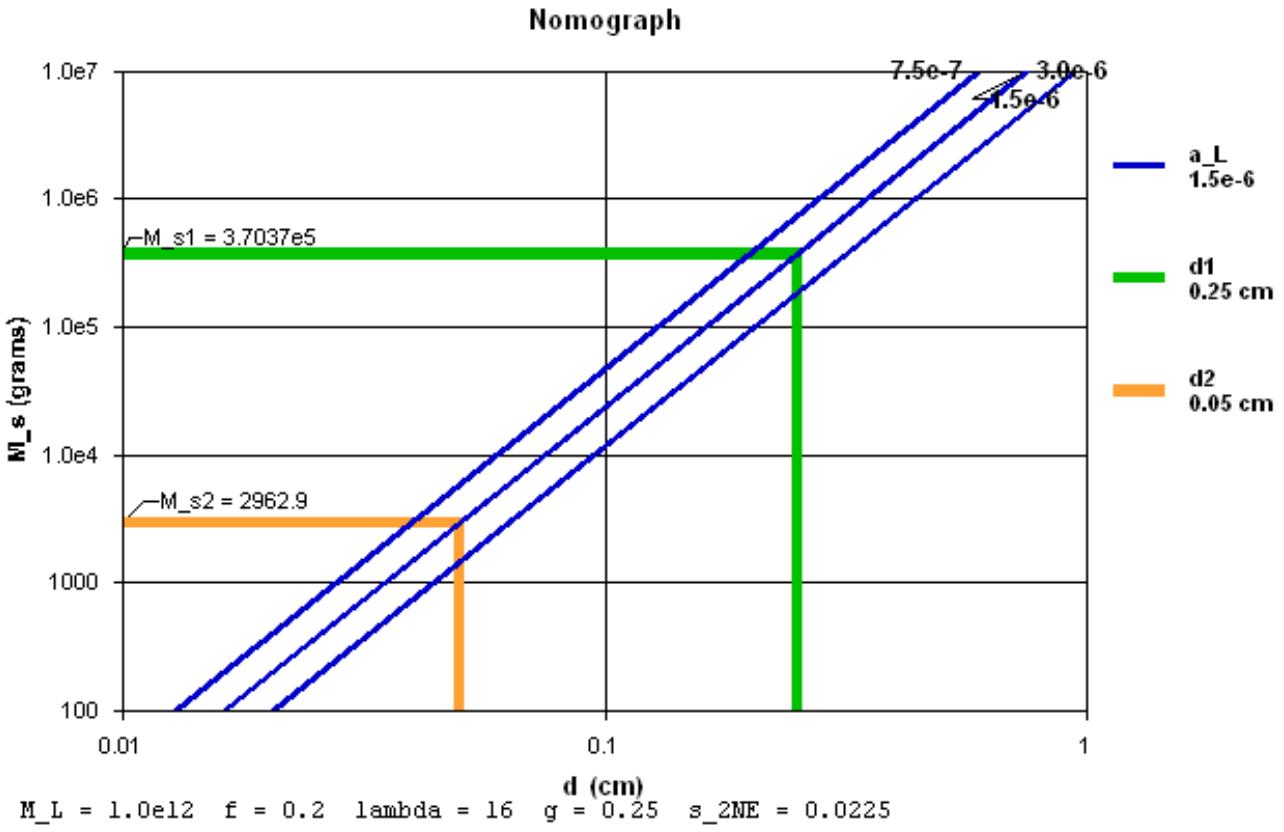


***Figure 16.5: Nomogram for nugget or equivalent cluster size vs. sample mass for coarse-grained and nuggety gold* (**[40]**Pitard, F, 2009.)**

The message from these examples is that it is essential to fully understand the mineral deportment in the materials being sampled. Sample mass depends critically on the size of the nuggets or cluster equivalents and without a proper understanding of how the minerals of interest occur, significant errors can be introduced because incorrect sample masses are collected. This will also affect the number of borehole intersections required from a specific reef if the mineralisation is particularly nuggety.

In regard to channel samples, it is essential to understand the gold deportment in order that sufficient sample material is collected. In the case of errors arising from the in-situ nugget effect, the size of channel samples is significant.

16.3 Determination of Low-Background Mineral Content *L*

Besides the nuggets which occur in an ore, a certain proportion of the mineral of interest may be an easy-to-sample, low-grade background type of mineralisation referred to as the low-background content *L*. Sometimes sampling protocols are selected in such a way that one is capable of effectively accounting for only the 2/3^{rds} low-background content, while substantial proportion 1/3rd remains far more elusive and possibly unaccounted for as illustrated in ***Figure 16.6.*** The problem with exploration drilling programmes in rocks mineralised in this way is that a boreholes intersection of nuggets will return very high grades. Because the balance of assay values is low, the high value is likely to be cut and the project could be made non-viable. In such cases it is essential to estimate low-background mineral content *L*.

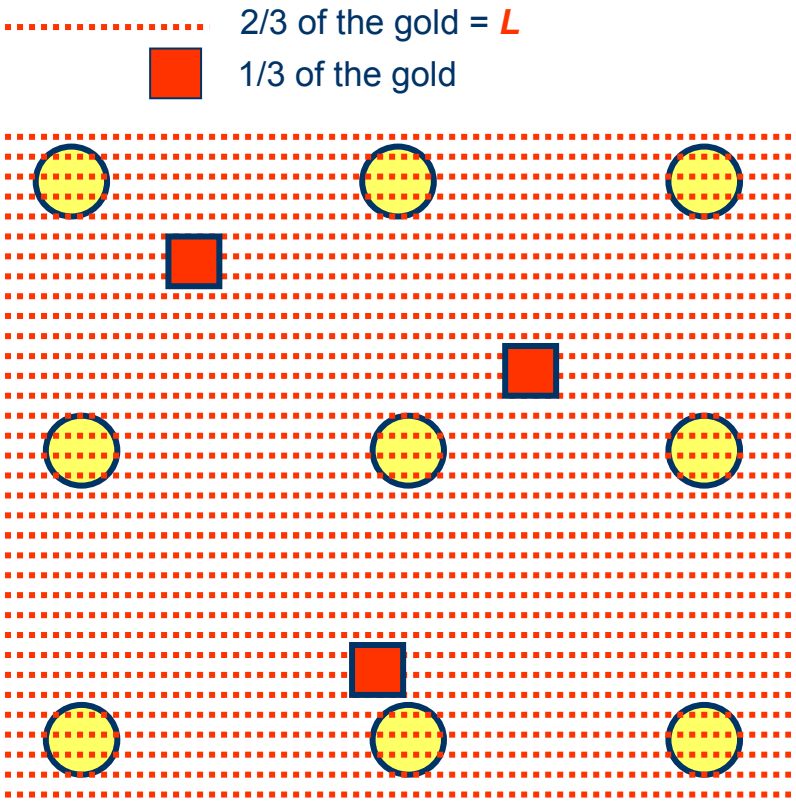


Figure 16.6: The low-background content accounts for nearly two thirds of the gold while the coarse grained gold for about a third. Drill patterns invariably miss most of the coarse gold leading to significant underestimation **([40]Pitard, F, 2009.)**

Method 1: Using the results of the heterogeneity test to plot the gold grade in g/t against the samples ordered by increasing % gold as shown in ***Figure 16.7***, reveals the inflection points in the curve generated in this way, and provides confirmation of the presence of low-background content *L* and an indication of the grade of the ore and of the coarse-grained gold.

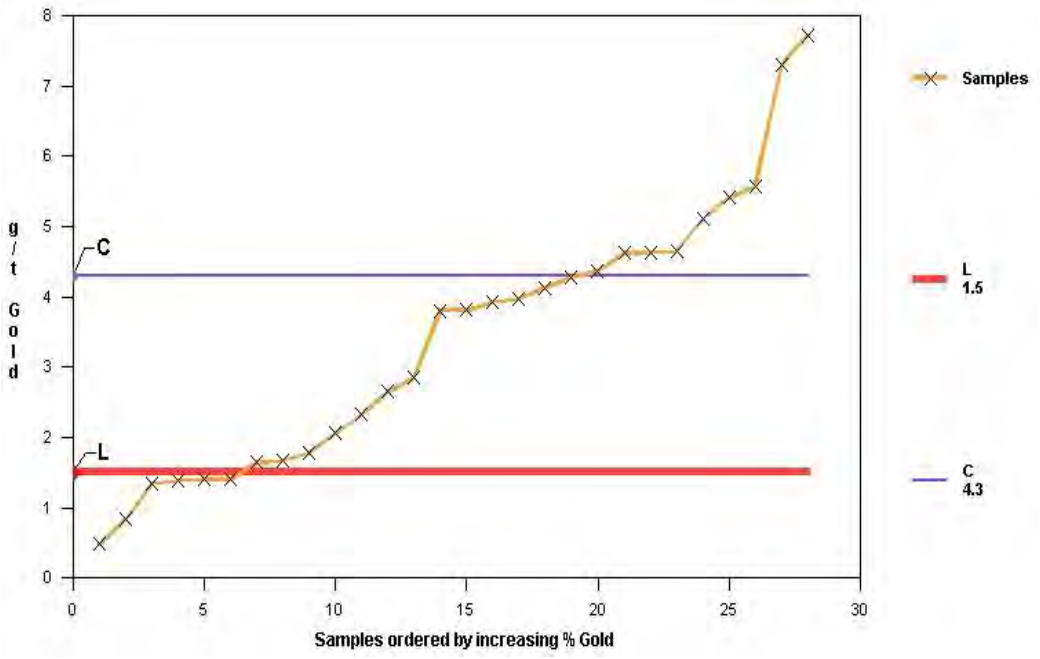


Figure 16.7: Low-background gold content using results from a heterogeneity test **([40]Pitard, F, 2009.)**

Method 2: A second method for estimating the low-background content *L* requires two series of samples, M_{S1} and $M_{S2,}$ with different masses. As the sample mass diminishes the assay results are more highly skewed and the mean and mode move farther apart. If the mode is taken as the most probable assay value **Y** of a single assay, two sets of assays using sample masses M_{S1} and M_{S2} will yield two modes, Y_1 and Y_2. Ingamells suggested the following formula as a solution to evaluating the grade of the low-background content *L*

$$L = \frac{Y_1 \cdot M_{S2}[X - Y_2] - Y_2 \cdot M_{S1}[X - Y_1]}{M_{S2}[X - Y_2] - M_{S1}[X - Y_1]}$$

Where:

Y_1 is the mode of the grades of the small sample mass, the most probable grade for the small samples

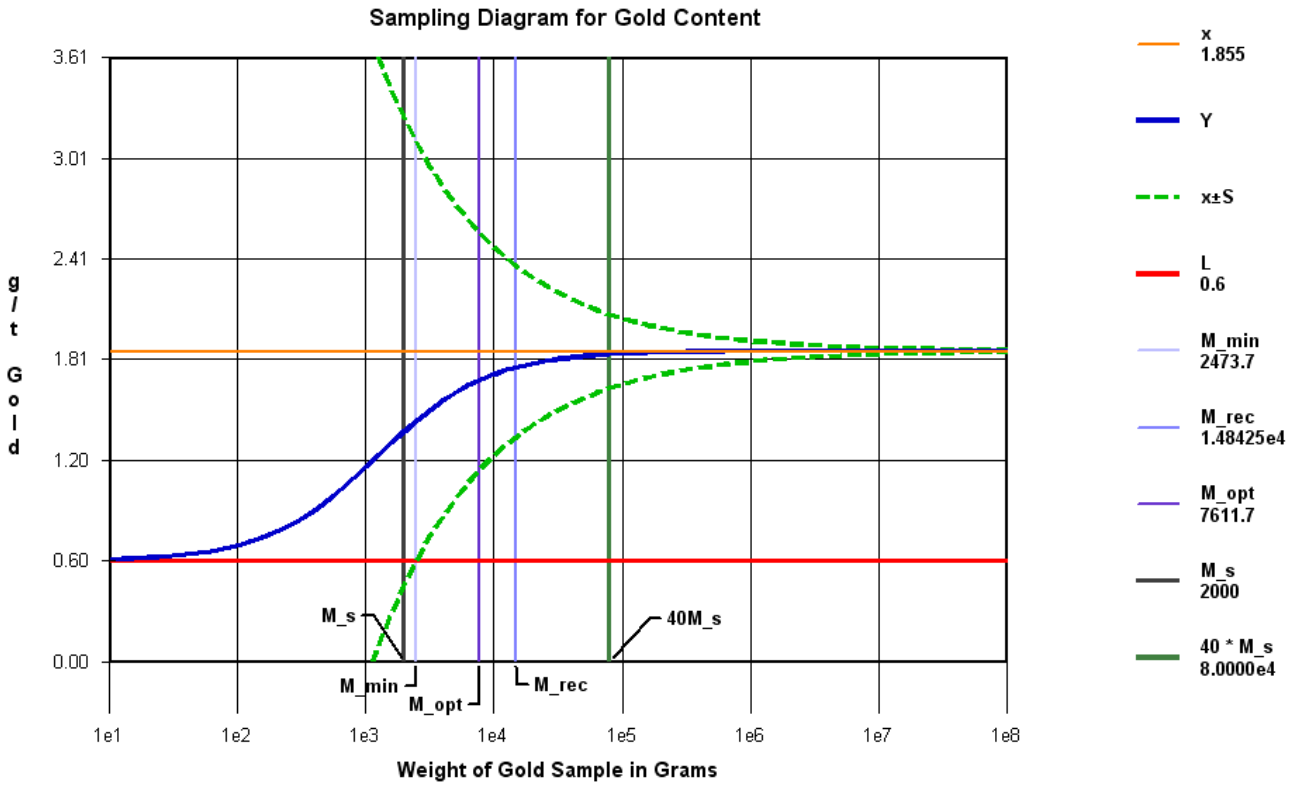
M_{S2} is the mass of the large samples

Y_2 is the mode of the grades of the large sample mass, the most probable grade for the large samples

M_{S1} is the mass of the small samples

X is the average for all samples

Values for Y_1 and Y_2 can be estimated visually from histograms of data from two sets of samples with different masses. It is preferable to derive values of Y_1 and Y_2 by calculating the harmonic means of the distribution.




```
Field variability for very large samples = 1.4309
N_samples = 40 and M_s = 2000 Grams
provides overall precision S(8.0000e4) = 0.2206 g/t and S_VR = 11.896%
M_s1 = 2000  S_1 = 1.5684  S_1R = 84.551%
```

Figure 16.8: Sampling diagram for gold content showing gold grade versus sample mass for material consisting of a low-grade background with higher grade nugget effect **([40]Pitard, F, 2009.)**

Using the above equation for L it is now possible to construct a Sampling Diagram, an example of which is shown in Figure 10.9 that shows the most probable result for a gold assay depending on the mass of sample material used for the assay. The dark blue line starts at a value of L for very small masses, about 1g, and before it reaches the mean value of the lot the required mass of the sample increases to 80kg, i.e. 40 x 2000, or 80kg (***Figure 16.8***). The dark blue line shows the most likely result to be returned by the assay lab. This graphic also indicates the minimum sample mass for different levels of precision of the assay result, and highlights the problem that if the sample mass is too small, it is possible that blocks above cut-off are sent to the waste dump and blocks below cut-off are sent to the mill.

16.4 Estimation of the Variance (V_{INE}) of the True INE

The variance at V[j = 0] in ***Figure 16.9*** represents all the accumulated variances associated with collecting a suite of samples for evaluation purposes taken in a mineral deposit. It also includes true INE which is variability affected by the volume of observation. For kriging to have meaning the ratio of V[j=0] to sill should not exceed 10% relative. If INE is greater than 30% of the total sill, warning bells should ring. Of course, such statement must be placed in the context of where the sill is. For example, if there is very little field variability V[j=0] may be close to the sill and it does not necessarily mean something is wrong with V[j=0].

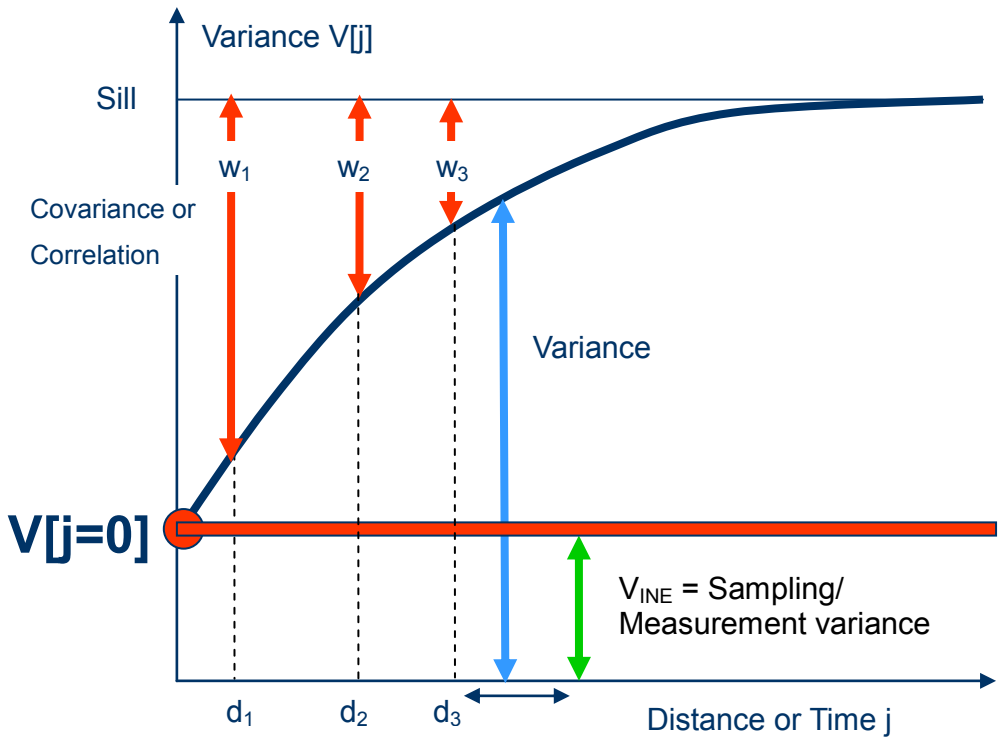


Figure 16.9: Semi-variogram showing INE and the co-variances that should be used as weights when grades are estimated based on distance estimation

The larger V_{INE} is, in terms of its contribution to V[j=0], the more likely it is that other variances contributing to V[j=0] will be large as well because of the cascading effect of heterogeneity. Having put in the required effort to establish a good protocol one would assume that: the protocol is optimised, the sampling equipment is correct and that AE is minimised. It should be remembered that whilst it may be possible to reduce V_0 it can never be eliminated. Although there are no hard and fast rules, the higher INE the less likely that the semi-variogram can be used for spatial estimation purposes. Furthermore, if INE constitutes a very high proportion of the total sill and relative to the average ore content, it is almost certain that there is a problem associated with the sampling system.

If V_{INE} and V_0 are left unresolved, serious economic consequences could follow and the modeling of a variogram is nothing other than an exercise in futility with kriging techniques that don't work well. One solution to the problem could be that evaluators cut high grades and as a result ignore the mean minus background [X-L] part of the resource. Reconciliation problems will be exacerbated and the optimum recovery of natural resources becomes an elusive task, the economic consequences of which are staggering.

Finally, there are some recommendations as to what can be done if V_{INE} is very large. Consider selecting as large a drilling diameter as possible. Quantify the changes in variance with the changes in block size of the smallest mining unit and any shortcomings in terms of the basic module mass of observation, such as N x M_S. Ensure that the geological ore body modeling input is sound, taking cognisance of natural boundaries and key indicators. For important "money-making" constituents make sure selected ore grade cut-off grades are below L. Rely on low-grade stockpiles. Do not cut any high grade for the calculation of the average overall grade. Learn to live with reconciliation problems and ensure that management is aware of the effects of precision ellipses.

17 GROUPING AND SEGREGATION ERROR (GSE)

17.1 Characteristics of GSE

GSE is an entirely natural phenomenon arising in lots and samples when materials of different densities in a mixture segregate under the influence of gravity. Many material-separation processes use this technique to achieve their objective; a technique whose effectiveness is directly proportional to the difference in density. In order to fully understand GSE it is necessary to go back to the concepts of CH and DH, especially DH, and in this case reference is made to the DH of small, movable lots only. It should be noted that homogenization can greatly reduce DH, but cannot change CH (***Figure 11.1***).

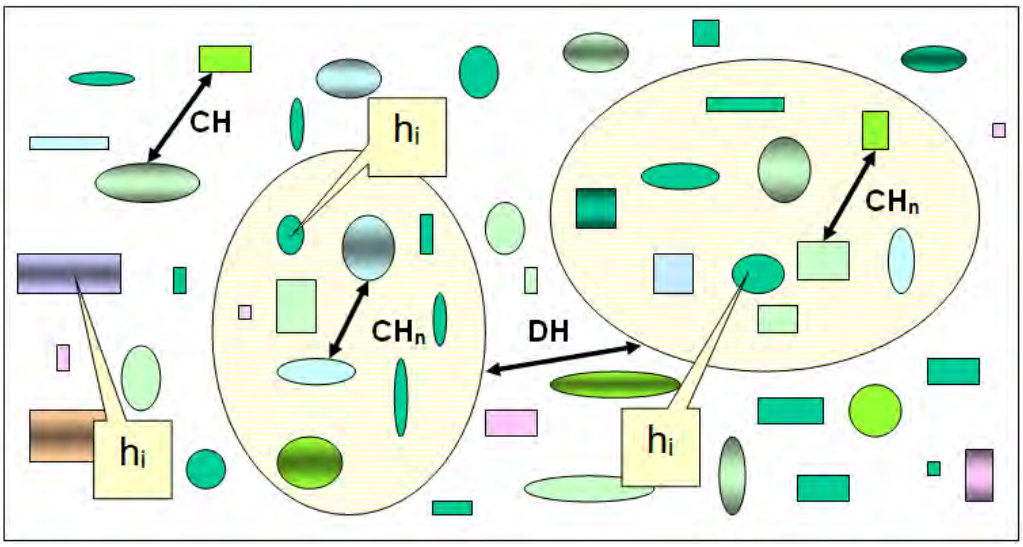


Figure 17.1: Comparison of constitution and distribution heterogeneity in a lot showing DH between more-or-less similar sized increments and the local CH within a lot **([40]Pitard, F, 2009.)**

With reference to the additivity of variances, it should be noted that the variability of fragments of a lot is equal to the variability of increments *plus* the variability between fragments within increments. The following definitions are relevant:

a_n : The content or grade of a given group of fragments
a_{nj} : The content or grade of a given fragment in a given group
h_n : The heterogeneity carried by a given group
h_{nj} : The heterogeneity carried by a given fragment in a given group

M_n : The weight of a given group
M_{nj} : The weight of a given fragment in a given group
N_G : The number of groups of a given size to empty the lot
N_{nj} : The number of fragments in a given group

First, calculate the heterogeneity h_{nj} carried by one fragment in one given group or increment. In exactly the same way as CH_L of a lot was defined, so too can the CH_n of a group of fragments as the relative, dimensionless variance of the heterogeneities h_{nj} generated by N_{nj} fragments within that group, be defined.

$$CH_n = s^2\left(h_{n_j}\right) = \frac{1}{N_{n_j}} \sum_{n_j} h_{n_j}^2$$

What is of interest is the average $\overline{CH}$, carried by all groups of fragments.

$$\overline{CH_n} = \frac{1}{N_G} \sum_n CH_n$$

Define the heterogeneity h_n carried by one group of fragments:

$$h_n = \frac{(a_n - a_L) M_n N_G}{a_L M_L}$$

The DH_L of the lot is defined as the relative, dimensionless variance of the heterogeneities h_n generated by N_G groups of fragments.

$$DH_L = s^2(h_n) = \frac{1}{N_G} \sum_n h_n^2 = \frac{N_G \sum_n (a_n - a_L)^2 M_n^2}{a_L^2 M_L^2}$$

$$\begin{array}{ccccc} CH_L & = & DH_L & + & \overline{CH_n} \\ & & \Downarrow & & \Downarrow \\ & & \text{Between} & & \text{Within} \\ & & \text{increments} & & \text{increments} \\ CH & \geq & DH_L & \geq & 0 \end{array}$$

At the limit $CH_L = DH_L$ when there is only one fragment. (CH_L in formula above)

17.2 DH_L Variability Domain

The maximum of DH_L is CH_L itself when $CH_n = 0$

$$(DH_L)_{max} = CH_L$$

The minimum of DH_L is defined as an absolute minimum $(DH_L)_{min}$ under which it is impossible to homogenize the material further. It is the inaccessible limit at which each group of a lot could be regarded as a random sample. This minimum is actually a random variable, the mean of which can be related to CH_L as follows when the number of groups and fragments are large numbers:

$$\text{Mean}(DH_L)_{min} = \frac{N_G}{N_F} CH_L$$

17.3 Definition of Grouping Factor Y

$$\frac{1}{Y} = \frac{N_G}{N_F}$$

This presentation becomes a problem when Y = 0. But, N_G and N_F are very often different values and Y is often a large number itself. Therefore, the following approximate presentation is chosen:

$$\frac{1}{1+Y} = \frac{N_G}{N_F}$$

$$\frac{1}{1+Y} CH_L \le DH_L \le CH_L = \frac{1+Y}{1+Y} CH_L$$

17.4 Definition of Segregation Factor Z

To calculate DH_L, multiply CH_L by a factor between 1 and 1+Y. It is written as YZ.

$$DH_L = \frac{1+YZ}{1+Y} CH_L = (1+YZ)\frac{N_G}{N_F} CH_L$$

$$DH_L = (1+YZ) N_G \sum_i \frac{(a_i - a_L)^2 M_i^2}{a_L^2 M_L^2}$$

17.5 Definition of GSE

GSE is defined by its mean and variance:

$$\text{Mean}(GSE) = \text{very small, often negligible}$$

$$s_{GE}^2 = YZ s_{FSE}^2$$

Total variance is the product of three individual variables. It is impossible to get rid of the variance of FSE as the segregation factor can never be eliminated. It *would* be possible to get rid of the grouping factor by selecting fragments, one at a time, an impossible task. The only practical answer is to minimise each of the three factors in the following order:

i. ***Minimise the variance of FSE*** by calculating sampling constants, drawing sampling nomograms and optimising sampling protocols.

ii. ***Minimise the Grouping Factor*** for a given sample weight by taking as many small increments as is practically possible, with respect to IDE, IEE and IPE. This is the simplest and most effective solution.

iii. ***Minimise the Segregation Factor*** by homogenizing the lot before sampling, (This is very difficult because of the effects of gravity).

17.6 Methods for Minimising GSE

The following are a variety of methods by which GSE can be reduced or minimised during sampling events.

17.6.1 Coning and quartering

This very old technique is usually restricted to the sampling of lots of minus 5cm materials weighing less than 1 ton. GSE for the sampling arrangement shown in ***Figure 11.2*** is given by the equation:

$$s^2_{GSE} = \frac{s^2_{SE}}{\sqrt{N}}$$

Where *N* is the number of increments collected. For example, if you were required to sample 10 000 grains with vastly different densities is would be far better to take 10 x 1 000 grain increments rather than 2 x 5 000 grain increments. When trying to homogenize a lot one should try first to reduce FSE through appropriate experiments and tests and then combine the results from taking as many increments as possible. One should never attempt to homogenize the lot as an alternative to taking increments. This principle can be illustrated by mixing 500g of quartz, with a density of 2.5, with 20g of chromite having a density of 4.8. Shake the mixture in a bottle to improve homogenization. Pour the mixture onto a flat piece of paper to see how quickly segregation takes place.

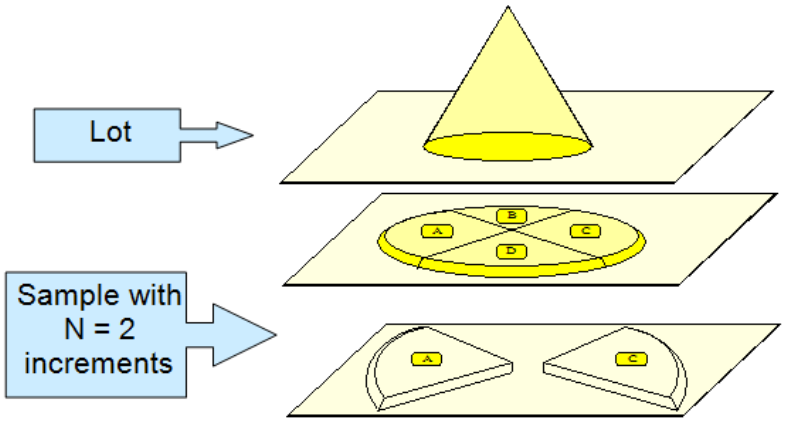


Figure 17.2: Coning and quartering **([40]Pitard, F, 2009.)**

17.6.2 Alternate shoveling

A faster, cheaper and better alternative is the alternate shoveling technique which simply splits a lot, increment by increment, into two equal samples. GSE is reduced according to the following equation:

$$s^2_{GSE} = \frac{s^2_{SE}}{\sqrt{N = ?}}$$

Where *N* is the number of increments into which a lot is divided.

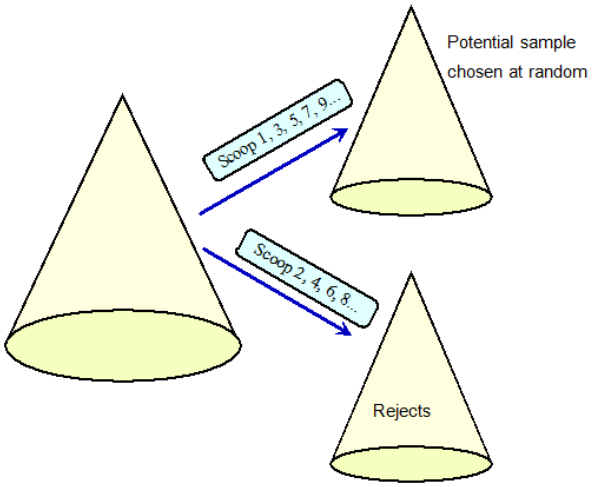


Figure 17.3: Alternate shoveling **([40]Pitard, F, 2009.)**

17.6.3 True fractional shoveling

This is done by splitting a lot into as many samples as possible on an equal-increment basis in order to increase *N* (number of increments) and thereby minimise GSE. One random sample is then selected.

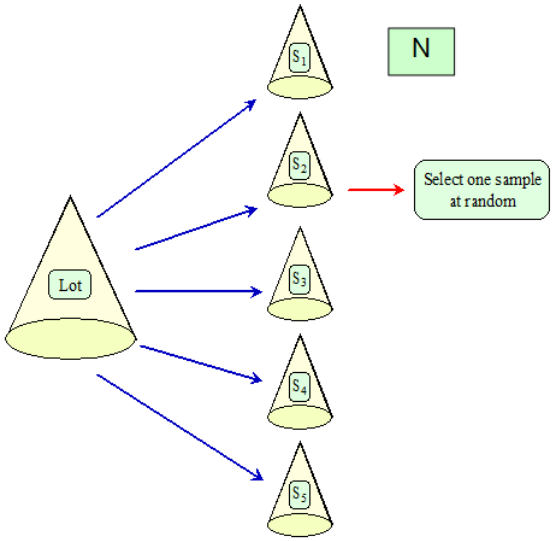


Figure 17.4: True fractional shovelling with a sampling ratio of 1/5 **([40]Pitard, F, 2009.)**

17.6.4 Degenerated fractional shovelling

This procedure is carried out on a sizable lot with a sampling ratio of 1/5 resulting in 20% of material in the sample and 80% in the reject pile.

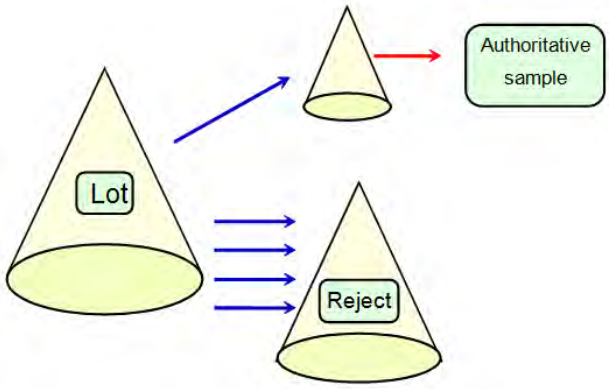


Figure 17.5: Degenerate fractional sampling with a sampling ratio of 1/5 **([40]Pitard, F, 2009.)**

17.6.5 Riffle splitter

The Riffle Splitter, also known as “Jones’ Riffle” or “Jones’ Splitter”, consists of an even number of identical adjacent chutes, usually between 10 and 20, forming a 45º or more angle with the horizontal plane. The chutes guide the cascading material alternately between the left and right buckets. The more chutes, the better. The riffle splitter is an excellent piece of equipment if used correctly provided a number of conditions are first met. Feeders that are too wide tend to over-feed the chutes at either end of the splitter. Using the receiving bucket to feed the riffle splitter is sure to introduce a delimitation bias. The riffle splitter must be fed slowly, down the middle of the chutes. The sides from which the rejects are chosen should be alternated. A cascade riffle splitter should not be used since particles accelerate through the splitter and GSE is reduced according to the following equation:

$s^2_{GSE} = \frac{s^2_{SE}}{\sqrt{N = ?}}$ Where *N* is the number of increments into which a lot is divided. For the splitter illustrated in ***Figure 11.6,*** *N* is half the total number of chutes i.e. it is the number of chutes feeding one bucket: *N* = 7.

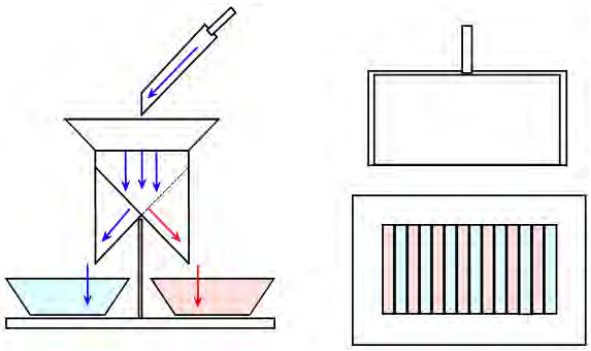


Figure 17.6: Riffle splitter **([40]Pitard, F, 2009.)**

Single stage riffle splitters are amongst the best mass reduction tools (Figure 17.7a). Cascading riffle splitters shown in Figure 17.7b should be avoided as they generally introduce significant bias.

Figure 17.7: a) Single stage riffle splitter, and b) Cascading riffle splitter (Photo by H Theart, Sampling and Analysis Conference, 2013)

17.6.6 Japanese slab-cakes

The process of making Japanese slab-cakes is illustrated in ***Figure 11.7.*** One should be able to make a clear distinction between GSE and the concept of an increment in IDE. Again GSE is reduced according to the following equation:

$$S^2_{GE} = \frac{S^2_{SE}}{\sqrt{N = ?}}$$

Where *N* is the number of increments into which a lot is divided, in this case 24:

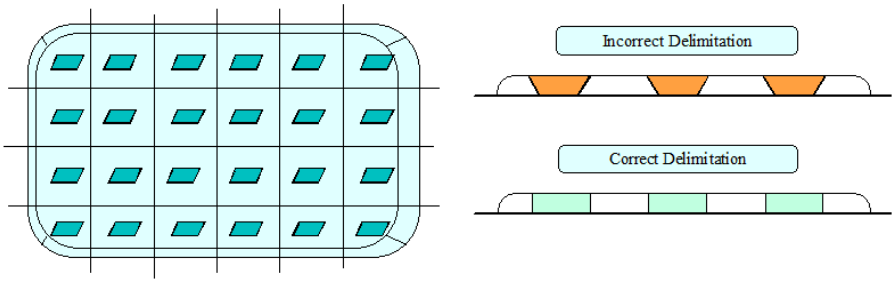


Figure 17.8: Japanese slab-cakes **([40]Pitard, F, 2009.)**

17.6.7 The V-blender

The V-blender (Figure 17.9 a and b) is used to mix the components of a lot and minimise the distributional heterogeneity; typically, the blender would run for about 10 minutes. In lots with a strong density difference between components segregation is likely to be induced when the hatch at the bottom of the blender is opened.

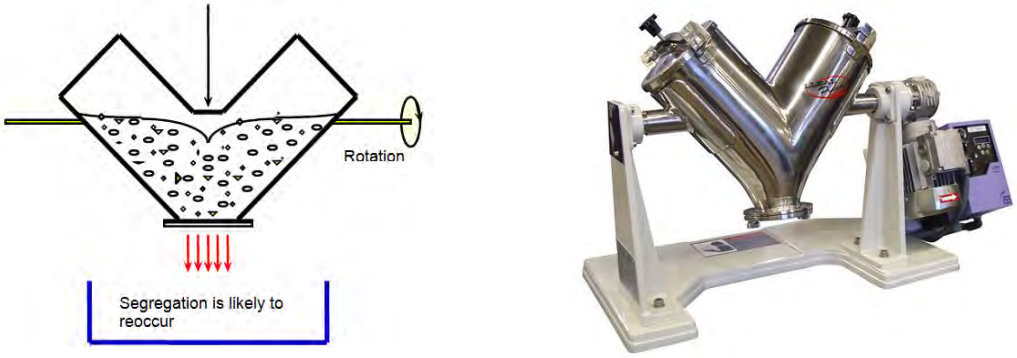


Figure 17.9: a) Section through a V Blender. The blender should not be filled above the axis of rotation **([40]Pitard, F, 2009.), and b) commercial V-blender** http://www.interempresas.net/Quimica/FeriaVirtual/Producto-Mezclador-de-solidos-en-V-de-laboratorio-66675.html

17.7 Demonstrating the Size of GSE

The following experiments show the potential enormity of GSE. A nickel ore lot is divided into 3 equal sub-lots. These sub-lots are used to perform the following tests with results expressed as % Nickel. Data for the exercise are listed in ***Table 17.1***.

a. **Test 1:** One sub-lot is not homogenized before sampling. It is evenly spread on a clean surface and divided into 16 equal strata that are assayed for nickel separately.

b. **Test 2:** The second sub-lot is divided into 16 splits using a riffle splitter, with no preliminary homogenization.

c. **Test 3:** The third sub-lot is homogenized using an alternate shoveling technique and then divided into 16 splits using a riffle splitter.

Firstly, from the experiment it is clear that DH_L cannot be larger than CH_L. However, this does not mean s^2_{GSE} cannot be larger than s^2_{FSE}. Secondly, if the recommendations made in "Theory of Sampling" are not followed, the variance of GSE and any other variances generated by other forms of segregation can become overwhelming. Thirdly, sampling by increments, for that is what the riffle splitter does, reduces the error variance considerably (S_{GE} for ***Test 2*** compared to ***Test 1***) and this is even further improved by the process of homogenization (S_{GE} for ***Test 2*** compared to ***Test 3***). The experiment shows that GSE is not to be underestimated and that while it is possible to improve a result it takes considerable effort to minimise GSE.

Table 17.1: Results of the nickel analyses

% Nickel	Test #1	Test #2	Test t#3
1	1.085	1.216	1.188
2	1.185	1.149	1.196
3	1.237	1.141	1.177
4	1.194	1.227	1.202
5	1.139	1.203	1.171
6	1.192	1.154	1.207

7	1.241	1.214	1.164
8	1.187	1.158	1.176
9	1.066	1.205	1.169
10	1.182	1.175	1.202
11	1.189	1.201	1.174
12	1.199	1.182	1.196
13	1.126	1.220	1.180
14	1228	1.209	1.195
15	1.228	1.214	1.206
16	1.177	1.161	1.159
Average:	**1.178%Ni**	**1.189% Ni**	**1.185% Ni**
S_R total:	**± 4.33%**	**± 2.43%**	**± 1.33%**
S_{FE}:	**± 1.08%**	**± 1.08%**	**± 1.08%**
S_{AE}:	**± 0.20%**	**± 0.20%**	**± 0.20%**
S_{GSE}:	**± 4.19%**	**± 2.17%**	**± 0.75%**

17.8 Inducing and Reducing GSE

17.8.1 Segregation is a relative concept that depends on the scale of observation.

Amounts of measured segregation from samples may be highly dependent on weight and the scale of observation as shown in ***Figure 17.10.*** In the total area shown the degree of segregation is clear, but at ¼ or ⅛ of the scale strong segregation is less evident. Therefore, collecting just a few, small samples may be misleading. If the objective is to map segregation, then the collection of many small samples is necessary.

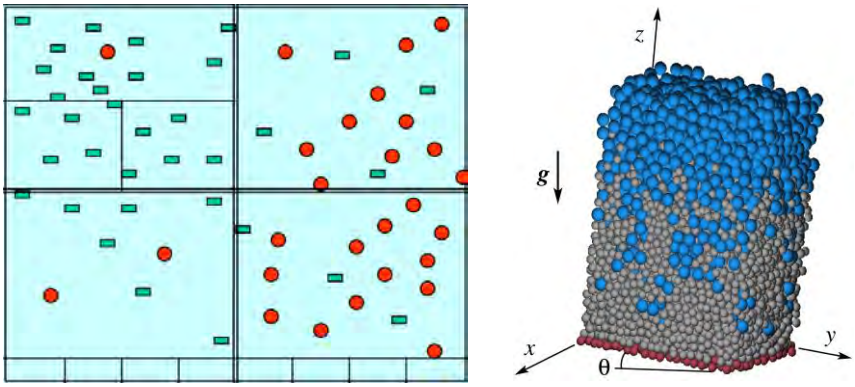


Figure 17.10: a) Segregation among materials of different densities at different scales of observation **([40]Pitard, F, 2009.), and b) Discrete Element Model of granular materials of different densities (D Khakhar, Department of Chemical Engineering, Indian Institute of Technology Bombay, Mumbai, India)**

17.8.2 Segregation is a transient phenomenon changing all the time

The extent of mineral segregation depends on the size of the difference between the densities of minerals in the lot, and the way in which the material is handled. This is particularly evident in materials at the point of discharge from a conveyor belt where the drag of the air and the speed of the materials coming off the belt combine to segregate higher and lower density materials as shown in ***Figure 11.10***. Once a stockpile of strongly segregated material is created in this way it is extremely difficult to take a representative sample even if incremental sampling is used.

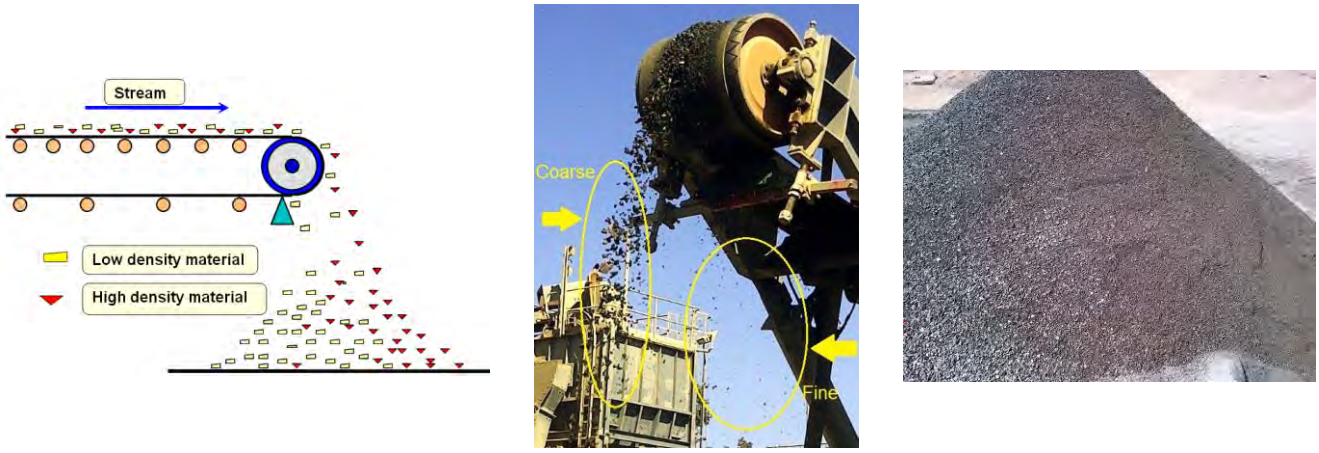


***Figure 17.11: a) GSE induced in a mixture of different density materials at the discharge point on a conveyor* (40Pitard, F, 2009.), b) segregation of coarse and fine materials at conveyor discharge point, c) resulting in segregation of coarse and fine materials in accumulating stockpile**

In classic segregation this problem is amplified if the materials are of different size fractions or not completely dry. A similar effect can be produced if materials conveyed as a falling stream pipes or chutes are significantly different in shape rather than density. Angular fragments may stick to the sides of a chute while the rounded fragments tend to move much more quickly. ***(Figure: 17.12).***

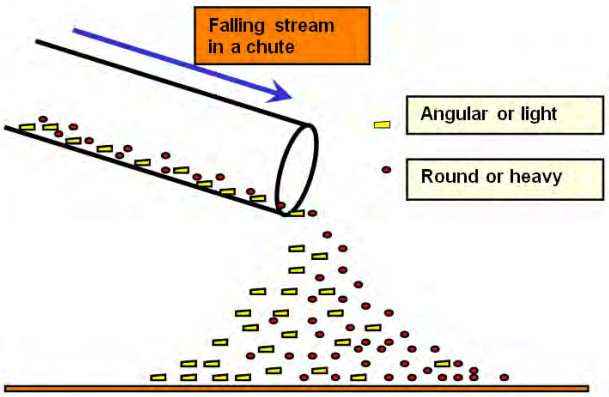


***Figure 17.12: Segregation induced in materials of roughly similar size because of density or significantly different shape.* (40Pitard, F, 2009.)**

In slow moving streams significant differences in shape may also lead to segregation because the angle of repose for rounded materials will be quite different to those which are angular. In all cases where particles are moving through the air the effect of air drag on trajectory is a function of particle size. Dry materials, having a long, free fall through the air at the end of very slow moving streams, tend to be winnowed so that the larger particles fall around the outer edges of the pile and the finer materials accumulate towards the centre of the pile. This is a major problem for all conical stockpiles and for which there is no solution. Once the pile is formed the only way a truly representative sample can be extracted is to sample the pile during reclamation.

The positioning of and correct location for a sampling system in a process stream is critical if samples are to be truly representative. Where a homogenously distributed stream of material is transferred from one

conveyor to another the stream may become highly segregated at the point of transfer. Cross-stream samplers are not sensitive to segregation within a stream, they merely grab samples. Those cross-stream samples which only sample part of a stream should be avoided.

To summarise, it is notable that segregation phenomena increase GSE and can be very detrimental to a process and confusing to an engineer in charge of process control. Furthermore, these phenomena show how critical increment delimitation correctness is and the greater the tendency to segregate, the greater the number of increments required when addressing the problem.

Figure 17.13: a) Sifting and winnowing of coarse and fine material in a conical stockpile (40Pitard, F, 2009.), and b) separation of coarse and fine materials on a stockpile http://www.rohstoffe.rwth-aachen.de/cms/Rohstoffe_und_Entsorgungstechnik/Forschung/Menschen/BBK_3/~skp/Feinstaub_in_Tagebauen/lidx/1/ ***c) segregation of coarse and fine materials in a stockpile*** *http://www.lignotechfeed.com/Pelleting-Aids/Ameri-Bond-2X/Field-Trials/Ameri-Bond-2X-reduces-segregation-in-turkey-feed*

17.8.3 Segregation in silos, bins and hoppers

Storage silos, bins and hoppers are containers drawn from at intervals or on a continuous basis. While the materials may be homogenous when first stored, the moisture content or design of these containers can induce segregation, particularly when withdrawing samples. Firstly, materials stored in silos, bins and hoppers must be extremely dry as the slightest moisture can affect their characteristics and the manner in which they are recovered. For many fine products and slurries it is critically important to maintain them in an unsegregated condition before the next step of a process, or before a sample is collected. Silo, bin and hopper technology, for the handling and storage of fine materials, is often empirical giving rise to many problems.

Mass-flow holding hoppers must be used between two sampling stages. If not, it will be necessary to completely empty the hopper containing one increment before the next increment is introduced. The list of potential problems is long.

17.8.4 Segregation at the entrance of a silo, bin or hopper

As a silo is being filled coarse material may fall off to the sides (if a cone is built inside the silo) as shown in ***Figure 17.14a.*** In addition, material along the sides may move slower than material at the center of the container resulting in an alternating flow of coarse and fine material at the draw point.

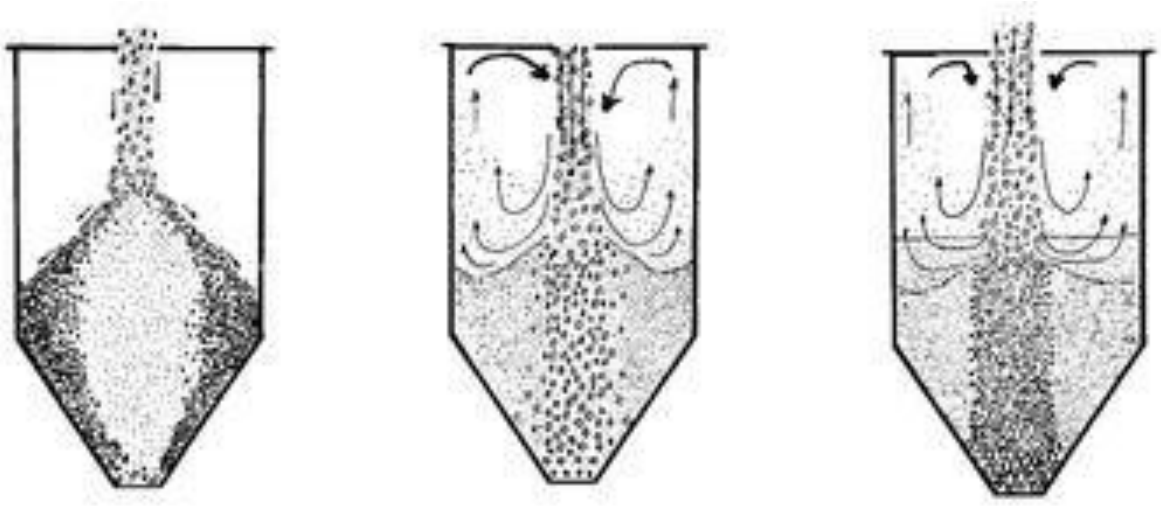
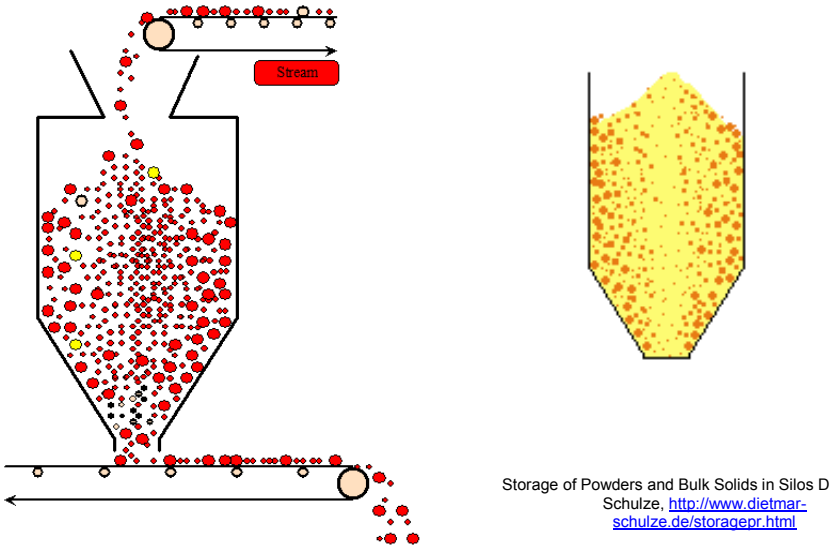


Figure 17.14: a) Coarse material accumulating around the interior perimeter of the silo, b) and c) depending on air flow and material density coarse materials may accumulate in the center of the silo



Storage of Powders and Bulk Solids in Silos D Schulze, http://www.dietmar-schulze.de/storagepr.html

Figure 17.15: a) Material segregation due to particle size in a silo, bin or hopper ([40]Pitard, F, 2009.), and b

Potential problems can be overcome by providing a multi-point feed into the container and ensuring that the internal slope of the container is 70º to 75º.

17.8.5 Percolation of fines between coarser particles

Voids between coarse particles tend to fill up with fines as shown in ***Figure 17.16***. The material may develop cohesive strength due to consolidation and as a result the flow-rate at the discharge of the bin may become erratic or even stop.

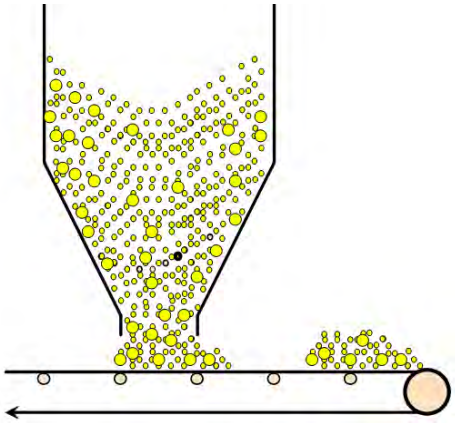


Figure 17.16: Percolation of fines between coarser particles **([40]Pitard, F, 2009.)**

To mitigate this problem ensure that the materials are very dry before entering the container and that the internal slope of the container at the discharge point is not too shallow.

17.8.6 Incomplete removal of previously charged fine material

If material contains even the slightest moisture or has a natural proclivity to stick to the sides of a container it is possible that the container will not be cleared entirely of the previous load before being filled again, as shown in ***Figure 17.17***. Such a build-up may continue over a long period of time reducing the surge capacity of the container and resulting in contamination of the new load by the old. Again, correctly designing the mass-flow bin and installing vibrators along the sides of the containers can overcome these problems.

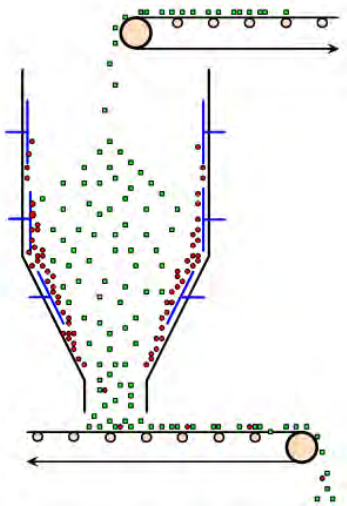


Figure 17.17: Incomplete removal of earlier material from the container **([40]Pitard, F, 2009.)**

17.8.7 Degradation of fine material by prolonged storage promoting caking and agglomeration

Mass flow in a silo is the ideal movement within a silo (Figure 17.18b). If the angle of the walls of a container is too shallow near the discharge point, fine material may build up and become consolidated or caked (stagnant portion) over time, as shown in ***Figure 17.18a.*** This leads to ratholing in the silo (Figure

17.18b). Sooner or later the material will be dislodged and large agglomerated fragments, rather than free flowing material, will be discharged.

Figure 17.18: a) Prolonged storage and build-up of stagnant material inside a container, b) example of ratholing in a silo. http://www.power-eng.com/articles/print/volume-117/issue-3/features/prb-coal-material-handling-challenges-and-solutions.html

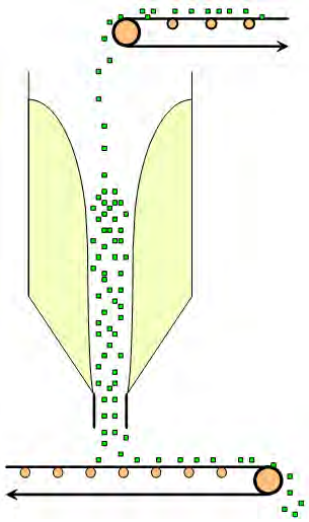


Figure 17.19: Piping or rat-hole formation during discharge from a silo **([40]Pitard, F, 2009.)**

If materials are damp or the container is poorly designed most of the material in the container becomes consolidated with only a pipe or rat-hole through the contents of the silo, as shown in ***Figure 17.19.*** Therefore most of the material remains in the hopper with the result that material last added is the first to be discharged and storage and surge capacity are completely ruined.

17.8.8 Creation of a stable arch

Where a stable arch is formed inside a silo, as shown in ***Figure 17.20***, it will interfere with the free-flow of fine material. Such problems arise from a variety of conditions including the fine-grained nature of the materials, dampness or because the diameter of the container itself is too small.

Figure 17.20: a) Stable arch formation in a silo containing damp fine-grained materials (40Pitard, F, 2009.), b) simulated example of arch formation, and c) schematic arch formation in silo

The problems of stable arch formation can be mitigated by ensuring that the discharge point is wide enough using a mass-flow bin and by aerating the container.

17.8.9 Development of rat-holes or piping

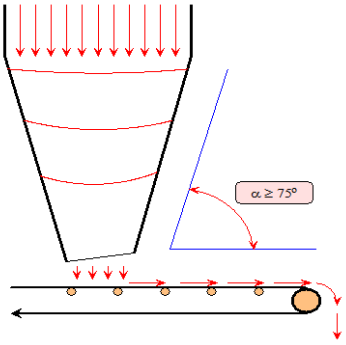


Figure 17.21: Correct design of a mass-flow bin **([40]Pitard, F, 2009.)**

Provided the material fed to the container is dry, most of the problems described in this section can be overcome by installing a correctly designed mass-flow bin as shown in ***Figure 17.21.*** The main criteria for the design of a mass-flow bin are that: the sides of the bin should be greater than 75º, and the mouth of the discharge point should not be horizontal, but rather at a slight angle to ensure that the flow of material is even.

17.9 Advantages of a Correctly Designed Mass-Flow Bin

There are several advantages to a correctly designed mass-flow bin:

- The internal surface of the inside of the bin is uniform.
- All welds along the seams of steel plate would be vertical rather than horizontal.
- Material first in is first out.
- Sample integrity is preserved.
- Segregation is minimised.

- Finer material will not plug the system.
- A continuous and uniform flow at the discharge is achieved.

However it should be remembered that even mass-flow bins can go wrong if the discharge system is poorly designed. Mass-flow bins may have a variety of different discharge systems including conveyor belts, screw feeders or vibratory feeders, each having their own problems if not properly designed as shown in ***Figure 17.22***. The angle of the discharge point must be at least 3.5º to the conveyor belt in order to provide an even flow of material. In addition, the profile of the horizontal cross-section of the discharge point must be radial rather simply rectangular. ***(Figure 17.22).***

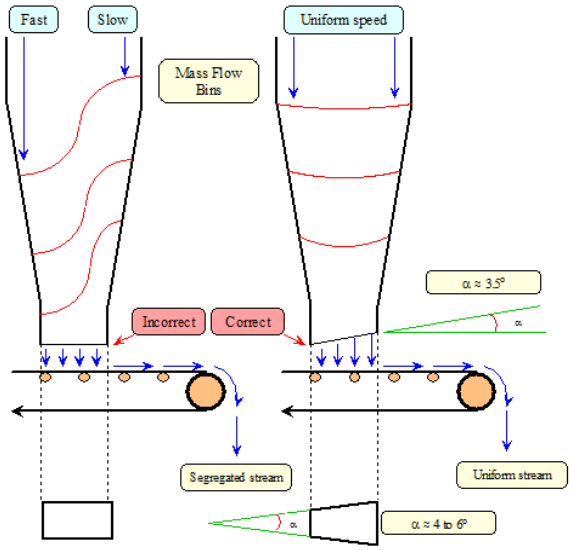


Figure 17.22: Problems associated with an incorrect design of the discharge mouth of a mass-flow bin **([40]Pitard, F, 2009.)**

Finally, it should be noted that material which becomes size-segregated in the feeder bins delivering it to a SAG mill can prove to be costly because of inefficiencies as shown in ***Figure 17.23.*** Inefficiencies arise because material segregated by size in a SAG mill is less than optimal and the amount of product delivered by the mill becomes cyclical, possibly outside specification limits.

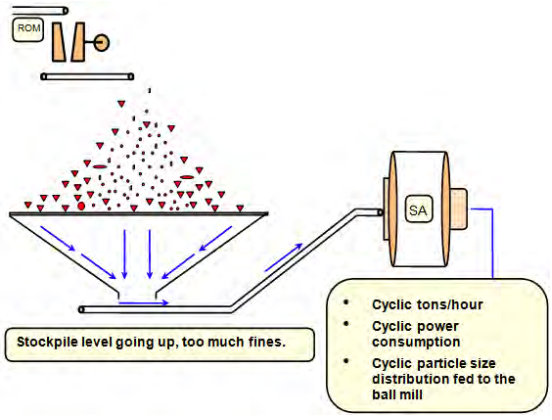


Figure 17.23: Size segregation in SAG mill feed **([40]Pitard, F, 2009.)**

PART 3: PRACTICAL IMPLEMENTATION OF A PROTOCOL

Thus far, issues surrounding the establishment of a sampling protocol by examining in detail INE, FSE and GSE have been dealt with. These three topics form the basis of a well-designed protocol that has taken account of volume of observation, mass and size of sample fragments and the methods and means of incremental sampling. The protocol must now be communicated clearly and effectively to all concerned. One method to achieve blanket communication would be to print the protocol in poster form for display in relevant and prominent areas from the mine/pit to the evaluator and grade control offices, right the way up to the laboratory.

It is now possible to focus on the problems associated with the implementation of the protocol. The challenge here is sampling correctness. This involves an awareness of the problems associated with IDE, IEE, IWE and IPE. IDE introduces the largest biases encountered in sampling. It is one thing to have a protocol that everyone agrees with, but entirely another to implement it so that the analytical results arriving on one's desk are the best that can be gathered. According to Pitard (2006) this is the area in which Pierre Gy made his best contribution, and which went unnoticed for too many years.

18

19 DELIMITATION ERROR (IDE)

19.1 Introduction

IDE is widespread and is a main generator of sampling bias. The principle of correctness is based on the statistical requirement that every fragment must have an equal chance with every other fragment of being included in a sample. In the sections which follow, IDE's occurrence in the field, plant and laboratory is identified and described. When dealing with INE, FSE and GSE there is some leeway regarding the protocol and its implementation. However, with regard to IDE, IEE, IWE and IPE, one is dealing with the practical implementation of the protocol and there can be no compromise. Actions performed during implementation are either right or wrong and if not performed diligently, will generate huge unpredictable biases and result in substantial invisible costs. If implementation is wrong, there is no opportunity to correct the situation. It is irretrievable.

All stakeholders must be considered when implementing a sampling protocol. The driller at the pit probably has a bonus based on the number of metres drilled per day or per month and therefore his primary concerns are not how well he clears the holes, whether or not the chips land in the sampling ring or if the cyclone is operating correctly. The production engineer is principally concerned with the rate of production and has little concern for those issues occupying the mind of the grade control officer. Yet, if implementation is incorrect, it doesn't matter how good the protocol may be, the battle is lost from the start. The principle of correct sampling requires that:

i. Every part of the lot has the same chance as every other part of the lot of being sampled: it is the first principle of equiprobability, and;
ii. The integrity of the sample is preserved during and after sampling: it is the second principle of equiprobability.

Pierre Gy's Theory of Sampling, verified by G. Matheron, demonstrates that perfect equiprobability cannot be achieved in sampling; however, under some stringent conditions it is possible to approach this inaccessible limit in a first order approximation. There are several actions which can be undertaken to reduce the chances of taking an incorrect sample.

i. Reduce the number of sampling dimensions, if possible. Correct sampling of three-dimensional lots is not practical so try and sample across one or two dimensions where possible.
ii. Define the correct sample by using the correct geometry for the sampling dimension, which is called an isotropic module of observation; a cylinder for two-dimensional sampling and a slice for one-dimensional sampling.
iii. Choose the right sampling tool which must be capable of taking a defined sample. The tool must be correctly used when collecting the sample. The tool must not be selective on what it is taking.
iv. Preserve the integrity of the sample.

A principle which must be adhered to is: reduce the number of sampling dimensions or shape of observation as far as possible. This can be done by considering a three-dimensional deposit as an integrated stack of two-dimensional slices, each slice being one bench height thick as shown in ***Figure 19.1.*** In this case, the ideal shape of observation is a cylinder which must represent the entire thickness of the bench. The position of the drill holes on the top bench indicates that they will intersect the deposit optimally, provided the mineralisation is horizontal and parallel to the bench surface. Most mineral deposits occur in rocks which are, at the least, dipping to some extent, reducing the effectiveness of the delimitation of a two-dimensional sample. If the fabric of the rock and its associated mineralisation is dipping vertically, results from the sampling of vertical RC blast holes are meaningless. To remedy this, drill inclined boreholes which will intersect the fabric at as high an angle as possible.

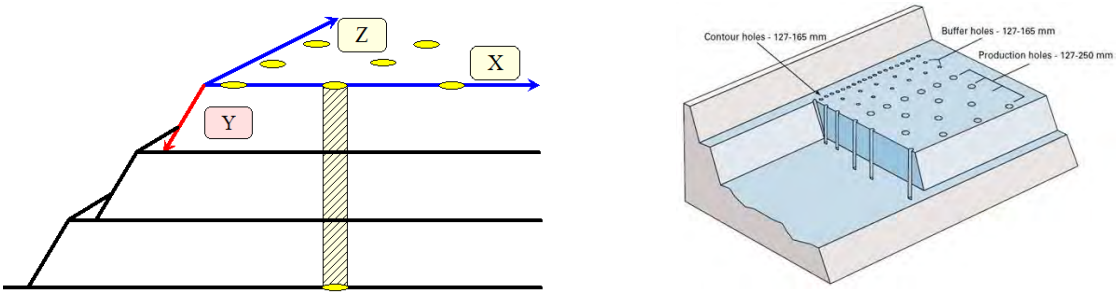


Figure 19.1: a) Three-dimensional mineral deposit as a stack of two-dimensional slices ([40]Pitard, F, 2009) and b) planned production bench for open pit mine http://www.geodrillinginternational.com/features/blasting-optimisation?SQ_DESIGN_NAME=print_friendly

Another type of delimitation bias is the drift in boreholes which are drilled into predominantly foliated sequences. This is usually accounted for and accommodated in the final calculation of reef thicknesses by a diligent geologist who will ensure that the boreholes are surveyed.

Figure 19.2: a) Blasthole drilling on mine benches miningandconstruction.com/mining/atlas-copco-pit-viper-drill-rigs-the-kings-of-collahuasi-1360/**and b) inclined blastholes on new bench** http://www.proactiveinvestors.com.au/companies/news/31328/hughes-drilling-is-undervalued-under-the-radar-31328.html

19.2 IDE at the Mine

19.2.1 Sampling borehole core

The method by which borehole core is sampled depends on the nature of the ore and on pre-determined objectives.

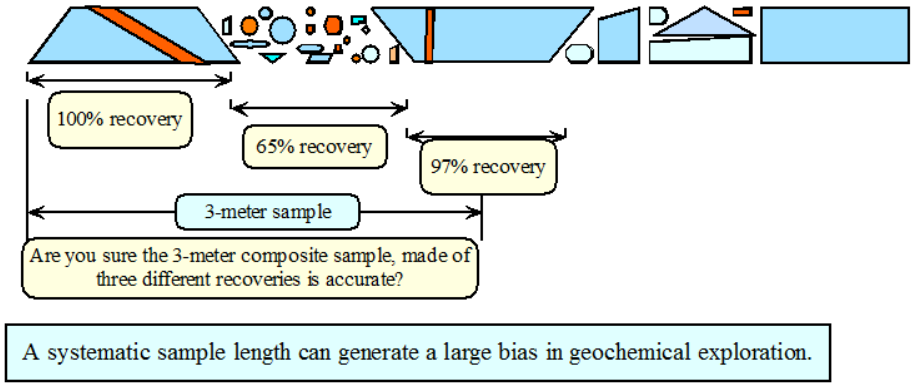


Figure 19.3: Using a standard length of core for sampling can generate large biases, especially where the core is extensively broken([40]Pitard, F, 2009.)

It is essential to ensure that core recovery exceeds 97 per cent in the ore zone **(Figure 19.3).** Alteration or weathering of core may make logging or sampling in an appropriate way impossible **(Figure 19.3b).**

Figure 19.4: a) Fresh core with high core recovery in the mineralised zone, and b) highly weathered core difficult to log and sample in the mineralised zone

Simply sampling at a constant length along the core may not be a good idea because core recovery in the area of mineralisation may be low despite it exceeding 95% in the rest of the hole. ***(Figure 19.4b).***

The Carbon Leader reef, one of the principle gold bearing reefs of the Wits basin in South Africa, is a case in point. The carbon-rich seams, which carry up to 80% of the gold, are extremely friable and easily break up when the pressure of a column of drill rods is applied to it. Here special care and attention has to be exercised if meaningful intersections of this portion of the core are to be recovered. Requirements with regard to the rate of core recovery must be stipulated in the drilling contract.

19.2.2 Channel sampling

When channel sampling, the outcome and value of the sample in terms of its representativeness is dependent on the diligence and consistency of the sampler. The sample should include all the material in the reef. Over-sampling of softer parts, in preference to harder parts, should be avoided.

19.2.3 Sampling Blast holes

Sampling of blast holes is generally not a good idea, yet because so many mines rely on it for grade control it is unlikely to disappear from practice. Furthermore, mine staff have used the method for so long with what appears to be acceptable results, that their confidence in the technique is unshakable.Bias associated with IDE is commonly introduced when sampling blast hole material at the mine. It has potentially devastating effects on both ore grade control and the long-term financial performance of the mine. The problems associated with blast hole sampling in a typical open-pit operation are illustrated in ***Figure 19.5a.*** Rock in the former sub-drill region, which is usually about 2m thick, is already broken up and so rock chips fail to come out during the air-flush process resulting in very poor recovery. Practically speaking, only the material above the current sub-drill should be collected as a sample. Collecting sample material from the sub-drill should be avoided as this material will only be delivered to the mill when the next bench is mined. The difficulty with this is that the control on the depth to which the hole is drilled just before collecting the sample is highly variable (***Figure 19.5b***)

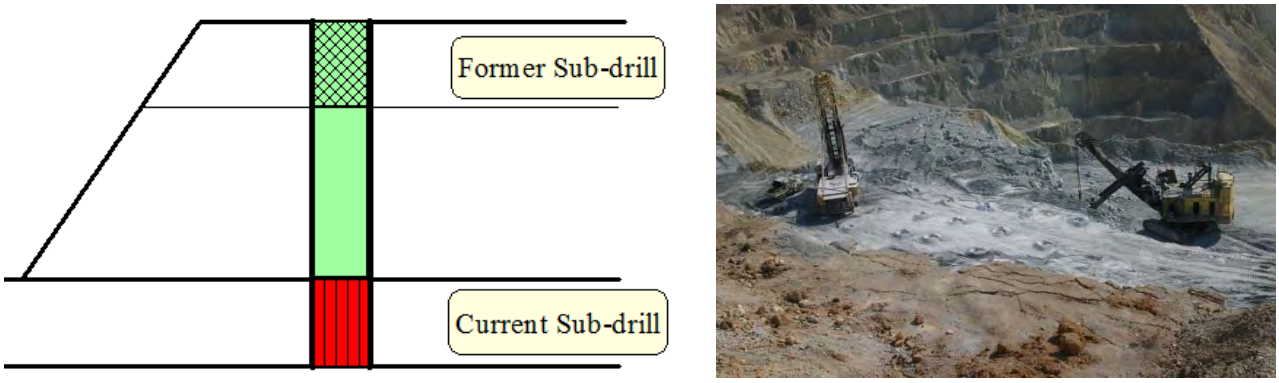


Figure 19.5: a) Increment delimitation errors in the blast hole drilling process([40]Pitard, F, 2009.), and b) Bor open pit mining drilling for blast, Bor District, Serbia. http://www.panoramio.com/photo/13828449

The main issue with regard to IDE is consideration of the sampling dimensions of a lot. It is entirely up to the sampler to consider a lot as being three-dimensional, two-dimensional or one-dimensional. However, the isotropic module of observation for IDE to be minimised, changes accordingly with the dimension of the lot under consideration. In a three-dimensional lot it is a sphere positioned at random anywhere within that lot if. However, in a two-dimensional lot it is a cylinder positioned at random systematic or stratified random representing the total thickness of the lot. In a one-dimensional lot it is a slice of uniform thickness positioned at random systematic or stratified random, representing the total width of the lot.

19.2.4 Sampling blast hole drill chips

The primary requirement for blast hole drill sampling is that the whole stream of chips must be intercepted. The use of poorly designed equipment may mean that equitable sampling is simply not achievable; not all size fractions are equally sampled. Such an example is shown in ***Figure 19.6.*** It should be noted that the term "RC drilling" refers to material conveyed up through the centre of drilling tubes resulting in almost all the chips reporting to the recovered sample. This is not the case for blast hole drilling where rock chips are blown out of the hole around the sides of the drill rod, leading to significant contamination and loss of rock cuttings.

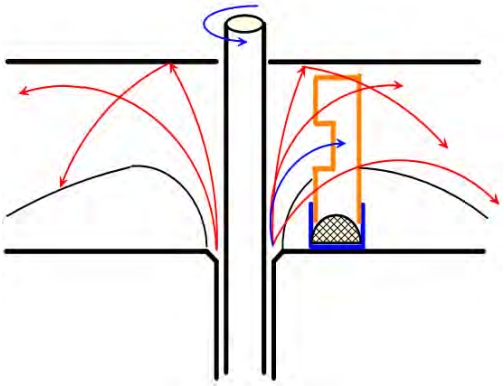


Figure 19.6: Window sampler contained in a sampling screen around the borehole. **([40]Pitard, F, 2009.)**

19.2.5 Blasthole sampling using a radial bucket

Radial buckets are so designed that they catch those rock chips discharged from a blast hole. The minimum opening of the radial bucket must be 3 times the top dimension recovered and should extend beyond the edge of the pile of cuttings. The height of the bucket should provide sufficient capacity that nothing is lost. The shape of the bucket must be perfectly radial for correct delimitation of the chip pile. These features are shown in ***Figure 19.7a, 19.7b, and 19.7c.***

Figure 19.7: a) Use of a radial bucket for RC drill cuttings ([40]Pitard, F, 2009.), b) radial bucket in place, and c) being emptied

The radial bucket is a somewhat primitive piece of equipment, but does the sampling job reasonably well if nothing else is available. More sophisticated sampling equipment is available and has been designed specifically for the purpose of blast hole drill samples and since the pile is not a perfect circle the system is reasonably proportional.

19.2.6 Sampling of stockpiles

Sampling a stockpile can be very difficult because it is a three-dimensional lot and one cannot fulfil the requirement that all fragments have an equal chance of being included in the sample. Depending on the material it may be augured but this is only applicable for moist, fine-grained materials (**Figure 19.8a**). Simply taking a grab sample of the material would violate the principles of the sampling dimensions of the lot and should be avoided (**Figure 19.8b**). Stockpiles of hard, lumpy materials may require dissecting by vertical channels using a front-end loader so that the internal walls of the stockpile can be sampled **(figure 19.8c).**

Figure 19.8: a) Stockpile of moist fine-grained material ideal for auguring ([40]Pitard, F, 2009.), b) grab sampling on moist precious metal concentrate, and c) sampling of hard, lumpy materials

19.2.7 Sampling underground

Correct delimitation of a face channel sample can be tricky especially where there is enrichment along mineralised contacts. When taking the waste sample one should stay away from the contacts to prevent contamination. Such was the case at a silver mine in Mexico **(*Figure19.9*)** where it was easy to confuse the ideal sample boundary (correct delimitation) with the actual collected sample (correct extraction).

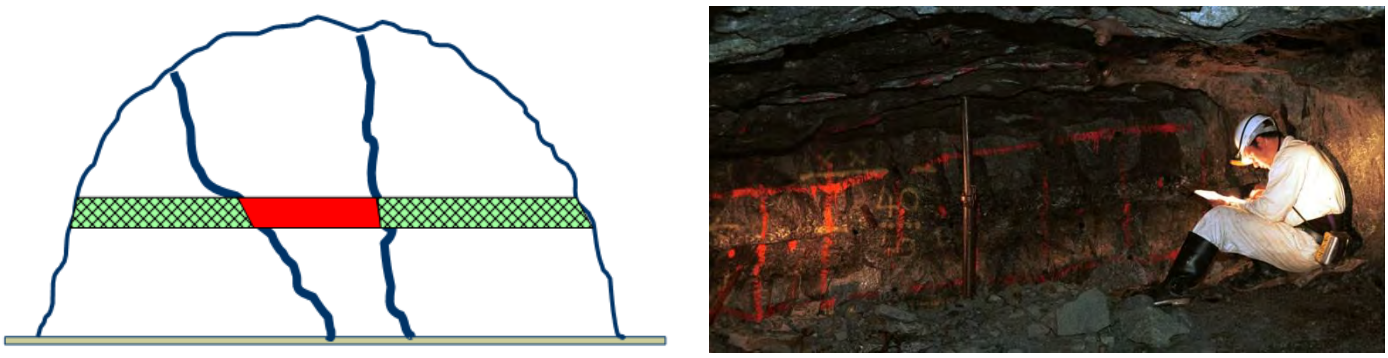


Figure 19.9: a) Sampling should be done at right angles to the mineralised vein and high- grade portions should be avoided when taking waste samples([40]Pitard, F, 2009.), and b) mapping and sampling a flat dipping reed on South African precious metal deposit

Sampling in steeply dipping mineralised ores

If there is a strong vertically dipping fabric, it is likely that the grade control map based on blasthole assays only could give impressively misleading information as shown in ***Figure 19.10a***. It is far better to sample horizontally across the floor of the pit and the fabric as indicated in ***Figures 19.10b, and 19.10c.***

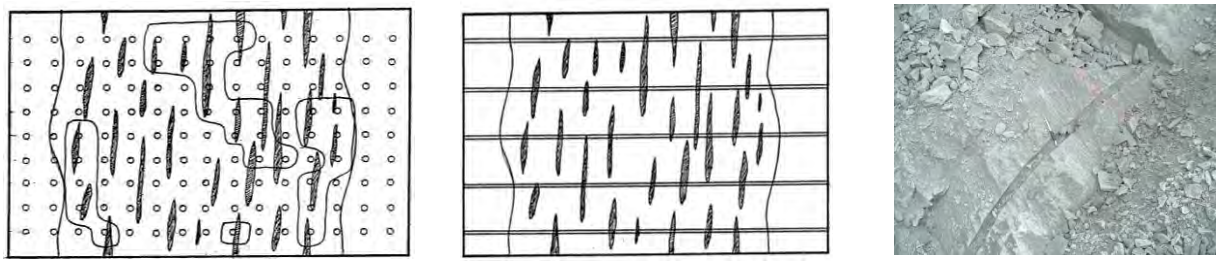


Figure 19.10: a) Hand contoured bench plan that misses much value in the pit ([40]Pitard, F, 2009.), b) horizontal channels across the floor of the pit at right angles to the strong shear fabric, and c) samples cut across the steeply dipping shear zone at the Lily mine, South Africa.

19.3 IDE in the Plant

IDE in the plant is centred mainly on moving streams of slurries or liquids and fragmented ores along conveyor belts or in pipes and chutes. According to the definition of representative sampling, the sampling equipment must sample a full cross-section of the stream at fixed intervals. Modern technology (***Figure 19.11***) has provided solutions, but more research is required in this area. Most inline stream samplers only sample part of the stream, either part of the time or all the time. Process streams are always segregated and represent three-dimensional lots that can be segregated in any one of the three directions shown in

Figure 19.11a. The challenge for the sampler is to define and extract one-dimensional samples (***Figure 19.11b***) from a flowing segregated stream.

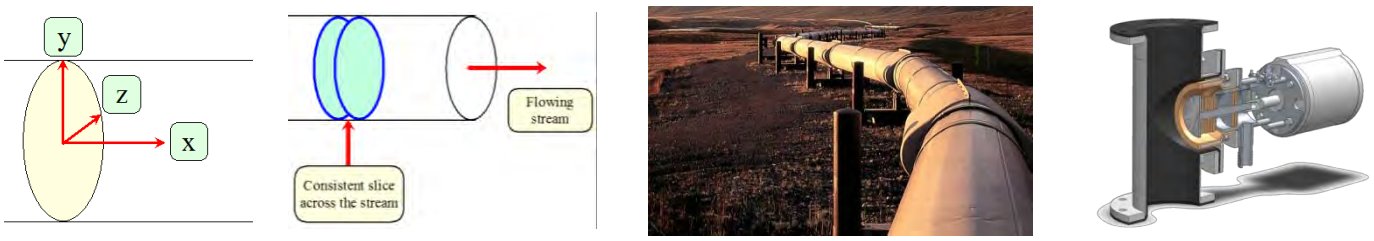


Figure 19.11: a) Increment delimitation in three dimensions([40]Pitard, F, 2009.), b) one dimensional sampling across a flowing stream([40]Pitard, F, 2009.), c) Canadian west-east oil pipeline,and d) an in-line stream sampler injector or thief sampler http://www.edart.co.za/

While it is clear that one-dimensional sampling is ideal and essential for accurate metallurgical accounting and effective process control, the question is: how to perform this? As shown in ***Figure 19.11a*** one-dimensional sampling involves taking a full and consistent "slice" across the stream. Samplers for this purpose can be accommodated on vertical or gravity downflow streams but not easily in pressurized, horizontal pipe columns (**Figure 19.11c**). A compromise is made at times in the absence of anything better, whereby poppet or injector samplers are mounted on side of pipe, to extract a small part of the stream part of the time (***Figure 19.11d***).

A three-dimensional lot could be a filled rail truck or a large stockpile. In both cases the worst possible time to sample would be after the truck is loaded or the stockpile stacked. Sampling should take place as the material is being put into place. If the material has already been loaded, the best option would be to wait for unloading of the truck or reclamation of the stockpile.

19.3.1 Dry Sampling Techniques

Of the three methods available for extracting a stream sample illustrated in ***Figure 19.12***, only one of them will deliver a 'correct' sample. Neither options B nor C will provide a correct sample. Only method A could, but even then this method can introduce bias if the sample taken is not consistent across the belt of the material stream as shown in red.

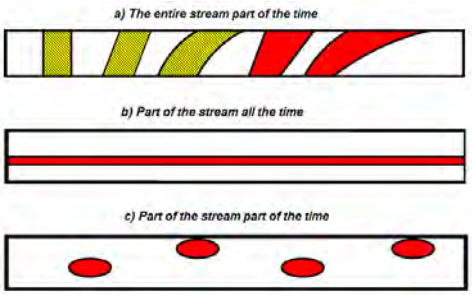


Figure 19.12: Sampling a moving stream([40]Pitard, F, 2009.), a) the entire stream part of the time (equi-probabilistic), b) part of the stream all the time, and c) part of the stream part of the time

It is essential that the width of the increment remains constant all the way across the stream. However, even though one may correctly delimit the increment, this does not necessarily mean that the sample will be correctly extracted.

To ensure that no bias is introduced, any frame placed on a stationary conveyer belt, need be placed correctly (***Figure 19.13a***). A bias test of a cross-cut or cross-belt sampler is ideally performed by collecting 50 sets of A and B samples using a fixed frame separated by several meters on a stopped belt. These pairs of reference samples are compared with one another and against the primary sample increments extracted between samples A and B by a primary cross stream or cross belt sampler (***Figure 19.13b***).

Cross-stream samplers must follow a straight path across a stream and must move at a constant speed. A cutter can become worn and therefore regular maintenance and cleaning of cutter edges is essential. Cross-stream samplers must have sufficient capacity and be powered by electric motors with sufficient power to carry a sampler across a stream at a constant speed. In the parked position the cutter should be well out of the way of the stream so that it cannot collect any material. If any part of the stream is missed by the sampler, a delimitation bias can be introduced.

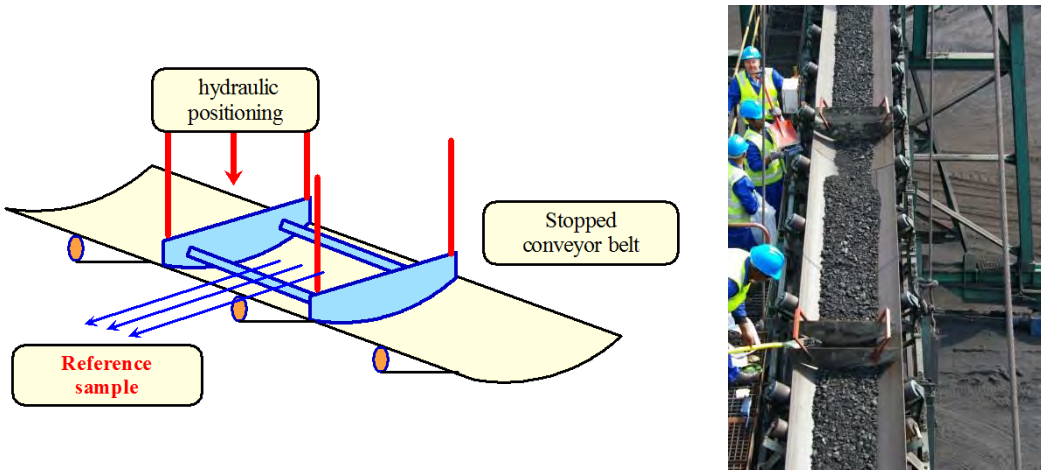


Figure 19.13: a) Sampling frame correctly positioned on the conveyor belt([40]Pitard, F, 2009.), and b) stopped belt bias testing at Richards Bay Coal Terminal (Allan Johns, Sampling and Analysis Conference 2013)

19.3.2 Cross-belt samplers

Cross belt samplers have been manufactured since 1964, the most common being the Siebtechnik hammer sampler that moves across the stream of material on the conveyor belt at right angles to the direction of travel and extracts a single increment: an example is shown in ***Figure 19.14a***. Typically the cutter blades should be separated by 3 times the nominal fragments size, the minimum being 30 mm wide, so the material being sampled by the unit shown in***Figure 19.14a*** is relatively fine-grained. Usually there is a brush, or better still a rubber wiper, at the tail end of the hammer or cutter that crosses the stream and this may work with varying degrees of effectiveness, but it is designed to clear the fine material at the bottom of the belt (***Figure 19.14b***). The cross-belt cutter located on the so-called go-belt in a typical gold plant shown in ***Figure 19.14c*** is somewhat wider than that for the iron ore plant because the material being extracted from the belt is considerably larger in size – up to 30cm diameter for RoM applications.

Figure 19.14: a) Cross belt sampler on a conveyor in coal industry b) brush or rubber wiper at the tail-end of the cross-belt cutter and c) a cross-belt cutter or go-belt in a gold plant (Multotec SA)

Research at the CSIRO in Australia by Robinson and Cleary has simulated the delimitation of the increment to be extracted using a cross-belt cutter (shown in green in ***Figure 19.15b***). The ability to track the path of the cutter has provided manufacturers with the ability to design a new cross-belt cutter which follows the trajectory of the material on the belt. An example of this new design cutter is shown in ***Figure 19.15c***.

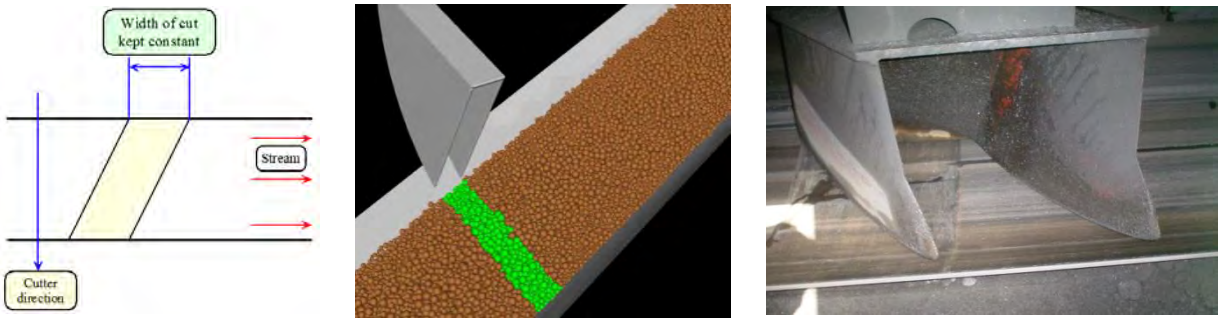


Figure 19.15: a) Cross-belt cutters follow a path at right angles to the movement of the conveyor, but it removes a diagonal strip of material from the belt, b) Discrete Element Model delimitation of increment to be extracted, and c) the skew or angled cross-belt cutter design that follows the trajectory of the material to be extracted (Multotec SA)

Claims by the manufacturers of the skewed or angled cutter are that it is firstly mechanically correctandcommercially unbiased even when material on the belt is segregated, secondly it removes only the increment material from the belt, and thirdly that it leaves no fines on the belt when correctly installed, commissioned and maintained. Cross-belt samplers with parallel side plates and that move across the belt at right angles have reduced integrity in that they extract too much material from the upper part of the load on the belt and that the bias is increased when the belt is heavily loaded (Materials Sampling Solutions, http://www.materials-sampling.com/HAMMERsampler.html).

19.3.3 Cross-stream samplers

Delimitation of the sample to be extracted from a falling stream has also been modelled by Robinson and Cleary the CSIRO, Australia. An example of their Discrete Element Model is shown at different stages of travel through the stream as shown in **Figure 19.16 a-c**. This model again illustrates clearly that the path of the cutter through the stream is a diagonal track across the path of the ore flow. Under the section dealing with the Extraction Error a number of points covering the important issues concerning cross-stream samplers are covered.

While the stream in this case leaves the belt with a trajectory of about 45 degrees the front edge of the cutter blades is at right angles to the flow of the stream.

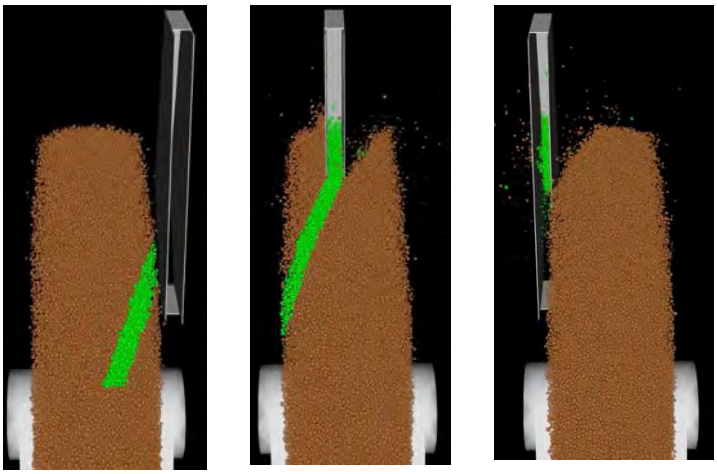


Figure 19:16: Discrete element model created by CSIRO Australia showing three positions of travel through the falling stream and illustrating the effectiveness of such cutters

For a vertically falling stream the principles of cutter operation are identical, and the cutter again moves at right angles to the flow of the stream. A schematic diagram showing the principles of movement is given if **Figure 19.17a and b**, The diagram in **Figure 19.17c** illustrates how the cutter moves back and forth through the vertically falling stream and collects an increment each time it passes and discharges it continuously. Many varieties of cross-stream samplers exist with continuous discharge or bottom dump applications where the sample is dumped at the end of travel.

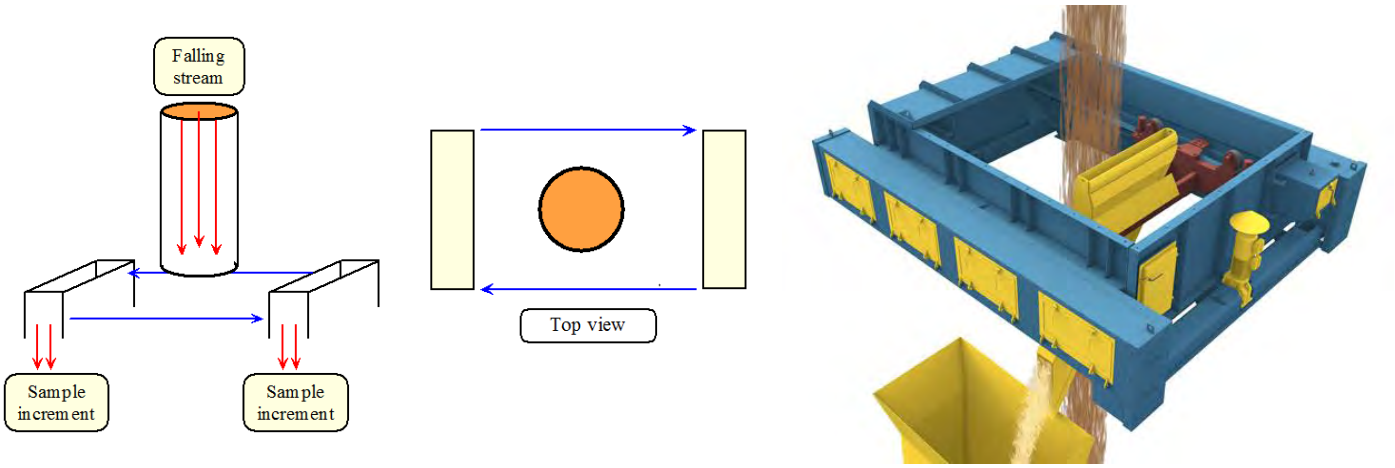


Figure 19.17: a) and b) The cross-stream sampler must cross the stream in a straight path and at a constant speed([40]Pitard, F, 2009.), and c) a commercially available cross-stream cutter with continuous discharge (ESSA, Australia).

19.3.4 The Dry Rotating Vezin sampler

This sampler, shown in ***Figure 19.18,*** is a good method for secondary and tertiary sampling. The advantage of the Vezin sampler is that it is small and compact, fairly inexpensive, sturdy and, most importantly, proportional. In order for the sampler to function correctly the stream must be centred over the

middle of the cutter and the cutter must rotate at a constant speed. The cutter must be radial with respect to the centre of rotation.

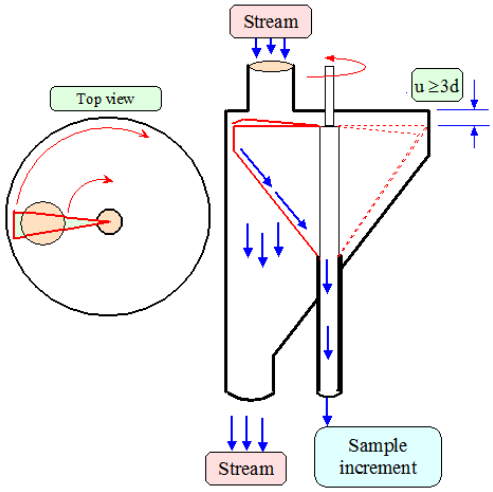


Figure 19.18: Rotating Vezin Sampler([40]Pitard, F, 2009.)

19.3.5 Stationary in-Stream Probes

There are a variety of stationary in-stream sampling probes, examples of which are shown in ***Figure 19.19***. These can be used, with great reservation, for process control but not for material balance. The major problem with these types of samplers is that they are based on the assumption that a stream is not segregated therefore collecting any part of the stream is good enough. Introducing turbulence in the stream may reduce segregation but it cannot eliminate it.

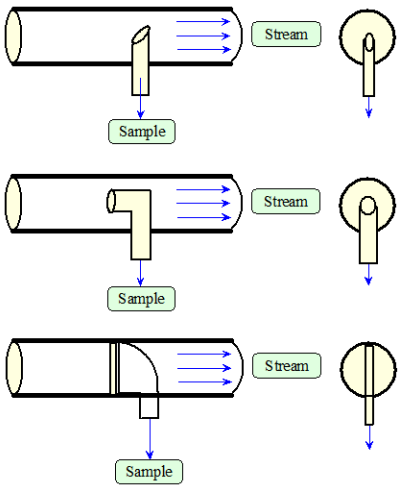


Figure 19.19: Stationary in-stream probes([40]Pitard, F, 2009.)

19.3.6 Diversion systems and flap samplers

Diversion and flap samplers divert the whole stream from which the increment is then taken, an example of which is shown in ***Figure 19.20.*** These systems use either flexible pipes or steel flaps but because they are not proportional, one half of the stream is over-represented and the other half under-represented. In a stream that is strongly segregated this could introduce huge biases.

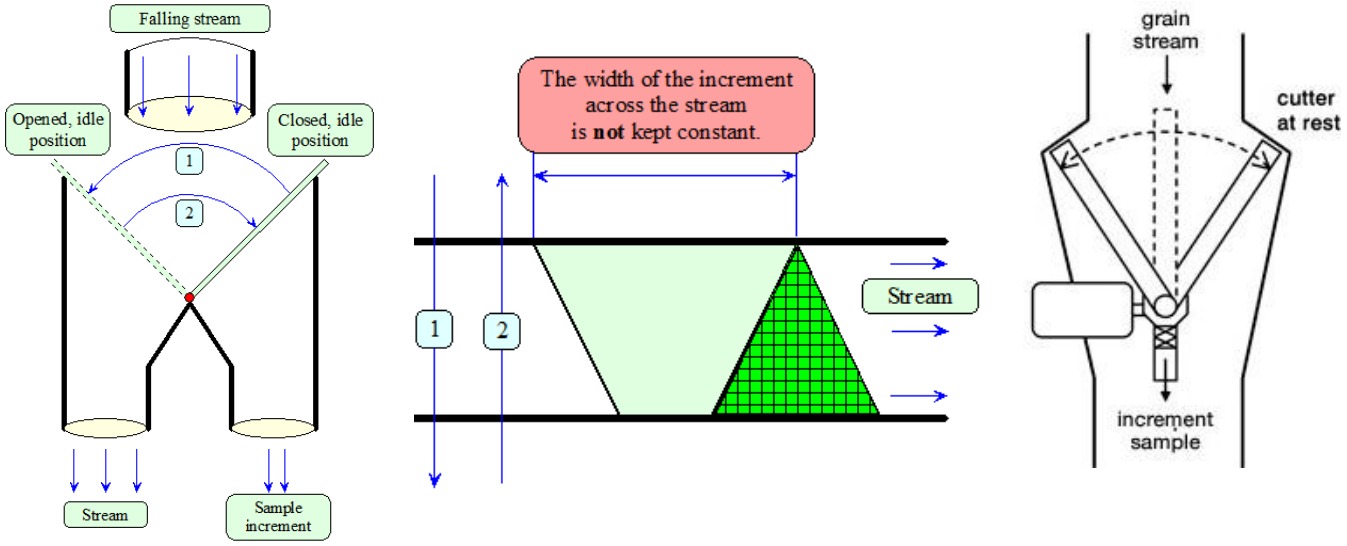


Figure 19.20: Diversion flap sampler([40]Pitard, F, 2009.)

20 19.4 IDE in the Laboratory

In the laboratory, as in the mine or the plant, optimal sampling is one-dimensional. In sampling a lot consisting of fine powder, the lot should be reduced to a two-dimensional layer and then sampled by taking channel samples across the layer. The correct design of scoops, spatulas and shovels having a flat base as shown in ***Figure 19.21,*** is essential.

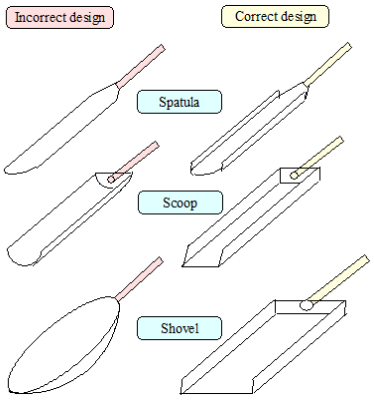


Figure 19.21: Examples of correctly and incorrectly designed spatulas, spoons and shovels ([40]Pitard, F, 2009.)

20 INCREMENTAL EXTRACTION ERROR (IEE)

IEE is an error of recovery. Even if a sample is perfectly (or imperfectly) delimited, it may not be possible to extract or recover it correctly.

20.1 IEE during Exploration Drilling

The key to minimising IEE in drilling programs is good core recovery, since 90 per cent recovery could result in a 50 per cent loss of gold in some types of mineralisation. Attempting to recover a sample in a damaged or broken core in the vicinity of a mineralised intersection, as shown in **Figure 19.4 a and b**, is an example of how IEE can introduce bias. In the case of exploration drill holes, good core recovery is essential to obtaining good samples. Furthermore, the choice of diamond drill bit is essential, as it may affect borehole core recovery. RC drilling is particularly prone to IEE, because perfect recovery of core, defined in the planning stage, is not possible.

Abrasion on the side of the core inside the barrel during normal diamond drilling leads to partial liberation or the so-called “plucking” effect. This leads to selective removal of soft minerals, particularly sulphides, from the surface of the core leaving it pitted to the touch. Copper analyses in core affected in this way are somewhat lower than they should be. Splitting the core with a diamond saw has the same effect. A similar effect occurs during RC drilling, when selective plucking of softer mineral grains from the wall of the borehole occurs; metal grades in sample material will be slightly higher. The difference in grade between rock recovered from diamond drill holes and RC drilling in a copper deposit because of the plucking effect is approximately 10%.

Although RC drilling is fast and cheap the main drawback is down-hole contamination as illustrated schematically in **Figure 20.1**. Softer sections of the sidewall, along the length of the hole, may collapse inwards and contribute to the sample, or portions of the sample are lost in crevices or fractures.

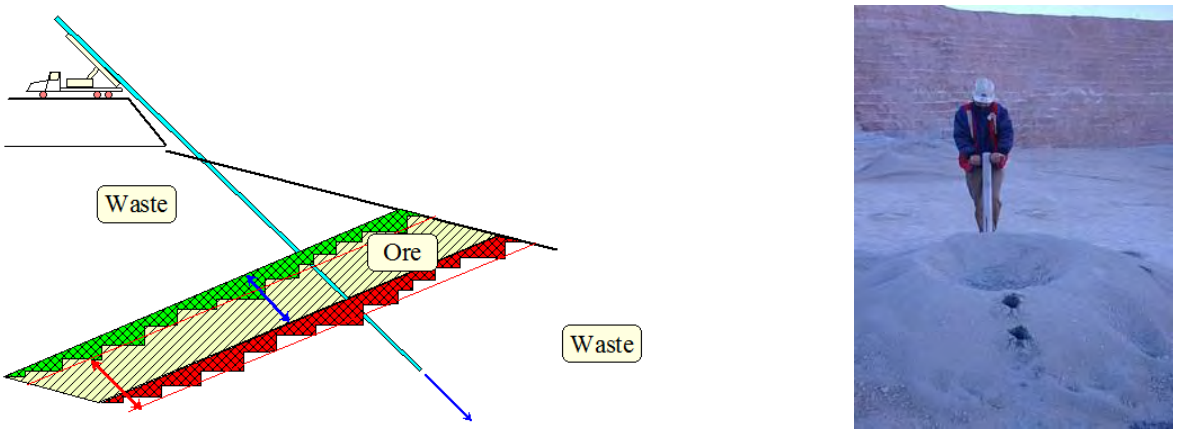


Figure 20.1: Down-hole contamination and smearing of grades([40]Pitard, F, 2009.)

Down-hole contamination smears the boundaries of the mineralised zones that falsely displaces the mineralised zone to deeper levels (***Figure 20.1***) in the geological ore body model. In the case of auger drilling the wide blades tend to push the coarse material aside and over-represent the fines.

Fugitive dust associated with RC drilling may be lost at the mouth of the borehole (***Figure 20.2***) or through the vent in the cyclone. Custom designed covers for the mouth of the borehole as designed by Ana Carolina Chieregati is shown in **Figure 20.2 b and c.** Secondary cyclones may also help to capture fugitive dust escaping from a RC drilling setup. Either way the loss of fines may result in a meaningful bias attributable to IEE. At and below the water table, local swelling may mean additional material contaminates the sample material in the hole. Insufficient air pressure to lift large particles, or loss of fines in the air or slurry could contribute to IEE. In particular, RC drilling provides little geological control on the position of changes in lithology and geological contacts, which in turn leads to poor geological control and modelling. Another problem associated with blasthole drilling is either the sorting of cuttings or segregation, as coarser fragments stay in the hole and the finer-grained material is blown out.

Figure 20.2: a) Example of measures to prevent loss of fines at the mouth of a blast hole(40Pitard, F, 2009.), b) and c) cover design by Ana Carolina Chieregati, Proactive reconciliation in the mining industry, 3rd WCSB, Brazil 2010

Although RC drilling has a lot of attractive features, it can introduce devastating extraction biases, during mineral exploration. Pressure at the bottom of the borehole may cause soft materials such as clays, soils and laterites, to be extruded up the borehole, leading to core recoveries of greater than 100%. If the range of fragment sizes is large, larger fragments could be rejected with finer materials being preferentially recovered as indicated in ***Figure 20.3.***

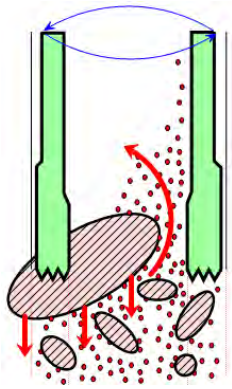


Figure 20.3: Selective and partial sample recovery([40]Pitard, F, 2009.)

20.2 IEE in the Mine

The sampling of draw points (***Figure 20.4***) is particularly difficult and can lead to very large IEE's.

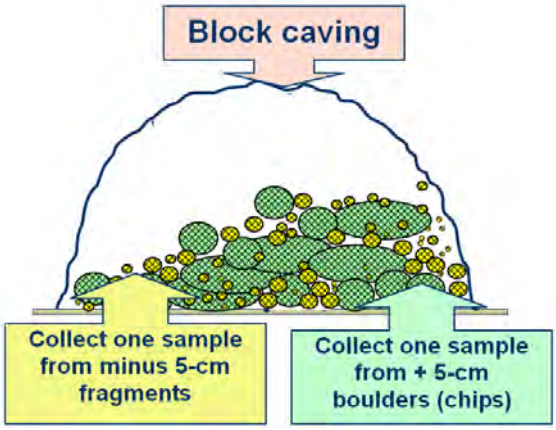


Figure 20.4: Sampling draw-points([40]Pitard, F, 2009.)

A suggested remedy, especially for grade control procedures in underground mining operations, is that for every 500t, two samples be collected, one from the minus 5cm fragment size range and one from the +5cm boulder (chips) fragment size range. The grade discharged from the draw–point can be monitored by collecting these samples at the mouth of the draw-point at regular intervals and plotted on a control chart with using a control chart for both types of samples. From these data control charts can be compiled as shown in Figure 20.5. Once the grade of the material from the draw-point falls below the draw-point over a given period of time or for a given tonnage the draw-point can be closed.

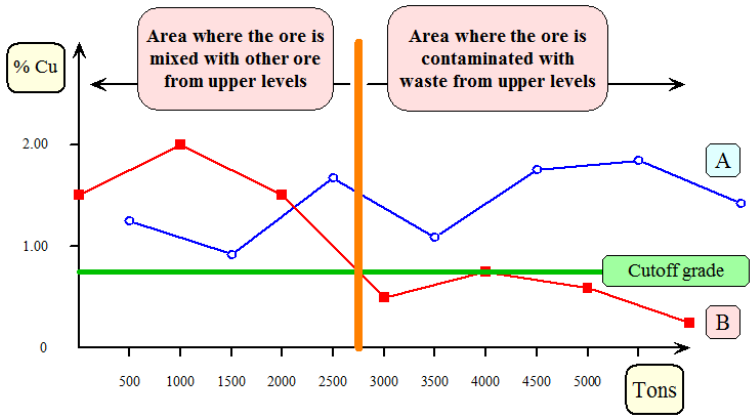


Figure 20.5: Control chart for draw-points; A: samples from -5cm fragments; B: Chip samples from +5cm boulders([40]Pitard, F, 2009.)

20.3 IEE in the Plant

Like IDE, IEE introduces some of the largest biases encountered in sampling. The rule of the centre of gravity, as illustrated in ***Figure 20.6,*** must be respected. All fragments, having their center of gravity inside the model-extended increment, belong to the model-extracted increment.

This aspect of IEE focuses on conditions of extraction correctness relative to cutter characteristics. Conditions likely to play an important role are the: straightness, thickness and shape of the cutter edges, length and inclination of the cutter opening, cutter width, speed, depth, capacity and general design as well as stream turbulences and velocity.

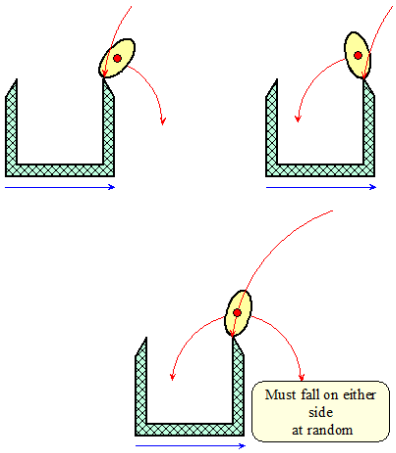


Figure 20.6: Rule of the Centre of Gravity. If CoG falls within the sample cutter, the fragment should be retained. If CoG falls outside the sample cutter, the fragments should be rejected. ([40]Pitard, F, 2009.)

Two examples of recommended cutter designs for samplers are shown for sticky material in ***Figure 20.7a*** and for normal materials in ***Figure 20.7b.***

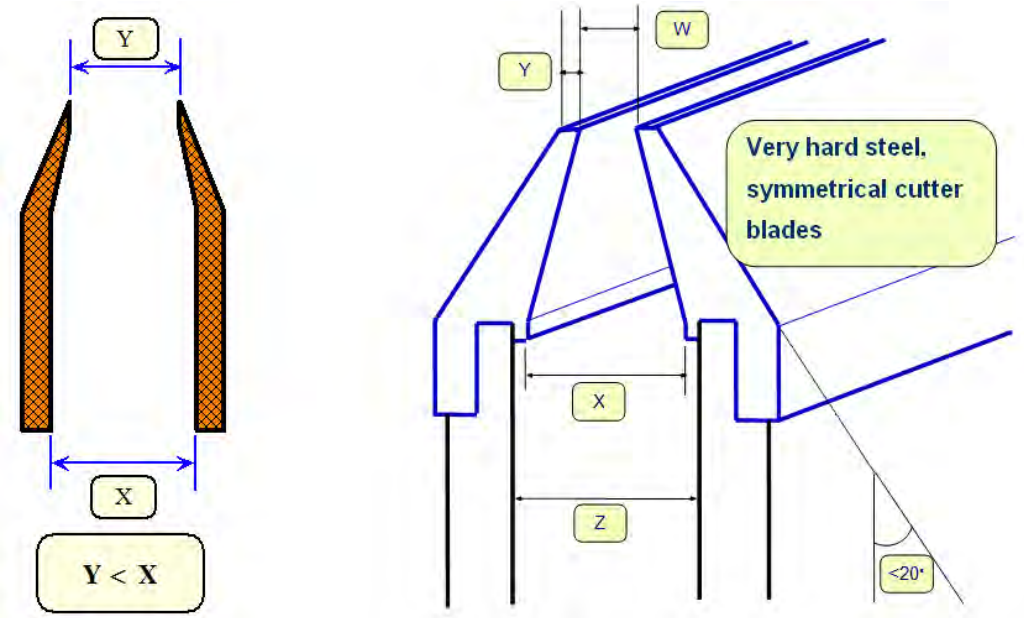


Figure 20.7: a) Recommended cutter design for sticky materials ([40]Pitard, F, 2009.), and b) recommended cutter design for normal materials ([40]Pitard, F, 2009.)

The Australian CSIRO[82] investigated sample cutter operations in detail and identified nine principles that must be applied to sample cutters in industrial sampling installations. The following principles apply:

- The sampler must intercept the entire stream of particles;
- The cutter must move at constant speed;
- The cutter blades must be sharp and straight;
- The cutter opening must have a constant width;
- The cutter must have sufficient capacity to hold the sample;
- There must be no loss or contamination of the sample;
- Bridging of material over the cutter opening must be avoided;
- Do not use vertical or near vertical cutter blades, and;
- Particles should not ever hit one cutter blade and bounce clear over the other cutter blade.

Other simple aspects of cutter operation that are all important include: routine maintenance of cutters, ensuring the cutters are driven by adequately powered motors, ensuring that the cutter have generously large apertures, and ensuring regular routine inspection of the cutter operation.

Pitard has listed several positions in the plant where IEE may arise, specifically for three typical dry material cross-stream sampling systems, namely a cross-stream sampler installed under the discharge of a conveyor belt, a cross-belt sampler installed above the conveyor belt, and a rotating Vezin sampler often used for secondary or tertiary sampling.

20.3.1 A cross-stream sampler installed at the discharge of a conveyor belt

The side-view of the cross-stream sampler illustrated in **Figure 20.8** is shown as a front-view in **Figure 20.9**. This is referred to as a 'bottom dump cutter' whose chief advantage is its simplicity. The points that Pitard makes in regard to the appropriate design, maintenance and operation of this type of cutter are as follows:

- Point 1: Electric motors must drive the cutters; hydraulic or pneumatic drives are not acceptable;
- Point 2: The cutter must intercept the whole of the falling stream;
- Point 3: The capacity of the bucket should be about twice the maximum increment weight at a maximum flow rate;
- Point 5: The belt must be equipped with a scraper, to capture all fine ore spilled during the return of the belt;
- Point 6: The cutter should be perpendicular to the trajectory of the intercepted stream;
- Point 7: Fragments bouncing on the cutter edges that belong to the increment, should be captured;
- Point 8: No part of the increment should get stuck on the slopes inside the cutter or in conveying chutes;
- Point 9: No spillage should occur at the discharge mechanism of the bottom dump cutter;

[82] Holmes, R. MCMXC. A video presentation entitled "Investigation of sample cutter operations". Australian Mineral Industries Research association Project P313: CSIRO Division of Mineral and Process Engineering..

- Point 10: Vertical and lateral movement of the cutter should not be hindered in any way;
- Point 11: For plant slurries in particular the stream velocity should not exceed exceeds 2 m/s;

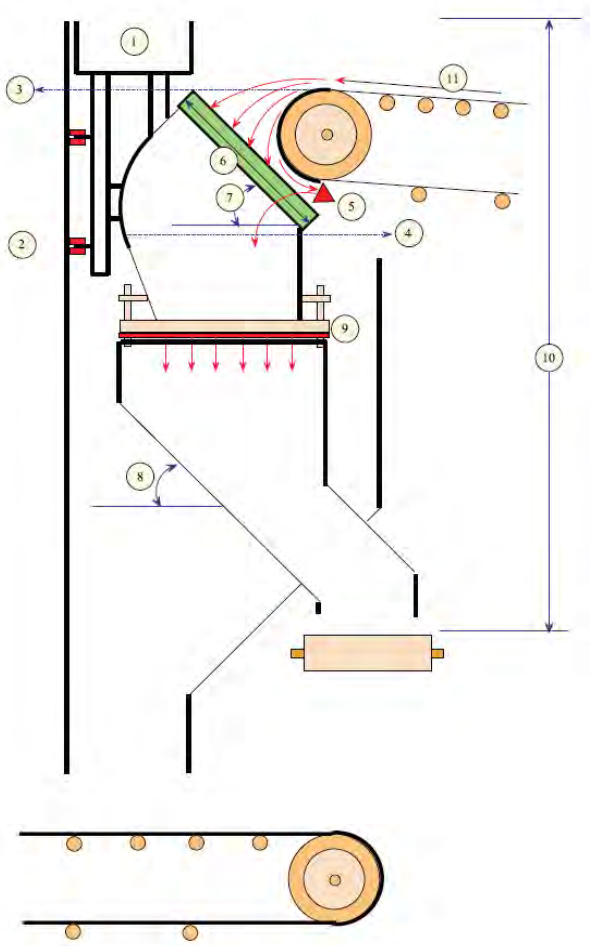


Figure 20.8: Side view with a bottom dump cutter (Source: Pitard)

Additional comments Pitard (2009) makes regarding the front view of the cutter shown in **Figure 20.9** continue as follows:

- Point 11: The cutter opening *W* must be generous; W ≥ 3*d* + 1 cm where *d* is the screen size that would retain no more than five per cent of the material;
- Point 12: The cutter velocity V should not exceed 45 cm/s;
- Point 13: The length of the cutter should be at least double that of the intercepted stream thickness, ensuring that the stream is well centred in the middle of the cutter;
- Point 14: The cutter must be travelling at full speed and constant velocity as it enters and passes through the stream.
- Point 15: The gate at the bottom dump cutter must: open completely when discharging the increment, and must close completely when collecting the increment;
- Point 16: A large inspection door, for daily sample cutter inspection must be present

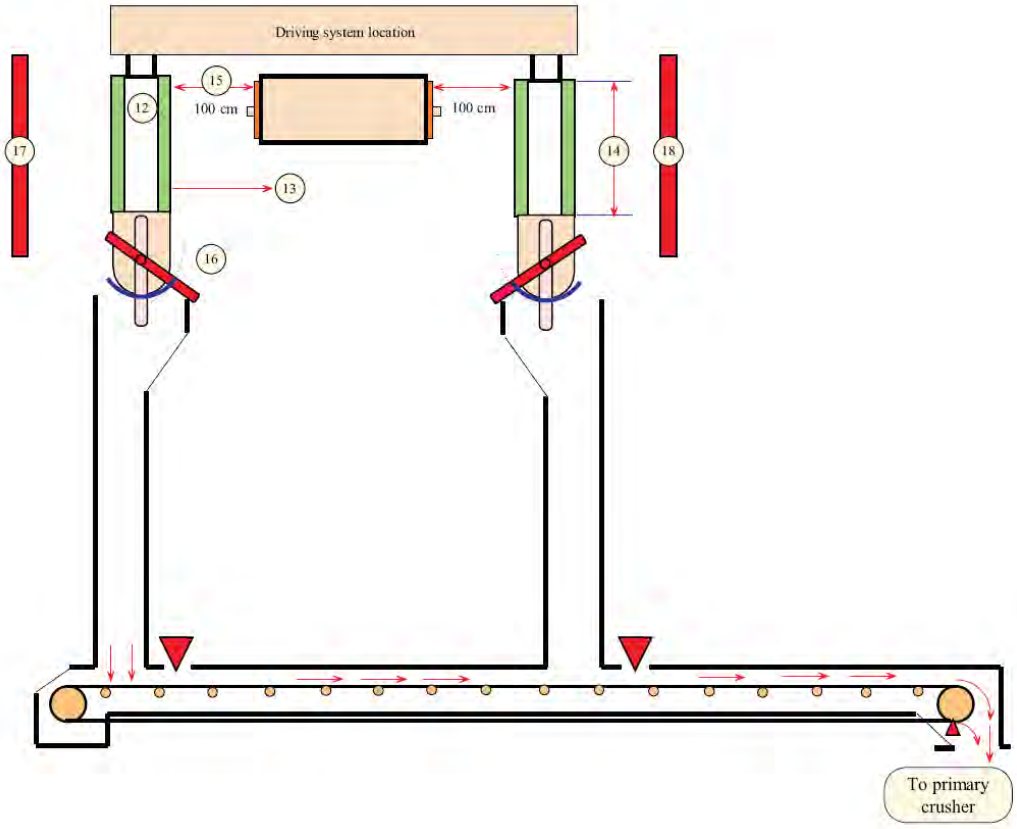


Figure 20.9: Front view of the bottom dump cutter sampler ([40]Pitard, F, 2009.)

20.3.2 A cross-belt sampler installed over the conveyor belt

Cross-belt type samplers, such as that illustrated in **Figure 20.10**, are popular in the coal, base metal , gold, and platinum industries. However the evidenced IEE associated with cross-belt samplers means they generally do not comply with the principles of sampling correctness. Pitard61 suggests that where possible, other samplers should be installed. Issues associated with such cutters include:

- Point 1: The imperative of an effective brush (or rather a rubber wiper arrangement) behind the cutter to sweep fine material off belt after the cutter has crossed the stream;
- Point 2: Frequent inspection , setting and/or replacement of the brush or rubber wipers;
- Point 3: Disturbed material (not delineated) swept to the side of the belt can introduce an extraction bias;
- Point 4: The capacity of the cross-belt cutter, must be compatible with the flow rate of material. Design of the cutter blades should ensure there is no damage to the belt. An exaggerated gap between the belt and the cutters introduces IDE. The velocity of the cutter may cause material to be lost either through bouncing or incomplete transfer to the sample chute (IPE);
- Point 5: Cross-belt samplers should be installed on a firm base in a horizontal section of the belt; preferably not on a slope;
- Point 6: The higher the cutter velocity (for straight cutters) the better, but this may introduce new problems when using angled cutters ; the cutter velocity must be constant;
- Point 7: The cross belt sampler must have a large, easy to open inspection hatch.

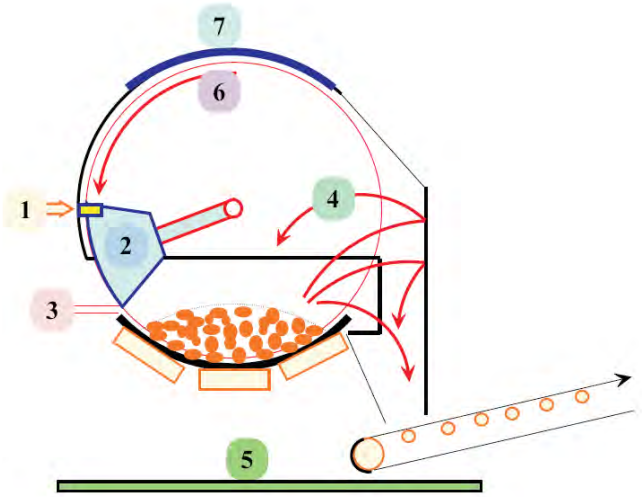


Figure 20.10: Generic cross-belt sampler. ([40]Pitard, F, 2009.)

Unlike the cross-stream samplers installed under the discharge of the conveyor belt, only some of the issues described for cross-belt samplers have acceptable solutions. As a result, the sampling standard should not readily promote the use of this kind of sampling system, even if industry considers it a popular choice. Furthermore, under no circumstances should this system be termed a proportional sampler.

A Discrete Element Model of a cross-belt cutter developed at the CSIRO, Australia by Robinson and Cleary indicates that although a large proportion of the material on the belt is extracted by the sampler (**Figure 20.11a and b**), there can be material that is left on the conveyor belt after the cutter has made its arc through the material on the belt (**Figure 20.11b**).

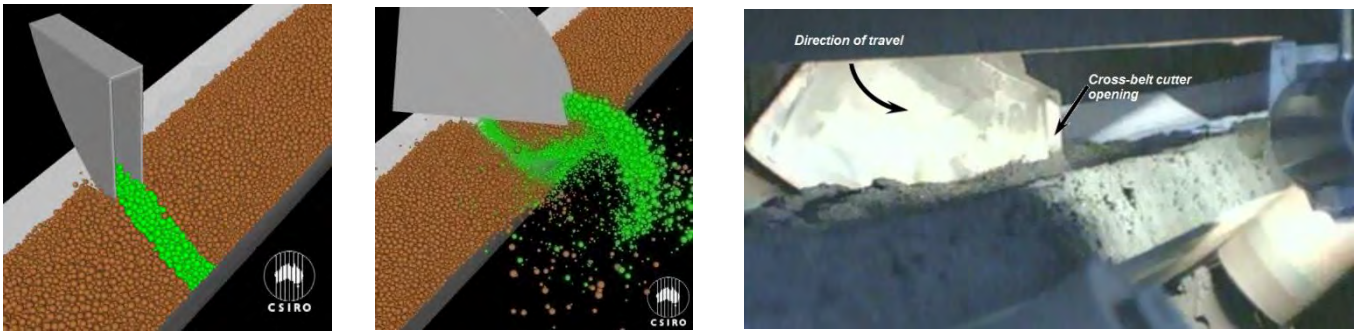


Figure 20.11: a) Discrete Element Model of a cross-belt cutter indicating the increment delimited, b) the increment extracted, and c) a high-speed camera picture of a cross-belt cutter extracting a reasonably good cross-belt sample increment (used by permission of the CSIRO, Australia)

In addition, material that was never delimited as part of the sample can end up being thrown off the belt and a component of the belt load originally delimited as part of the sample can be scattered by the cutter and not captured as part of the increment. For cross-belt samplers with straight cutters, a set of curtains immediately adjacent to the cutter at stream exit point can minimise misplacement of material.

20.3.3 Dry Rotating Vezin sampler

Rotating Vezin samplers are often used for secondary or tertiary sampling and are highly recommended as they are cheap, robust, compact, and perfectly proportional. A plan and side-view projection of the rotating Vezin cutter is shown in **Figure 20.12** and a horizontal projection of the sampler is shown in **Figure 20.13**.

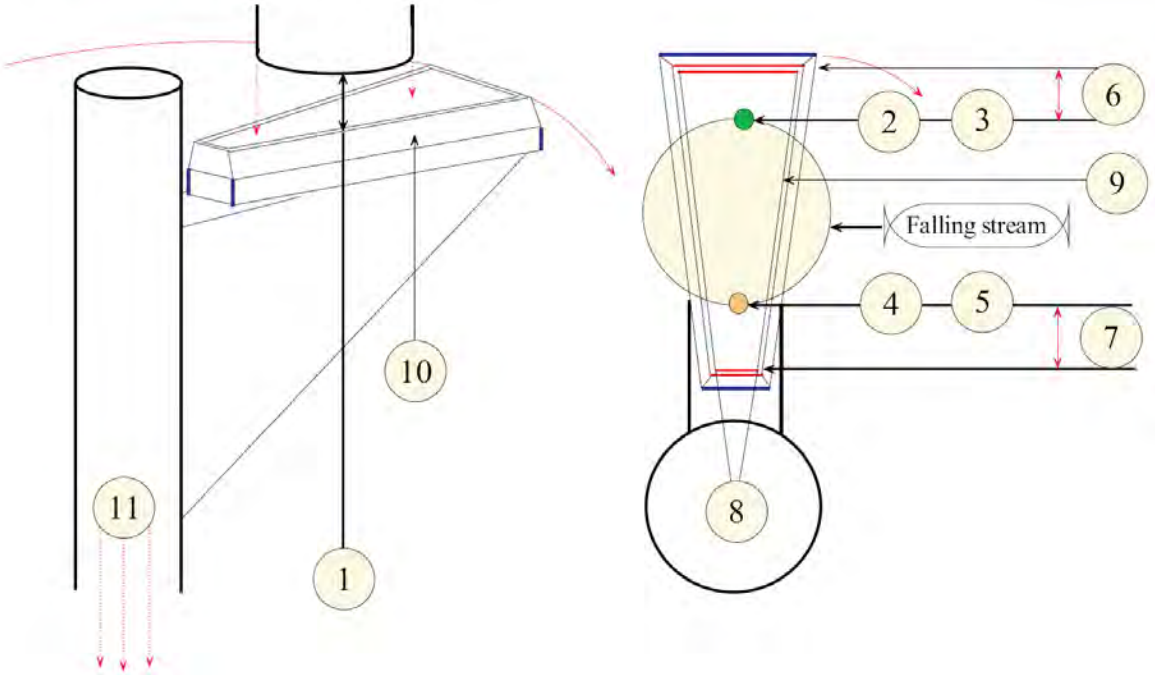


Figure 20.12: Side-view and plan projections of the Rotating Vezin cutter ([40]Pitard, F, 2009.)

Pitard (2009) emphasises that in order to ensure correctness attention is required in the following areas:

- Point 1: The distance *D* between the stream discharge and the cutter edges should be relatively small, following the rule: $D \geq 3d + 2$ cm where the screen size *d* retains no more than five per cent of the material;
- Point 2: The speed V of the outer perimeter of the cutter should not exceed 45 cm/s for larger diameter (60 cm), and no more than 30 cm/s, for smaller diameter rotating Vezins.
- Point3: At the farthest point from the rotation centre, where the cutter intercepts the stream, the cutter opening is always conservative.
- Point 4: At the closest point from the rotation centre, where the cutter intercepts the stream, the cutter opening *W* must fulfil the requirement $W \geq 3d + 1$ cm where *d* is the size of a screen that would retain no more than five per cent of the material.
- Point 5: At the closest point from the rotation centre, where the cutter intercepts the stream, the cutter speed is always conservative.
- Point 6: The distance between the farthest point from the rotation centre, where the cutter intercepts the stream, and the outer end of the cutter opening, must be large enough to recover fragments bouncing on the cutter edges that belong to the increment. A strict minimum distance is 5 cm. This would prevent an extraction bias from taking place.
- Point 7: The distance between the closest point from the rotation centre, where the cutter intercepts the stream, and the inner end of the cutter opening, must be large enough to recover fragments

bouncing on the cutter edges that belong to the increment. A strict minimum distance is 5 cm. This would prevent an extraction bias from taking place.

- Point 8: The cutter edges must be perfectly radial with respect to the centre of rotation. Small deviation from that rule is likely to introduce a delimitation bias.

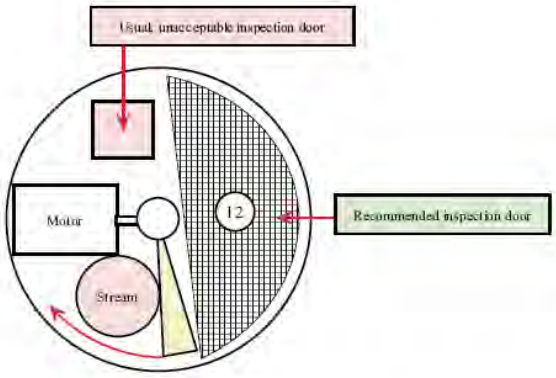


FIG 21 - Recommended inspection door for a Vezin sampler.

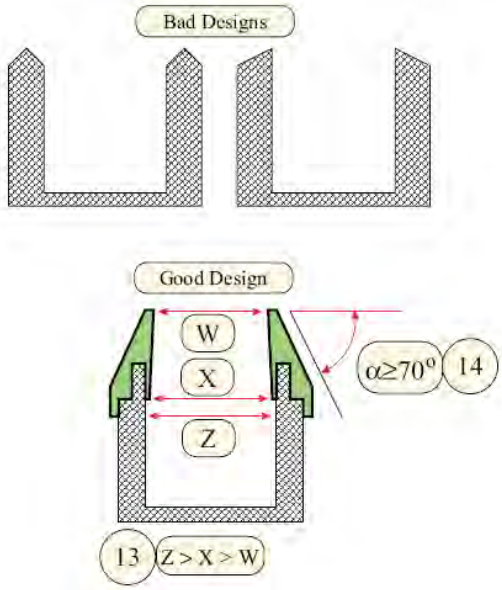


Figure 20.13: Plan view of Vezin cutter (top), sections through good and bad cutter designs (middle and bottom)([40]Pitard, F, 2009.)

- Point 9: The top of the cutter edges must show a narrow, flat area (eg 0.5 to 1 mm) to prevent quick deformation of the cutter edges that may lead to both a delimitation bias and an extraction bias.
- Point 10: The cutter blades must be perfectly symmetrical, mirroring each other. Adjustable blades are an invitation to deviate from that important rule. Any small deviation, sometimes not obvious with the naked eye, may introduce a which are both bias generators. It is highly recommended to engineer the cutter as a single cap, so there is absolutely no doubt about the symmetrical correctness.
- Point 11: The exit of the Vezin cutter must be large enough so no material may accumulate inside the cutter and make it overflow. The exit of the Vezin chamber should be large enough so the material from the stream does not accumulate or interfere with the rotation of the cutter.

- Point 12: Many Vezin samplers on the market have ‘postage stamp’ size inspection doors. Any sampler that does not have a large inspection door, easy to open and close, and easily accessible is not an acceptable sampling system and should be rejected unconditionally by the sampling standard. Install a screen on the inspection door and a safety switch to prevent the sampler from rotating when the door is opened.
- Point 13: The inner width of the cutter, and especially the cutter chamber, must always be larger than the effective opening of the cutter, preventing material from sticking to the inner walls.
- Point 14: The outer slope of the cutter blades must be at an angle at least 70°, so no material can find its way through the cutter opening by climbing along the leading cutter edge.

20.4 Slurry cross stream sampling

20.5 Introduction

The objective is to make suggestions for automated slurry sampling systems to allow for reliable metallurgical accounting as a process plant imperative. Taking the opportunity of this work, a guideline is given, so clients or end users can prepare an in-house standard series of basic requirements for manufacturers of sampling equipment and engineering firms to fulfill for any new project, or for modernizing slurry sampling systems in existing operations in precious and base metal operations in particular and mineral industry in general.

20.6 A necessary commitment from end user Management or executives

This guideline would be ludicrous without emphasizing how important it is for upper management to fully endorse correct sampling (i.e. unbiased sampling). The report would be incomplete if there was no mention of the important role sampling can play in risk identification, risk assessment, and risk treatment. **Good sampling practice has a direct effect on the economic health of a company.** Therefore management, board of directors, and shareholders should be extremely motivated to study a reliable database in order to create believable cost-benefit analyses. In this endeavour, stakeholders must be able to see the value of quality sampling: it can be done if the in-house sampling standard is very clear about the role of good sampling to perform reliable statistical studies, which in turn allows managers, directors, and shareholders to implement quality management.

The “three-legged table” concept

What is wrong with a three-legged table? If one leg is broken, there is no table left! Like it or not, the market value of a company is all about money. We could compare the top of the table to the capability of a company to make good benefits, to effectively create added stakeholder value, therefore to make the market willing to pay a higher price for its shares. For this scenario to take place, the table must have three solid legs as shown in figure 20.14:

A proactive management style emphasizing causes of problems instead of emphasizing effects of problems (e.g., bad sampling versus bad conciliation).

A clear commitment to good sampling and laboratory practices (e.g., use of optimum sampling protocols relative to well defined Data Quality Objectives, invest in correct sampling systems, and invest in good laboratory practices), [1].

A capability to understand variability (e.g., train staff on geostatistics and chronostatistics), to perform reliable statistical studies based on good samples and good assays, so management can have access to many costs generally invisible to the ordinary accountant, [2].

Another way to look at the requirement for correct sampling systems is to clearly understand that there is a direct correlation between the market perception of the share value of the company and the reliability of its sampling/analytical capabilities during exploration, mining, and milling. It would be a devastating mistake to underestimate the critical importance of that connection.

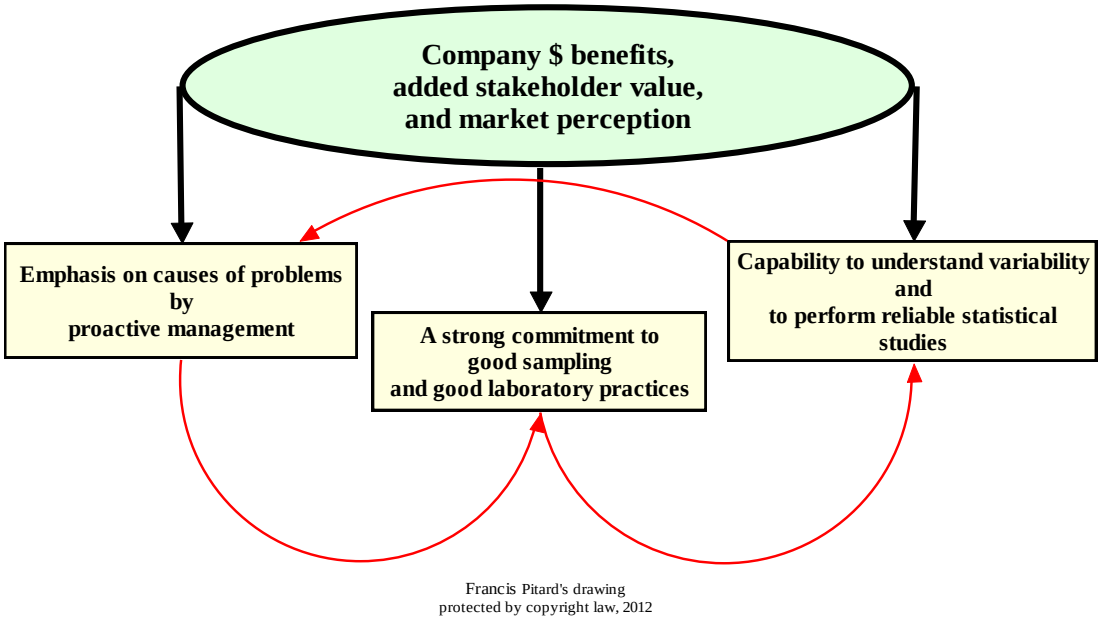


Figure 20:14. A management strategy founded on three solid, steady pillars

20.7 Suggested Sampling Systems for Mill Cyclone Overflow, Leach or Flotation Feed, and the CIP, Scavenger, Cleaner or Final Plant Tails

The slurry is often 95% passing 150 microns. It is recommended to collect a composite sample every 8 hours, small enough to be sent to the laboratory.

The following suggestions are the fundamental requirements to make sure slurry cross stream sampling stations will be correct in all details. Failure to comply can generate a non-representative sample. It does not show the basic engineering work necessary to install the various components on site, which is the entire responsibility of plant management, the manufacturer of sampling equipment, and the engineering firm.

Try using recommended similar conceptual designs for slurry sampling stations, being aware the sampling stations for some may be slightly oversized. However it will present the advantage of having the same spare parts for all stations.

20.8 THE PRIMARY LINEAR CROSS-STREAM SAMPLER

A straight-path cross-stream sampler like the one illustrated in **Figure 20.15** is a recommended suggestion. Important points to address are as follows (For points 1-16, see **Figure 20.15**):

Point 1: If the recommended diameter of the main process falling stream is, say 50 cm, for the various applications, this diameter should not increase above the indicated value, without a complete redesign of the primary sampler.

Point 2: Assuming the stream is well centered on the trajectory of the cutter, the length of the cutter opening should be at least 80 cm, providing a safety margin of about 15 cm at each end to ensure complete recovery of any splashing material on the cutter that would belong to the increment.

Point 3: The cutter blades must be built with very hard stainless steel, to ensure a reasonably long life.

Point 4: The distance between the exit of the 51cm diameter pipe and the top of the cutter, should not exceed 5 cm: Anywhere between 2.5 and 5 cm is acceptable.

Point 5: The angle of the discharge slope of the cutter chamber can be anywhere between 30 and 45^{o}. Too steep a slope would feed the launder and the secondary sampler too fast. Any welding seam cross the path of increments inside the cutter, along the slopes must be avoided.

Point 6: The distance between the bottom of the cutter, in the alignment of the stream trajectory, should not be smaller than 2/3 the length of the cutter. It is imperative that no material can splash out after entering the cutter.

Point 7: It is critically important to insure the perfect stability of the cutter as it crosses the stream. Under no circumstances should the cutter slightly flip on impact with the falling stream. Wheels and rails can be provided to help achieve perfect stability.

Point 8: The cutter must be driven by an electric drive. A hydraulic or pneumatic drive is not acceptable. The motor torque must be very generous so the cutter does not slow down as it crosses the stream.

Point 9: As the cutter crosses the falling stream, the level of liquid/slurry inside the cutter should under no circumstances reach the upper level of the cutter for two reasons:

- To prevent overflowing that would result in an extraction bias.
- To prevent the cutter from getting too heavy that may result in a delimitation bias.

Point 10: The opening allowing the exit pipe of the cutter to feed the launder should not be a source of spillage or sample contamination. Two important features can prevent this problem from taking place:

- A fixed rubber curtain, with a slot allowing the pipe to slide inside during the trajectory (i.e., the blue part in figure 2, near point 10).

- A metal shield, welded to the pipe, preventing any material following the pipe from entering the launder (i.e., the red part in figure 2, near point 10).

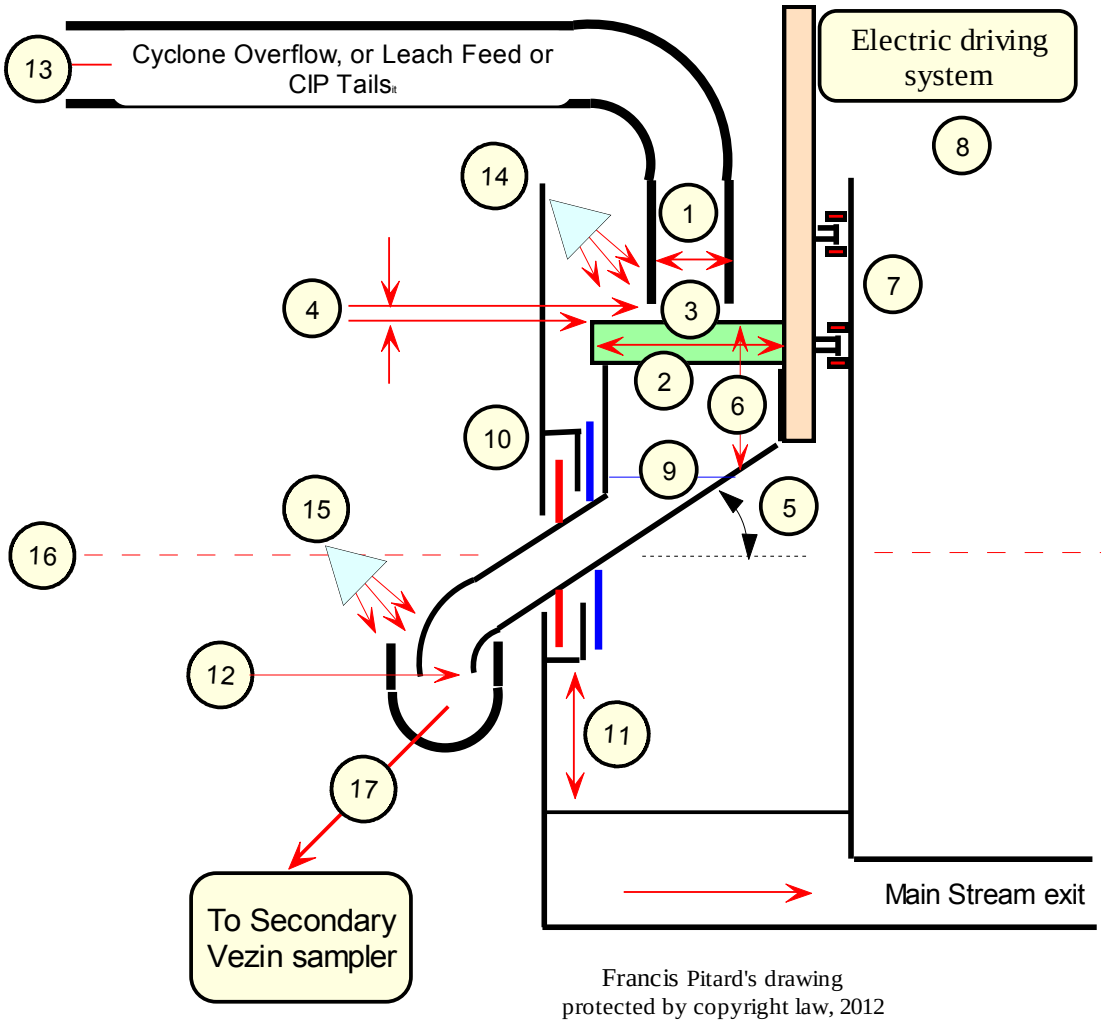


Figure 20.15: Side view of the recommended primary sampler for the Cyclone Overflow, Leach and Flotation or Plant Feed and the CIP, Rougher, Cleaner, Scavenger or Final plant Tails

- Point 11: The level of material in the main tank feeding the CIL circuit should not rise anywhere close to the opening leading to the launder. This is a very important engineering detail.
- Point 12: The diameter of the exit pipe of the cutter should be such that the condition required in point 9 is fulfilled.
- Point 13: The velocity of the stream should remain below 2-meter/second for the cutter to work satisfactorily. If the stream has too much energy, excessive splashing, and cutter damage may result. At a maximum flow rate of 1700 m3/hour (for 51cm diameter pipe column), the speed may slightly exceed 2 meters per second. Any flow increase would absolutely require a larger diameter pipe to slow down the stream.
- Point 14: At both parking places, an automated water spray should be installed to keep the cutter clean after each cut is completed. The material inside the cutter, washed by the sprayer, must be sampled by the continuous Vezin sampler.

- Point 15: The launder must be washed with an automated water spray installed to keep the launder clean after each cut is completed. The material inside the launder, washed by the sprayer, must be sampled by the continuous Vezin sampler.
- Point 16: It is of the utmost importance the primary sampler be surrounded by a platform for easy access, so the operator can inspect all the parts of the sampling system at any time. Large inspection doors, one at each parking place, equipped with 2.5-cm screen for safety, are essential for ensuring the correct functioning of the sampling system.

For points 17-23, see **Figure 20.16**.

Points 17 and 18: The parking places of the cutter should be at least 25 cm away from the stream, to prevent contamination, and to allow enough space to reach cruising speed before the cutter enters the stream.

Points 19 and 20: At each cutter parking place, a large inspection door, easy to open and close, should be provided for effective, daily inspections.

Points 21 and 22: Behind each inspection door, a 1-inch mesh screen should be installed for safety reasons.

Point 23: The maximum speed across the stream is 45 cm/s (ASTM). Recommend a speed V = 40 cm/s as preferable.

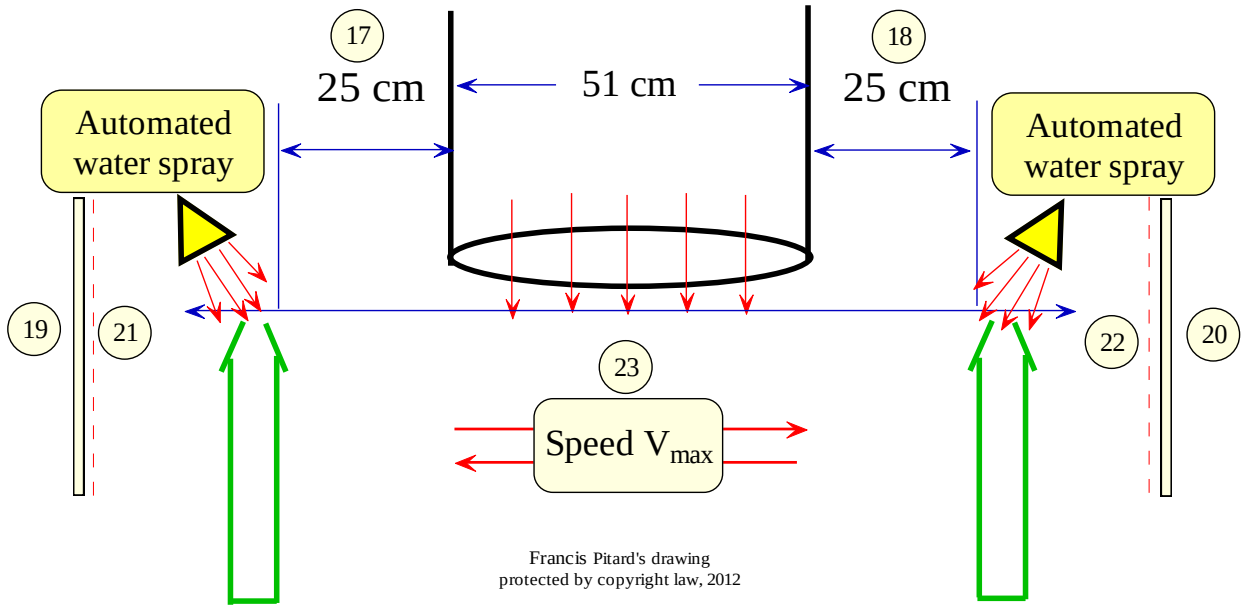


Figure 20.16. Front view of the recommended primary sampler

For points 24-29, see **Figure 20.17**.

Point 24: For a maximum flow rate of 1700 m^3/h , say, a minimum 1.5-cm cutter opening is necessary to prevent an extraction bias, as illustrated in figure 10. Recommend a 2-cm cutter opening W.

Point 25: To prevent fast deformation/ wear of the cutter edges, recommend a 1-mm flat surface at the top of the edge. Under no circumstance should that flat surface be at an angle (i.e., the flat surface must be perfectly perpendicular to the intercepted stream).

Point 26: Any cutter chamber width, at X or Z, should be larger than the effective opening W of the cutter.

Point 27: The angle of the cutter blades, relative to the falling stream should not exceed 20°, so any material falling on the leading blade when crossing the stream cannot enter the increment that would introduce a devastating extraction bias.

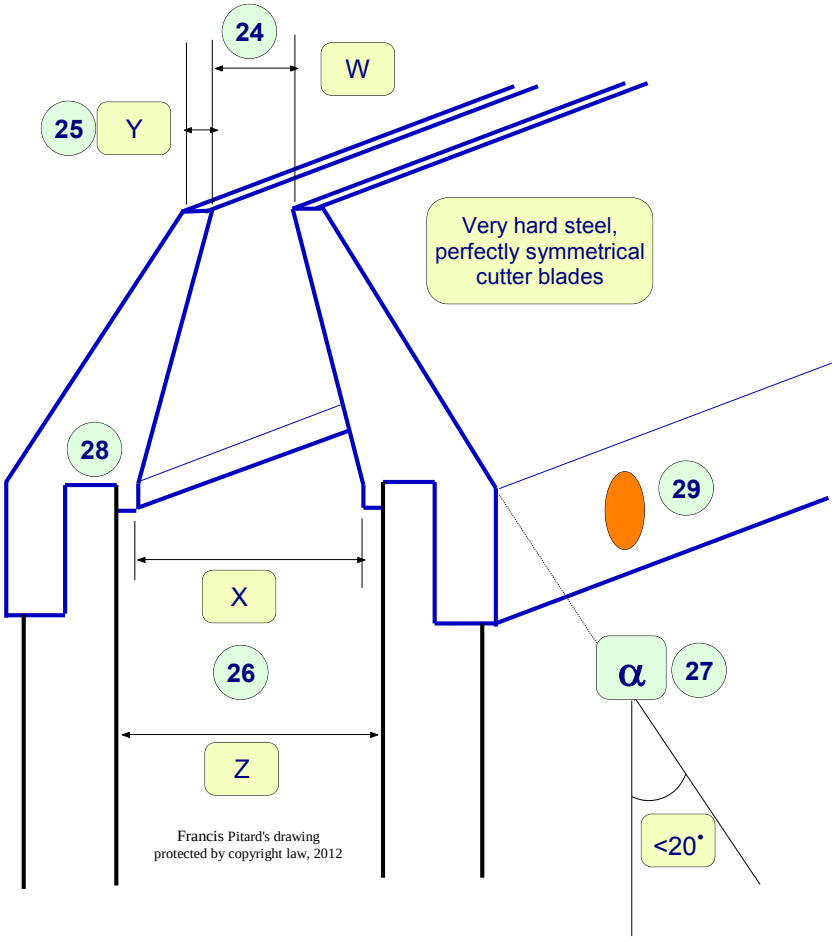


Figure 20.17. Recommended cutter design

Point 28: The cutter blades should be engineered in such a way that there is only one possible position to install it. This would insure perfect symmetry, the blades mirroring each other. This is always a problem with adjustable blades, because operators do not appreciate how important this detail is.

Point 29: Any bolt to fix the blade to its socket must be as far away/down from the cutter edge as practically possible, or an extraction bias may take place.

If the recommendations in points 23 and 24 are applied, we may calculate the volume V_I of one increment when crossing the falling stream:

$$V_I = \frac{1700000x2}{40x3600} = 23{,}6liters$$ for a maximum flow rate

Finally, make sure the timer activating the primary sampler can be adjusted anywhere between 30 seconds to 30 minutes. Make sure that an option exists to choose a certain stratum/interval and then an additional one to select a random time within that interval to cross the stream. Stratified random sampling is necessary when cycles are expected.

21 SAMPLE COLLECTION LAUNDER

Figure 20.18 shows important features to insure the launder located between the primary and secondary samplers works correctly.

Point 1: The entire trajectory of the primary cutter discharge must be well contained inside the launder.

Point 2: The level of slurry inside the launder should not interfere with the trajectory of the primary cutter.

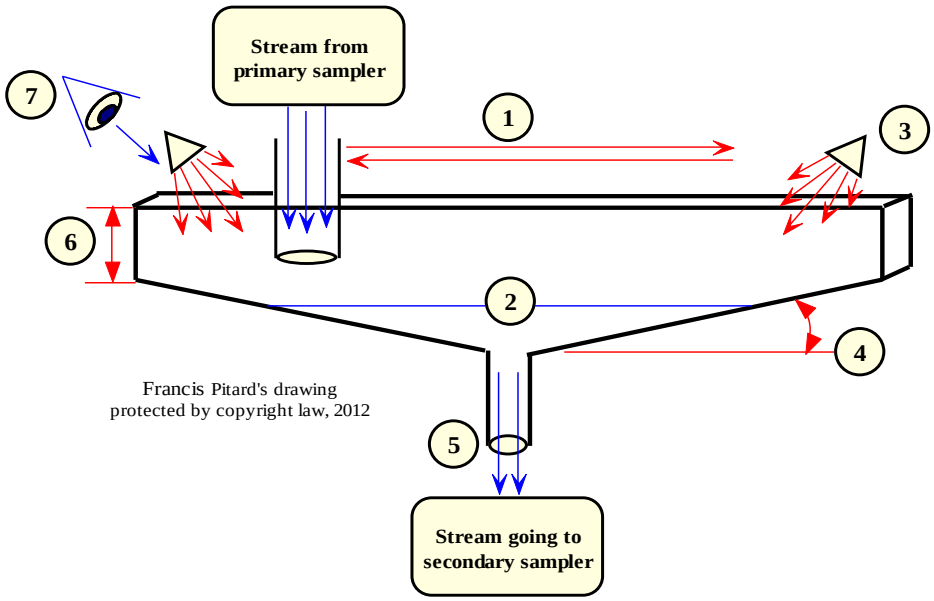


Figure 20.18: Illustration of important features for the launder

Point 3: On each side of the launder, an automated water spray should be installed to keep the launder clean after each cut is completed. The material washed by the sprayer inside the launder must be sampled by the continuous Vezin sampler.

Point 4: The angle of the launder bottom should be about 15° to slow down the stream going to the secondary sampler.

Point 5: The diameter of the launder exit should be slightly smaller than the diameter of the primary cutter, so the primary increment is not fed to the secondary sampler too fast.

Point 6: The capacity of the launder should be such that it is impossible for it to overflow.

Point 7: The launder must be easily accessible for visual inspection and for cleaning, after each working shift is completed.

22 SECONDARY ROTATING VEZIN CROSS-STREAM SAMPLER

Suggest a continuous rotating Vezin cross-stream sampler like the one illustrated in Figure 20.19. A Vezin with 60-cm inner diameter is recommended. A 6% sampling ratio, divided by three 2% radial cutters is recommended. Important points to address are as follows (For points 1-10, see figure 6):

Point 1: The entry to the Vezin sampler should under no circumstances be farther than 1 meter from the exit of the launder for the following reasons:

To prevent too much material to remain on the walls, along the chute.

To prevent the stream to get too much momentum before reaching the Vezin cutters. The quieter the stream to be sampled the better.

Point 2: The motor to drive the cutters must be electric, should be well protected from the environment, and should not interfere with the installation of a large inspection door.

Point 3: The inspection door should cover at least 30% of the top surface of the Vezin sampler.

Point 4: The distance between the exit of the stream coming from the launder and the top of the cutter blades should be 3d + 2cm, d being the size opening of a screen retaining no more than 5% by weight of the solids. Since d is very small, a 2-cm distance is recommended.

Point 5: The exit of the stream from the launder must not be exactly flush with the top of the Vezin to prevent some of the slurry from going sideways along the ceiling of the chamber.

Point 6: The angle of the bottom part of the cutters should be anywhere between 50 and 60^{o} to allow quick evacuation of increments. Under no circumstances should any welding seam cross the path of increments inside the cutters, along the slopes.

Point 7: All parts of the stream to be sampled must have exactly the same chance of being selected. This requires two critically important conditions:

Cutters must cross the stream at constant speed.

Cutter edges must be perfectly radial relative to the center of rotation.

Point 8: Under no circumstances should the exit diameter of the Vezin be smaller than the entry diameter shown in point 5.

Point 9: The closest stream point from the center of rotation is the location to measure the minimum cutter opening W. Assuming the Vezin has a 60-cm inner diameter, a 8-cm diameter stream centered as shown in

Figure 7, the opening of the cutters at that point is about 2.1 cm, which is conservative: The strict minimum requirement at this point is 1 cm.

Point 10: The farthest stream point from the center of rotation is the location to measure the maximum cutter velocity V. Assuming the Vezin has a 60-cm inner diameter, a 8-cm diameter stream centered as shown in figure 7, the velocity of the cutters at that point should be no more than 30 cm/s (i.e., no more than 12 rotations per minute). There is little compromise possible with this requirement as proven many times by experience with many clients. Most Vezin samplers around the world rotate much too fast.

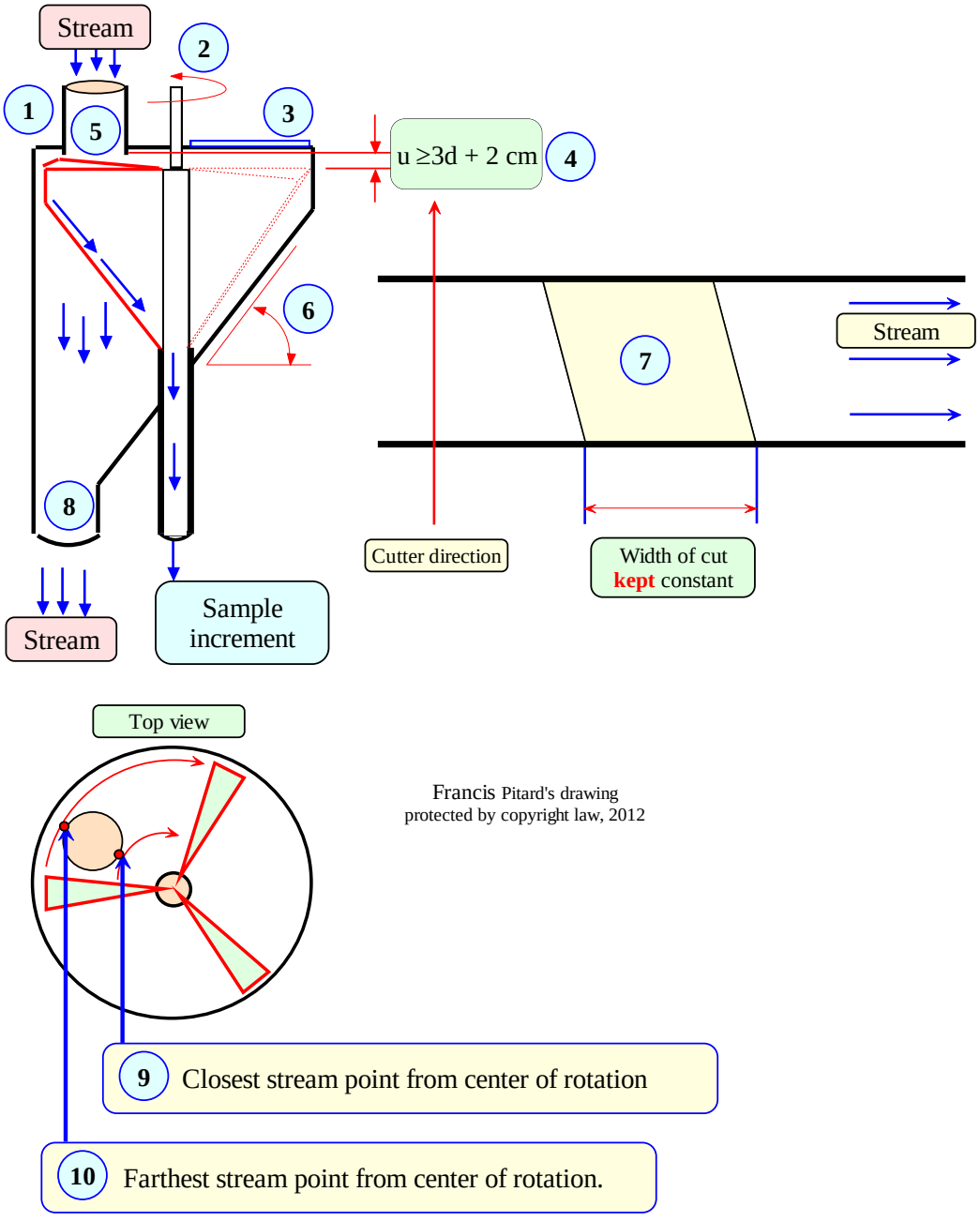


Figure 20.19: Illustration of a typical, correct Vezin sampler

For points 11-15 see **Figure 20.20**:

Point 11: The stream must be centered in such a way that a minimum 5-cm safety margin can be provided on the inner and outer sides of the stream for the minimum length of the cutters. There is little compromise possible with this requirement.

Point 12: A better view of the argument described in point 10.

Point 13: A better view of the argument described in point 9. Also, 3 radial cutters are recommended, so the Vezin sampler can take at least 5 to 7 increments from each primary increment.

Point 14: An automated water spray should be installed to keep the cutters clean after each primary cut has been sampled. The material washed by the sprayer inside the cutters must be sent to the final sample.

Point 15: A large inspection door, easy to open and close, should be provided for effective, daily inspections. It should cover a minimum of 30% of the entire surface of the top of the Vezin. Behind the inspection door, a 1-inch mesh screen should be installed for safety reasons. Furthermore, a switch should be installed so that when the inspection door is opened, the cutters stop rotating: This is a mean machine for the hands of a careless operator.

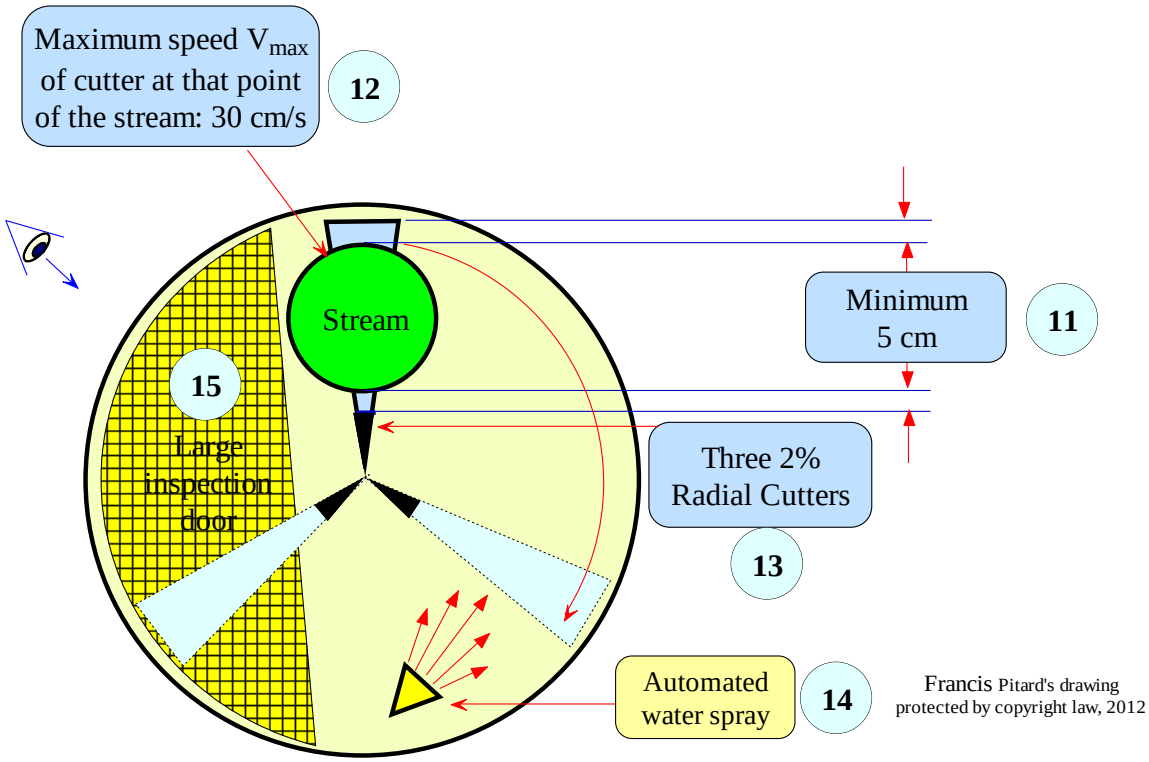


Figure 20.20: Top view of the recommended Vezin sampler

For points 16-18 see **Figure 20.21**:

Point 16: The cutter should be made of a single metal piece that could be inserted on the top of the cutter chamber. The cutter must comply with the rules illustrated in **Figure 20.17**.

Point 17: Slopes on the sides of the cutter must be no more than 20° relative to the vertical axis (i.e., 70° relative to the horizontal plane).

Point 18: The top of the cutter edges can have a 1-mm flat surface for longevity

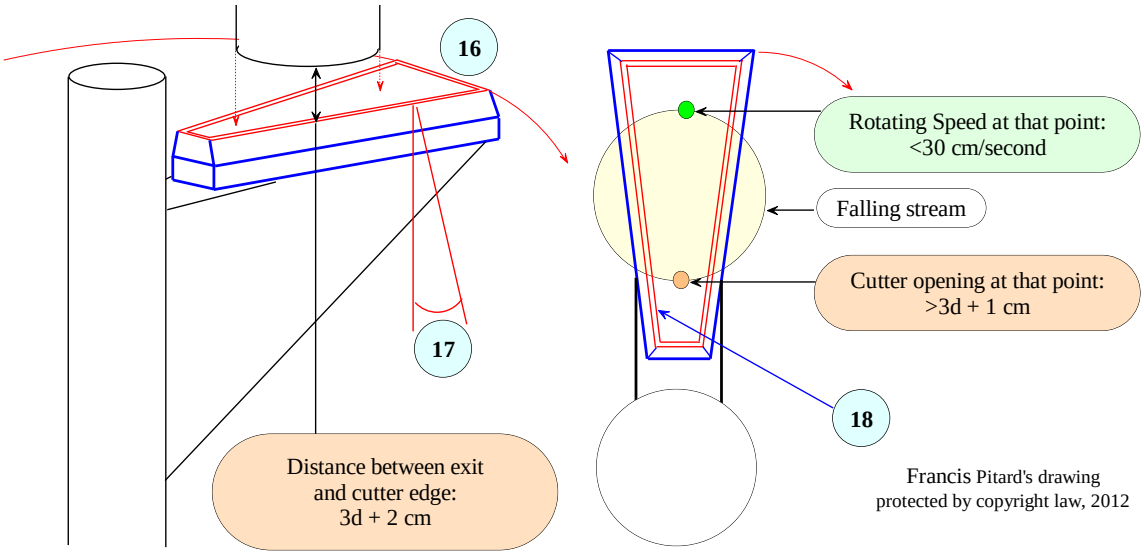


Figure 20.21: Illustration of one of the best Vezin cutters in the world:

22.1 OPTIONAL SAMPLE FILTRATION SYSTEM

Instead of waiting until the end of an 8-hour shift to press filter samples, which is a long operation to perform, you may explore the possibility of vacuum filtering samples continuously all along the 8-hour shift. Suggest a setup like the one described in **Figure 20.22**. You cannot find such systems easily on the market. For each sampling station, two filtering units are necessary. At the end of one 8-hour period, you connect the sample stream to the second unit, while the first unit is still filtering the solution from the last cuts. Perhaps an automated press filter system could also be engineered.

The entry to the filtering system should under no circumstances be farther than 1 meter from the exit of the Vezin sampler to prevent too much material to remain on the walls, along the chute. If no filtering system is used, the sample bucket should not be farther than 1 meter below the exit of the Vezin sampler.

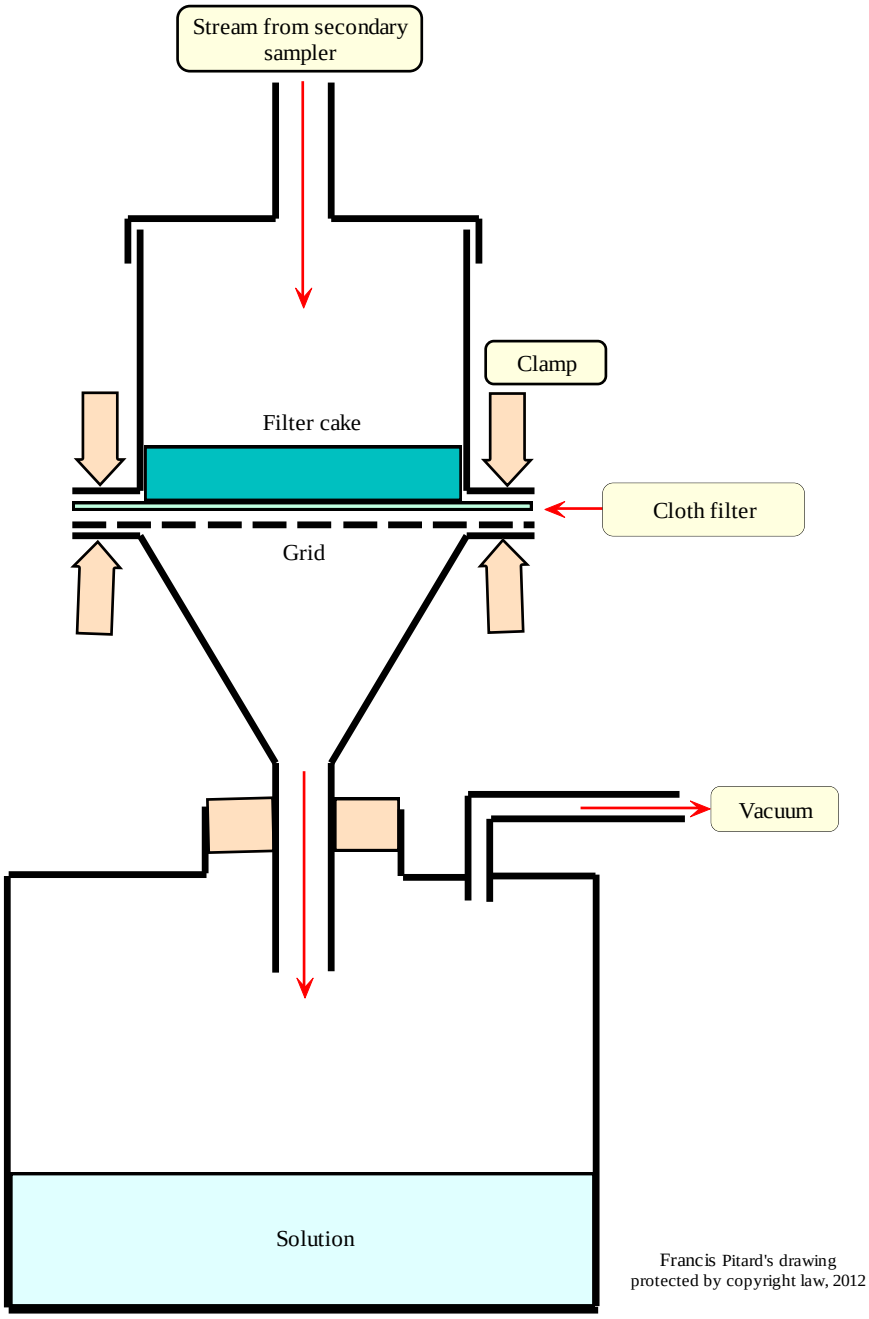


Figure 20.22: Suggested automated filtering system to be installed at the sampling stations

22.2 Final volume for the Cyclone Overflow sample

Assuming a composite sample is collected every 8 hours, and one increment is collected every 15 minutes across the primary stream, providing 32 cuts per shift which are subsampled with a continuous 6% Vezin sampler, the final volume of the sample is as follows:

$$V_S = \frac{1700000x2x32x6}{40x3600x100} = 45{,}33liters$$ for a maximum flow rate

These volumes are about what they should be for eg. gold, and yes it is a lot of work to dry them, crush the lumps, split then, and pulverize them. If these volumes are judged excessive, then you could install a Vezin sampler with only two radial 2% cutters instead of three.

22.3 Available vertical space

In the eventuality the necessary vertical space is not available, the entire location where sampling stations will be installed must be redesigned with the engineering firm; this a Cardinal rule that must never be broken:

In a new project, never do the engineering first, then ask for sampling stations; ask for sampling stations, then and only then, proceed for the necessary engineering.

Breaking this rule is evidence of no compliance with the basic requirements of due diligence.

22.4 Necessary experiments to optimize a sampling station after its commission.

After the correct sampling station is installed and working properly, a variographic experiment is necessary to select the appropriate sampling mode and sampling interval. A 15-minute interval and a systematic sampling interval are not necessarily the right thing to do. The following experiment should be performed by bypassing the secondary Vezin sampler. In other words, use the primary sampler only, collect the total volume of each increment at the reject exit of the Vezin sampler that is stopped, and make sure no cutter is aligned with the stream.

- Take 30 samples at 30-second intervals using the primary sampler. Obviously, you must be well prepared with 30 5-gallon buckets ready and tagged properly, 1-30. The chronology in which samples are taken is critically important.

- Continue to take 60 samples across the stream at 4-minute intervals, using the primary sampler. For this part of the experiment, you need another 60 5-gallon buckets numbered from 31 to 90.
- Filter, dry, break lumps, split 1000 grams, pulverize, and assay each sample.
- Send results to F. Pitard (or others) for statistical interpretation, and optimization of the sampling interval and the selection of the appropriate sampling mode, either random systematic, or stratified random.

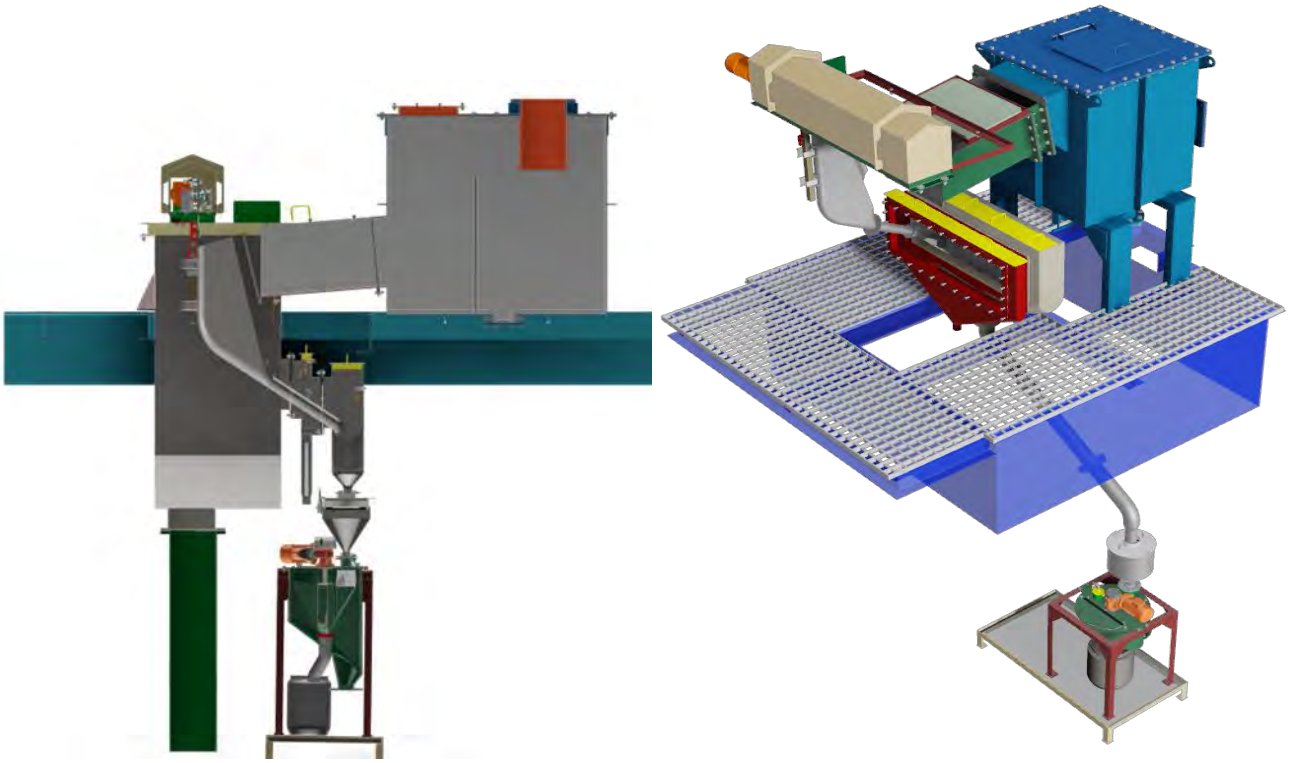


Figure 20.23: Combination Launder and Vezin sampling system

22.5 Check list for the operator responsible for the sampling station

First and above all: under no circumstances should people in charge of sampling stations be production people. Sampling stations belong to the Quality Assurance/Quality Control department. The maintenance, cleaning, operation of sampling stations belong the QA/QC department. Of course, maintenance of sampling equipment must be coordinated with the main maintenance department, however a maintenance contract with the manufacturer is a much better idea. Sampling stations and sampling equipment must be painted in a different color, so only people with proper sampling training are allowed to maintain and operate sampling stations. If this policy is unacceptable, then you may as well forget spending a lot of money to build correct sampling stations.

Every working shift, the operator in charge of collecting the 8-hour sample must take 10 minutes to review and clean the sampling station as follows:

- Connect the stream going to the vacuum filter or filter press to the second unit to start a new sample.
- Collect the filter cake into an appropriate, labeled container.
- Inspect the primary cutter, and water hose the blades if material accumulated on them.
- Inspect the launder, and water hose the channel if material accumulated on it.
- Inspect the Vezin cutters, and water hose the cutters if material accumulated on them.

- Inspect cutter blades condition: You should expect to replace them at least once a year, therefore always put them in your budget.

- Report all anomalies to the supervisor, using the checklists shown in figures 2 through 7, for items subject to change with excessive wear, deformation, or corrosion.

22.6 Guidelines for selecting a cutter width

The graph illustrated in **Figure 20.24** is solely based on F. Pitard's experience. There is very little information on this subject in the literature. If the stream to be sampled travels at a velocity superior to 2 m/s, the following graphic quickly becomes invalid.

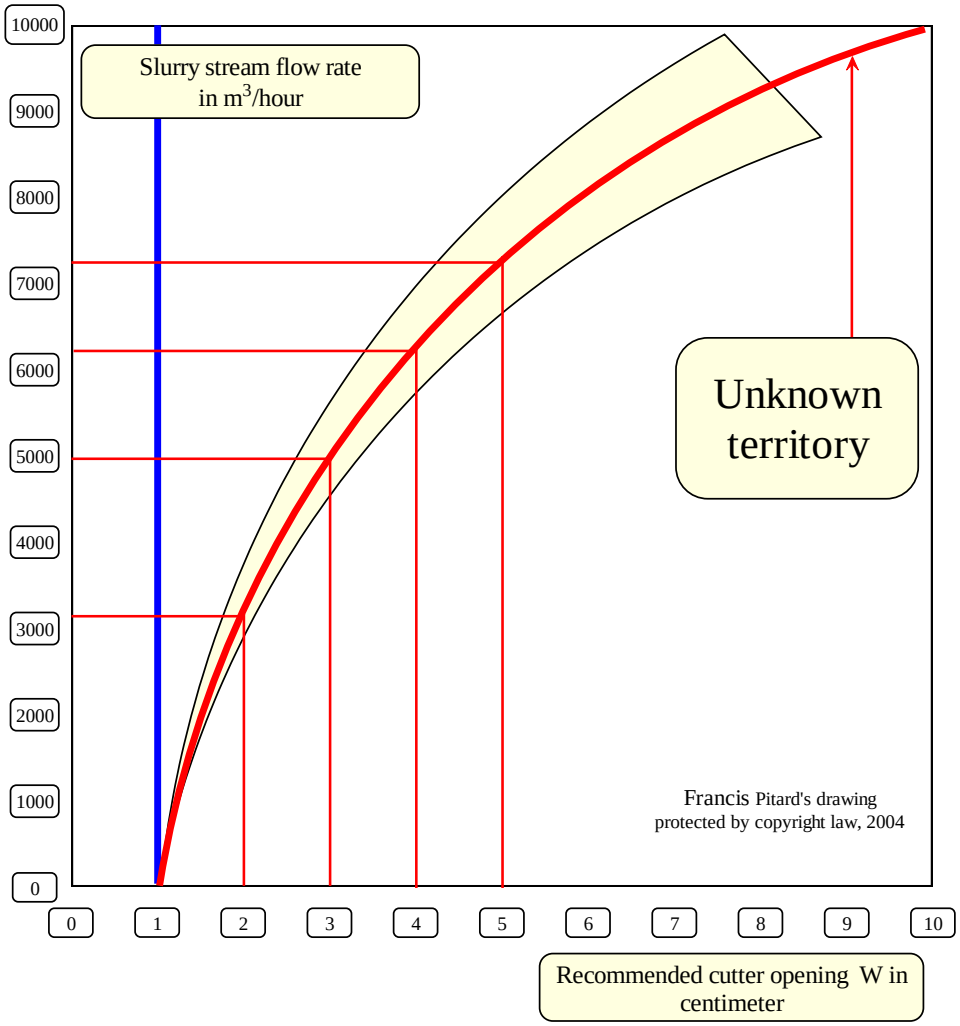


Figure 20.24. Recommended graph for selecting a cutter opening for slurries, based on F. Pitard's experience for streams traveling at a speed less than 2 m/s.

For flow rates above 5000 m^3/hour, many factors may play an important role, such as the speed, shape, and angle of the stream. For these special cases, it is important to rely on the sampling expert's experience.

For solids, the following rule applies up to 1000 tons/hour, with W being the actual cutter opening, and W_{min} being the minimum acceptable cutter opening:

$$W \geq W_{\min} = 3d + 1cm$$

Contrary to what many people may believe, W_{min} is not necessarily the recommended cutter opening. It should instead be understood as the limit beyond which big trouble may begin. Therefore, it would be wise to remain above that limit.

Above 1000 tons/hour, many factors may play a very important role such as flow rates, humidity, density, etc… For these special cases, it is important to rely on the sampling expert's experience.

22.7 Guidelines for selecting a cutter speed

The following rules are based on F. Pitard's experience, and they are more conservative than P. Gy's recommendations.

Straight-path cross-stream samplers

The velocity V of the cutter should not be superior to a maximum V_{max} recommended in the old ASTM guidelines:

$$V \leq V_{\max} = 45cm/s$$

Rotating samplers

$V \leq V_{\max} = 40cm/s$ for large diameter samplers (e.g., diameter larger than 60 cm at the farthest point of encounter with the stream, from the center of rotation)

$V \leq V_{\max} = 30cm/s$ for small diameter samplers (e.g., diameter less than 60 cm at the farthest point of encounter with the stream, from the center of rotation)

23 TWO-IN-ONE ARCUAL SLURRY SAMPLERS

These popular units for two stage slurry sampling as an alternative to the primary linear cross cut sampler are equally reliable provided they meet the design principles as laid out to date for slurry sampling and the cutter designs, in particular, adhere to the needed requirements as specified above. TIO samplers save costs on slurry boxes with overflow weirs, etc

Figure 20.25 shows cutter opening and cutter speed requirements for the primary half-arc rotating sampler. There is little compromise possible with these requirements if you want to prevent several sampling biases from taking place (i.e., delimitation, extraction and weighting biases).

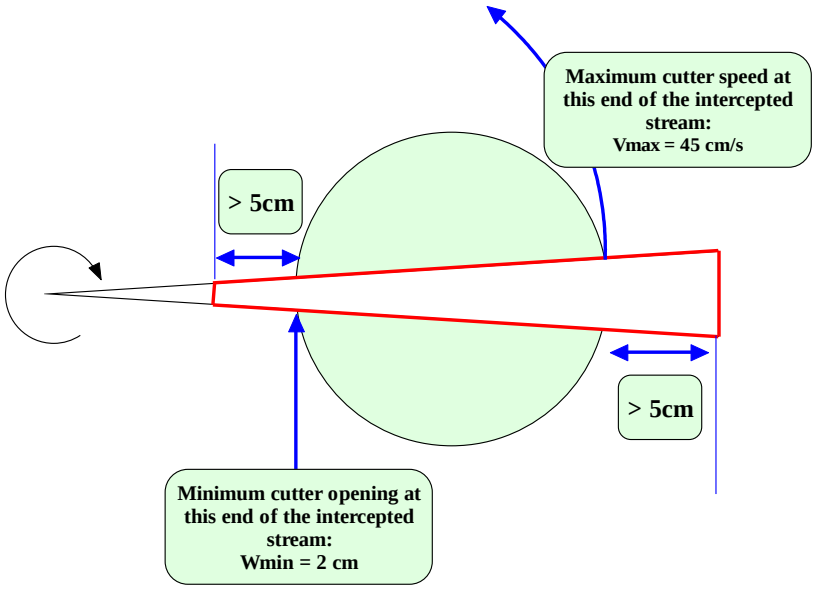


Figure 20.25: requirements for flow rates between 500 and 1500 m^3/hour for main process stream

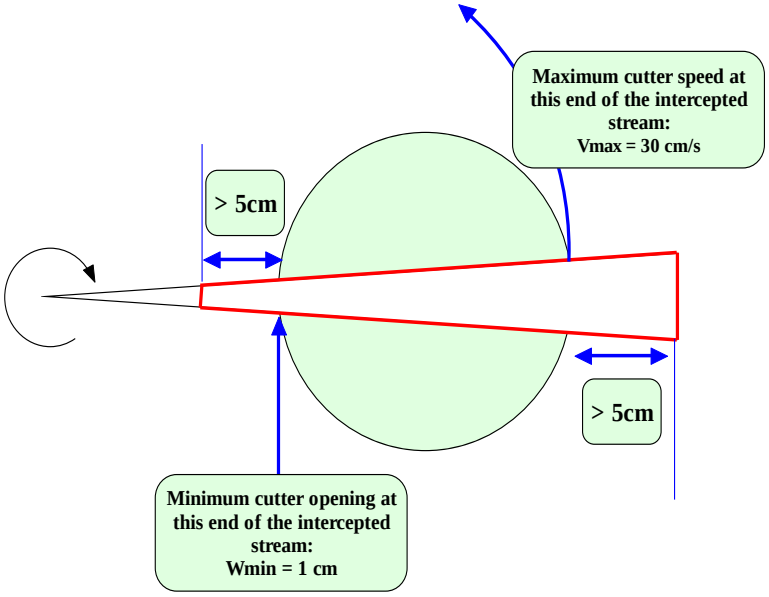


Figure 20.26: requirements for flow rates between 50 and 500 m^3/hour for main process stream

The discharge flange diameter is typically two pipe sizes (i.e. 100mm) larger than the inlet flange to prevent flooding of unit at excessive flow rates. The unit can be entirely supported by its inlet flange to vertical pipework in an in-line or tank mount position and / or by additional brackets on side of housing for larger units. Generous inspection and access covers are available and necessary air intake slots on top of housing.

The primary section of the 2-in-1 sampler is constructed of mild steel, with all wetted surfaces being rubber lined. The unit makes use of reliable dual proximity sensors to control the cutter travel between the two 'parked' positions on either side of the sampler inlet flange.

The secondary section of this unit is an integrated vezin sampler, which has multiple (4) cutters of pre-determined widths. This makes the 2-in-1 sampler extremely versatile, enabling an ergonomically sized final sample. The secondary Vezin sampler inlet has a conical polyurethane insert to provide a primary sample retaining feature. This ensures multiple cuts of each primary increment can be taken via the 20 or 30mm ∅ outlet aperture feeding the Vezin sampler.

Both the primary and secondary cutter hoods are radially tapered and fabricated from 304/316 stainless steel, which ensures sharp cutting edges and long life. Optional features for non-standard units are spray / flush water systems, replicate /dual final samples and a surge funnel with aeration and trash screen to feed the secondary Vezin sample. See Figure 20.

If there is no holding tank/launder between the primary sampler and the secondary sampler; as a result each primary increment is fed to the secondary rather quickly. However, in order to obtain a representative sample the secondary must take at least 7 cuts from each primary cut; therefore it is an absolute must to use the vezin sampler with four cutters and a conical retaining feature in inlet.

You must use the 4 cutters for the secondary sampler, and there is no compromise possible with this.

If the sample is too large at the end of the 12-hour shift, then collect samples every 6 hours, say.

Conclusion: If sampling stations are far from being correct and they are not properly used, it is very likely that most reconciliation problems would be minimized after addressing the aforementioned.

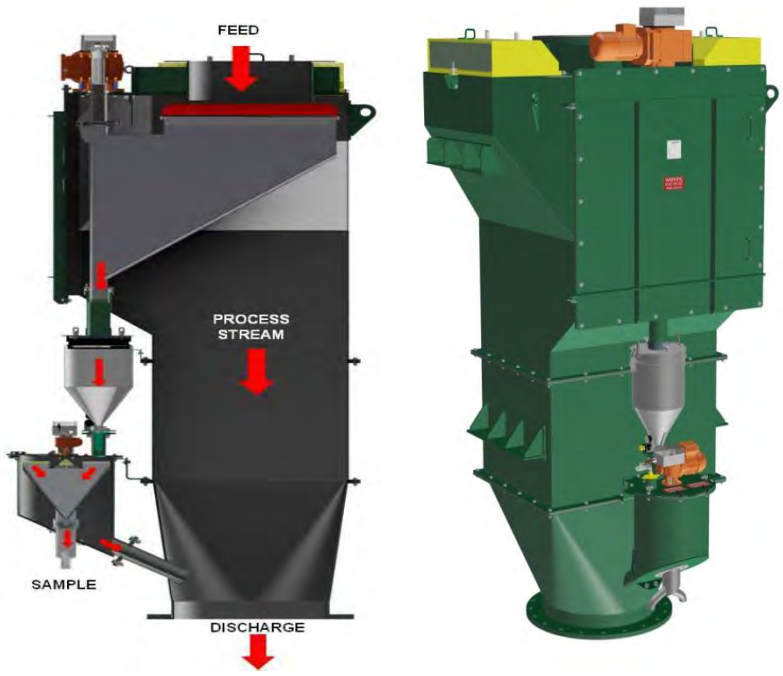


Figure 20.27: Two-In-One slurry Sampler a) cross sectional view and b) isometric view (Multotec SA)

24 BASIC DATA CONCERNING THE INSTALLATION OF AN AUTOMATED SAMPLING STATION

The following list is the required information end user (owner) should provide to any manufacturer of sampling equipment or engineering firm who would be contracted to design and build a new sampling station. The indicated list does not necessarily apply to your product. However, all the necessary information can be gathered to make a relevant list for the manufacturer.

24.1 The product to be sampled

- slurry
- solid
- liquid
- gas
- product temperature

24.2 If the product is a slurry:

- % solids by weight
- give physical characteristics concerning the solid and liquid phases.

24.3 If the product is a solid:

- Nature of this solid:
    - unsalted ore
    - concentrate obtained by…(e.g., floatation)
    - residue obtained by…
    - chemical product
    - metallurgical product
- Approximate mineralogical or chemical composition
- Size distribution analysis, or at least, dimension d of largest lumps, d being defined as the opening of a screen retaining no more than 5% of the material by weight.
- Dimension of largest, accidental lumps.
- Form of these lumps
- Is the solid adhesive? Indicate minimum slope of chutes with respect to horizontal.
- Does the solid flow easily?
- Is the solid fluidized?

- Is the solid dusty?
- True density
- Expanded density
- Mean humidity of product
- Maximum humidity of product
- Minimum humidity of product
- Is there a size class of fines with greater humidity?
    - Which class?
    - Maximum humidity of this class

24.4 If the product is a liquid:

- Approximate chemical composition or nature
- Density

24.5 Purpose of Sampling

- Measurement of one or more content values (Indicate element to be analyzed.)
- Measurement of humidity
- Determination of size distribution (Indicate size classes.)
- Other measurements (Give details.)
- Indicate final particle size of the desired sample.
- Is the sample for process control, material balance, both process control and material balance, or for commercial purpose?

24.6 Throughput

- Hourly throughput of material to be sampled
    - By weight
    - By volume
- Operation of the plant (1, 2, or 3 8-hour shifts per 24 hours)
- Is the throughput constant?
    - If the throughput is not constant, indicate minimum and maximum values, and corresponding periods of time.
- Is throughput continuous?

    - If the throughput is not continuous, indicate periods with or without throughput of the product.
- For how many hours of operation is the sample to be taken, or for what quantity
    - By weight
    - By volume
- Weight or volume of the desired sample (In case of a slurry, indicate weight of solids.)

24.7 POSITION OF SAMPLER

The sample is taken at discharge from:

- A chute
    - Width
    - Slope, with respect to the horizontal
- A conveyor belt
    - Width
    - Speed
    - Head drum diameter
- A bucket elevator
    - Bucket width
- A pipe
    - Diameter
    - Slope, with respect to the horizontal
    - Is it the delivery of a pump?
    - Of a gravity feed?
- Air-slide or any other air-driven system
- Other cases (Give details.)
- Location of sampling point
    - In closed building
    - In building with no side paneling
    - Not under cover

24.8 Special Construction of Sampler

- Must the unit be dust-proof? (e.g., heavily dust-laden atmosphere)

- Yes
- No

- Is corrosion possible on the unit? (e.g., atmosphere containing corrosive vapor or smoke. In the affirmative case indicate nature of vapor or smoke.)
  - Yes
  - No
  - Nature of recommended protection
- Is corrosion possible on that part of the sampler in contact with the material to be sampled?
  - Yes
  - Which part?
  - No
  - Nature of the material recommended for construction of parts in contact with the material to be sampled

24.9 Electric power supply available

- Power
  - Nature (DC, 3-phase, 2phase, etc…)
  - DC voltage
  - AC inter-phase voltage
  - Neutral conductor included, and if so, what is phase/neutral voltage?
  - Number of periods
- Relaying
  - Nature (DC, 1-phase,…)
  - DC voltage
  - AC voltage
  - Number of periods
- Signaling
  - Nature (DC, 1-phase,…)
  - DC voltage
  - AC voltage
  - Number of periods

- Is tropicalization of electrical equipment required?

- Timer

  - Is the option of taking increments at a stratified random sampling interval necessary? If so, indicate possible ranges of selected stratum. Indicate also if a certain amount of time between strata, during which no increment should be collected, preventing overflowing the sampling station, is necessary.

24.10 LOCATION

- Country
- Region
- Altitude
- Nearest town
- Distance
- Orientation
- General climatic conditions (temperature, rainfall, snowfall, wind, humidity, near seashore, etc…)
- Enclose a drawing of that part of the installation where the sampler is to be installed. Give details about lateral and vertical space available.
- Special local conditions (Give details).

25 MANUFACTURERS OF SAMPLING EQUIPMENT

Some manufacturers, such as Multotec, Ludowici, Techpromin and there are others, know Pitard well, and should be willing to make proposals complying with the enclosed guidelines. You can be assured, that not many manufacturers will be receptive to these conditions.

You must insist on sampling systems complying with the enclosed requirements unconditionally. Systems “off the shelves” are not acceptable.

Francis Pitard Sampling Consultants, L.L.C. has used its best efforts to perform consultation and recommendations with that standard of care, skill, and diligence using sound and professional principles and practices in accordance with normally accepted industry standards. You are at liberty to accept or reject all or any part of the recommendations without liability to Francis Pitard Sampling Consultants, L.L.C.

INCREMENT PREPARATION ERROR (IPE)

25.1 Characteristics of IPE

All non-selective operations performed on a lot, selected increments or selected samples, between sampling events, are referred to as preparation stages. These operations include:

- Transfer of the increment from a sampling to a conveying system
- Transfer from a conveying system to the next sampling system
- Comminution stages
- Wet or dry screening operations
- Drying of solids
- Filtration of pulps
- Homogenization stages, etc.

In any preparation stage the weight of the material should not change and the integrity of the increment should be preserved. Contamination of or changes in weights between preparation stages not directly linked to the purposes of sampling introduces IPE. IPE's are inherent in the design, operation and maintenance of sampling systems and are the result of:

- Ignorance
- Carelessness
- Clumsiness
- Unintentional errors
- Fraud and sabotage

IPE's can be responsible for very large sampling biases and the prevention of such errors requires awareness and careful attention. IPE's belong to several categories:

- Contamination errors
- Loss errors
- Errors generated by a change in chemical composition
- Errors generated by a change in physical composition
- Errors generated through poor training
- Errors resulting from fraud and sabotage

Each of these sources of errors is discussed in the following text.

25.2 IPE's resulting from Contamination

It is virtually impossible to extract, crush, grind, pulverize or screen materials without introducing some contamination either from the equipment used or from other equipment in the vicinity of operations. The best preventive action against IPE's is to ensure that potential contaminants are excluded or, at the very least, present in such small quantities as to be considered negligible. Which potential contaminants can be tolerated and which cannot depends on the objectives of the sampling event.

It is not enough to simply list those general conditions necessary for the minimisation of contamination. Primarily an operator must be aware of the purpose of sampling to prevent him/her from using equipment incompatible with the sample. Sampling with purpose will heighten the awareness of potential contamination problems.

25.2.1 Contamination by dust

Sampling of materials containing large amounts of fine and dry particles generates complex contamination problems which are often impossible to solve satisfactorily. However, some steps can be taken to reduce contamination by dust such as:

- Reducing, to a minimum, the free-fall of the material being sampled by minimising the lengths of chutes from conveyor belts, using vibratory feeders for small lots and by feeding riffle splitters slowly.
- Enclosing all sources of dust inside hoods with a slightly negative air pressure.
- Reducing ventilation - too much ventilation is always detrimental.
- Protecting cutters in idle positions with covers to prevent dust collection.

A popular, but somewhat useless, construction in plants or laboratories is the sampling tower. The material falling through this mechanism is gravity-fed and usually accelerates as it falls. A good sampling station should be compact and allow all components to be as near to each other as is reasonably possible. Contamination of the sample by material and dust present in the sampling circuit can occur in two different situations.

i. The same materials are submitted to a sampling circuit at regular intervals in which case it is important to ensure that the content of the constituent of interest does not change much between each sampling operation. Quick cleaning between each sampling event is necessary. In geochemical exploration, or for blast hole sub-sampling, it is advisable to keep an identical collection sequence of samples through all subsequent operations. In this particular case it is important that the samples should not be randomised to ensure that cross-contamination is prevented.

Keep in mind that there is a close correlation between the increase in contamination and the decrease of the cost of sample preparation, so selecting the cheapest laboratory may not be the most economic choice. The role of competition in the market could mean the possible exclusion of better, but more expensive laboratories from the market, which in turn leads to poor provision of services to the industry.

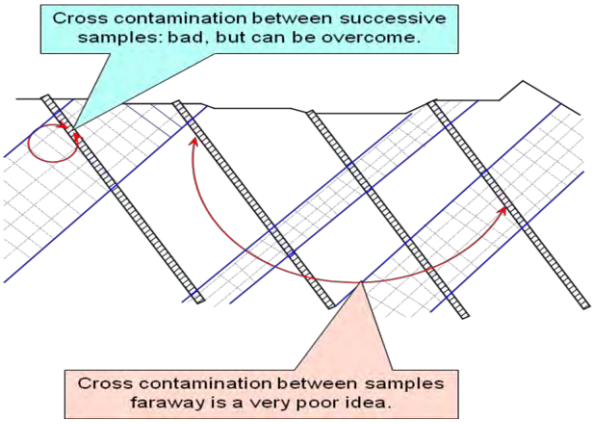


Figure 14.1: Cross-contamination between successive samples along borehole core can be detrimental but is preferable to the cross-contamination that can occur between samples that are very far apart or from a different borehole. The action of randomising samples is not a good idea here ([40]Pitard, F, 2009.)

ii. Different materials are submitted to the sampling circuit at regular intervals. In order to prevent cross-contamination it is essential that feeds, concentrates and tailings be processed with different equipment installed in different rooms. This may be considered an unaffordable luxury and, if not possible, the sampling circuit must be thoroughly cleaned between each operation. In this case the circuit must be fed with a pre-sample of material similar in composition to the next real material to be sampled and prepared. All materials collected in the course of preventative cleaning must be rejected. There is also less risk of contamination by pulverizing tailings, feeds and concentrates.

25.2.2 Contamination by abrasion

Crushing, grinding, pulverizing, screening and, to a lesser, extent all handling operations performed on abrasive sample materials are likely to introduce contamination through the small amounts of material abraded from the equipment during its use. There are numerous examples of minor contaminants that can be introduced and thus ruin a sample:

- Iron contamination of raw materials used for processing valuable white glass or of high purity zircon used in the manufacture of ceramics
- Iron contamination of an iron ore shipment sample.
- Copper, zinc, nickel or iron abraded from sieves or iron and nickel abraded from the materials used in ring and puck pulverizes.
- Zinc abraded from galvanized plates used for drying geochemical samples collected during the exploration of a zinc deposit.
- Trace amounts of chlorine in geochemical samples which have been physically handled.
- Phosphorus contamination by cleaning glass equipment with detergents.

25.2.3 Contamination by corrosion

Corrosion of sampling and preparation equipment is likely to take place with chemically aggressive materials such as:

- Wet materials developing acid reactions (i.e. ores containing sulfides)
- Pulps containing acid reagents in floatation plants.
- Slurries containing strong acids in hydrometallurgical plants.
- Processing plants involving sea water or other alkaline solutions.
- Rust due to moist air.

In most cases problems can be alleviated by using sampling equipment made from high quality stainless steel.

25.3 IPE's resulting from Loss of Material

25.3.1 Loss of fines as dust

Loss of fines as dust occurs as soon as dry materials are transported, transferred or sent into free-fall towards the cutter of a sampler. Sustained free-fall of material before it reaches the next sampling stage must be avoided since this may introduce IPE (i.e. IPE = non-selective process), into the process. Another source of loss is during the use of crushing, grinding or pulverizing equipment. Losses are amplified when these pieces of equipment are enclosed inside dedusting systems working under a strong vacuum. Dedusting systems should use minimum vacuum with a small airflow and be carefully controlled by an operator. Hammer mills rotating at high speed and generating strong turbulence are not recommended. For pulverizing samples up to 5kg, completely enclosed systems are best, such as vibratory ball mills, ring mills, puck mills etc.

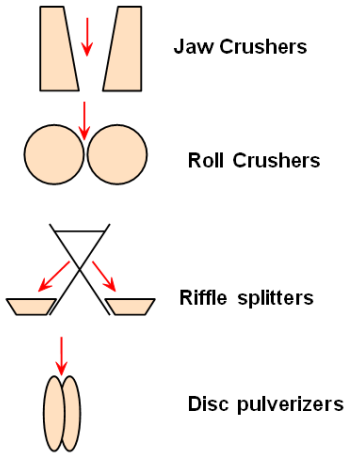


Figure 14.2: Losses generated by the use of old equipment in a laboratory ([40]Pitard, F, 2009.)

The use of outdated technology can also be problematic if dust is lost at each stage of preparation. The lack of dust control in older equipment may mean it is obsolete. ***Figures 14.2*** and ***14.3*** illustrate the types of technology that can introduce IPE's.

A more acceptable arrangement of modern equipment that can be cleaned and which is more consistent with best practice is shown in ***Figure 14.3***. This type of system can handle up to 150 samples in 24 hours. However, it is essential that if technology is to perform well there must be continuous monitoring of

equipment. It should not be left to perform without surveillance. This type of setup is cleaned by passing a pre-sample through the system before the actual sample is submitted.

25.3.2 Loss of material left in the sampling and preparation circuit

After each sampling operation all sampling and preparation equipment must be cleaned. All the material left in the circuit must be recovered and sent back to where it belongs, either to the rejects, or to the sample, depending on from whence it came.

Loss of specific fractions of a sample: This may occur because of mechanical or electrostatic properties in the equipment used. Examples include:

- Loss generated by careless screening may achieve a perfect segregation of some important constituents. Such segregation is actually used to our advantage when we perform “metallic” screened gold assays. However, the loss of one gold particle stuck in a mesh can ruin everything. Screening usually destroys the natural heterogeneity and homogeneity of material and means trouble if there is no clear objective aimed for;
- Loss generated by the smearing of the important constituent. Typically, trying to pulverize coarse gold particles generates substantial gold losses through smearing of the metal on the sides of the pulverizer;
- Loss generated by the electrostatic properties of the important constituent. Tungsten mineral “scheelite”, occurs as tiny flakes which stick electrostatically to the sides of plastic containers.

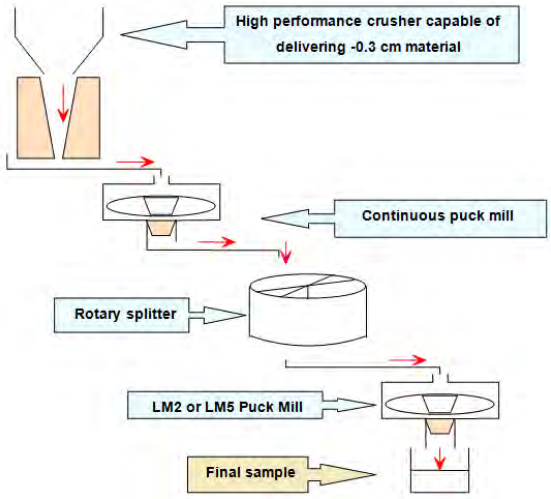


Figure 14.3: Modern equipment in a compact arrangement that allows for easy cleaning and maintenance. (*[40]*Pitard, F, 2009.) *(Source: Pitard, F, Sampling Theory and Methods Course notes 2006).*

25.4 IPE’s resulting from the Alteration of Chemical Composition

IPE’s may also arise because the chemical composition of a material is altered as a result of mechanical effects or temperature changes, (i.e. a sulfide becoming a sulfate, or a carbonate becoming an oxide). Such changes can be identified by measuring the critical content of a sample using the formula:

$$\text{Critical Content} = \frac{\text{Weight of Critical Component}}{\text{Weight of Active Components}}$$

Estimation of the critical content changes each time the critical or active components or both are changed. As with physical fragments, defects affecting the probability of selection of some constituents directly alter the sampling accuracy in a broad sense.

25.4.1 IPE's resulting from Chemical Subtraction or Addition

Examples of these processes include the elimination or fixation of constitution water, crystallization water, carbon dioxide and volatile elements or constituents, such as mercury, selenium, germanium and many organic compounds. In addition, the oxidation of sulfides may affect their sampling characteristics and composition.

25.4.2 IPE's resulting from the Alteration of Physical Composition

In the mineral industry the physical characteristics and chemical composition of a mineral is a function of its moisture content and/or its particle size distribution. Problems can arise from:

- Addition/subtraction of absorption water or occluded water.
- Creation or destruction of a given granulometric class by screening materials for different lengths of time
- Sublimation of native sulfur, etc.

25.4.3 IPE's resulting from Unintentional Mistakes

For psychological, political or economic reasons sampling within the estimation circuit is a most neglected area. This is true for most countries to the point that sampling theory is not even taught at universities or colleges. As a result the sampling operator is unaware of the most elementary principles of sampling correctness. As he blindly follows a vague recipe based on empirical observations, or on bizarre traditions, he necessarily introduces unintentional mistakes such as the dropping of samples followed by incomplete recovery, mixing of fractions belonging to different samples, mixing of labels, poor maintenance of sampling equipment and contamination and losses.

25.4.4 IPE's resulting from Fraud or Sabotage

The fact that so few people are familiar with the many subtleties of sampling theory means that fraud is perpetuated, especially during the trade of commodities. The Bre-X scandal is a case in point. Other examples include:

- The moisture determination of a copper concentrate during commercial sampling
- So-called "salting" by introducing gold particles to crushed core samples sent to a commercial laboratory

- Money-making laboratories which receive thousands of environmental samples collected at great expense, and which ignore the basic rules of correct sub-sampling
- Someone knowing that a given sampling protocol is wrong, but accepting the *status quo* in the name of a flawed standard
- Operators who don't have the time or inclination to seriously consider sub-sampling and preparation problems because the contract is always given to the cheapest laboratory. Cheap sampling can lead to financial disaster in the long run.

26 INCREMENT WEIGHTING ERRORS (IWE)

26.1 Characteristics of IWE

Readers familiar with the Theory of Sampling may notice the author chose to include the Increment Weighting Error in the family making up the Materialisation Error[61]. The reason is that IWE is the direct effect of how the sampling system collects increments. If the sampling system is not proportional, IWE is likely to be significant. While, if the sampling system is proportional, IWE may be negligible. However the flow rate fluctuations of a stream, which is part of the large-scale variability, may also greatly affect IWE; this is precisely the point, and the sampling system should be designed to take this into account during the materialisation phase. The variance of the preparation errors IPE is a separate component introduced between sampling stages.

The variance of IWE is a function of fluctuations in a stream flow-rate from which the increments making up the sample are collected. If IWE causes a bias the sample is not correct. The question here is one of equivalence between mass and time in the stream flow-rate: should the sample be collected every 500t or every 10 minutes? Some manufacturers of sampling equipment have implemented a systematic selection mode of increments based on a constant tonnage rather than on a constant time interval. This is an expensive, high maintenance solution that can be analysed by examining the mass of the increment:

$$M_i = \frac{W \times R}{V}$$

- M - is the mass of the increment
- W - is the constant width of the cross-stream cutter
- V - is the cutter velocity
- R - is the main stream flow-rate

Sampling theory requires that the mass of the increment must be proportional to the amount of material on the conveyer belt passing a given point. If sampling is to be proportional to tonnage then the cutter velocity must be proportional to the mass on the belt, which is easier said than done. A much better solution is to make the mass proportional to the flow-rate. For example if W = 10cm, R = 1000t/hr and V = 45cm/sec., the mass of the increment M_i will be:

$$M_i = \frac{10\ \text{cm} \times 1000000\ \text{kg}}{45\ \text{cm/sec} \times 3600\ \text{sec/hr}} \approx 62\ \text{kg}$$

If one selects a systematic selection of increments based on a constant tonnage, IWE cancels if and when the increment weight is also constant. Since W is constant, the increment weight is constant too, only if the cutter speed V can be set proportionally to the flow-rate R. Constant tonnage sampling systems can be classified into two categories:

26.1.1 Systems using a constant cutter speed

This system is useless as it only results in moving the problem elsewhere in the event.

26.1.2 Systems using a proportional cutter speed

In theory this is a correct system but in practice presents enormous difficulties:

- Measuring the instantaneous flow-rate at the sampling point with accuracy is nearly impossible.
- Setting the cutter speed V proportional to the flow-rate R is very difficult.
- Realising such a reliable system at an acceptable cost is nearly impossible.
- Maintaining such reliability is costly.

26.2 Minimising IWE

If money is to be spent in an effort to reduce IWE it is cheaper and far more effective to regulate the flow-rate during extraction of the sample representing the entire lot. If the flow-rate is going to change drastically, just start collecting a new sample. It is cheaper and more effective to rely on a constant time interval between increments if the following guidelines are respected:

- The variance of IWE is acceptable when the fluctuations of the flow-rate are maintained within ±10% relative at the 68% level of confidence.
- The variance of IWE is negligible when the fluctuations of the flow-rate are maintained within ±5% relative at the 68% level of confidence.

For a zero weighting error the use of a correct Vezin sampler, shown in ***Figure 15.1***, should be considered as it is perfectly proportional: the sample mass is proportional to the cutter opening and therefore perfectly proportional to the flow-rate. Many sampling systems are not proportional. It is possible to calculate a mass ratio to monitor changes in the procedures.

$$\text{Mass ratio} = \frac{M_s \text{ (mass of sample)}}{\text{Tonnage in the process}}$$

26.3 Problems Associated with Weightometers

The ***weighing error*** is introduced during the weighing procedure, either due to incorrectly transcribing figures, due to incorrect calibration, or due to a mechanical and/or electronic malfunctioning of the scale during weighing. This is not a sampling error and should not to be confused with the Increment Weighting Error IWE, which is also a problem indirectly related to sampling issues. Another method by which discrepancies in the sampling weighing systems can be monitored is through a relative difference (RD) plot of results from two weightometers that “see” the same tonnages at different places in a plant.

$$\text{Relative Difference (RD)} = \frac{W_1 - W_2}{\left(\frac{W_1 + W_2}{2}\right)}$$

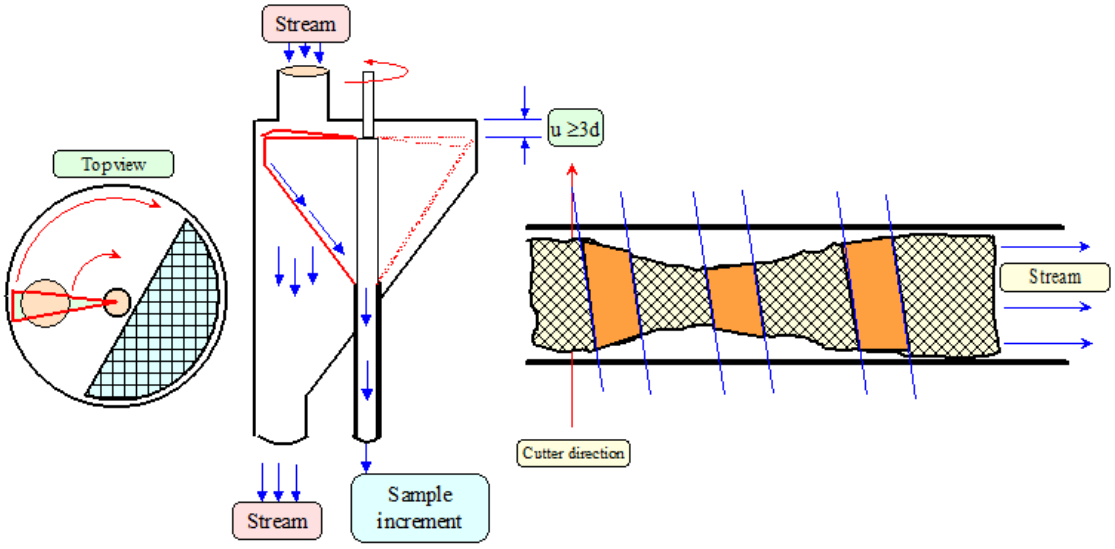


Figure 15.1: For all Vezin samplers the sample mass extracted is proportional to the flow-rate. ([40] FF Pitard)

For example, weightometers W1 and W2, shown in ***Figure 15.2,*** see the same tonnages, but at different places in the plant. The %RD for readings from both weightometers every 30 minutes for a monthly period should provide a stable graph with minimal variance around the zero position. The %RD plot should also be overlaid by a one-day or one-week moving average that will allow any biases in the system to be identified.

Another useful tool for monitoring the performance of weightometers is to sort the pair data by increasing grade and plot the %RD against the pair numbers of increasing grade rather than on a grade scale. In this way it is possible to identify any conditional biases in the system. Such biases arise as a consequence of a structural problem rather than a consequential problem. Generally, weightometer installations in plants are fraught with problems because basic issues have not been appreciated or adhered to.

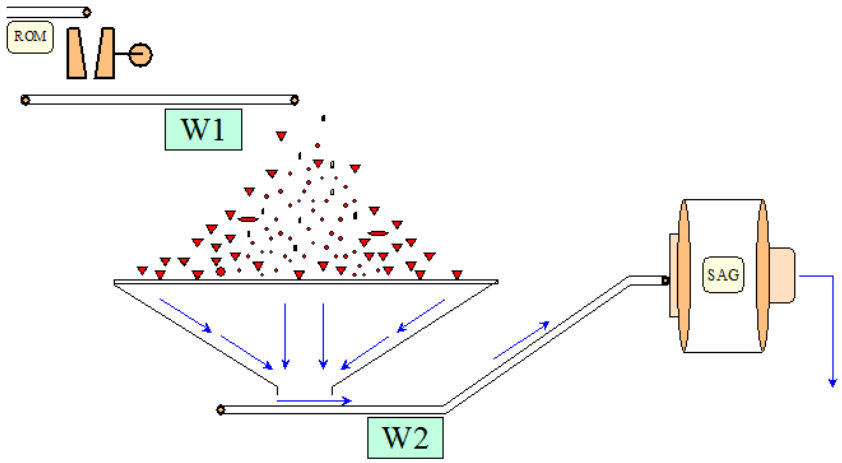


Figure 15.2: Two weightometers installed at positions W1 and W2 must "see" the same tonnage to be effective. ([40] FF Pitard)

The accurate determination of tonnages entering a plant is critically important as weightometers are notorious sources of reconciliation problems. The accuracy of many weightometers is doubtful because of the location in which they are installed and the manner in which they are cleaned, maintained and calibrated. Their accuracy depends on many factors and field experience proves the specifications to be far less optimistic than manufacturers guarantee. Nevertheless, if a few precautions are taken, weighing accurately with weightometers is possible. There are three basic rules of compliance in order to successfully implement tonnage monitoring with weightometers:

- The initial installation of the weightometer needs to be correct.
- A logical test program needs to be implemented.
- Deviations in behaviour and potential biases must be monitored on %RD plots.

A recommended setup for weightometers is shown in ***Figure 15.3.***

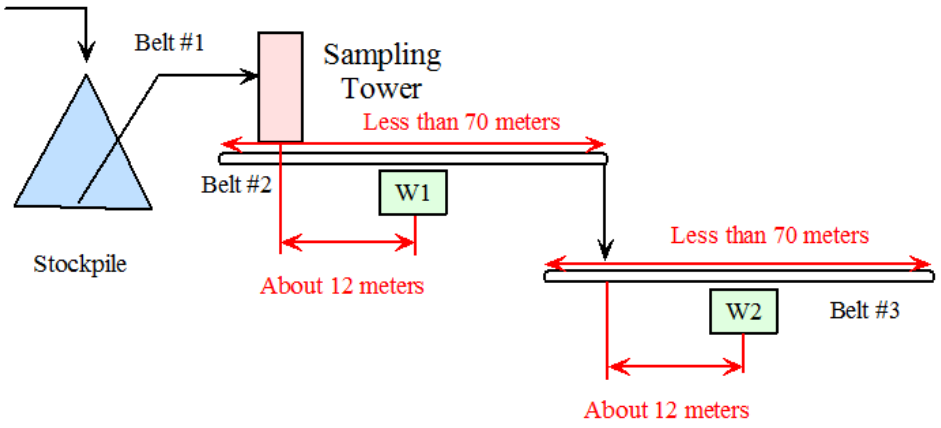


Figure 15.3: A recommended weightometer set-up. ([40] FF Pitard)

Installations of weightometers must comply with the following issues:

- Weightometers and conveyors cannot exist independently of each other. It is imperative for an engineering firm to build them as an integrated system.
- They must be installed on a stable concrete floor subject to zero movement and minimal vibration
- Weightometers must be installed where the conveyor is horizontal, plumb and level because the belt tension is much lower and more predictable in horizontal conveyors. Those installed on inclined conveyers never function correctly.
- They must be calibrated when the belt is empty. Calibration chains or weights should be selected to permit calibration at about 75% of scale capacity.
- The conveyor belt should be relatively short, no more than 70m long. Any length greater than 70m means the tension on the belt is too high for accurate, consistent weightometer measurements.
- The location at which weightometers are installed is critical as their correct functioning depends on the tension of the belt. They should be about 12m (between 7-17m) from the loading point of the

conveyor belt, the point where tension is minimised. If placed too close to the loading point the weightometer over-estimates the mass of material on the belt.

- Skirting and trailing idlers should not be located closer than 7m from the scale location.
- The portion of the conveyor belt supported by the weightometers should have under-cover protection from dust and heat. Weightometers should not be exposed to huge changes in temperature.
- Retrofitting a weightometer to a conveyor belt after it has been built is unlikely to give satisfactory results.
- For a weightometer to work well, run-out of idler rolls must be minimised.
- The angle of troughing idlers must be very accurate.
- Deflection of conveyor stringers must be minimised.
- The layout of the weighing system should be such that a quantity of the material that passed or is to be passed over the belt can be weighed, if required, in a separate weigh hopper or in a railroad truck.

There are many other rules of compliance to be observed if weightometers are to function correctly. Additional information can be found in the excellent work of Colijn[83].

[83][83] Hendrik Colijn. 1975. Weighing and Proportioning of Bulk Solids. Distributed worldwide by CH-4711 Aedermannsdorf. Switzerland. 1975.

PART 4: APPLICATIONS OF VARIOGRAPHY TO PROCESS CONTROL

27 THE MOVING AVERAGE

27.1 Correct Use of the Moving Average

A simple moving average, as opposed to a central- or weighted-moving average, is generally calculated over a set span of readings in a time series data set, say a 5-point or 10-point moving average. The span over which an average is calculated at any point, say d_1 is fixed, but it moves forward sequentially one data point at a time, including the next data point and discarding the previous one. The series of d_1 to d_n mean values is joined to give the moving average, a smoothed rendition of the otherwise highly variable data that reveals the overall trend. The wider the span, the more the data are smoothed. Wider spans include more data meaning that the relative change from one point to the next is not very large, because even if the values between the first and last data values was large the weight of the intervening values would tend to suppress the variation. The principle purpose of the moving average is to smooth out short term variability and to minimise the effect of random, irrelevant variability in a set of data i.e. to smooth out *V[0]*, the variability that does not exist in the system, or to minimise the effect of a random residual component *V4[j]* in a variogram. Applying a moving average to a time series data set allows the underlying or longer term trends, variability, or cyclicity to emerge. Moving averages allows us to see the bigger picture, the larger scale trends and variability. The more data one has the larger should be the span of the moving average, depending what aspect of the data one wishes to emphasise. The effective use of the moving average involves four graphics:

- The original data a_m (shown in ***Figure 22.1***) the integrity of the original data should not be tampered with;
- The Moving Average (MA, shown in ***Figure 22.2***); the choice of the window is at the users discretion;
- The Random Noise RN: RN = am – MA; provides some measure of validation that the window is appropriate (shown in ***Figure 22.3***), and;
- The Corrected Data (CD) relative to the Real Data Average (RDA), shown in ***Figure 22.4***:
  CD = RDA – MA or relative to a Targeted Average (TA): (RDA = Real data average) CD = TA – MA

In a diagram of the moving average, original data is paramount and should not be tampered with. The size of the moving average can be chosen but depends on the objectives one is trying to achieve which should smooth out V[0], the variability that does not really exist in the system. Therefore, the moving average minimises V[0]. The graphic of RN is really a validation of the window.

27.1.1 First Graphic: The original data $a_{m:}$

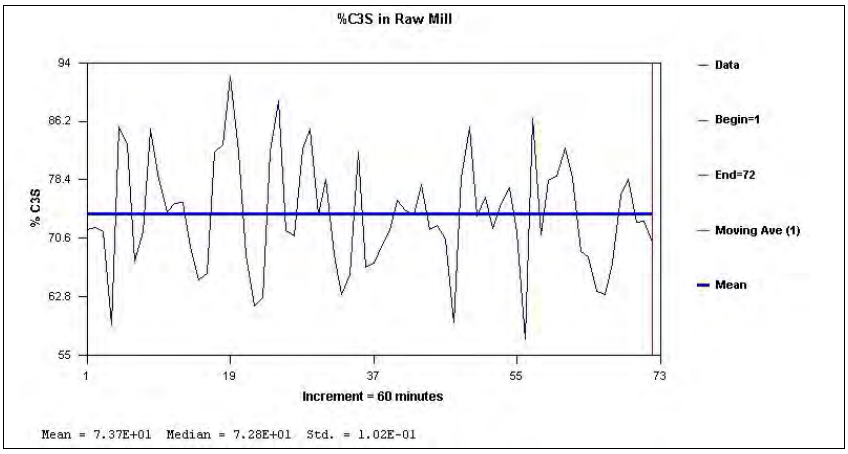


Figure 22.1: Original Data a_m with the thick horizontal line being the arithmetic average

27.1.2 Second Graphic: The moving average (MA)

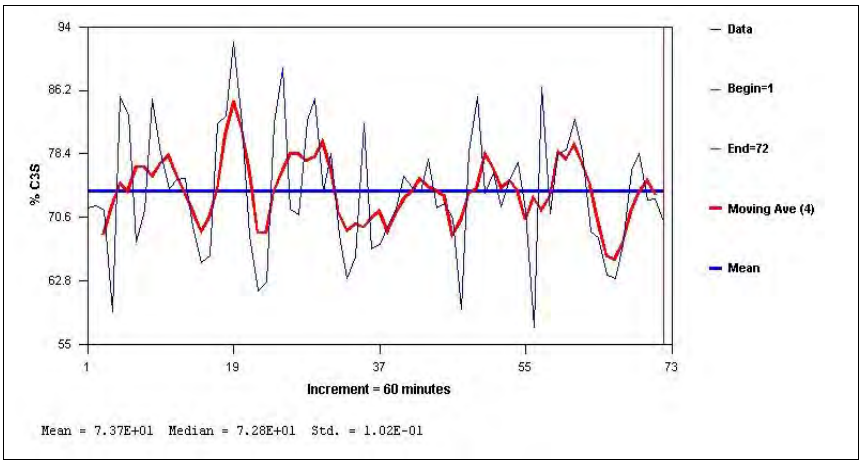


Figure 22.2: A 4 point window for the Moving Average of %Chemical Index in the raw mill. N is the number of points used in the moving average (^{40} FF Pitard)

In the variability of the original data and the moving average are shown together in the second graphic (***Figure 22.2***).

27.1.3 Third Graphic: Random noise and corrected data (very useful in process control):

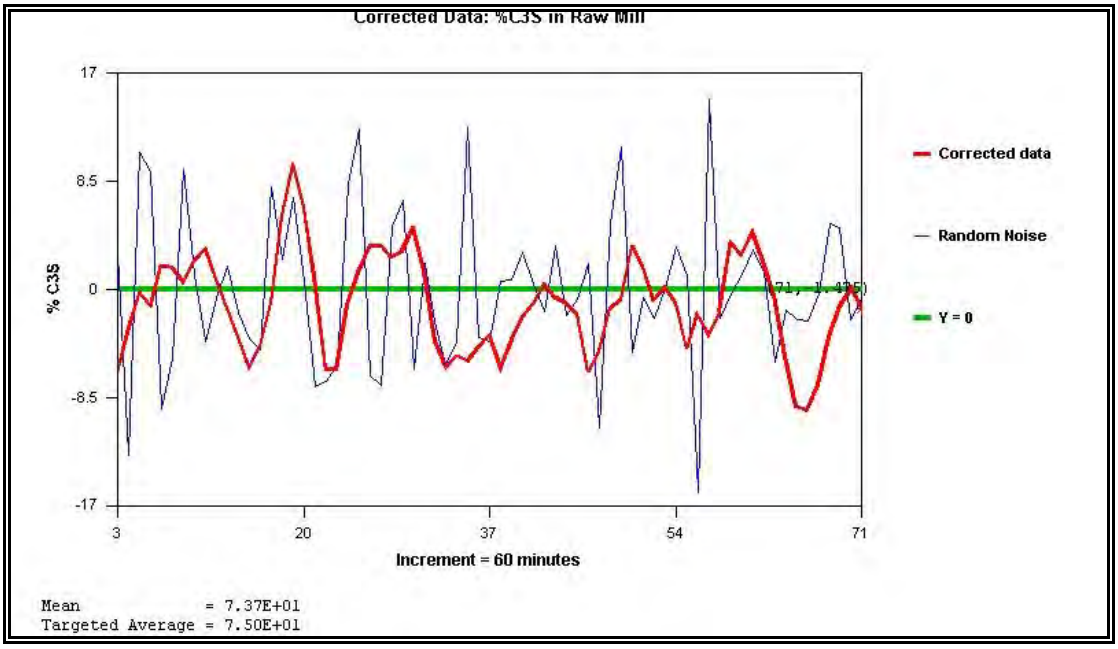


Figure 22.3: Moving average superimposed on the Random Noise ([40] FF Pitard)

A continuous curve for Random Noise at a steady level without any indication of a trend is the ideal result. If there is any indication of a cyclical trend in the RN across the zero line then there is a possibility that the window is too large and should be reduced.

27.2 Incorrect Use of the Moving Average

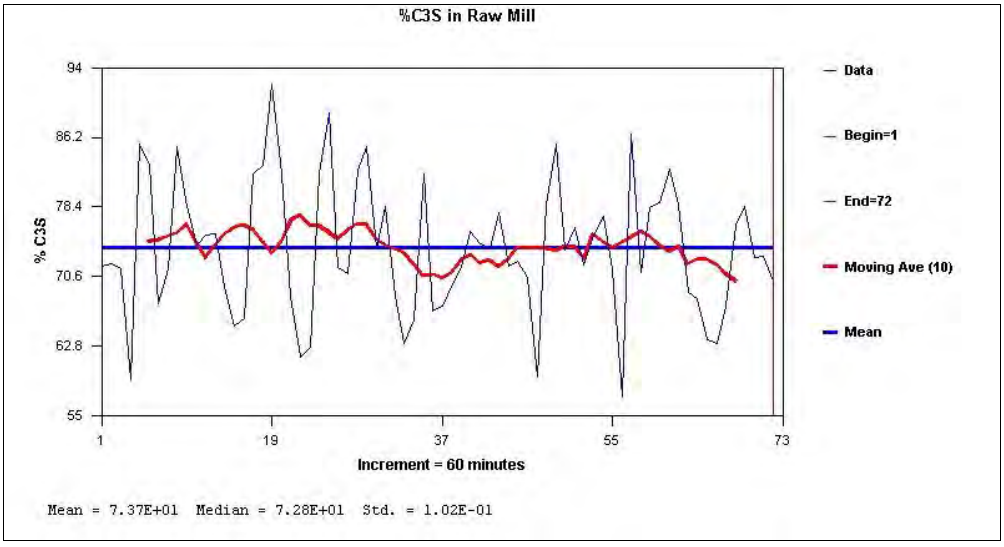


Figure 22.4: Cyclical trends that move across the zero line indicate that the window of the moving average is too wide and must be reduced. ([40] FF Pitard)

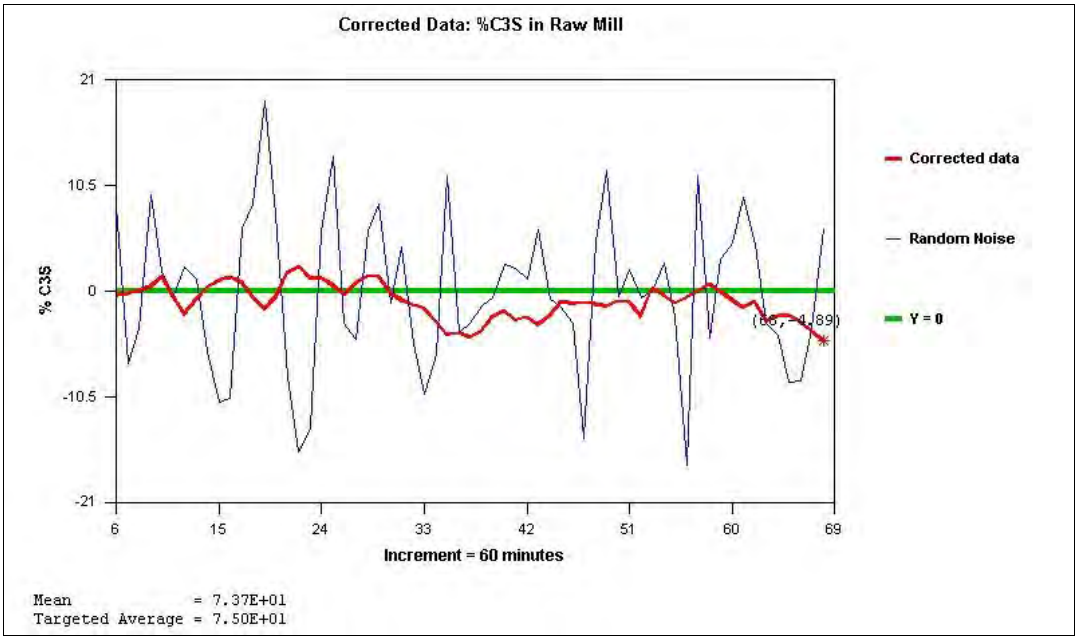


Figure 22.5: Deviation from the zero line indicates the amount of correction that must be added to the system. At zero no process correction is required. ([40] FF Pitard)

A corrected data curve is derived by subtracting the moving average curve from the raw data (RN = a_m – MA). The curve should vary around the arithmetic average in a consistent manner. If there is any indication of a cyclical trend then the moving average is too wide and one must start again.

27.3 An Application of the Moving Average: The Relative Difference Plot

The relative difference plot is a powerful yet simple graphic tool where one can utilise duplicate sets of samples. On such graphs it is easy to detect biases, discuss their significance and follow their evolution. Check assays should be taken every 20th sample resulting in sufficient data being available within a short space of time. A thousand samples will give 500 pairs, adequate for a stable analysis. The plot has the following features:

a. **Step 1:** The relative difference between two measurements being compared i.e. two laboratories or duplicate analyses, A and B, are plotted on the vertical axis.

$$\%\ Relative\ \ difference = 100 \times \frac{a-b}{(a+b)/2}$$

The average of the two values A and B, i.e. [A + B] / 2 is sorted by chronological order, or by increasing values of [A + B] / 2 and is plotted on the horizontal axis.

b. **Step 2:** The most important feature, the moving average (N = 20) is then plotted.

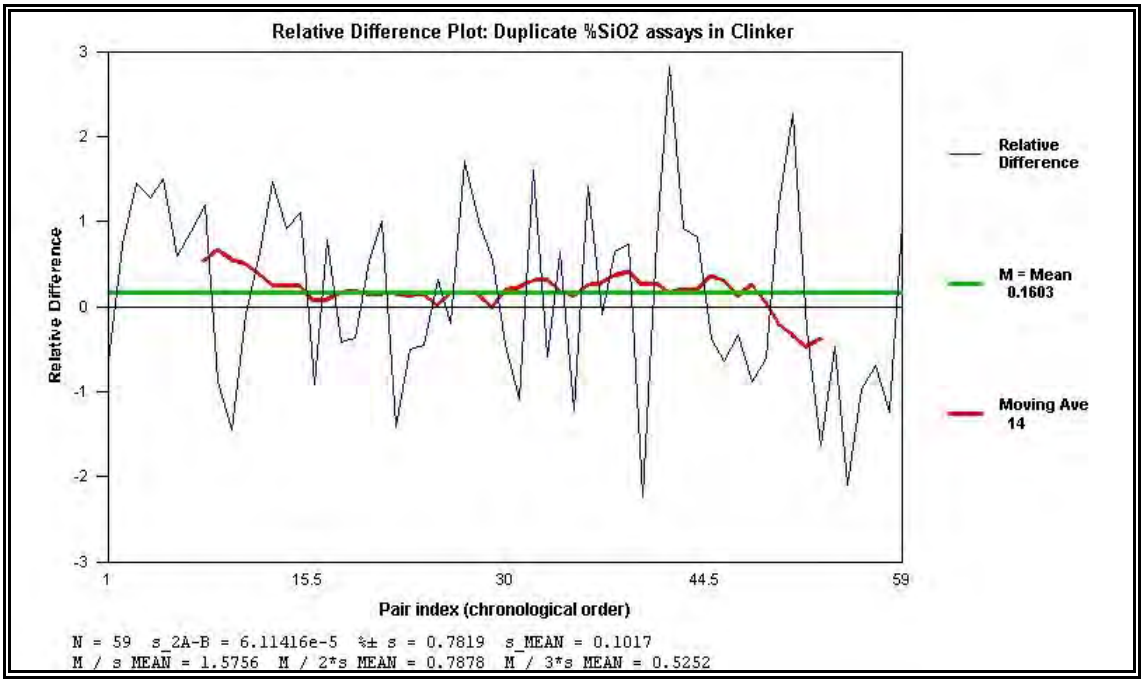


Figure 22.6: Example of a relative difference plot; a bias is clearly indicated ([40] FF Pitard)

The downturn in the tail of the curve in ***Figure 22.6*** exactly compensates the upturn at the beginning of the cycle. If the horizontal axis is sorted by increasing content, the curve behaves as shown. The mean value (shown in green in ***Figure 22.6***) lies above the zero line, providing clear evidence of a bias, particularly a conditional bias, associated with the higher grades. One might ask: "What is a good correlation coefficient? Is it 0.94, 0.97 or 0.98?" Using the formula:

$$A = \sqrt{1-(\rho)^2} = \sqrt{1-(0.97)^2} = 0.17$$

Thus about 17% of the pairs are suspicious. If 25% are suspect at ρ = 0.97 there is a major problem with the precision ellipse. For this formula to work correctly a fairly large range is required.

27.4 Application: Beware of Correcting Factors

Incorrect sampling and poor measurements may introduce biases into a data base as indicated in the Relative Difference plot shown in ***Figure 22.7***. It is tempting to adjust biased data by using correcting factors based on observations and experiments. Unfortunately, there is no such thing as a constant bias in sampling. Therefore, filtered information, elimination of embarrassing figures and the use of correcting factors are symptomatic of poor management. Correcting factors give a false sense of security and merely hide the problem if one attempts to make adjustments.

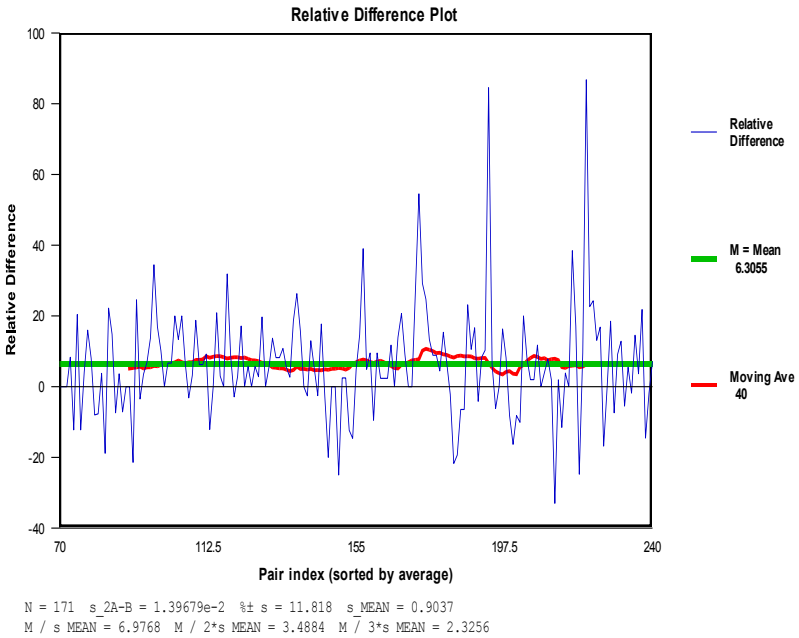


Figure 22.7: Relative difference plot showing an example of an analytical bias (^{40} FF Pitard)

If the moving average stays fairly close to the mean value without veering off in either direction, as in ***Figure 22.7,*** the bias is more or less constant and is likely to be an analytical bias. On the other hand, where the moving average takes major swings around the mean value there is almost certainly evidence of significant sampling bias or several different types of bias superimposed on one another.

Evidence of a sampling bias is shown in the Relative Difference plot shown in ***Figure 22.8***. This is clearly not the footprint of an analytical bias, but rather the indication of a sampling bias. The variability in the moving average plot is a clear indication of this.

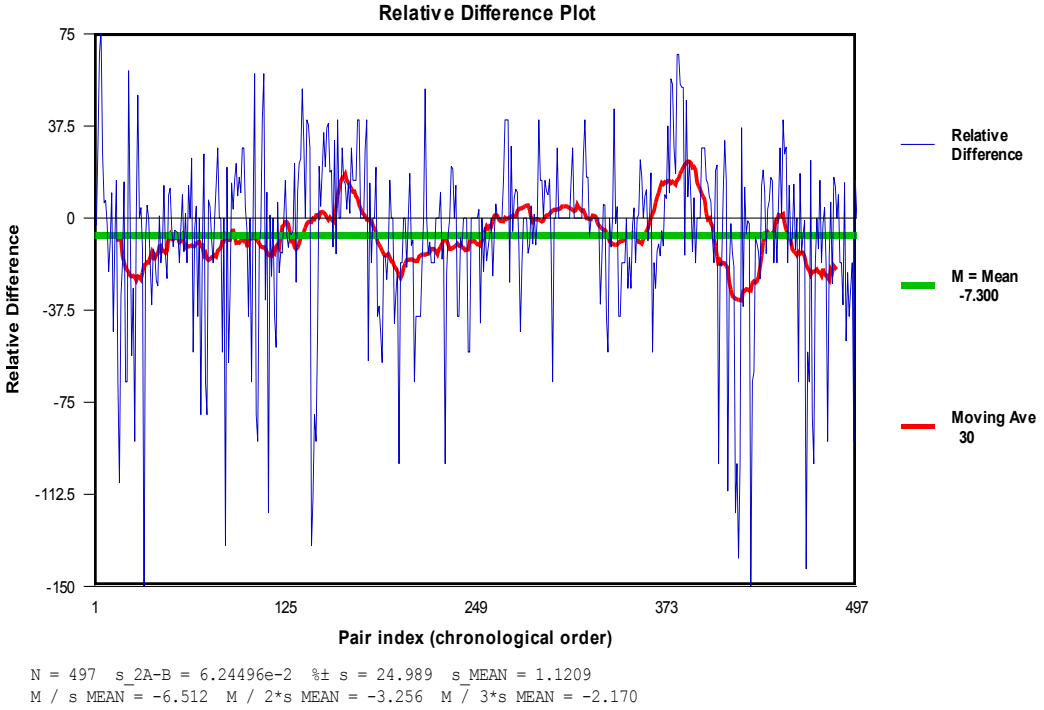


Figure 22.8: Relative difference plot showing an example of a sampling bias (^{40} FF Pitard)

During process control the system is almost always under-sampled giving rise to major fluctuations around the mean. Metal recovery and run of mine ore grades display a variability which is correlated. However, at maximum capacity the plant will have poor recovery at the spikes because it has no capacity to absorb these. In a copper plant the following data was collected:

Before adjustments to the plant		
Range, (variability)	± 50%	±150%
Recovery	82%	65%
After adjustment to the plant		
Variability (Range)	25%	75%
Recovery	85%	±80%

28 LARGE-SCALE VARIATIONS AND VARIOGRAPHY

28.1 Introduction

Although the analysis of chronological data is dealt with in depth in many useful texts chronostatistics (Pitard, 2006), is particularly useful for the analysis of time series data in metallurgical and hydrometallurgical process plants. Pierre Gy (Pitard 1993) demonstrated how the large-scale stream heterogeneity and variability of chronological data compiled as a variogram can be quantified using the semi-variogram. Conventional statistics fails to identify the sources of process stream heterogeneity and variability. These can be identified in a variogram and may reveal spatial-temporal continuities that provide insight into the factors responsible for process behaviour.

Chronologically related large-scale variability is most easily discerned in one-dimensional process streams or spatial settings including conveyor or rail transport of solid fragments, slurries in pipes or a chronologically ordered set of drums as shown in Figure 23.1.

www.dunlopconveyorbelting.com/ www.news.carrentals.co.uk www.tradekey.com www.flickr.com/nuclear awareness

Figure 23.1: Examples of the range of moving streams that could be sampled([40] FF Pitard)

The analytical data for the variable of interest are plotted as a variogram and the specification limits, the mean, and the target value for the variable of interest are compared against the variability of different aspects of the process as identified in the variogram. Plotting the information on a variability diagram provides an indication of the alterations that are required in order to bring the plant processes under control and ensure that the product lies within the specifications limits.

The work of Pierre Gy (1992) in developing variographic analyses of chronological data was extended by Pitard (1993) using the alumina content of clinker used to manufacture speciality cement. Minnitt and Gluck, (200?), Minnitt and Pitard (200?) and Minnitt (200?) provide other examples of variographic analyses of manganese in a hydrometallurgical plant and in the production of iron ore. More recently the subject has been examined by Francois-Bongarçon (2012).

28.2 Components of Variability

The variogram allows the different components of variability in a chronological set of data that may otherwise be masked by one another, to be resolved. The total heterogeneity (h_T) in a chronologically

ordered data set is the integrated accumulation of three sources of heterogeneity as shown in ***Figure 23.2***, such that:

$$h_T = h_1 + h_2 + h_3$$

where,

h_T = Total heterogeneity,

h_1 = Random, discontinuous heterogeneity that is a property of the materials

h_2 = Non-random, continuous heterogeneity that is a function of time

h_3 = Cyclic, continuous heterogeneity that is a mechanical function of the system

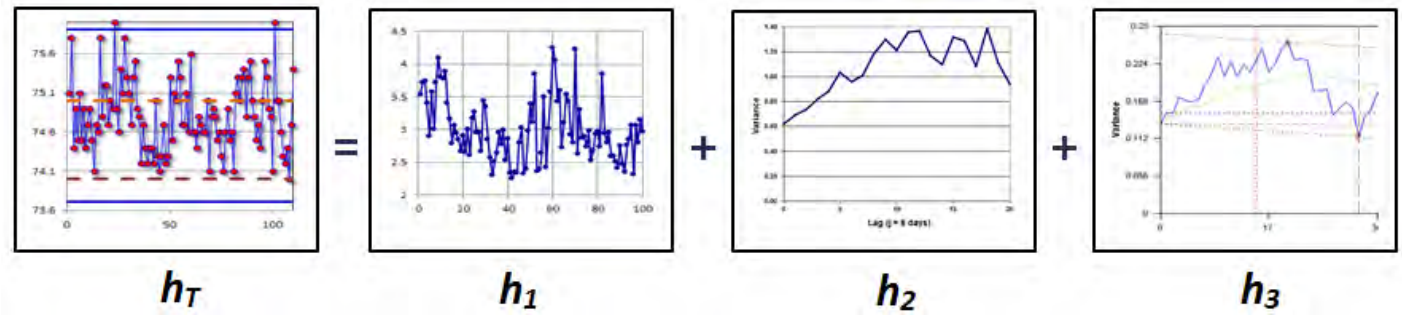


Figure 23.2: Total heterogeneity (h_T) in any system is a combination of random discontinuous heterogeneity (h_1), non-random continuous heterogeneity (h_2), and cyclical continuous heterogeneity (h_3) ([40] FF Pitard)

The combined effect, or total heterogeneity, is most easily represented by a variogram. The simplest way of comparing two values such as $\mathbf{a_1}$ and $\mathbf{a_2}$ representing the content of a component of interest in unit $\mathbf{U_1}$ and unit $\mathbf{U_2}$, respectively, separated by a given interval in space or in time **j**, is to consider their difference *d* many times, so that $\boldsymbol{d = a_1 - a_2}$ as shown in ***Figure 23.3.***

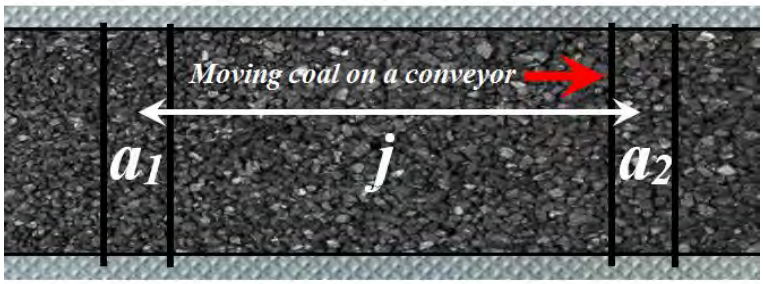


Figure 23.3: A moving conveyor belt with coal analysed at point's a_1 and a_2 separated by distance j (after FF Pitard)

The average difference $\bar{d}$ between the content of the component of interest of [N_u - *j*] pairs of units a given interval *j* apart:

$$\bar{d} = \frac{1}{N_u - j} \sum_{n=1}^{m} \left[a_{m+j} - a_m \right]$$

where N_u is the number of data and *j* is the number of pairs.

Assume there are five successive across-the-stream samples, collected at regular intervals *j = 1 = 10* minutes as shown in ***Figure 23.4.***

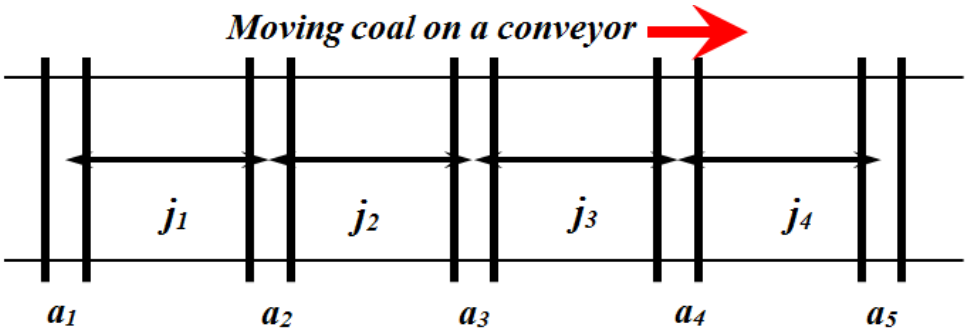


Figure 23.4: Five successive cross-stream samples (a_1 to a_5) collected at 10 minute intervals (j_1 to j_4)

For N_u = *6* samples and the lag *j = 1*, then the number of pairs is N_u *- j = 6 - 1 = 5*

For N_u = *6* samples and the lag *j = 2*, then the number of pairs is N_u *- j = 6 - 2 = 4*

The average difference between many pairs of samples for a given space or time lag is shown in ***Figure 23.4.*** The average difference tends towards zero as *[N_u - j]* increases, so a new difference $\bar{D}$ is defined of which the average of the squared differences is:

$$\bar{D} = \frac{1}{N_u - j} \sum_{n=1}^{m} \left[a_{m+j} - a_m \right]^2$$

In order to better compare this variance with the variance of conventional statistics, the semi-variogram $V[j] = \bar{D}/2$ is defined.

$$V[j] = \frac{1}{2[N_u - j]} \sum_{n=1}^{m} \left[a_{m+j} - a_m \right]^2$$

This formula is used to calculate the absolute variogram, details of the variogram construction being developed in other texts by Journel and Huijbregts (1978), Isaaks and Srivastava (1989) and Journel and Huijbregts (1978). “A variogram is a plot of the average variance between two sample values as a function

of the distance between them. It is used to quantify the spatial variability of spatial phenomena[84]". First-order variability is the squared average of the differences between adjacent data points (j = 1). Second-order variability (j = 2) is the squared average of the differences between alternate data points and so on. A variogram is a plot of average variance (V) against the order (j) at which they are calculated. In this application variographic analysis examines systematic variations in chronologically ordered data and is illustrated in Table 23.1.

Table 23.1: Six samples analysed sequentially for %Fe and the calculation of the variogram for these data

%Fe	Distance (m)	2	4	6	8	10	12
66.3	2m	0.16	1.21	1.44	2.89	2.89	1.96
65.9	4m	0.49	0.64	1.69	1.69	1	
65.2	6m	0.01	0.36	0.36	0.09		
65.1	8m	0.25	0.25	0.04			
64.6	10m	0	0.09				
64.6	12m	0.09					
64.9	**Sum**	1.00	2.55	3.53	4.67	3.89	1.96
	Semi-variance	***0.08***	***0.26***	***0.44***	***0.78***	***0.97***	***0.98***
	Distance (m)	***2***	***4***	***6***	***8***	***10***	***12***

The results of the variogram calculated in Table 23.1 are plotted in ***Figure 23.5*** for six sequential data points.

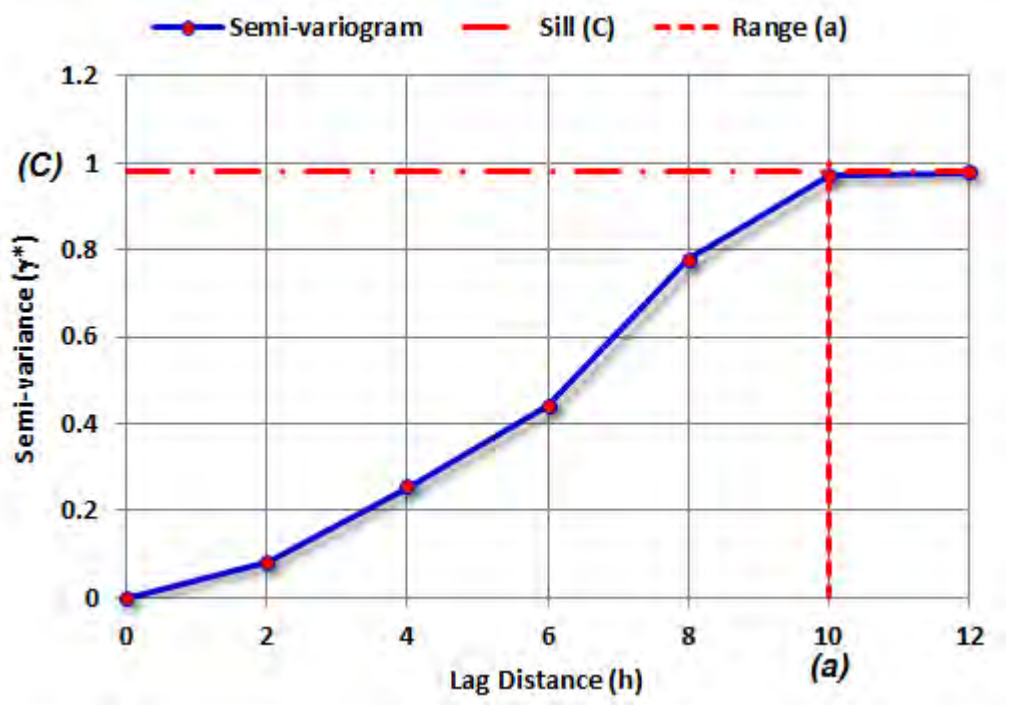


Figure 23.5: Variogram for the six sequential data points listed in Table 23.1

[84] Nel, F. Variography, Section 4 in Unpacking the Geostatistical Toolbox, GEOSTATISTICAL ASSOCIATION OF SOUTHERN AFRICA. http://www.gasa.org.za. Page 8.

The shape of the variogram (Figure 23.5) may vary considerably especially at very short ranges. In this case the variogram, which is always zero at lag distance = 0, rises steadily to reach the sill at a semi-variance of 0.98, and then flattens off. The variogram reaches the sill at a distance referred to as the range of influence (a).

1. Chronostatistics: Analysis of Chronological Variability

The silica content (SiO_2) of iron ores listed in Table 23.2 has been measured every 12 hrs, over an 84 day period. Usually the upper and lower specification limits (USL, LSL), the mean of the analyses, and some preferred target average (TA) is shown with the two- or three-times standard deviation limits on the variability plot. The variability of these data is shown in ***Figure 23.6*** and while there is a clear indication that there is a strong cyclical heterogeneity superimposed on these analyses, this type of diagram reveals relatively little about the process. A large proportion of process control may be founded on just such variability diagrams, with adjustments to processes based entirely on the plant superintendent's interpretation of plant-performance from the variability diagram. The system avarge is 2.87 %SiO_2, but the Target Average is consistently lower at 2.84 %SiO_2. The variability plot of ***Figure 23.6*** shows that seven of the 168 analyses lie well beyond the 2x standard deviation (97.5%) control limit and that there is a large amount of variability within the upper and lower control limits.

Table 23.2: Sequential %SiO_2 analyses from an iron ore plant

Sample No	*SiO_2*	*Sample No*	*SiO_2*	*Sample No*	*SiO_2*	*Sample No*	*SiO_2*	*Sample No*	*SiO_2*	*Sample No*	*SiO_2*
1	2.85	31	2.92	61	2.82	91	2.90	120	2.99	150	2.82
2	2.93	32	2.85	62	2.88	92	2.88	121	2.92	151	2.90
3	2.88	33	2.88	63	2.80	93	2.85	122	2.87	152	2.81
4	2.83	34	2.90	64	2.86	94	2.84	123	2.80	153	2.92
5	2.83	35	2.92	65	2.81	95	2.87	124	2.83	154	2.87
6	2.85	36	2.85	66	2.86	96	2.93	125	2.83	155	2.88
7	2.77	37	2.87	67	2.84	97	2.90	126	2.86	156	2.84
8	2.83	38	2.88	68	2.88	98	2.88	127	2.83	157	2.90
9	2.94	39	2.84	69	2.88	99	2.84	128	2.86	158	2.95
10	2.90	40	2.96	70	2.78	100	2.84	129	2.89	159	2.92
11	2.84	41	2.99	71	2.87	101	2.87	130	2.86	160	2.94
12	2.92	42	2.92	72	2.81	102	2.94	131	2.88	161	2.90
13	2.88	43	2.85	73	2.86	103	2.82	132	2.89	162	2.85
14	2.91	44	2.80	74	2.88	104	2.85	133	2.88	163	2.83
15	2.87	45	2.81	75	2.88	105	2.84	134	2.90	164	2.87
16	2.88	46	2.83	76	2.89	106	2.86	135	2.88	165	2.87
17	2.85	47	2.81	77	2.84	107	2.93	136	2.89	166	2.87
18	2.90	48	2.82	78	2.91	108	2.87	137	2.91	167	2.83
19	2.92	49	2.86	79	2.91	109	2.88	138	2.85		
20	2.90	50	2.79	80	2.92	110	2.84	139	2.84		
21	2.99	51	2.80	81	2.84	111	2.79	140	2.86		
22	2.91	52	2.87	82	2.98	112	2.90	141	2.88		
23	2.94	53	2.92	83	2.86	113	2.85	142	2.91		
24	2.87	54	2.87	84	2.88	114	2.86	143	2.88		
25	2.82	55	2.88	85	2.88	115	2.87	144	2.81		
26	2.82	56	2.88	86	2.83	116	2.87	145	2.83		
27	2.87	57	2.87	87	2.89	117	2.90	146	2.87		
28	2.85	58	2.92	88	2.87	118	2.87	147	2.82		
29	2.87	59	2.96	89	2.83	119	2.96	148	2.82		
30	2.84	60	2.98	90	2.82	120	2.87	149	2.83		

While operators in the plant may be aware of the problems, mechanisms to control the silica levels either do not exist or they are too erratic for even skilled operators to correct. One may argue that the specification limits are too narrow, but there are two assay points that lie outside the two standard deviation limits suggesting that these points are in some way anomalous. In general the difference in silica values of samples, immediately following those outside the 97.5% limits, is large suggesting that attempts to adjust the system led to over-correction.

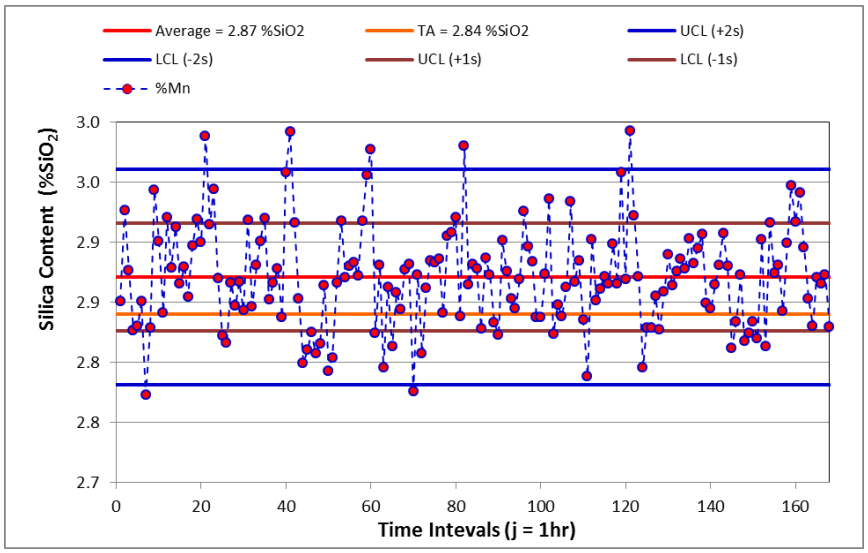


Figure 23.6: Silica content of 168 iron ore analyses sampled every 12 hrs for 84 day period, the average silica content (2.78%), upper and lower control limits for 1x and 2x standard deviations, and a target average are also shown

The presence of cyclical variability in the data can be emphasised through the application of a moving average as shown in the 5-point moving average for the example of silica content in iron ores used here, ***Figure 23.7***. The cycles have a very strong period of 240 hours or 10 days that is probably related to mining activity in a specific area or portion of the deposit as extraction proceeds.

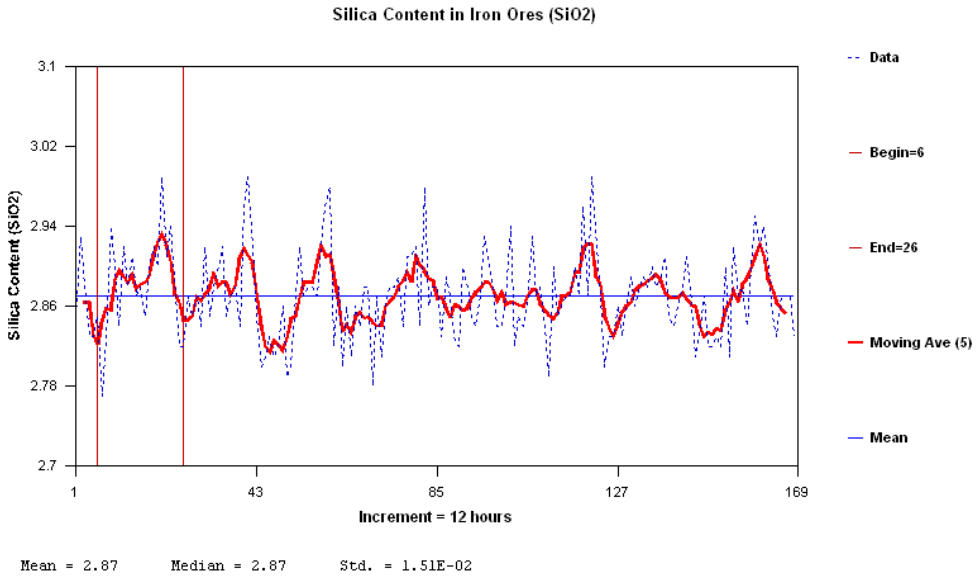


Figure 23.7: A 5-point moving average for the silica content in iron oresshowing a period of 10 days

28.3 Use and Application of the Variogram

Two variogram types, the absolute- and relative-variograms can be compiled for sequential data. The absolute variogram for the $\%SiO_2$ data listed in ***Table 23.2*** is shown in ***Figure 23.8.***

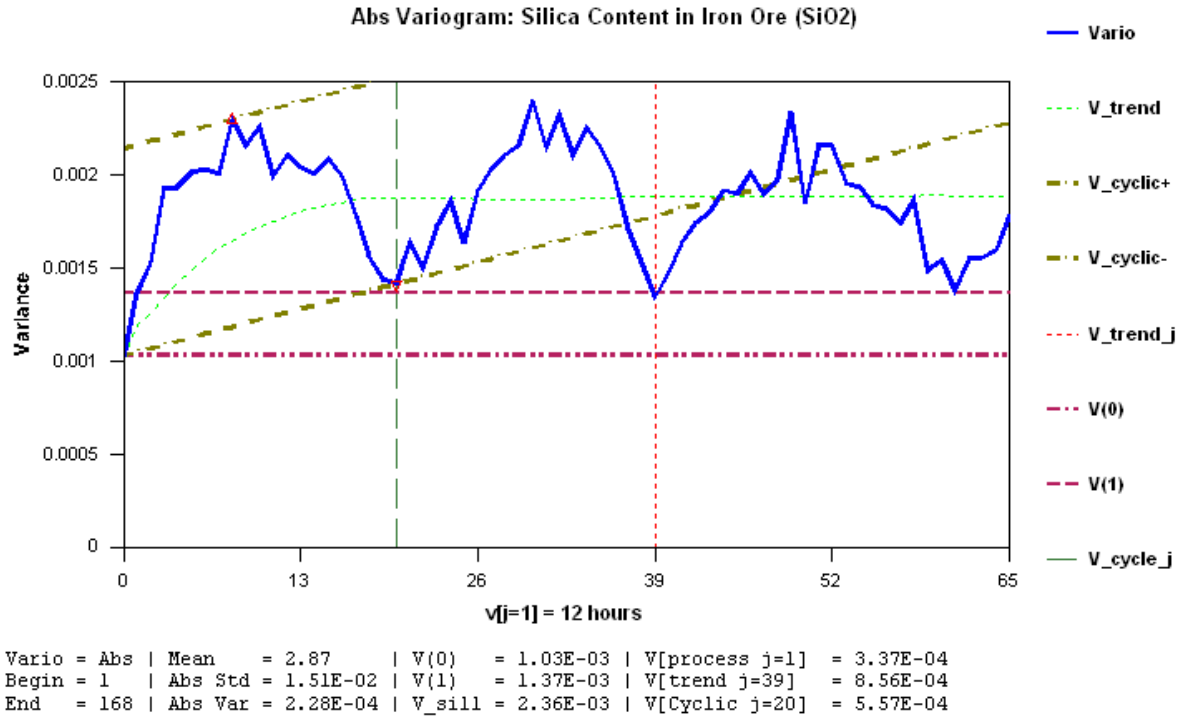


Figure 23.8: Absolute variogram of the silica content in iorn ores for a 32.5 day period (65 x 12hr lags)

The absolute variogram, which reveals no more than the relative variogram, indicates that the twelve-hour resolution of the process is fairly low, but that it is quite stable in that the cyclical component is well defined. The variogram clearly indicates the presence of three robust cycles at a regular period of about 240 hrs (10 days). The regular rise and fall of the variogram over the 65 lags shown in Figure 23.8 is due to cyclical behaviour that was also demonstrated in the moving average plot of Figure 23.7. Clearly the first cycle is the most stable and accurate as it uses more data points than the later cycles.

The absolute variogram is important when trying to establish absolute values for the components *V[0]* and *V[1],* as shown in Figure 23.9. *V[0]* is the variance at a lag of zero. This is usually referred to as the Nugget Effect and includes variability from all sources of sampling error and bias including FSE, GSE, IDE, IEE, IWE, IPE and AE. In fact all forms of variance which are not process-related and which have accumulated during the sampling procedure are represented here. In addition to the accumulated sampling errors and bias *V[0]* also includes a component of random variability – the real Nugget Effect - that arises from the heterogeneity of the ores themselves. It is variance unrelated to large-scale in-plant processes, but related only to the inherent small-scale variability of the material and the sampling procedure.

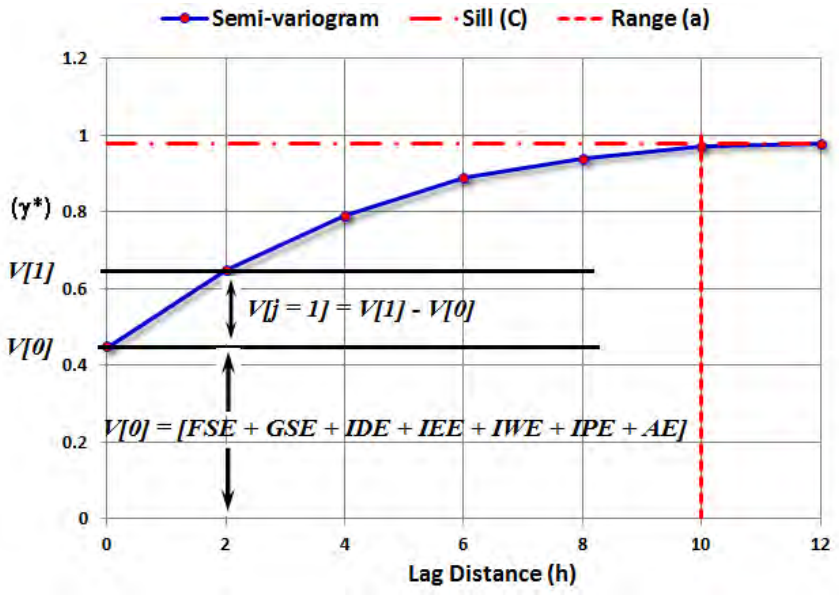


Figure 23.9: Components of the variogram showing the Nugget Effect (V[0])as a combination of FSE, GSE, IDE, IEE, IWE, IPE, and AE, the total sill, and the range of influence

The typical or average variability between one sample and the next is measured by *V[j = 1]* (Figure 23.9). This is the variability in the process, one lag apart, for any given lag and is represented by:

$$V[1] = V[j = 1] - V[j = 0]$$

The two variances *V[j=0]* and *V[j=1]* must also be explained in context of the variability plot. This is the only use of the absolute variogram.

28.4 Limitations to the Use of Variograms

Generally one should not calculate a variogram with a lag that goes beyond half the data available, i.e. the number of pairs [Nu - j] should be larger or equal to Nu/2. If one has only 100 data points there should not be more than 50 points on the variogram. The average of the squared differences is known with good precision only if the number of pairs available is sufficient. One should not use fewer than 20 points to draw a variogram as the stability of the variance will then deteriorate quickly. The guideline is:

N_u - j	Level of Precision
30	Very good precision
25	Good precision
20	Acceptable precision (recommended limit)
15	Poor precision
10	Meaningless

Data that obviously belongs to different series or different populations as illustrated in ***Figure 23.10,*** should not be mixed. The data sets must be separated before variography is attempted. For the variogram to be robust, the variability of first differences should remain constant through time. For a given, well-established sampling protocol this hypothesis usually holds, but should not be taken for granted.

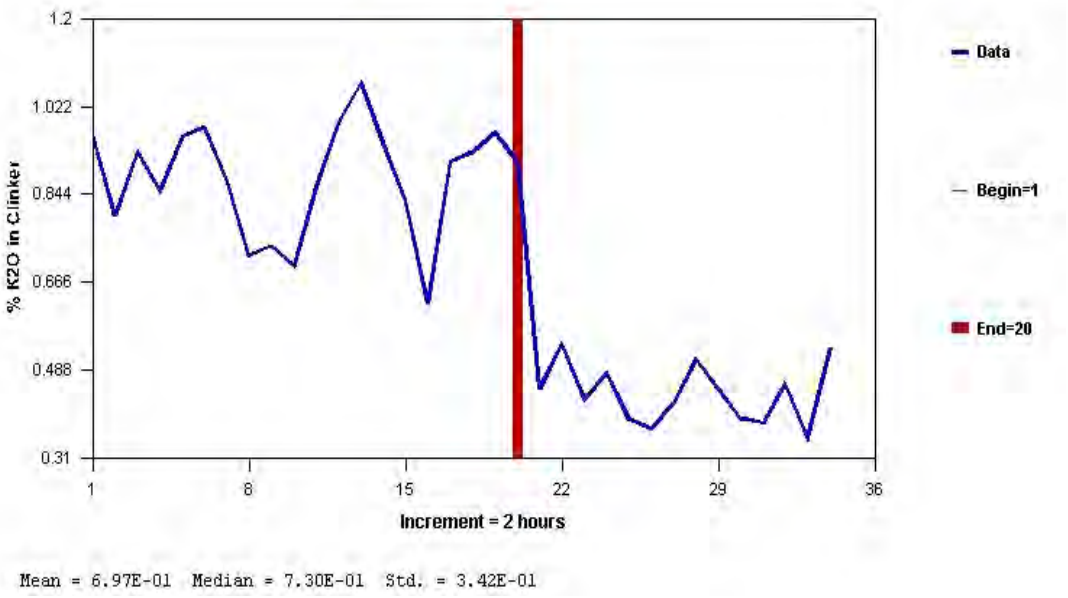


Figure 23.10: %K20 in clinker - beware of mixing two populations (^{40} FF Pitard)

(Source: Pitard, F, Sampling Theory and Methods Course notes 2006).

28.5 Long Range Variography

28.5.1 The relative variogram

For variography to be useful, sampling intervals should be relatively short. Furthermore, variograms from different experiments, or different time-frames, are often easier to compare if they are relative and dimensionless. If a_L is the estimated average from all data involved in the calculations, the recommended formula is as follows:

$$V[j] = \frac{1}{2[N_u - j]a_L^2} \sum_{j=1}^{m} [a_{m+j} - a_m]^2$$

An example of such a variogram is shown in ***Figure 23.11***.

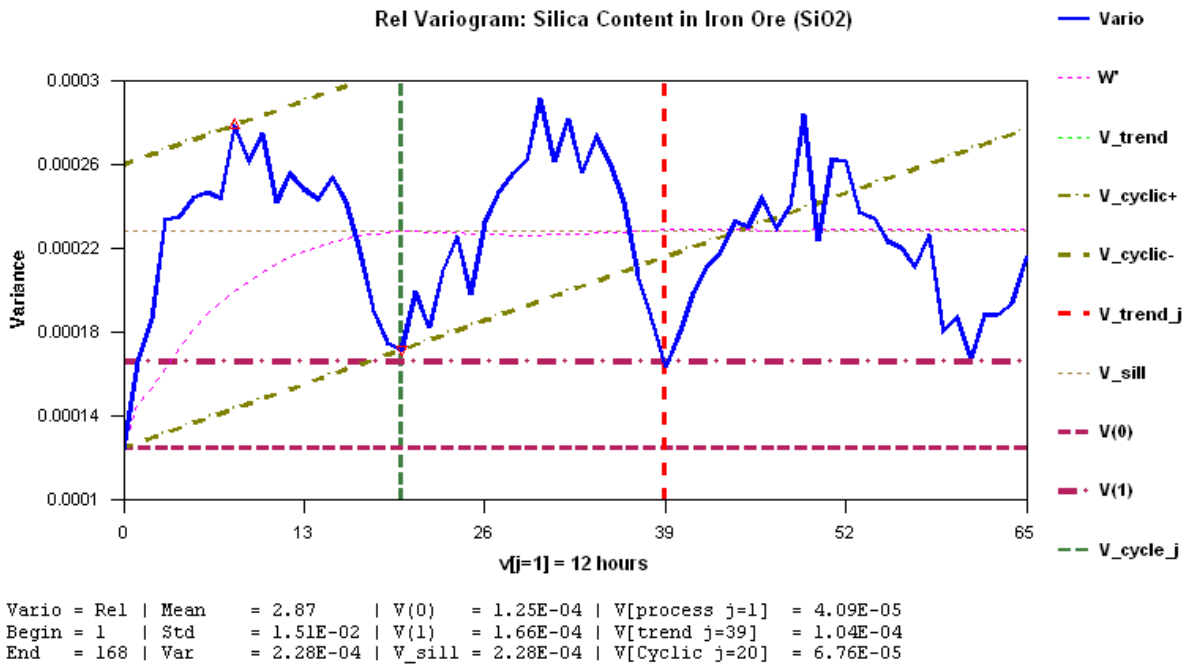


Figure 23.11: Relative dimensionless variogram of SiO_2 content in iron ores

The value of the relative variogram is that it allows one to compare the variability of data from different time series.

28.5.2 Compiling and Interpreting the Variogram

The following points are evident in the variogram (Figure 23.11) and are summarised in Table 23.3.

Table 23.3: The symbol, source, and amount of variability in the Absolute Variogram

Symbol	Source of Variability in Absolute Variogram for Silica	Variance ($\%SiO_2^2$)
Mean	2.87% SiO2	
Abs Std:	Absolute Standard deviation	0.0151
Abs Var:	Absolute Variance	0.000228
V[0]	**Nugget effect**. This short-range variability has nothing to do with the process. It is a combination of inherent heterogeneity (Fundamental Error (FE) or Grouping and Segregation Errors (GSE)), and all uncontrolled sampling error arising from a poor sampling protocol. The NE to Sill ratio is about 44%%. Since this is a bit more than a third of the overall variation, it suggests that the sampling errors associated with the Nugget Effect (NE), the Grouping and Segregation Error (GE) and the Fundamental Error (FE) of the sampling protocol, could be problematical and are worthy of further consideration. Upper and Lower Control Limits (UCL and LCL) are set in the Control Chart by multiplying the standard deviation of V[0] by 3 in order to cover the 99.7% confidence interval.	V[0] = 0.00103 √V[0] = 0.0321 S = ±0.0321 **UCL** = Mean + 3S = 2.87 + 3*0.0321 = 2.9663 $\%SiO_2$ **LCL** = Mean - 3S = 2.87 – 3*0.0321 = 2.7737 % SiO_2
V[1] V[process]	This is the non-random **process variability** that occurs in the plant between two consecutive analyses. V[0] is about 16% lower than V[1] so it is unlikely that there are major problem associate with the process variation. This process variability can be controlled and it appears that the sampling interval is appropriate. This measures the typical difference between V[1] and V[0] at the first lag point in the variogram. It is non-random variability due to the sampling process related to the Delimitation Error (DE), the Extraction Error (EE), the Preparation Error (PE) or the Analytical error (AE) and can be eliminated through establishing a sound sampling protocol.	V[1] = 0.00137 √V[1] = 0.037 **UCL'** = 2.87 + 3S + √V[1] = 2.87 + 3*0.0321 + 0.037 = 3.003 % SiO_2 **LCL'** = 2.87 – 3*S - √V[1] = 2.87 – 3*0.0321 – 0.037 = 2.7367 $\%SiO_2$ V[process] = V[1] – V[0] = 0.00137 – 0.00103 = 0.0003 √V[process] = 0.0173
V[cyclic]	The value for V[cyclic] is half the total amplitude of the process cycle, between the highest and lowest points on the variogram, usually associated with the first cycle. It is a non-random variable related to specific activity in the process. In the case of the short-range analysis, the amplitude is spread over three cycles. UCL" = Mean + 3√V[0] + √(V[1] - V[0]) + √V[cyclic] = 2.87 + 0.0963 + 0.0173 + 0.0236 = 3.0072 $\%SiO_2$ LCL" = Mean - 3√V[0] - √(V[1] - V[0]) - √V[cyclic] = 2.87 – 0.0963 – 0.0173 - 0.0236 = 2.7328 $\%SiO_2$	V[cyclic] = 0.000557 √V[cyclic] = 0.0236 **UCL''** = +3S = 3.0072 $\%SiO_2$ **LCL''** = -3S = 2.7328 $\%SiO_2$

V[sill]	V[sill] is a measure of the average variability of the process measured across all the data. This should be the same as V[trend] since they measure the same thing. V[sill] is measured across the entire variogram whereas V[trend] is the value of the sill at a given lag.	
V[trend]	V[trend] can be measured at any lag distance, but usually at the specific lag point where the variogram reaches a maximum. This component is due to some mechanical or human intervention that takes place approximately every 2 to 3 days and introduces variability into the system. Generally the trend of the variogram is upwards until a point (the range) is reached, beyond which the variogram is level or declines.	V[sill] = V[trend] = 0.000856 %SiO_2

The data derived from the variogram are transferred to the Control Chart and a variogram-based hierarchy of Control Limits is defined in Table 23.4, which shows how the various control limits are defined and calculated.

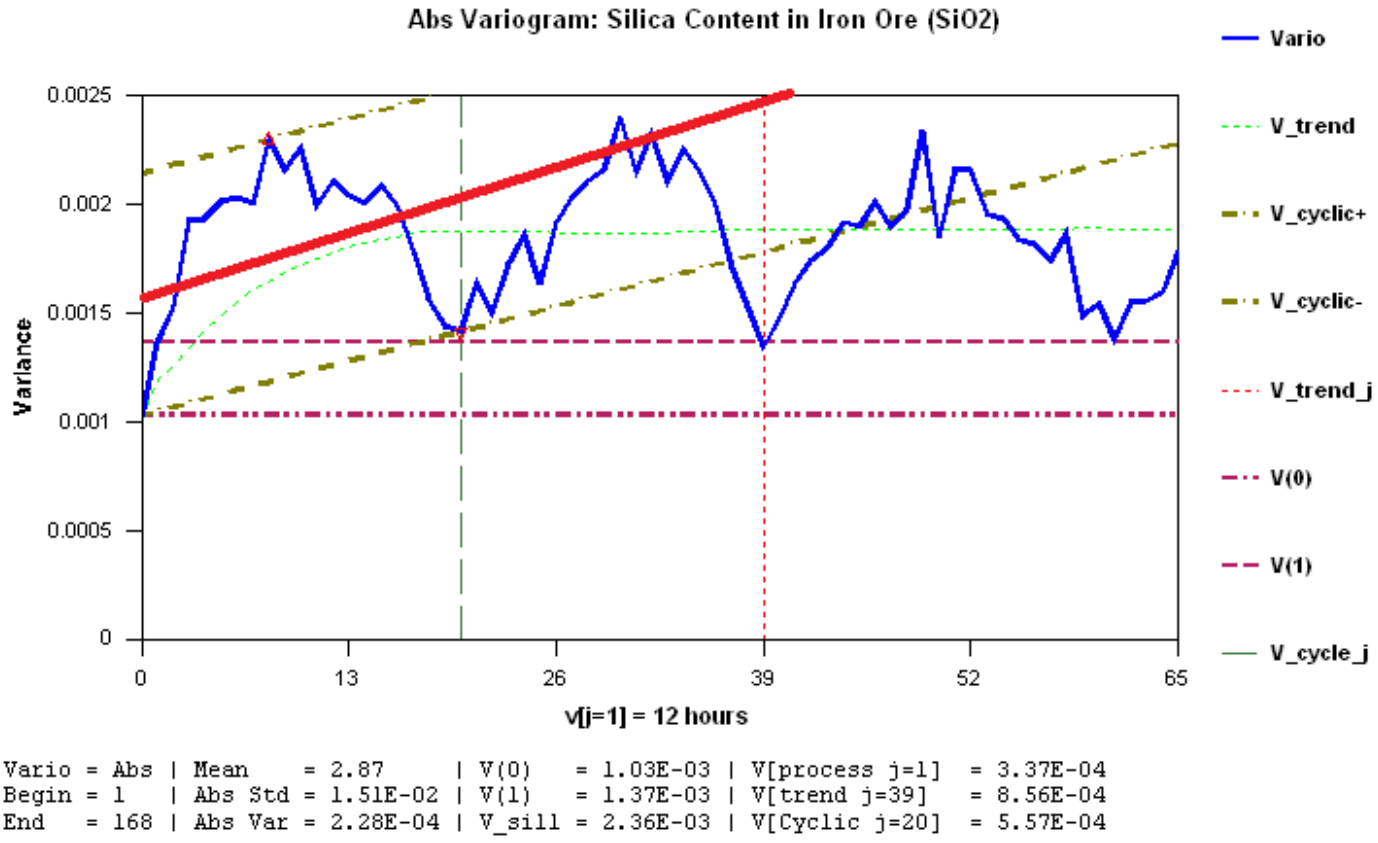


Figure 23.12: Absolute variogram for the silica content in iron ore

Table 23.4: Hierarchy of Control Limits derived from the variogram

Component of Variability	Variances	Control Limits
V[0], the combined NE, FSE and GSE: UCL and LCL = Mean ± 3√V[0]	V[0] = 0.00103 S = √V[0] = 0.0321 3S = 0.0963 %SiO_2	**UCL** = 8.98 + 3S = 9.23 %SiO_2 **LCL** = 8.98 - 3S = 8.73 %X
+Plus Process Allowance: UCL' and LCL' = UCL ±√V[1] and LCL ± √V[1] = ± { (3√V[0]) + √V[1] }	3x√V[0] = 0.2544 %X + √V[1] = 0.0927 = 0.3471	**UCL'** = +3S + V[1] = 8.98 + 0.3471 = 9.33 %X **LCL'** = -(3S + V[1]) = 8.98 - 0.3471 = 8.63 %X
+Plus Cyclicity Allowance: UCL" and LCL" = ± { (3√V[0]) + √V[1] + √V[cyclic]}	3x√V[0] = 0.2544 + √V[1] = 0.0927 + √V[cyclic] = 0.0941 = 0.4412	**UCL''** = 3x√V[0] + √V[1] + √V[cyclic] = 8.98 + 0.441 = 9.42 %X **LCL''** = 3x√V[0] + √V[1] + √V[cyclic] = 8.98 - 0.441 = 8.54 %X

The data listed in Table 23.4 can now be plotted on the Control Chart for Metal X as a guide to what the sample variability is likely to do within the given constraints, of the is shown in ***Figure 23.13***.

28.6 Sampling and Analytical Variability *V[0]*

The short-range heterogeneity or random variability that ***is not part of the process*** is defined as *V[0]*. This variability arises from combined sampling error and bias, and is not relevant to the process variations one is interested in monitoring and controlling. However *V[0]* may become large enough to mask the variability in the routinely generated data, the information derived from the variogram is no longer useful for effectively controlling a process. Therefore *V[j = 0],* simplified to *V[0]* notation, is an estimation of the true, unknown random variability affecting the set of data under study, and it must be minimized.

As can be seen in the illustration of accumulated sampling errors (***Figure 23.13***), *V[0]* measures the combined effect of the FSE, the GSE, and the AE, random variability that can never be eliminated because of the inherent heterogeneity of the material itself – it has nothing to do with the process. *V[0]* is however often higher than this minimum due to the additive nature of errors arising from IDE, IEE and IPE, that inflate the true value of *V[0]*. *V[0]* does see the sampling bias directly. However, sampling bias is never

constant and because of the transient nature of segregation it may have a short-range variability its own, which the variogram sees as an expanded value for *V[0].*

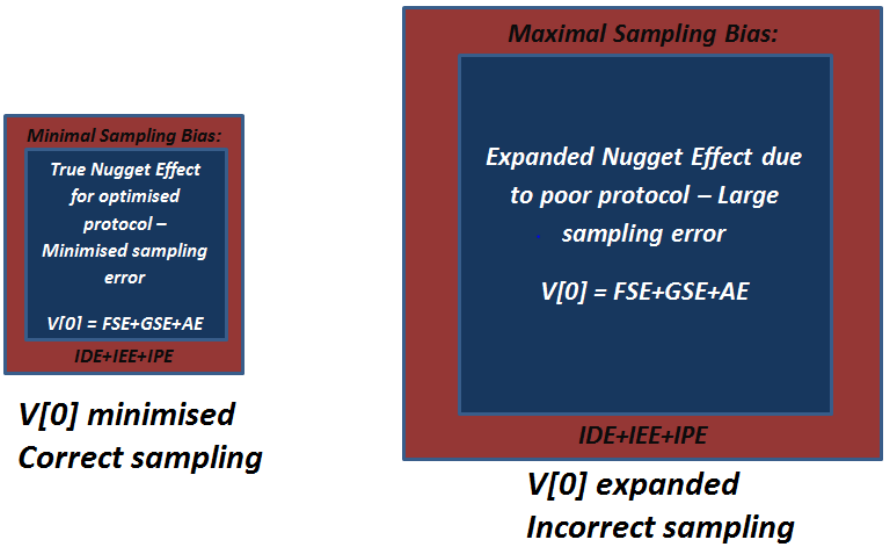


Figure 23.13: Expected short-range random variability V[0] can be minimized by correct sampling but may be expanded due to incorrect protocols and bias introduced by IDE, IEE, and IPE

The expanded variance due to incorrect sampling (Figure 23.13) can be reduced by an improved sampling protocol, by short term averaging, compositing, or by improving the sampling equipment. The size of *V[0]* determines the extent to which this may be necessary. The rule for calculating the standard error of the mean applies.

28.7 Process Variability V[j]

The ***process variability*** *V[j]* associated with a flowing stream can be represented in terms of a variogram, and is an aggregate of three components of variability represented as:

$$Process\ variability: V[j] = V_1[j] + V_2[j] + V_3[j]$$

where:

- $V_1[j]$ = Short-range, random, discontinuous variability within the process
- $V_2[j]$ = Long-range, non-random, continuous variability within the process
- $V_3[j]$ = Periodic, continuous variability within the process

Information from the variogram informs decisions concerning the adequacy of sampling and measurement protocols, cost/benefit analyses on projects to eliminate identifiable sources of variability through engineering modifications, decisions concerning sampling frequency, the definition of process control strategies and experimental design decisions.

28.7.1 Short-range, Random Term $V_1[j]$

$V_1[j]$ is the short-range, random, discontinuous variability within the process; it is the average, over all samples used in the variogram, of the difference in grade between two consecutive samples. Because it is the difference between any two consecutive data points, it is measured as the difference between V[0] and V[j = 1], at the first lag in the variogram (***Figure 23.14***). We should also note that when j tends towards zero, V[j = 1] tends to V[0].

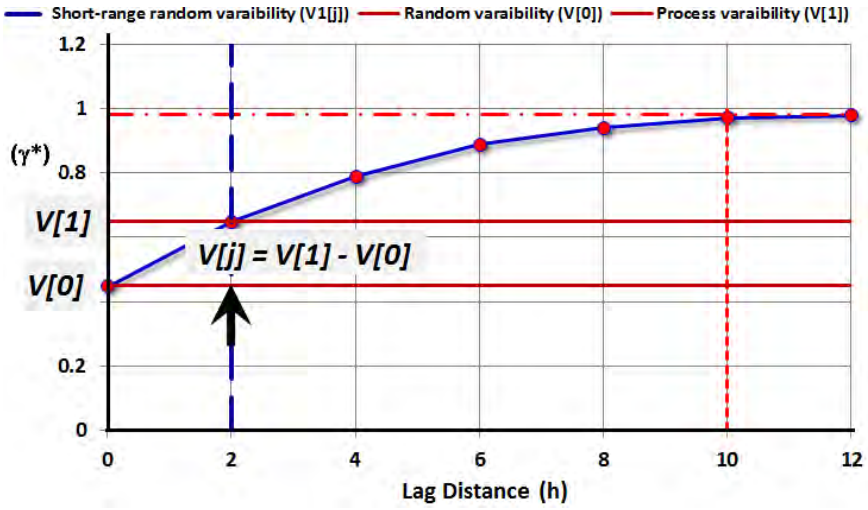


Figure 23.14: The absolute variogram illustrating the V[j=1] and V0 terms

This is identical to the interpretation for the absolute variogram shown in Figure 23.9, except the values are different because here we use the relative variogram.

28.7.2 Long-range, Non-random Term, $V_2[J]$

The long-range, continuous, non-random variability is a trend that is introduced into the process by variability that arises from changes in the primary inputs from the mine or quarry, from raw materials in stockpiles and silos feeding the plant, from mills and kilns within the plant, by the control room, and from the addition of chemical reagents etc. It is variability which one tries to control and hence it is essential that there are well-defined DQO's and clarity about expectations of the process, as well as the capability of the process.

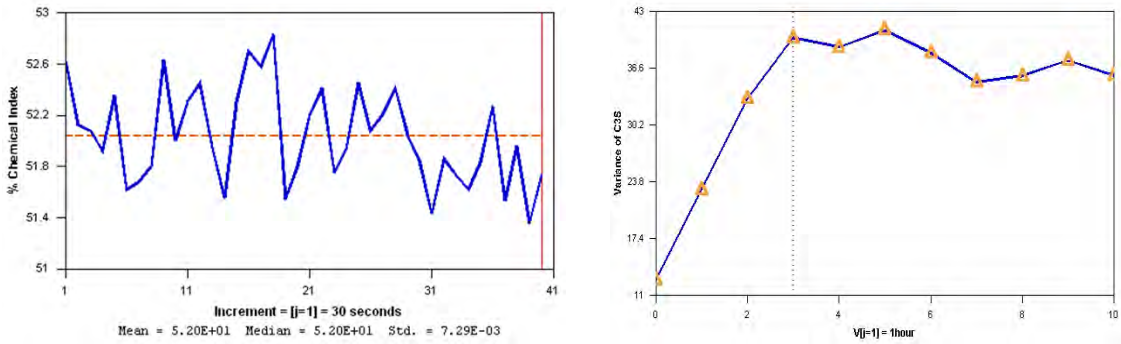


Figure 23.15: (a) %Chemical Index in raw mill, (b) Absolute variogram for %Chemical Index in raw mill ([40] FF Pitard)

The data variability is shown in ***Figure 23.15*** with a corresponding absolute variogram shown in ***Figure 23.16.***

Data is collected every hour and although there is considerable variation in the results on an hourly basis, the absolute variogram (***Figure 23.16***) indicates that there is very little to be done to improve the situation. The variogram levels off after about three hours indicating that the process stabilises very quickly. Had the variogram continued to climb after three hours, one may begin to suspect that there is a problem at the plant. Problems in the system are identified by taking the value for $\sqrt{V_{Sill}}$ and adding it to the variability diagram. Provided $\sqrt{V_{Sill}}$ lies within the control limits the plant is operating correctly.

28.7.3 Continuous Periodic Variability within the Process $V_3[j]$

Cyclical activities, either known or unknown, such as the weekly change of reagents, the extraction of ores from a certain part of a mine, certain long term behaviour patterns amongst staff or plant personnel, may be the cause of a continuous periodic variability that imposes cycles on the process. These can usually be identified in a variogram, and if the cause is unknown will require careful time and motion study to identify.

Depending on the frequency and amplitude of such cycles, the products from process plants could move beyond the limits specified by the customer.

28.8 Interpreting and Applying Information from the Variogram

Step 1: The target: The first step in interpreting the variogram is to establish both the ***mean*** and the ***expected target value*** for the product stream. In the example of the %Chemical Index the target lies somewhat higher than the mean as shown in ***Figure 23.17***.

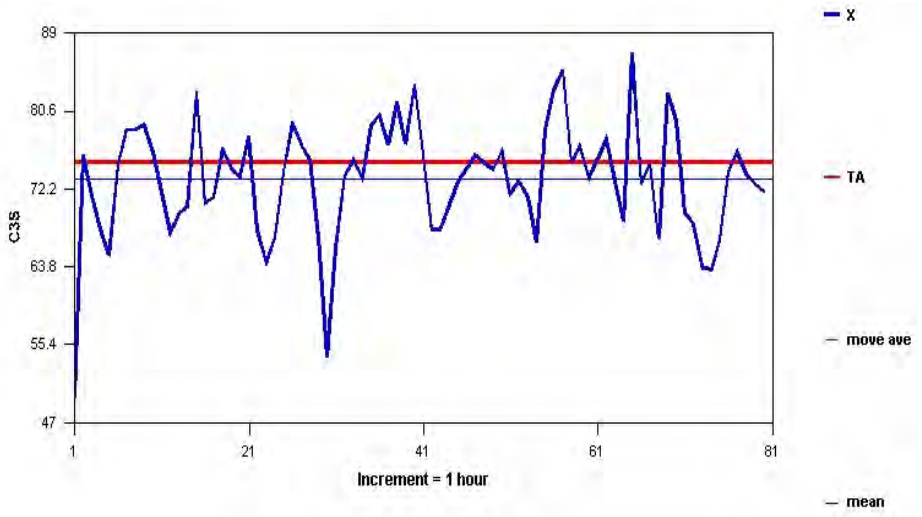


Figure 23.17: %Chemical Index in raw mill before homogenising silo: Target 75+-12 (2 sigma) showing the mean and the target or expected value for the stream (^{40} FF Pitard)

Step 2: The specification limits are usually defined on technical or economic factors and represent the ideal range within which the variability is constrained. The upper and lower specification limits, shown as

US and LS in ***Figure 23.18***, may not be equidistant from the mean and could be defined differently for different reasons. The next and most important step is to take the information from the variogram and superimpose it on the variability diagram in order to see how the systems are performing relative to the mean value, the target value and the specified limiting values (***Figure 23.18***). In this particular case there is only one point that definitely lies beyond the lower limit; the bulk of the points lie within the upper and lower specification limits with a variability that is acceptable. There may be periods when the plant processes approach the limits, but they do not actually breach them. The process is easily controllable.

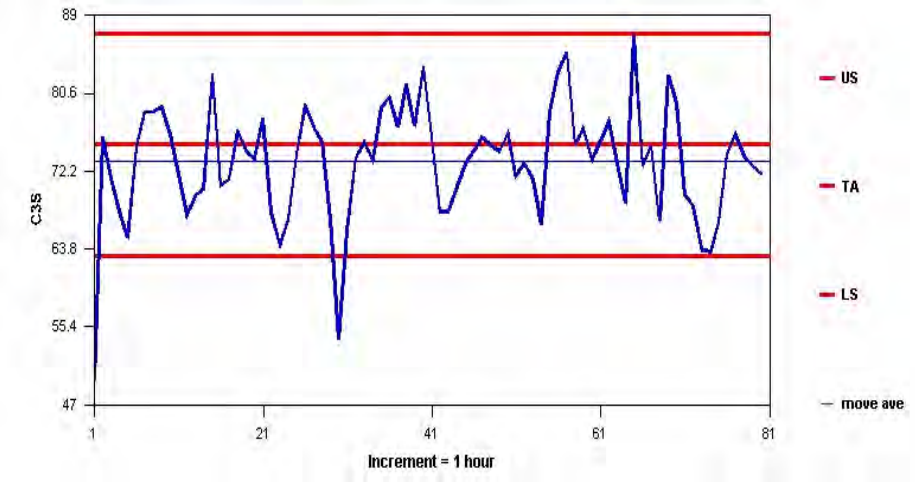


Figure 23.18: %Chemical Index in raw mill before the homogenising silo: Target 75+-12 (2-sigma) showing the mean, the target and the upper and lower specification limits. (^{40} FF Pitard)

Step 3: The sampling + analytical variability *V[0]*: This is determined by the size of V[0] and is represented on a variability diagram as Upper and Lower Control Limits about the mean or the targeted value depending on the requirements of the product. It is calculated as follows: Sampling + analytical variability = Mean ± 2√V[0]. This is essentially twice the standard deviation of this variance or a 95 percent confidence interval about the range of values, since V[0] is a random variable. These values are plotted on the variability diagram as shown in ***Figure 23.19*** and are then compared to the specification limits and the overall variability of the process. The important and relevant question here is: "Relative to your Data Quality Objectives, as defined by the specification limits, how is the variability in the process and the plant affecting your product?"

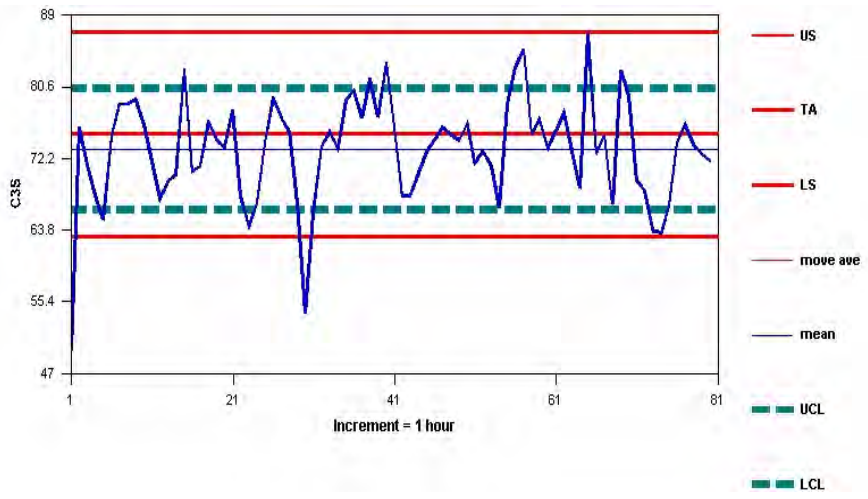


Figure 23.19: %Chemical Index in raw mill before homogenising silo: Target 75+-12 (2-sigma) showing the mean, the target, the upper and lower specification limits and the upper and lower control limits ([40] FF Pitard)

The upper and lower control limits (UCL and LCL) which define what the plant is capable of in terms of sampling protocol, lie within the upper and lower control specification limits (US and LS), suggesting that the variability due to sampling and analytical variability does not create problems for the plant superintendent. However, the gap between the control limits and the specification limits is not large and therefore does not provide much leeway with regard to variability between control limits and specification limits. Ideally one would want the gap between the control and specification limits to be as small as possible.

Step 4: The sampling interval variability V[1]: This is non-random variability in the plant and is measured on the variogram by V[1] = V[j=1] – V[0]. The notation V[1] is an arbitrary notation. We chose to reserve the notation V[j=1] for the true process variability. It is a measure of typical non-random drift in the process between two typical samples in the plant and is represented on the variability diagram, ***Figure 23.20*** as the Upper and Lower Control Limits shown a s the thick blue lines with a prime sign, UCL' and LCL'.

Since the lower specification limit LS and the Lower Control Limit LCL' lie on top of one another (***Figure 23.20)*** there is a clear indication that, in combination with the sampling and analytical variance, the process is in fact "out of control". Again, ideally one would want the gap between the prime control limits (UCL' and the LCL') and the upper and lower specification limits (US and LS) to be as large as possible. Generally the distance between the specification and limit lines on the variability diagram should be no more than a third of the specification limit from the mean or targeted value.

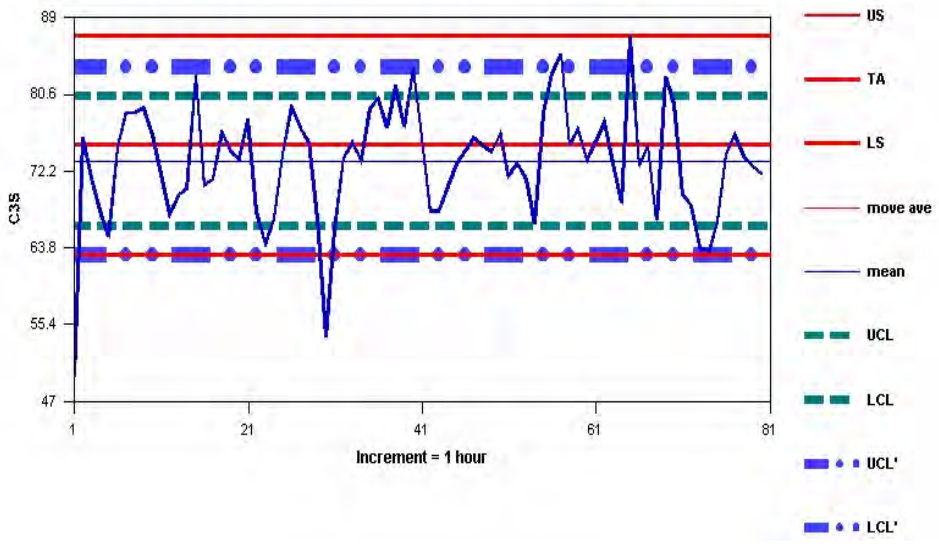


Figure 23.20: %Chemical Index in raw mill before homogenisation silo: Target 75+-12 (2 sigma) showing the component of non-random variability in the plant ([40] FF Pitard)

28.9 Periodic, Continuous Term $V_3[j]$

Cycles are extremely important and it is critical that the presence of cycles be detected and correctly interpreted. Understanding and interpreting cycles correctly always provides an opportunity to minimise a cost, either visible or invisible. A large majority of variographic experiments or variograms calculated with existing chronological data show cycles. The regularity of the amplitude and period shown in Figure 16.9 can only arise as a consequence of equipment related cyclical influence.

28.9.1 Components of the Periodic Term

The cyclic component of the chronological data results in a cyclic component in the variogram. The period T of the cycle in the variogram is equal to that of the cyclic component in the data. Consider a sine curve h_{3t} in a set of data at any instant t, with an amplitude h_3 and a period T and calculate its variogram:

$$h_{3t} = h_3 \sin[2\pi t / T]$$
$$V3[j] = \frac{h_3^2}{2}[1 - \cos(2\pi j / T)]$$

Note that the amplitude of the variogram

$$V_3 = \frac{h_3^2}{2}$$

is half the peak from minimum to maximum. This makes the amplitude of the cycle on the variogram equal to the variance of the cyclic component in the data.

28.9.2 Interpretation of cyclical features

Perhaps the most important feature of any variographic analysis is DQO since this gives an indication of where the system is expected to operate. Without a clear understanding of DQO there is no possibility of making meaningful adjustments to the system. The lack of clear DQO indicates a lack of clear management and understanding of the capacity of the plant processes. It should always be remembered that the identification of a cycle means that there is an opportunity to improve the processes in the plant and reduce large-scale variability. There are several kinds of cycles:

28.9.2.1 ***The perfect cycle; regular amplitude and a regular period****: This is usually related to a specific piece of equipment which is responsible for the cycle. If the period of the cycle is shorter than j = 1 then there may be a cycle within a shorter cycle. It is important to look at the general trend of the variogram since the slope of the variogram tells one how fast the system is changing and sooner or later it should reach a maximum. If possible take a suite of samples between the shortest lag distances in an attempt to identify any other cycles.*

28.9.2.2 ***Regular amplitude, erratic period****: Generally this type of behaviour in the variogram can be related to problems in the control room. The operator is required to maintain a certain parameter between two limits and when the system approaches the limit the operator over-corrects and causes an equally severe problem, but at the other end of the scale.*

28.9.2.3 ***Regular period, but erratic amplitude****: This is usually related to operator or people related problems rather than equipment indicating that training of operating personnel is required. The cycle is usually related to a seven day manpower cycle or changing of shifts.*

28.9.2.4 ***Erratic period and amplitude****: This is clearly more difficult to deal with than the other types of cycles and may be a combination of problems. Such cycles originate in the quarry and may be due to segregation in stockpiles and silos.*

29 IMPORTANT APPLICATIONS OF ADVANCED VARIOGRAPHY

29.1 The Random Residual Component of the Variogram *V4[j]*

It has been demonstrated that the variance of the random term is represented by V[0]. Because data available to calculate a variogram is often limited, the precision of every calculated point on the variogram is affected by V[0]. This precision, or random residual component, affects the shape of the variogram and can become a problem for effective variogram modeling. Furthermore, when such background noise affects the variogram, it can be confused with a periodic component for those not well acquainted with variography. Therefore, the total variogram can be written:

$$V[j] = V_1[j] + V_2[j] + V_3[j] + V_4[j]$$

and $V_4[j] \sim 0$ when [Nu – j] increases

29.2 Modeling the Variogram

Complex periodic phenomena and the random residual component V4[j] may render the modeling of the variogram difficult, if not impossible. To circumvent this one can use three **auxiliary functions** directly derived from the variogram:

- The first order integral
- The second order integral
- The moving average

In an effort to remain pragmatic, it is suggested one completely bypass the mathematical modeling of the variogram. *"There are no mathematical functions that can claim a divine right to represent a variogram."* Dr. Michel David (1945-2000), Professor of Geostatistics.

In light of David's statement the model must fit the data and not be wishful thinking. The key to a pragmatic approach is a point-by-point interpretation of the variogram. The advantage of this approach is that it does not alter what is known from the few calculated points representing the variogram by proxy. Using the Auxiliary Functions of the variogram provides the following advantages:

- It allows the variogram to be extrapolated to V[0] with greater precision and accuracy than by simply extrapolating the model semi-variogram;
- It allows the residual random variability V4[j] to be smoothed out;
- It allows the cyclic variability to be smoothed out;
- It provides a means of calculating the variance s^2(PIE) of the total, continuous Process Integration sampling Error (PIE), and;
- It provides a means of better control of processes by emphasizing the relevant variability.

29.3 Point-by-point interpretation of the variogram

The most effective point-by-point interpretation of a variogram is to give to the center values an interval of influence equal to 1, *j = 1, 2, 3,…, n-1* and the extreme values V(j=0) = V_0 and V(j = n) an interval of influence equal to ½ as shown graphically in ***Figure 24.1.***

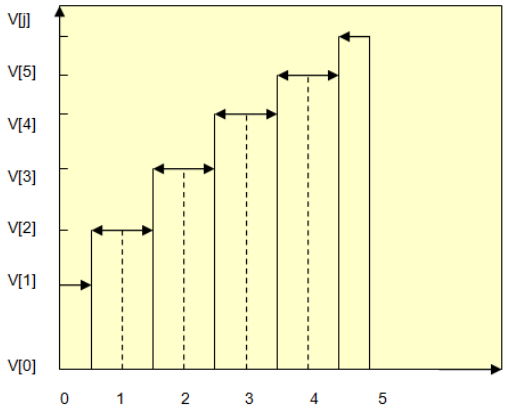


Figure 24.1: Point-by-point interpretation of the variogram (40 FF Pitard)

The point-by-point interpretation of the variogram requires that the surface area for each segment of the variogram be determined. A series of surfaces is defined, S_0, S_1, S_2, S_3, S_4 and S_5 as shown in Figure 24.2. Calculate S[j] defined as:

$$S[j] = Estimate\left[o_0^j V(j')dj'\right]$$

Or, in a pragmatic way:

$$S[5] = \frac{V[0]}{2} + V[1] + V[2] + V[3] + V[4] + \frac{V[5]}{2}$$ or

S[0] is nil by definition.

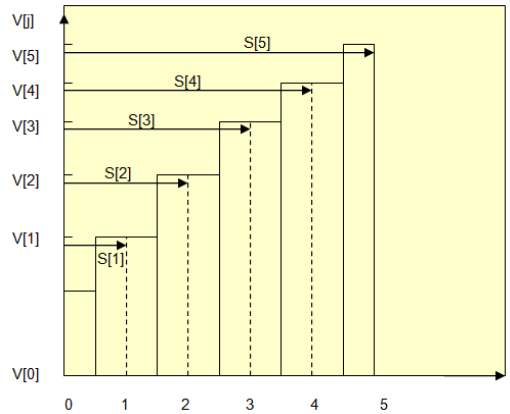


Figure 24.2: Variogram defined by surfaces S[0] to S[5] (40 FF Pitard)

In addition the point-by-point interpretation of the variogram requires a generalization for all values of j ≥ 1:

$$S[j] = S[j-1] + \left\lfloor \frac{V(j-1)}{2} \right\rfloor + \left\lfloor \frac{V(j)}{2} \right\rfloor$$

29.4 Point-by-point Calculation of the First Order Integral, W[0] = V[0]

Point-by-point calculation of the First Order Integral, W[0] = V[0] is shown in ***Figure 24.3***. By definition:

$$W(j) = \frac{1}{j}\int_0^j V(j')dj' = \frac{S_j}{j}$$

where S_j is the surface and j is the total lag and the integral is the surface divided by the lag

$$For \quad j = 0,\ S(0) = 0 \ and \ W(0) = V(0)$$
$$For \quad j > 0,\ W(j) = \frac{Sj}{j}$$

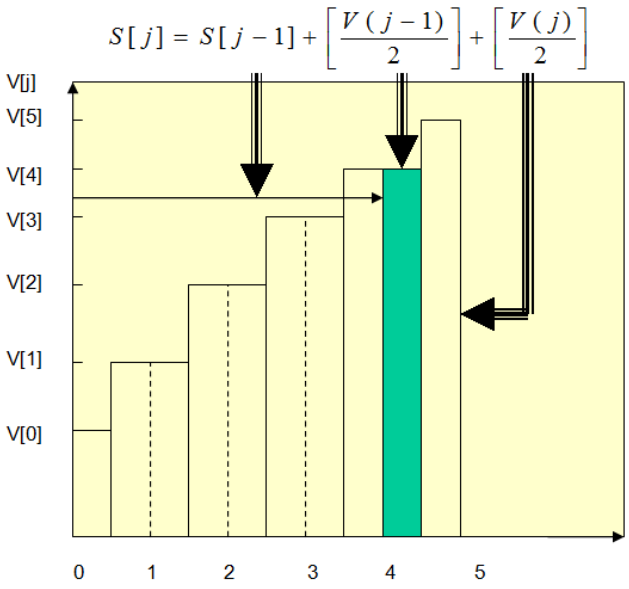


Figure 24.3: The generalized variogram for all values of j≥1 ([40] FF Pitard)

Once generalized values for all j ≥ 1 have been established the random residual $V_4(j)$ and the cyclic $V_3(j)$ components cyclic $V_3(j)$ can be smoothed away. An example having no cycle is shown in ***Figure 24.4***.

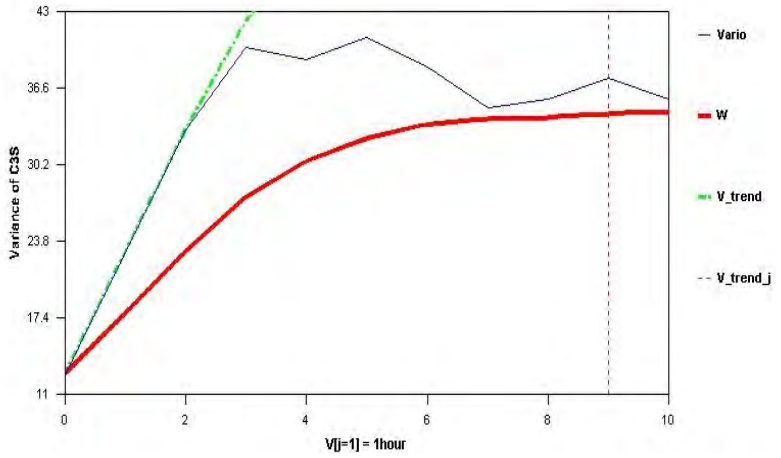


Figure 24.4: In a variogram having no cyclical component the First Order integral W of the variogram shown by the green line smoothes out the random residual component. ([40] FF Pitard)

The principle aim of establishing the First Order integral is so that we obtain a unencumbered and accurate value for V[0]. An example of a variogram with cyclical characteristics such as the example of K_20 in clinker, is shown in ***Figure 24.5.***

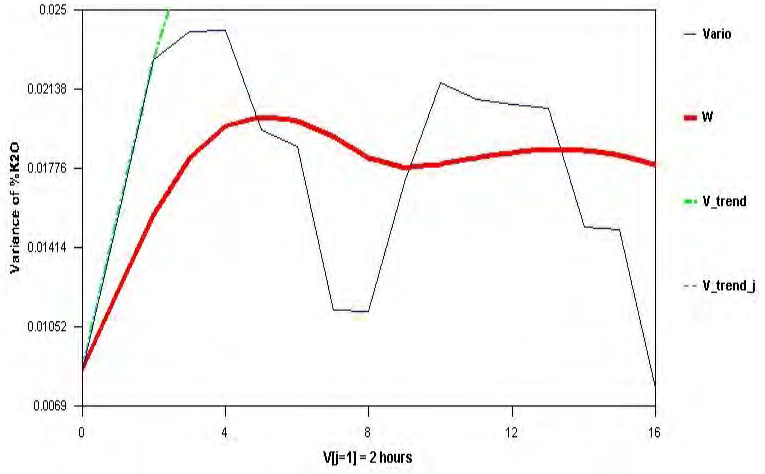


Figure 24.5: Variogram with cyclical characteristics is smoothed out by the First Order integral shown as a green line; a Second Order integral smoothes everything out ([40] FF Pitard)

29.5 The Point-by-point Second Order integral of the variogram W'(j)

By definition: The Second Order integral of the variogram W'(j) has the following form:

$$W'(j) = \frac{1}{j}\int_0^j W(j')dj'$$
$$= \frac{2}{j^2}\int_0^j dj' \int_0^j V(j'')dj''$$
$$= \frac{2}{j^2}\int_0^j j'W(j')dj'$$
$$= \frac{2}{j^2}\int_0^j S(j')dj'$$

Define:

$$S'(j) = \int_0^j S(j')dj \quad then$$
$$W'(j) = \frac{2}{j^2}S'(j)$$

For j = 0 we have S'(0) = S(0) = 0 and W'(0) = W(0) = V(0)

Since W'[0] = W[0] = V[0] are all the same we can do a clean extrapolation of the variogram to the y-axis, such that for j > 0 we have:

$$S'(j) = S'(j-1) + \frac{S(j-1)}{2} + \frac{S(j)}{2}$$

In a further example of %Chemical Index in a Raw Mill before the homogenizing silo, the usefulness of the First and Second Order integrals to smooth away the random residual V4(j) and the cyclic V3(j) components, can be demonstrated.

a. ***Example having no cyclical characteristics***: is shown in Figure 24.6, the points worth noting being that all the curves have the same sill, and they all intersect V[0] on the y-axis at the same point.

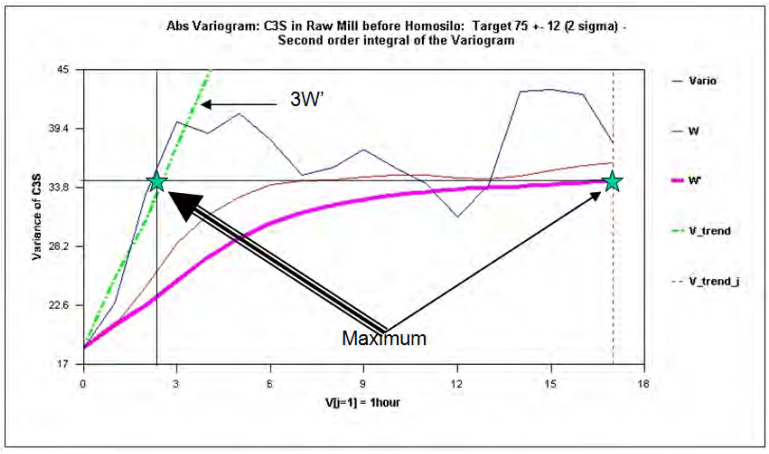


Figure 24.6: Clear definition of V[0] using the Second Order integral ([40] FF Pitard)

b. *Example with cyclical characteristics*: is shown in ***Figure 24.7*** in which any cyclicality has been completely smoothed out by the Second Order integral, and provides a clear indication of the value for V[0].

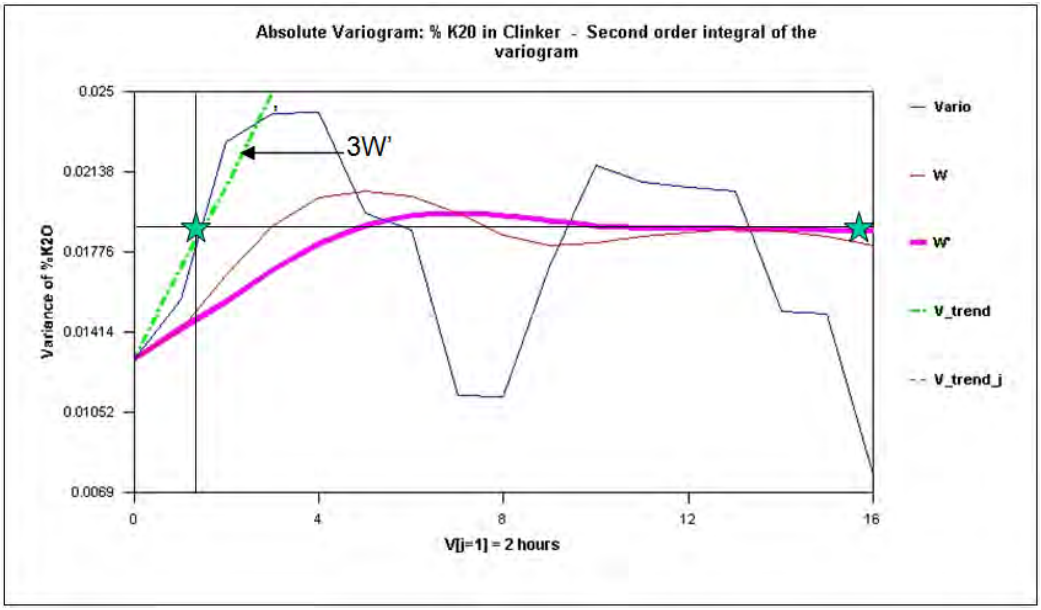


Figure 24.7: Second Order integral completely smoothes out any cyclical characteristics and provides a clear definition for the value of V[0]. ([40] FF Pitard)

29.6 Use of First (W) or Second Order integral (W') to extrapolate the variogram to V[0]

The extrapolation of the variogram is accurate only if [j = 1] is a very short lag: a few seconds, minutes, centimeters or meters. In an example of poor extrapolation shown in ***Figure 24.8*** it is difficult to know where the intersection with the y-axis is or to clearly define V[0].

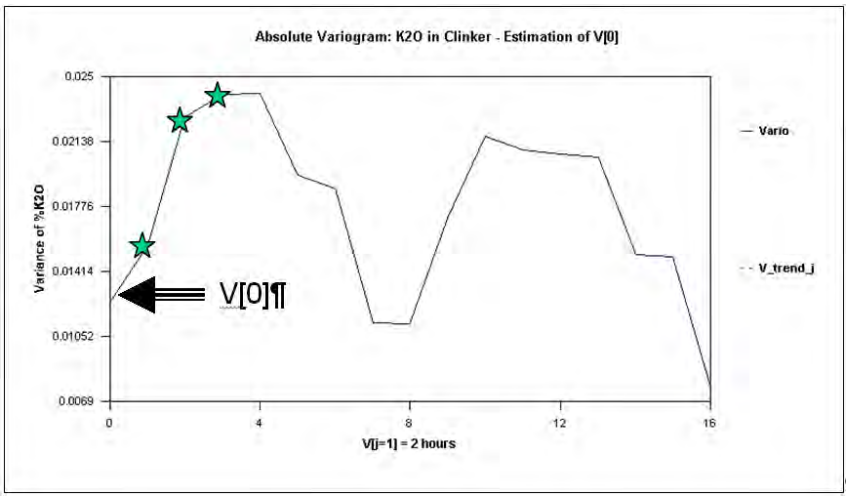


Figure 24.8: V[0] is poorly defined because of the large interval between points on the variogram ([40] FF Pitard)

In an example of extrapolation that clearly defines the intersection with the y-axis shown in ***Figure 24.9***, the points on the variogram are relatively close together.

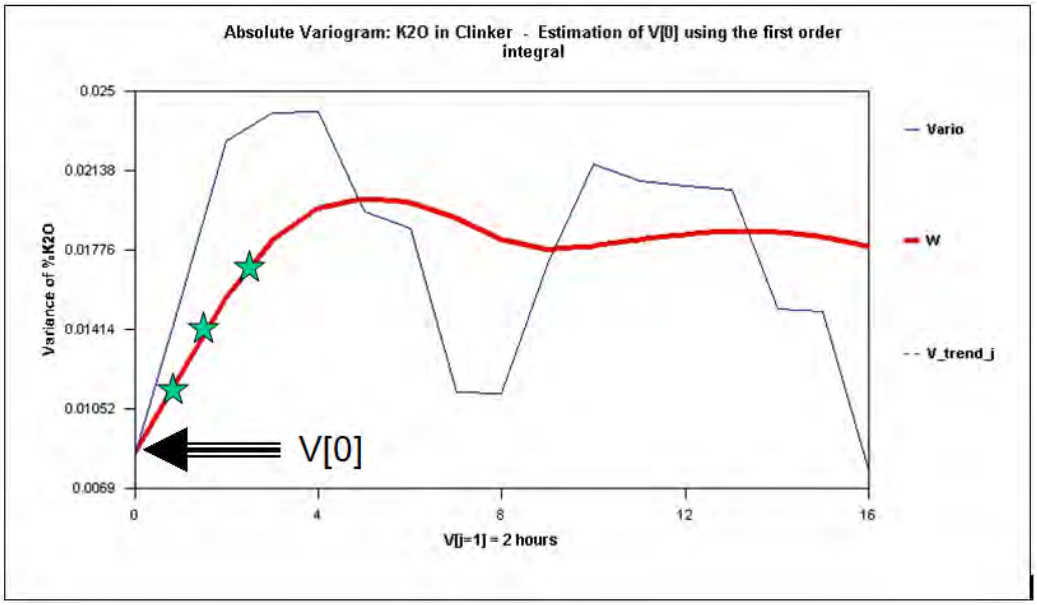


Figure 24.9: Points close together on the variogram make for accurate extrapolation to V[0] ([40] FF Pitard)

Extrapolation is acceptable if the interval is small, but if V_1 is 50m or 100m, extrapolation is wishful thinking. In such cases additional experimentation, taking samples at fairly short intervals may be necessary. The point at a lag = 0 can be determined using down-the-hole variography in a mineralised portion of core, which will allow one to interpolate confidently between the last two points.

Where there is insufficient data in the last segment of the variogram there may be a need to conduct an experiment to gather information at a very short range. For example when feeding the plant one can safely collect an 8hr composite for material balance purposes. However, the short-range variogram will require a sample to be taken at the sampling station every 20 minutes (j = 1 = 20min). At the end of an 8hr shift this will give 8 x 3 = 24 samples which will allow one to compile the variance at 20min. Do the same experiment taking samples every five minutes (that is with j = 1 = 5min), which at the end of an 8hr shift will give 40 samples. This approach could be used until sufficient information is gathered in order to be confident about the position of V[0].

Uncertainty associated with a single sample taken every 8hrs is simply the variance divided by the number of samples. Thus, the variance of a composite is deduced by dividing variance V_4 = j = 20min by 24 to give the uncertainty for one increment. Therefore:

$$Uncertainty = \sqrt{\frac{V_4}{24}}$$

If variance associated with the sample preparation and assay is not divisible by 24 one is stuck with it. In fact it may be a dominant error if one has reduced the error of uncertainty to near zero.

THE ERROR ASSOCIATED WITH CONTINUOUS SELECTION FROM A MOVING MATERIAL STREAM

The aim of this section is to establish the error or standard deviation associated with a single sample taken every 4 hours. The sample is a composite of 27 increments, each increment being extracted from the material stream at 9 minute intervals.

1. **Definition of Error Generators in Relation to Variograms**

Heterogeneity in material streams results in a variety of sampling errors and observing and interpreting the variability arising from heterogeneity in a stream may enable one to iidentify technical difficulties, minimise productivity losses, and minimise operational costs. The following terms are defined:

- L: a given one-dimensional lot
- S: a sample representing L by proxy
- Q: the number of increments making S
- **sy**: notation for systematic sampling
- **st**: notation for stratified random sampling
- **ra**: notation for random sampling
- *j* is the basic sampling interval.
- CSE: the Continuous Selection Error

There are a number of means by which errors are generated and can be identified using the variogram. Principally the errors are generated as a function of the *sampling selection mode* which could be due to systematic sampling, stratified sampling, or random sampling.

Systematic sampling: the applied pattern of sample selection is such that samples are collected ***at regular intervals***, and may include Simple Systematic and Random Systematic sampling. The error generated in this way is: $Z\,j_{sy}$: An error generator created by systematic sampling $s^2\ CE_{sy} = \frac{Z\,j_{sy}}{Q}$

Stratified sampling: The applied pattern of sample selection ensures that a random portion of each group in the population or lot is sampled, and includes Simple Stratified Random and Authoritative Stratified Random sampling. The error generated in this way is:

$Z\,j_{st}$: An error generator created by stratified random sampling $s^2\ CE_{st} = \frac{Z\,j_{st}}{Q}$

Random sampling: This obeys the probabilistic principle that each fragment has the same chance as every other fragment of being in the sample. **Strict Random Sampling:** From a practical standpoint, this sampling selection mode should be rejected because it never provides a better alternative than Stratified Random Sampling. The error generated in this way is:

$Z\,j_{ra}$: An error generator created by random sampling $s^2\ CE_{ra} = \frac{Z\,j_{ra}}{Q}$

Where Z(j) is the variability for ***one increment*** and Q is the number of increments. The mathematical terms for describing these errors has been provided by by G. Matheron for the estimation of variance and extension of the Continuous Selection Error (CSE)[85].

c. ***Systematic sampling (SS)***: Assume j = 20cm. Uncertainty of the composite sample expressed as a variance is given by:

$$\frac{Z(j)_{\text{Systematic Sampling}}}{Q \text{ increments}} = \frac{Z(j)_{sys}}{24}$$

$$Z\ j_{sy} = 2W\ j/2\ - W'\ j$$

The important issue here is that it helps one answer the question *"How often must one take a cut of a stream?"* This can only be determined once the sampling station is installed because only then can the appropriate experiments in the plant be performed, but it may also depend on the nature of the material being sampled.

d. ***Stratified random sampling (SRS):*** $Z\ j_{st} = W'\ j$. In this case the uncertainty associated with a composite sample is expressed as a variance given by:

$$s^2(CE)_{SRS} = \frac{w'(j)}{Q \text{ increments}} = \frac{w'(j)}{24}$$

e. ***Random Sampling (RS)***: $Z\ j_{ra} = s^2\ h_T\ = CH_L =$ Constant in lot L

CH_L = Sill of the Variogram

2. Problem description and proposed experimental solution

This example is presented by Francis Pitard (2007, 2008, 2009) and consists of feed to a cement silo that is sampled every four hours, the four-hour sample being made up of 26 increments, each increment being collected every 9 minutes using a systematic sampling mode. In order to maximise the sampling interval one has to know the variance (total variability) of the Continuous Selection Error (CSE) affecting the %C3S determination within the four-hourly period during which the composite is collected, and more importantly to investigate the variability of the stream in the 9-minute interval between increments.

The solution to this problem is found by dramatically reducing the sampling interval in order to investigate the behavior of the system at very short intervals in the 9-minute period between increments. The aim here was to collect samples every 30 seconds to give eighteen increments in the sampling interval of 9 minutes. A variographic experiment was performed at the sampling station, taking one increment (sample) every 30 seconds and the variability of the stream plotted as a variogram provided a solution.. Each increment was sub-sampled and assayed using the usual routine procedure. In this particular case the term increment is used synonymously with the word sample. ***NB:*** *In fact a*

[85] Matheron, G. 1970. "The regionalized variables theory and its applications." Center of Mathematical Morphology. Ecole Nationale Supérieure des Mines de Paris. Textbook #5, pages 66, 72 and 73. 1970.

sample usually consists of a number of increments, but when applied in variography the smallest unit being considered is the increment, even though several increments may be composited to form the sample.

Table 1: Comparison between the current sampling protocol and the experimental increment protocol

	Current sampling protocol	**Experimental increment protocol**
Sampling selection method	Systematic sampling	Systematic sampling
Increment recovery rate	Every **9 minutes**	Every 30 seconds
Number of increments	26 increments	18 increments
Sampling interval	Every 4 hours	**9 minutes**
Time to collect 40 samples	10 hours	20 minutes

The protocol in Table 1 indicates that the current protocol consists of 4-hourly samples comprising 26 increments, each increment being collected every 9 minutes. In order to investigate the as yet unknown variability in the material stream in the 9 minute interval between increments, it is necessary to collect samples in the 9-minute window. The experiment ran for 20 minutes and takes a sample every 30 seconds, so that a total of 40 samples were collected. Thus 18 samples are collected for every 9 minute interval. This means that the behavior of the variance of the system at very short intervals can now be examined. Data from the experiment are listed in Table 2.

Table 3: Forty analytical results of sampling cement %C3S at 4.5 minute intervals

Sample #	**%C3S**	**Sample #**	**%C3S**
1	52.61	21	52.2
2	52.13	22	52.41
3	52.08	23	51.75
4	51.92	24	51.93
5	52.35	25	52.45
6	51.62	26	52.08
7	51.68	27	52.21
8	51.8	28	52.41
9	52.63	29	52.05
10	52	30	51.85
11	52.31	31	51.43
12	52.45	32	51.86
13	51.95	33	51.73
14	51.55	34	51.62
15	52.3	35	51.83
16	52.7	36	52.27
17	52.58	37	51.53
18	52.83	38	51.96
19	51.54	39	51.35
20	51.8	40	51.73

The variability diagram in Figure 1shows the variation in the data about the mean of 5.20%C3S over a 3 hour period.

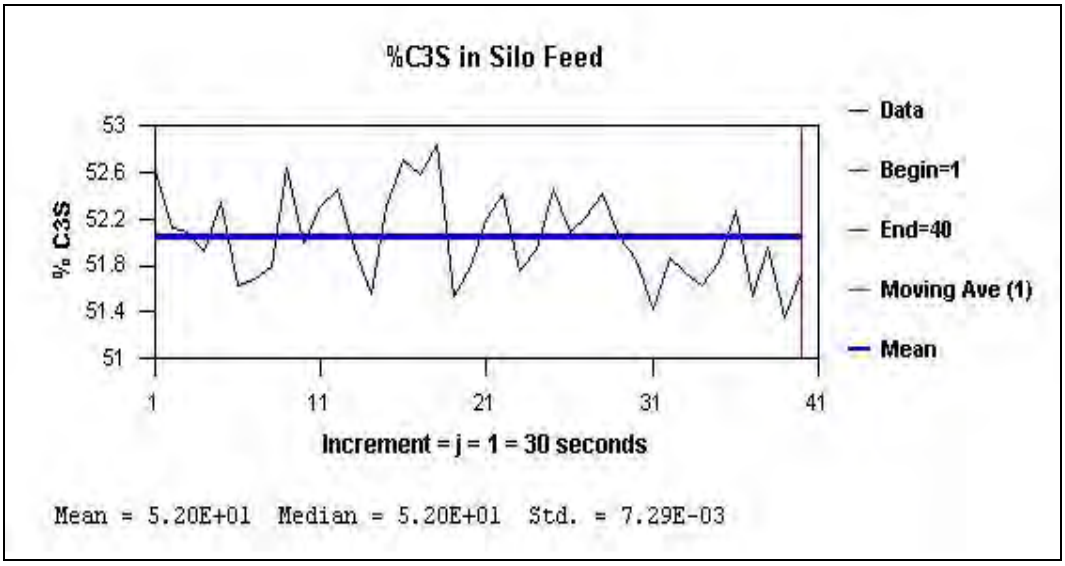


Figure 1: Variability diagram for %C3S in silo feed over a 20 minutes period

For this experiment 40 samples were collected at 30 second intervals, so the 9-minute increments in the plant are now represented by eighteen 30-second samples .

Variography

The absolute variogram for the data presented in Table 3 is shown in ***Figure 2*** together with curves for the First Order (W) and Second Order (W') integrals. An enlarged version of the variogram is shown in ***Figure 3*** to magnify the area and points of interest.

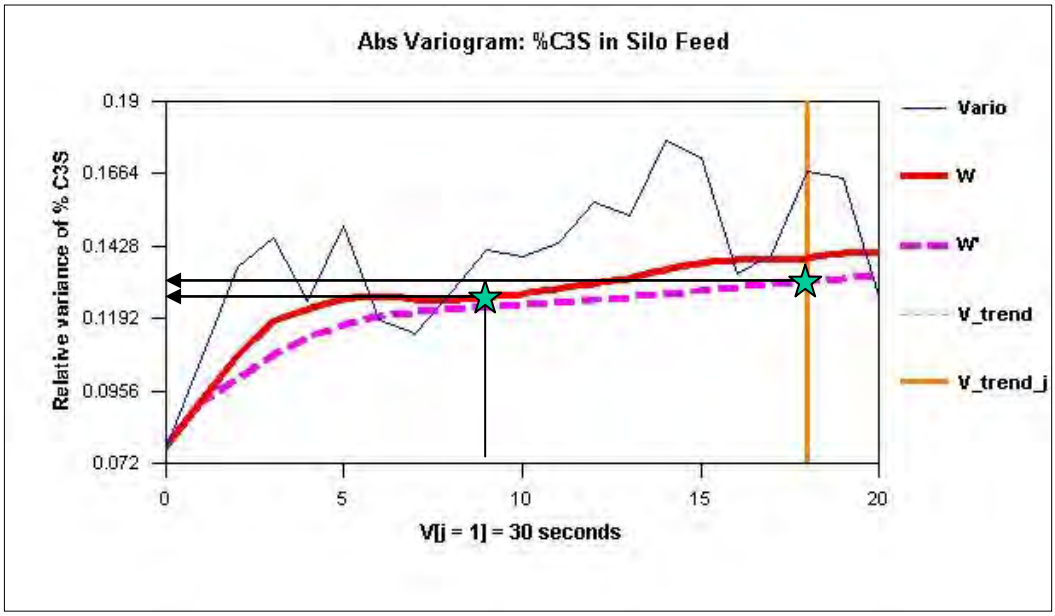


Figure 31: Absolute variogram for %C3S showing derivation of the variance (on y-axis) for First and Second Order integrals

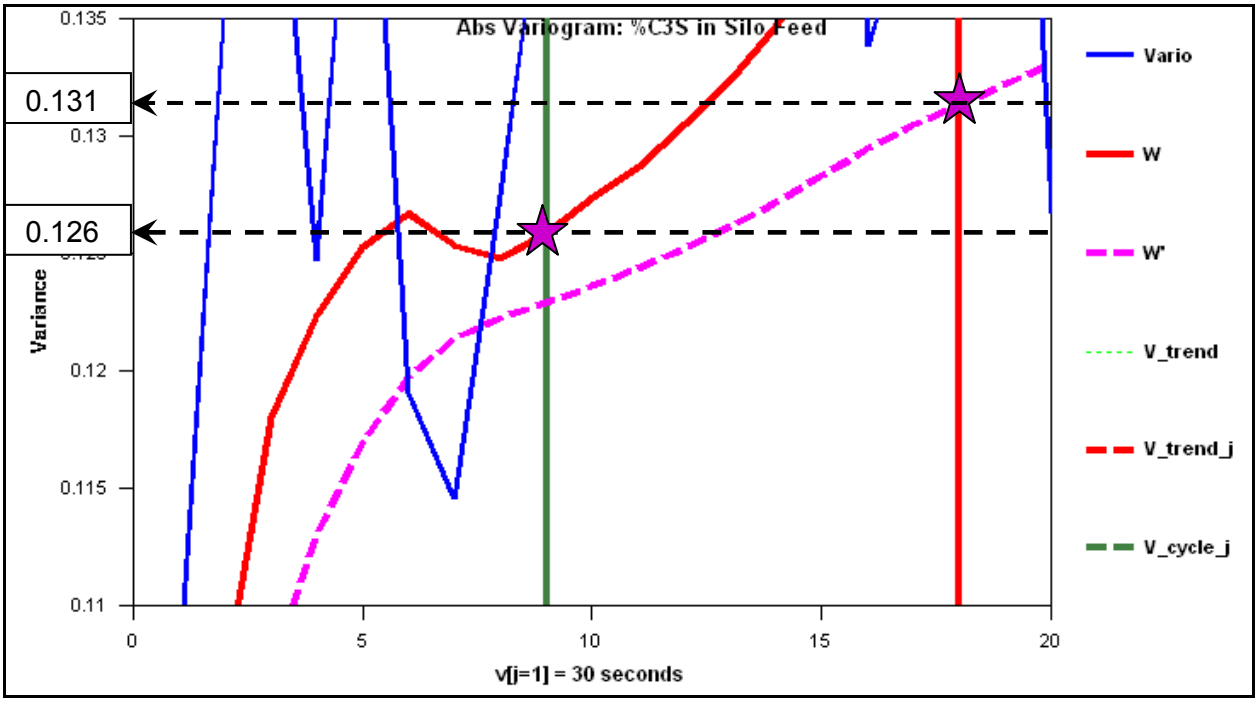


Figure 32: Area and points of interest in Figure 31 magnified

Determination of the Minimum Sampling Interval for Systematic Sampling Interval (using s^2[CE]

In order to calculate the variance associated with the given sampling interval the values of the Second Order integral at the 9-minute increment, and the First Order integral at half the lag distance i.e. 4.5 minutes is required.

$$Z\ j_{sy} = 2W\ j/2\ - W'\ j$$

$$\frac{Z(j)_{\text{Systematic Sampling}}}{Q\ \text{increments}} = \frac{Z(j)_{sy}}{26}$$

The first value is read from the Second Order integral W'(j=18) in the absolute variogram (Figure 3) at the 9-minute lag, i.e. 18 x 30sec = 9min, and gives a variance of 0.131 for%C3S. The second value is read from the First Order integral W(j=9) in the absolute variogram at half the lag distance, i.e. at 9 x 30sec lags = 4.5 minutes, to give a variance of 0.126 for%C3S. Since we know the equation for the variance we can make the substitutions and calculate a variance for s[CE] to provide a standard deviation that can be converted to 90 or 95 percent confidence intervals about the sample grade at any point. The maximum sampling interval is calculated using the following equation:

$$s^2[CE] = \frac{Z(j)_{\text{Systematic Sampling}}}{Q\ \text{increments}} = \frac{Z(j)_{sys}}{26}$$

$$Z\ j_{sys} = 2W\ j/2\ - W'\ j$$

$$s^2[CE] = \frac{2W\ j/2\ - W'\ j}{26}$$

$$s^2[CE] = \frac{2(0.126) - 0.131}{26} = 0.00465\ \%C3S^2$$

and taking the square root gives s[CE] = ± 0.0682 %C3S.

Conclusions

This is a very important result as it indicates that the variability is on the belt between consecutive cuts is ±0.068 %C3S. It is essential that the impact of this value be considered in the context established by DQO set for the particular plant or system. It should be clearly understood that Q = 26 divides only the sampling error. Since the composite is assayed only once, it does not divide Analytical Error by Q. If one is not comfortable with a value of ±0.068 %C3S, change *Q* - the number of increments.

In order to calculate the maximum sampling interval the standard deviation can be transferred to the variability chart and see what its relationship is to the other limits and constraints in the sampling system. If the limits set by the Continuous sampling Error is small then it might indicate that the rate of increment recovery is too fats. In this case the rate of increment extraction could be reduced to say one increment every 20 minutes and one sample every six hours may be an acceptable interval. This would require some careful experimentation to establish what the maximum sampling interval should be. In order to establish the error (or standard deviation) associated with any one 4-hour sample taken from a stream we need to consider the following. For the lumpy ore, a sample made up of 10 composited increments, taken every four hours, the variability in the iron ore stream in the 24 minute interval between increments needs to be examined. The 24-minute period between increments is the opaque region where we have no idea of what is happening. An experiment that specifically targets that time space must be designed. A sample every two minutes (12 samples per 24-minute interval) and run the experiment for at least 80 minutes – you must have about 40 samples to work with in the variogram.

30 THE MOVING AVERAGE

30.1 Correct Use of the Moving Average

Although the moving average has many convenient applications it must not be misused. Its principle purpose is to minimise the effect of random, irrelevant variability in a set of data i.e. to smooth out V[0] the variability that does not exist in the system, or to minimise the effect of a random residual component V4[j] in a Variogram. The effective use of the moving average involves four graphics:

- The original data a_m (shown in ***Figure 18.1***) the integrity of the original data should not be tampered with;
- The Moving Average (MA, shown in ***Figure 18.2***); the choice of the window is at the users discretion;
- The Random Noise RN: RN = am – MA; this provides some measure of validation that the window is appropriate (shown in ***Figure 18.3***), and;
- The Corrected Data (CD) relative to the Real Data Average (RDA), shown in ***Figure 18.4***:
  CD = RDA – MA or relative to a Targeted Average (TA): (RDA = Real data average) CD = TA – MA

In a diagram of the moving average, original data is paramount and should not be tampered with. The size of the moving average can be chosen but depends on the objectives one is trying to achieve which should smooth out V[0], the variability that does not really exist in the system. Therefore, the moving average minimises V[0]. The graphic of RN is really a validation of the window.

a. First Graphic: The original data a_m:

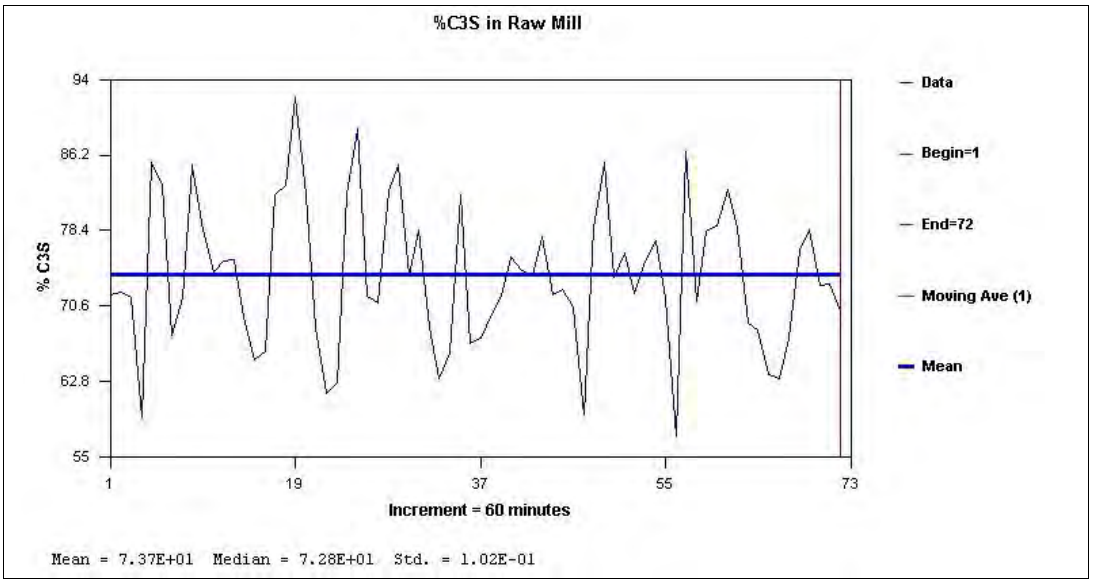


Figure 18.1: Original Data a_m with the thick horizontal line being the arithmetic average

b. **Second Graphic: The moving average (MA)**

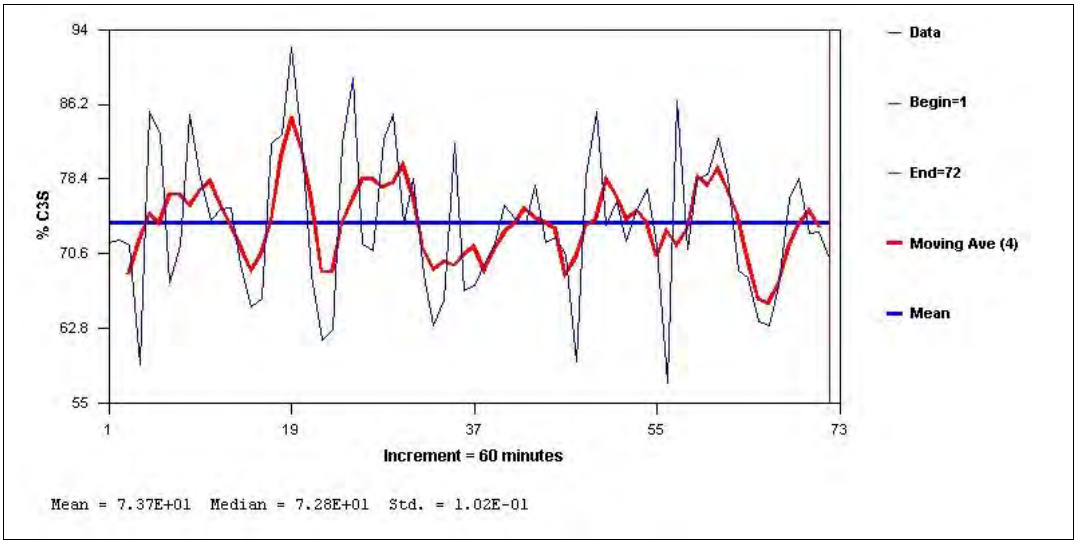


Figure 18.2: A 4 point window for the Moving Average of %Chemical Index in the raw mill. N is the number of points used in the moving average (40 FF Pitard)

In the variability of the original data and the moving average are shown together in the second graphic (***Figure 18.2***).

c. **Third Graphic: Random noise and corrected data (very useful in process control):**

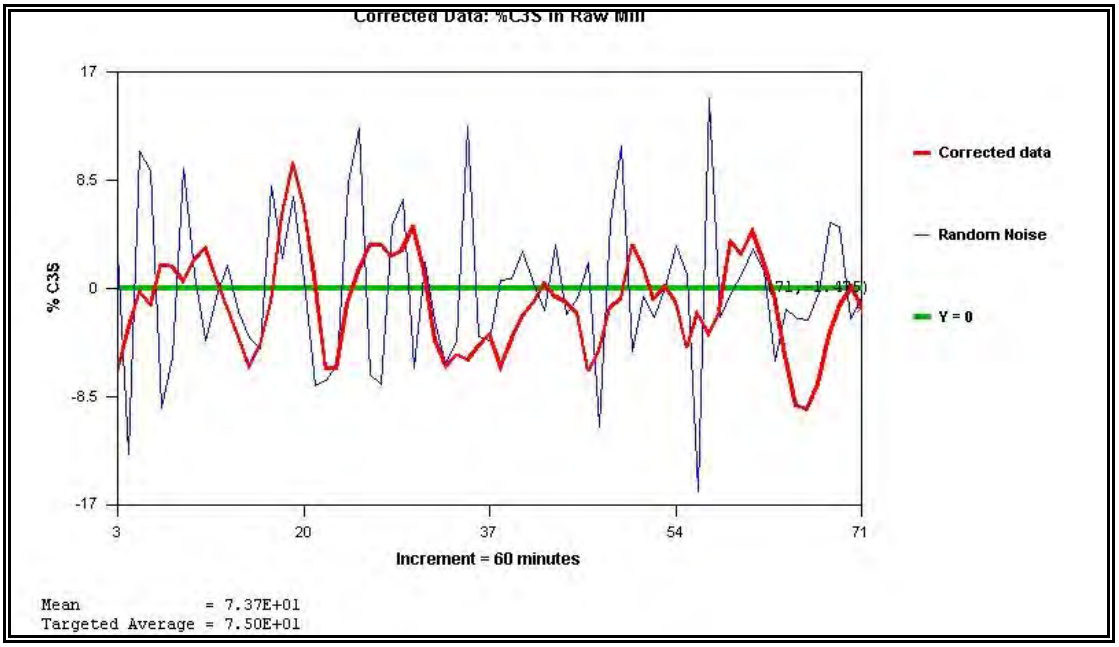


Figure 18.3: Moving average superimposed on the Random Noise (40 FF Pitard)

A continuous curve for Random Noise at a steady level without any indication of a trend is the ideal result. If there is any indication of a cyclical trend in the RN across the zero line then there is a possibility that the window is too large and should be reduced.

2. Incorrect Use of the Moving Average

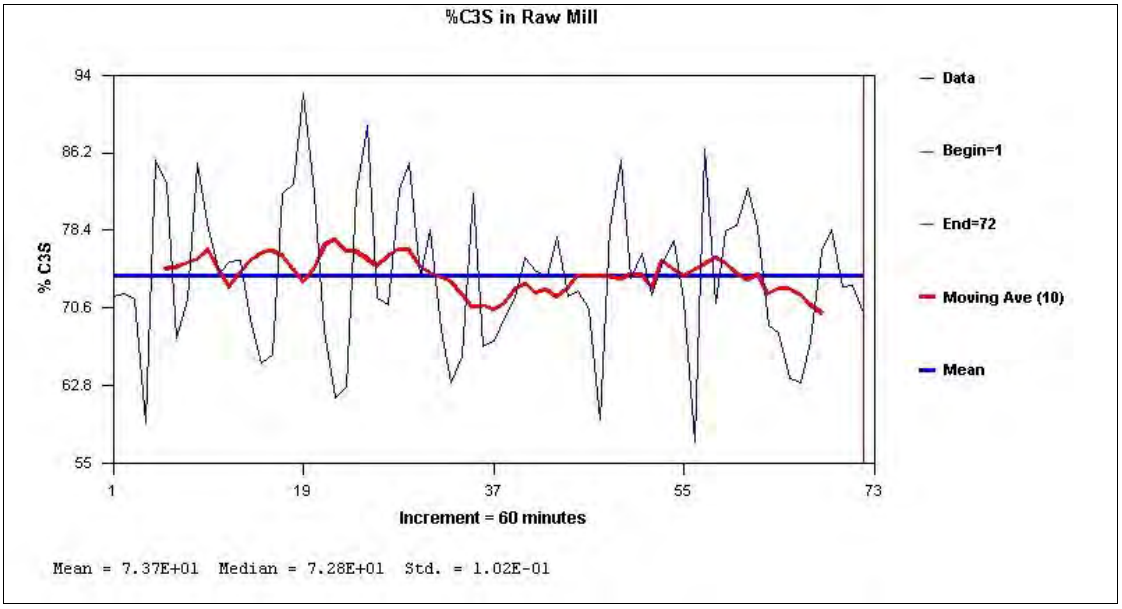


Figure 18.4: Cyclical trends that move across the zero line indicate that the window of the moving average is too wide and must be reduced. ([40] FF Pitard)

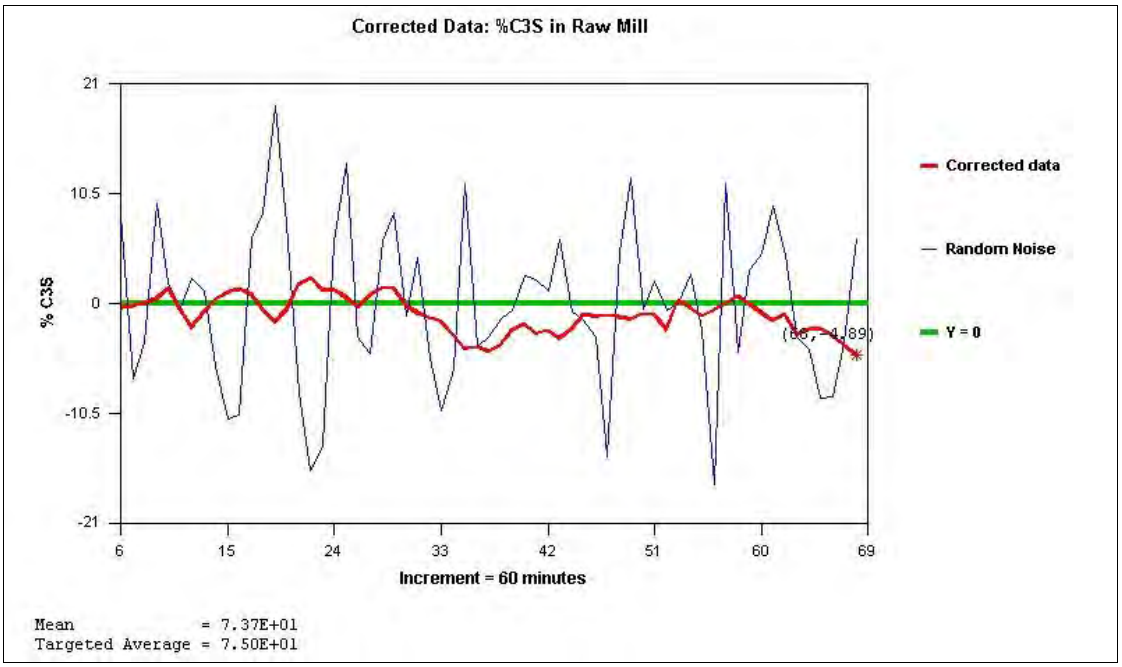


Figure 18.5: Deviation from the zero line indicates the amount of correction that must be added to the system. At zero no process correction is required. *([40] FF Pitard)*

A corrected data curve is derived by subtracting the moving average curve from the raw data (RN = a_m – MA). The curve should vary around the arithmetic average in a consistent manner. If there is any indication of a cyclical trend then the moving average is too wide and one must start again.

3. An Application of the Moving Average: The Relative Difference Plot

The relative difference plot is a powerful yet simple graphic tool where one can utilise duplicate sets of samples. On such graphs it is easy to detect biases, discuss their significance and follow their evolution.

Check assays should be taken every 20th sample resulting in sufficient data being available within a short space of time. A thousand samples will give 500 pairs, adequate for a stable analysis. The plot has the following features:

a. **Step 1:** On the vertical axis lies the relative difference between two measurements being compared i.e. two laboratories or duplicate analyses, A and B.

$$\%\text{Relative difference} = 100 \times \frac{a-b}{\frac{a+b}{2}}$$

On the horizontal axis lies the average of A and B: [A + B] / 2 is sorted by chronological order, or by increasing values of [A + B] / 2

b. **Step 2:** The most important feature, the moving average (N = 20) is then plotted.

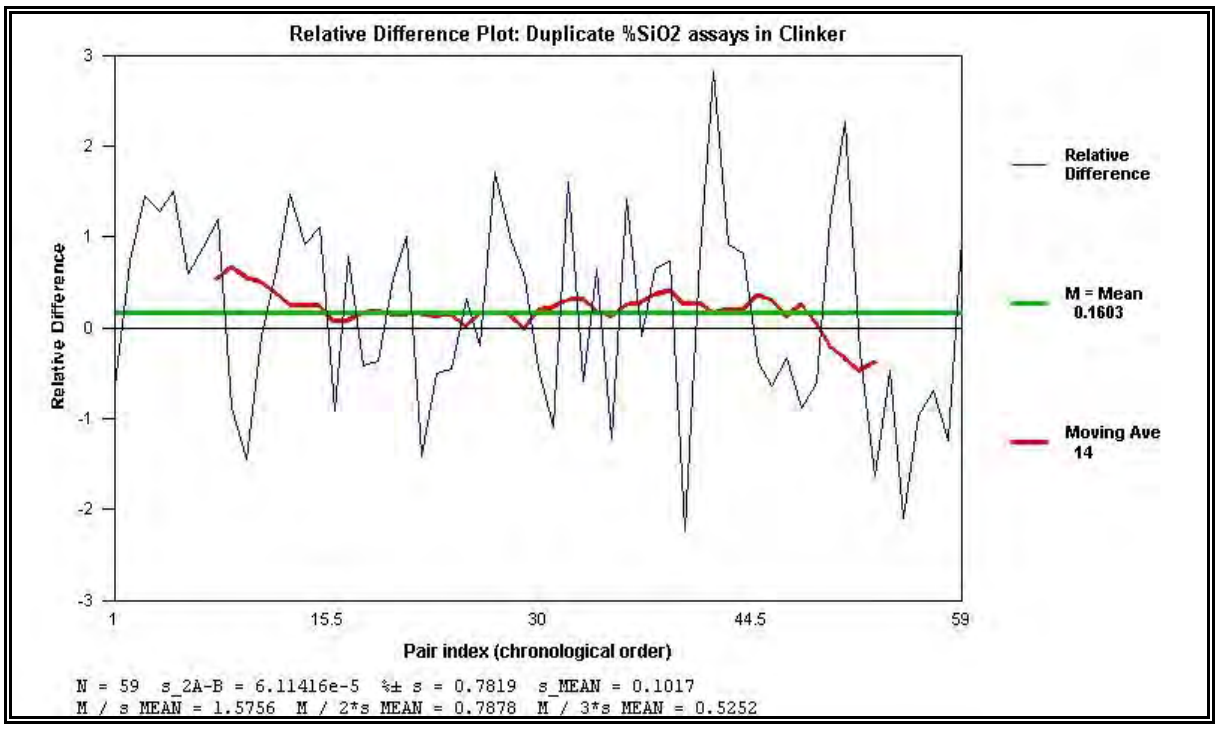


Figure 18.6: Example of a relative difference plot; a bias is clearly indicated *([40] FF Pitard)*

The downturn in the tail of the curve in ***Figure 18.6*** exactly compensates the upturn at the beginning of the cycle. If the horizontal axis is sorted by increasing content, the curve behaves as shown. The mean value (shown in green in ***Figure 18.6***) lies above the zero line, providing clear evidence of a bias, particularly a conditional bias, associated with the higher grades. One might ask: "What is a good correlation coefficient? Is it 0.94, 0.97 or 0.98?" Using the formula:

$$A = \sqrt{1-(\rho)^2} = \sqrt{1-(0.97)^2} = 0.17$$

Thus about 17% of the pairs are suspicious. If 25% are suspect at ρ = 0.97 there is a major problem with the precision ellipse. For this formula to work correctly a fairly large range is required.

4. Application: Beware of Correcting Factors

Incorrect sampling and poor measurements may introduce biases into a data base as indicated in the Relative Difference plot shown in ***Figure 18.7***. It is tempting to adjust biased data by using correcting factors based on observations and experiments. Unfortunately, there is no such thing as a constant bias in sampling. Therefore, filtered information, elimination of embarrassing figures and the use of correcting factors are symptomatic of poor management. Correcting factors give a false sense of security and merely hide the problem if one attempts to make adjustments.

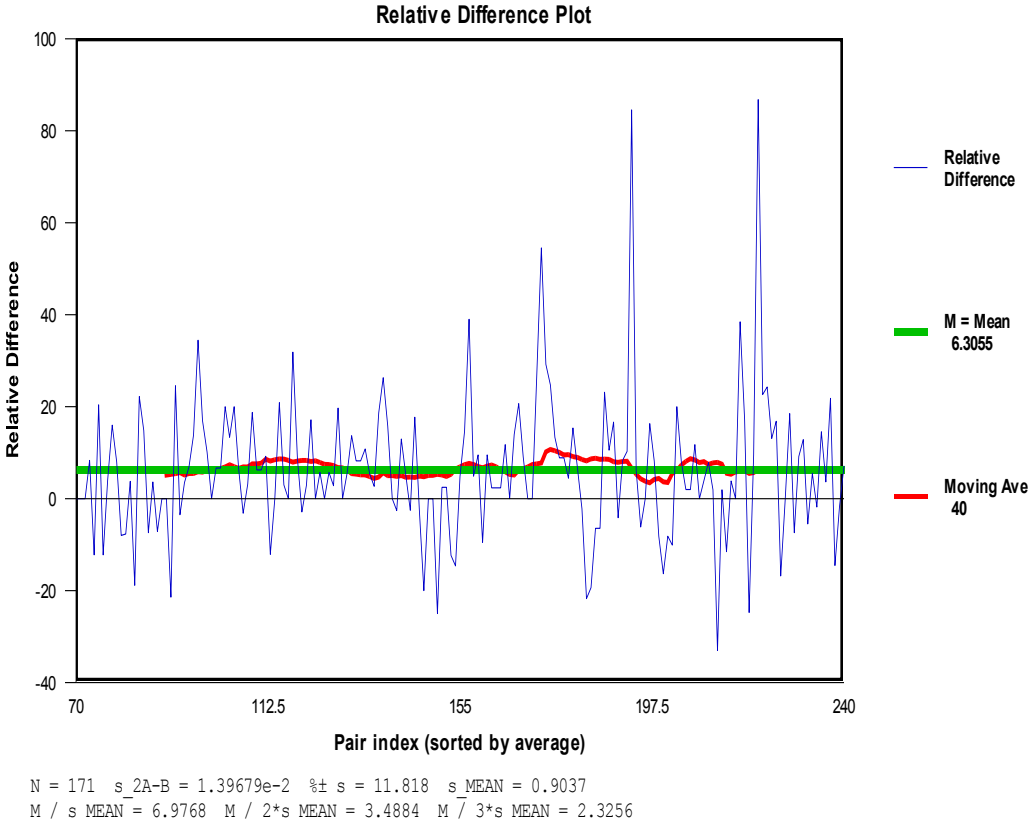


Figure 18.7: Relative difference plot showing an example of an analytical bias *(^{40} FF Pitard)*

If the moving average stays fairly close to the mean value without veering off in either direction, as in ***Figure 18.7,*** the bias is more or less constant and is likely to be an analytical bias. On the other hand, where the moving average takes major swings around the mean value there is almost certainly evidence of significant sampling bias or several different types of bias superimposed on one another.

Evidence of a sampling bias is shown in the Relative Difference plot shown in Figure 18.8. This is clearly not the footprint of an analytical bias, but rather the indication of a sampling bias. The variability in the moving average plot is a clear indication of this.

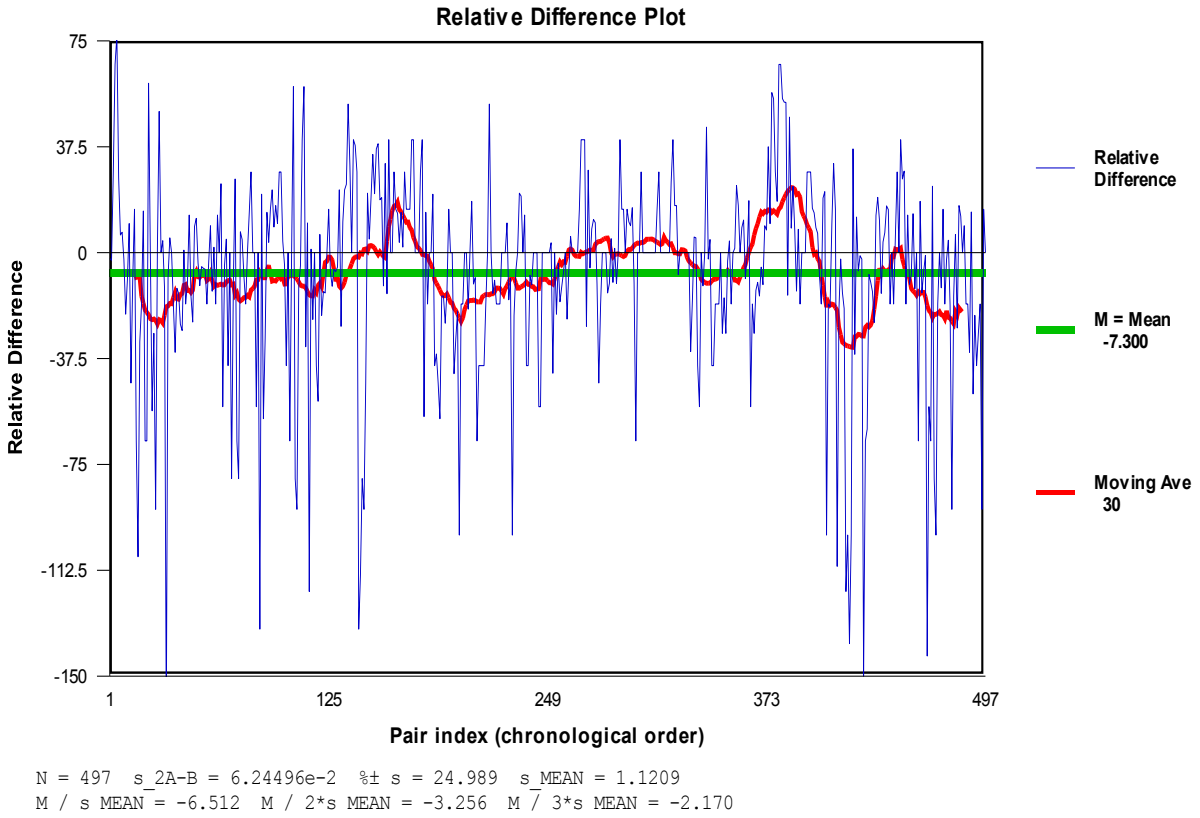


Figure 18.8: Relative difference plot showing an example of a sampling bias *(^{40} FF Pitard)*

During process control the system is almost always under-sampled giving rise to major fluctuations around the mean. Metal recovery and run of mine ore grades display a variability which is correlated. However, at maximum capacity the plant will have poor recovery at the spikes because it has no capacity to absorb these.

In a copper plant the following data was collected:

Before adjustments to the plant		
Range, (variability)	± 50%	±150%
Recovery	82%	65%
After adjustment to the plant		
Variability (Range)	25%	75%
	85%	±80%

CHAPTER 19 - INTRODUCTION TO VARIOGRAPHIC STATISTICAL PROCESS CONTROL (SPC)

1. Chronostatistics

Variographic SPC is a natural result of sampling theory based on the important concept of material heterogeneity. There is no effective process control possible without addressing all the different kinds of heterogeneity. This is the superiority of variographic SPC over conventional SPC. This new science is called Chronostatistics and is based on information gleaned from variograms resulting in a variogram-based hierarchy of control limits:

Limits quantifying pure noise: UCL and LCL = $\pm 3\sqrt{V[0]}$

+ process trend allowance: UCL' and LCL' = $\pm \{ (3\sqrt{V[0]}) + \sqrt{V_2}[j=1] \}$

+ process cycle allowance: UCL" and LCL" = $\pm \{ (3 \sqrt{V[0]}) + \sqrt{V_2}[j=1] + \sqrt{V_3} \}$

Rationale: Investigate the various sources of variability

Dr. W. Edwards Deming clearly demonstrated in several experiments that one should not react to variations within $\pm 3\sqrt{V[0]}$. Indeed, there is no logical reason why anyone should react to variability that does not exist in a process (i.e., UCL and LCL). This concept allows us to optimise the sampling/measurement protocol. An active on-going reaction philosophy should be established to correct all true process movements, when they are large enough to show, through random noise. For these movements one must be ready to apply corrective methods.

Variations characterised by $V_2[j=1]$ are true process movements, predictable over a certain time frame and potentially correctable. Therefore, one doesn't want to let variations become larger than $\pm \{(3\sqrt{V[0]}) + \sqrt{V_2}[j=1]\}$ without reacting (i.e., UCL' and LCL'). However, adding $\sqrt{V_2}[j=1]$ to the limits UCL and LCL, to obtain UCL' and LCL', makes a calculated allowance for process movements that are outside one's control. Indeed, one is unable to react at time intervals shorter than j =1. This concept allows optimisation of the sampling/measurement interval.

Adding $\sqrt{V_3}$ to the limits UCL' and LCL', to obtain UCL" and LCL", makes a calculated allowance for a process cycle beyond one's control. It is very difficult to effectively correct a cycle. If this addition becomes too large, it is necessary to investigate the causes of the cycle. Sometimes engineering modifications of a process are necessary to eliminate a cycle. Sometimes a cycle is introduced through the manner in which one's work is organised. Whatever the case it is essential that one should know the cycles. They are always an opportunity to minimise a visible or invisible cost or improve the process.

2. Case study at a cement plant;

This example appears in the 1992 work of Pitard[Error! Bookmark not defined.], in which he describes a cement plant in the USA producing small quantities of a highly specialized product. Every now and then the company

has to reject a full day's production because it is out of specification, but they didn't understand why. The cement plant preparing special cement must comply with the following three specifications:

- The raw mill must have an average of 5.50 %+63μ particles.
- The upper specification is 5.80%.
- The lower specification is 5.20%.

The process is controlled by taking a composite sample every two hours, the results of which are shown in Table 19.1. It is necessary to perform a full variability investigation of what happens within a typical 120 hour time window. An interesting point feature of this data is that not one point fails the specification criteria so it is essential to graph the data (***Figure 19.1***) in order to see the main problems of the chronological data and to resolve the components of variability. Understanding these components is critical.

i. Interpret the raw data using the table of data.
ii. Interpret the raw data using a chronological plot.
iii. Study the absolute variogram, step by step.
iv. An experiment is necessary: Find out which.
v. Study the variographic control chart, step by step.

Table 19.1: Analyses of cement grain size: J = 1 = 2 hours; Results in % +63μ

Sample	%	Sample	%	Sample	%	Sample	%	Sample	%	Sample	%
1	5.61	11	5.61	21	5.52	31	5.55	41	5.41	51	5.48
2	5.69	12	5.6	22	5.55	32	5.52	42	5.44	52	5.48
3	5.66	13	5.55	23	5.52	33	5.49	43	5.47	53	5.49
4	5.57	14	5.5	24	5.59	34	5.49	44	5.55	54	5.42
5	5.56	15	5.49	25	5.61	35	5.43	45	5.57	55	5.41
6	5.6	16	5.51	26	5.61	36	5.48	46	5.58	56	5.37
7	5.53	17	5.57	27	5.55	37	5.4	47	5.53	57	5.36
8	5.58	18	5.52	28	5.52	38	5.41	48	5.52	58	5.4
9	5.53	19	5.51	29	5.57	39	5.45	49	5.48	59	5.36
10	5.49	20	5.51	30	5.57	40	5.43	50	5.44	60	5.33

a. **Existing chronological data:**

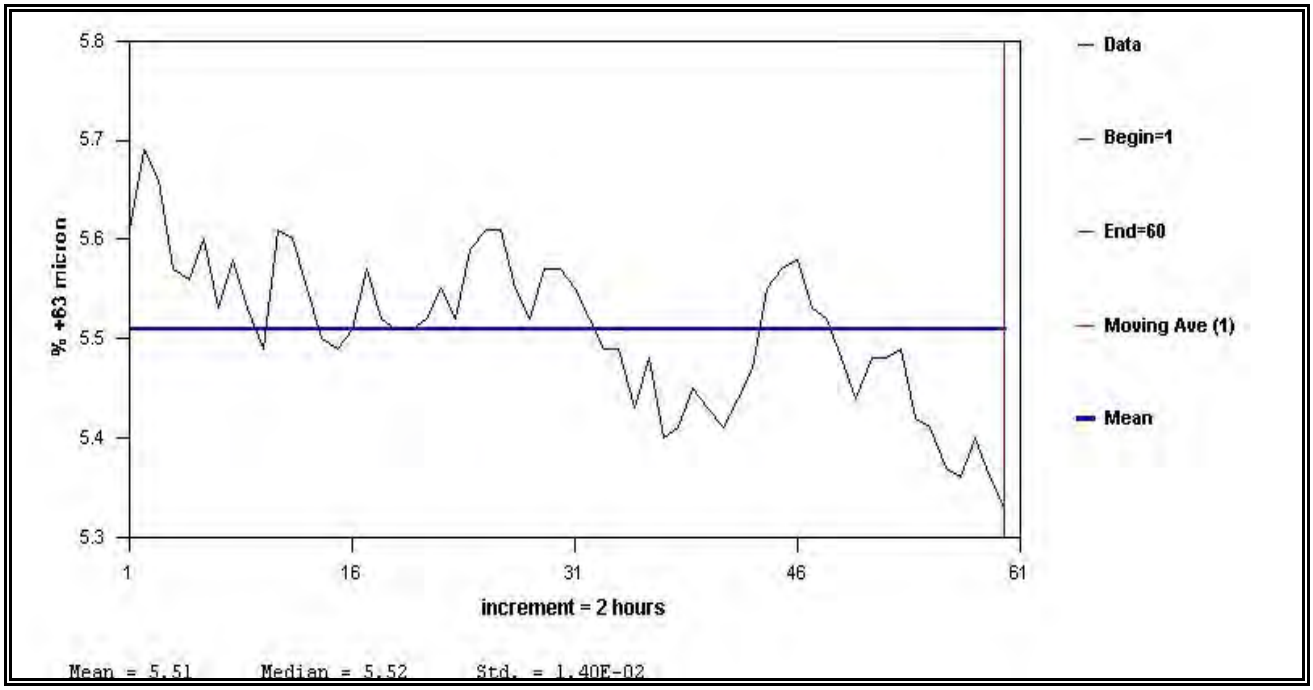


Figure 19.1: %+6 3micron particles in a special cement ([40] FF Pitard)

b. **Estimation of V[0], V[1] and V[process j = 1]**

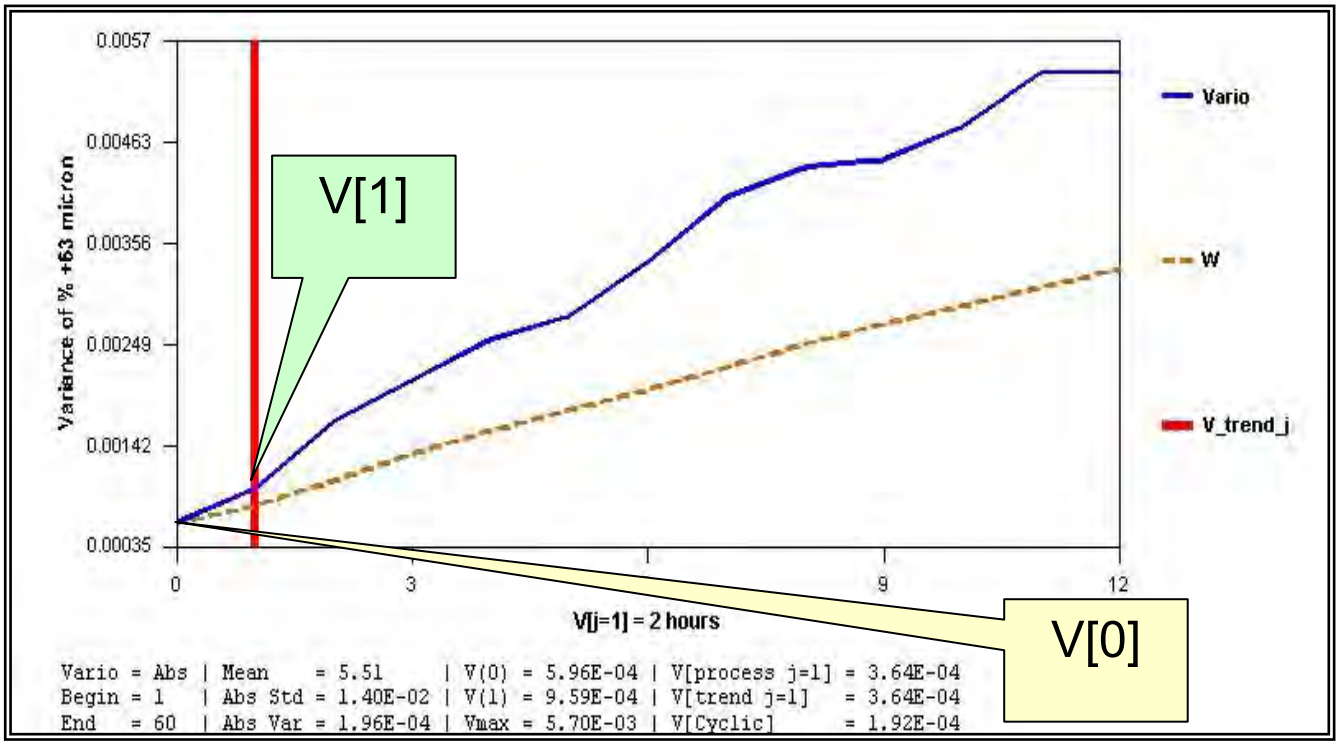


Figure 19.2: An absolute variogram for a 24h period: %+63 microns particles in special cement showing the First Order integral (W) that gives V[0], This information is used later. ([40] FF Pitard)

c. **The cyclic component: Estimation of V_3**

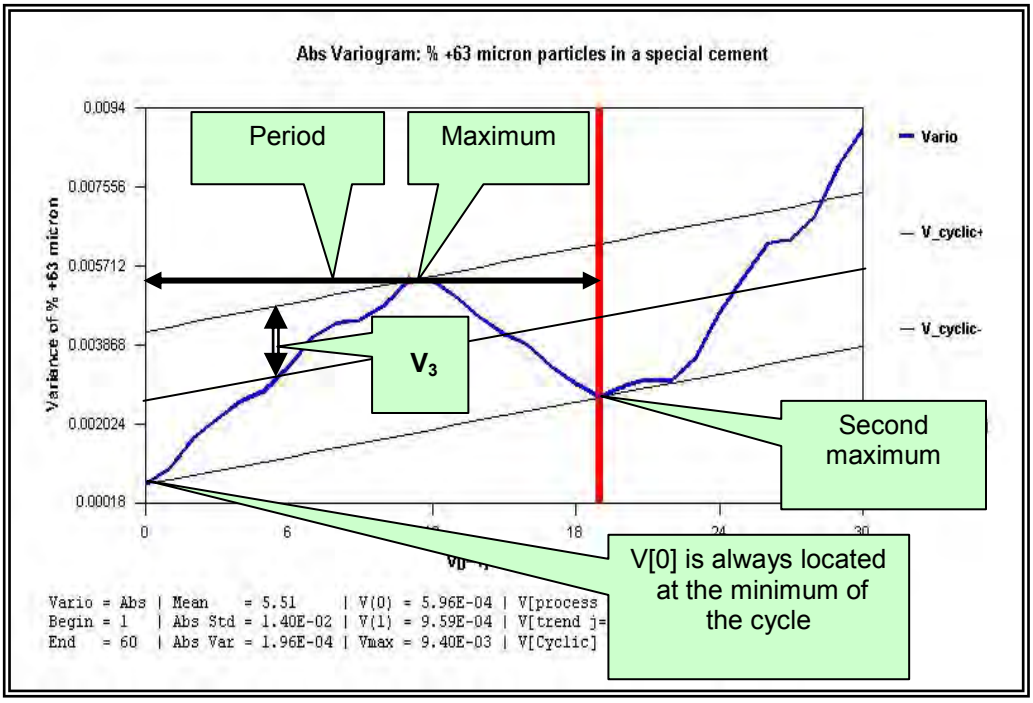


Figure 19.3: Absolute variogram showing the data to be perfectly cyclical ([40] FF Pitard)

When there is a cycle V[0] is, by definition, at the minimum point in the cycle. The Process Trend V_2 [j] is a relative concept that depends on the time scale of observation. The scale is defined by the value given to j.

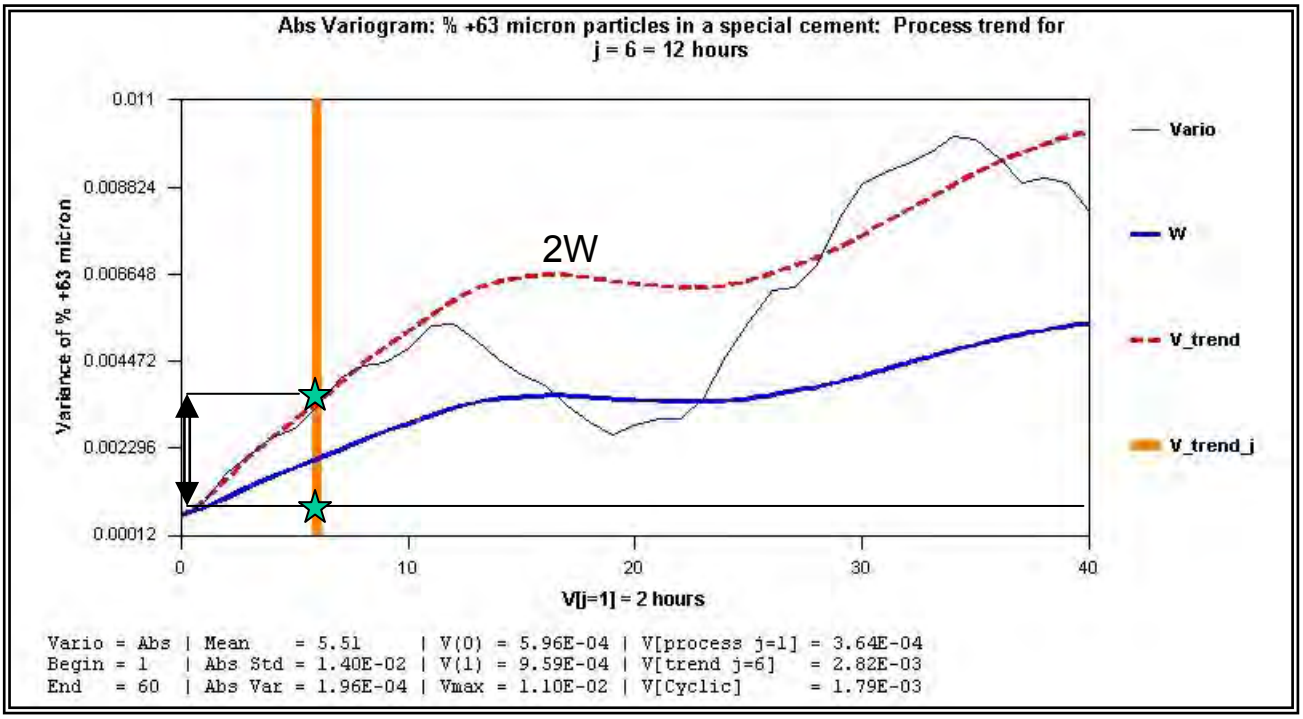


Figure 19.4: The difference between INE and the variogram indicates the process drift ([40] FF Pitard)

When the variogram increases rapidly for a given value of j, the curve 2W or 3W'is the best estimate of the process trend. For values of j, beyond which the variogram reaches its maximum, the curves 2W and 3W' become meaningless and should not be used. It is then advisable to use W or W'. There is obviously a

transition zone in which the software user must be cautious since curves 2W or 3W' would lead to an over-estimation of the trend and curves W or W' would lead to an under-estimation.

d. **Process trend V_2[j] estimation: Limitations of curves W and 2W:**

The same could be said about curves W' and 3W'.

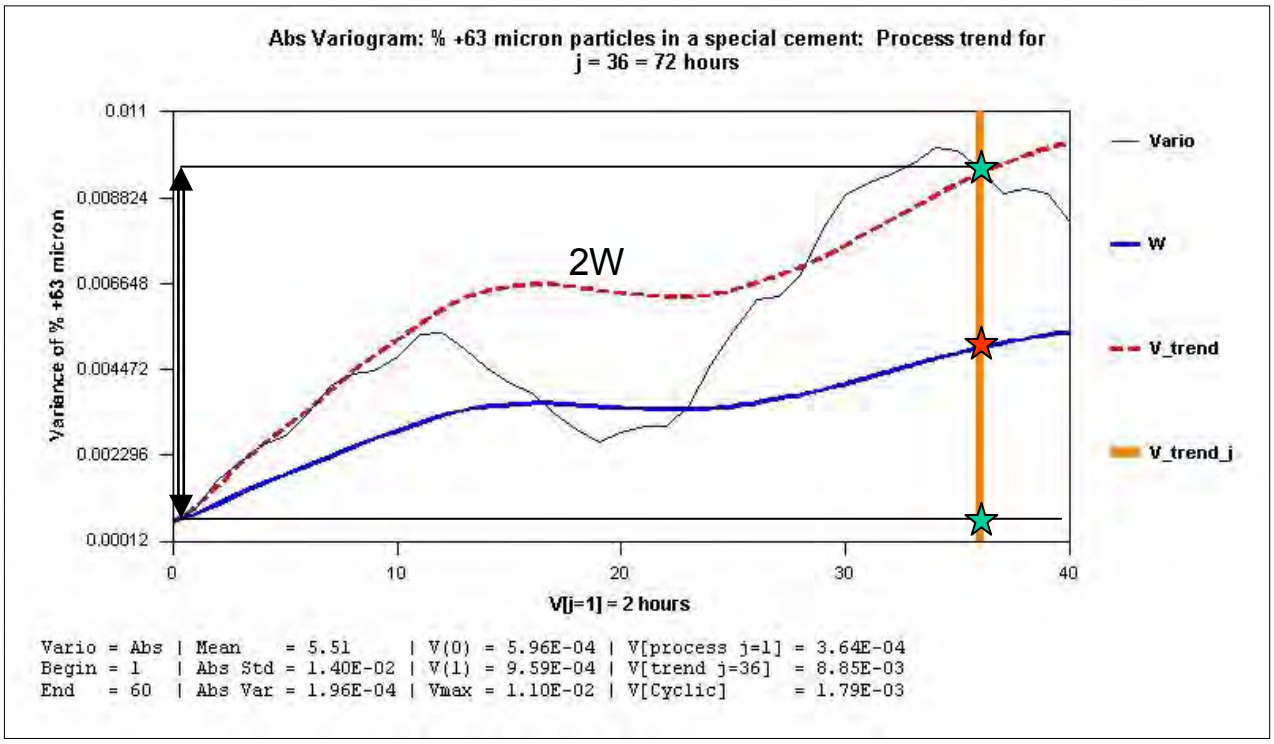


Figure 19.5: Process trend V2[j] estimation: Limitations of curves W and 2W ([40] FF Pitard)

In this case, one may approach the maximum of the variogram at j=36. Therefore the estimation of V_2[j=36] may be slightly over-estimated when using curve 2W, whilst curve W is obviously under-estimating. All the variance observations are summarised in the pie chart (***Figure 19.6***).

e. **Summary of Information: The Pie Chart**

The pie chart shown in ***Figure 19.6*** simply provides a means of comparing the relative contribution of the various sources of variability as identified in the variogram. This problem arises because plant superintendents try to control the process without understanding that there is a cycle within the process. Attempts are made to correct the process, but this occurs at the worst possible time resulting in an over-correction. If a cycle is not well understood it is very difficult to control variability in the process and ultimately the trend cannot be controlled because there is no awareness of the cycle: V[trend] / V[cycle].

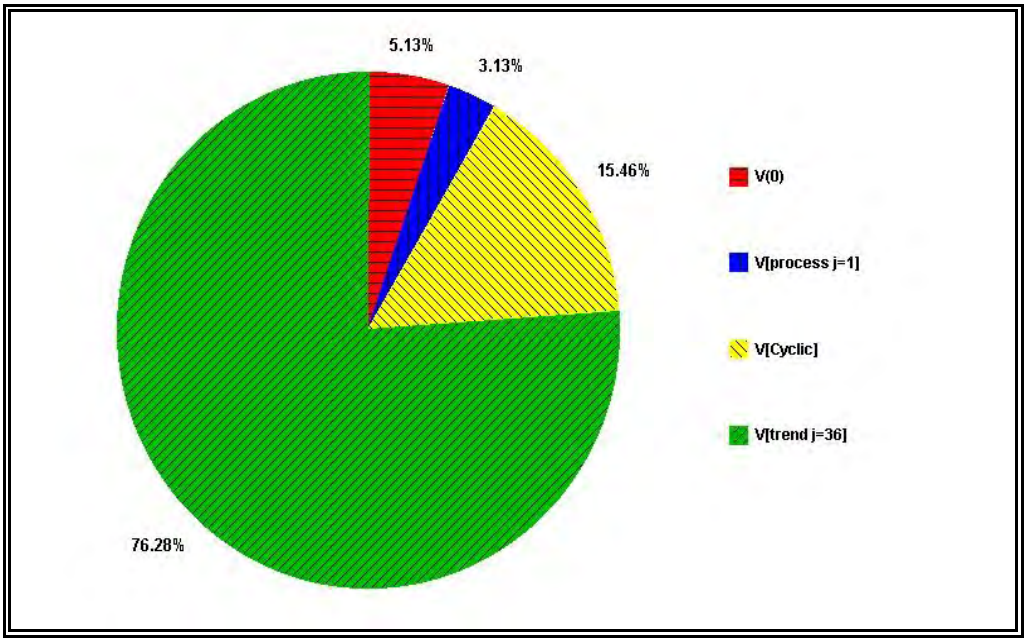


Figure 19.6: Variability components %+63 micron particles in special cement (^{40} FF Pitard)

3. Variographic Experiment: Chronological Data

Why is control of the process trend such a problem in this case study? An experiment to verify the accuracy of V[0] and V_2[Process j =1] was conducted, the data being given in Table 19.2, in an attempt to quantify the variability that was occurring in the 2hr interval between sampling events. To calculate the true random variability V[0] 30 samples are collected at ten-second intervals, under the exact same conditions as for routine samples. Sub-sampling and analytical protocols are also kept exactly the same as for routine samples. The results in ***Table 19.2*** were obtained and the data variability is depicted in ***Figure 19.7.*** The variogram of the data is shown in ***Figure 19.8.***

Table 19.2: Data collected from 30 samples

Sample #	% +63µ	Sample #	% +63µ	Sample #	% +63µ	Sample #	% +63µ	Sample #	% +63µ	Sample #	% +63µ
1	5.45	6	5.52	11	5.47	16	5.46	21	5.52	26	5.48
2	5.52	7	5.51	12	5.5	17	5.5	22	5.47	27	5.51
3	5.46	8	5.47	13	5.52	18	5.53	23	5.51	28	5.5
4	5.45	9	5.52	14	5.54	19	5.49	24	5.5	29	5.53
5	5.47	10	5.48	15	5.48	20	5.48	25	5.45	30	5.47

4. Plot of raw data versus time

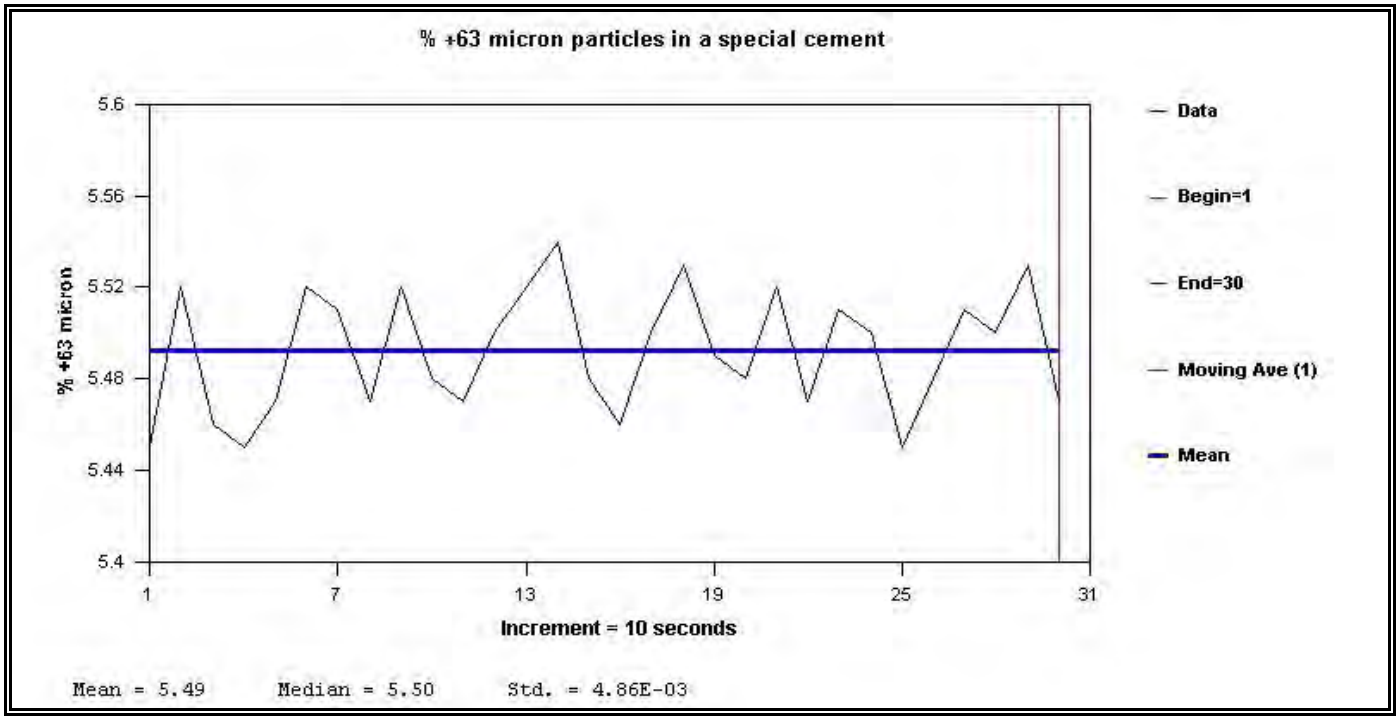


Figure 19.7: %+63 micron particles in cement: Results of the 10sec interval sampling experiment. No cycle is evident. Variability is due to random background noise only ([40] FF Pitard)

5. Short-term variographic experiment

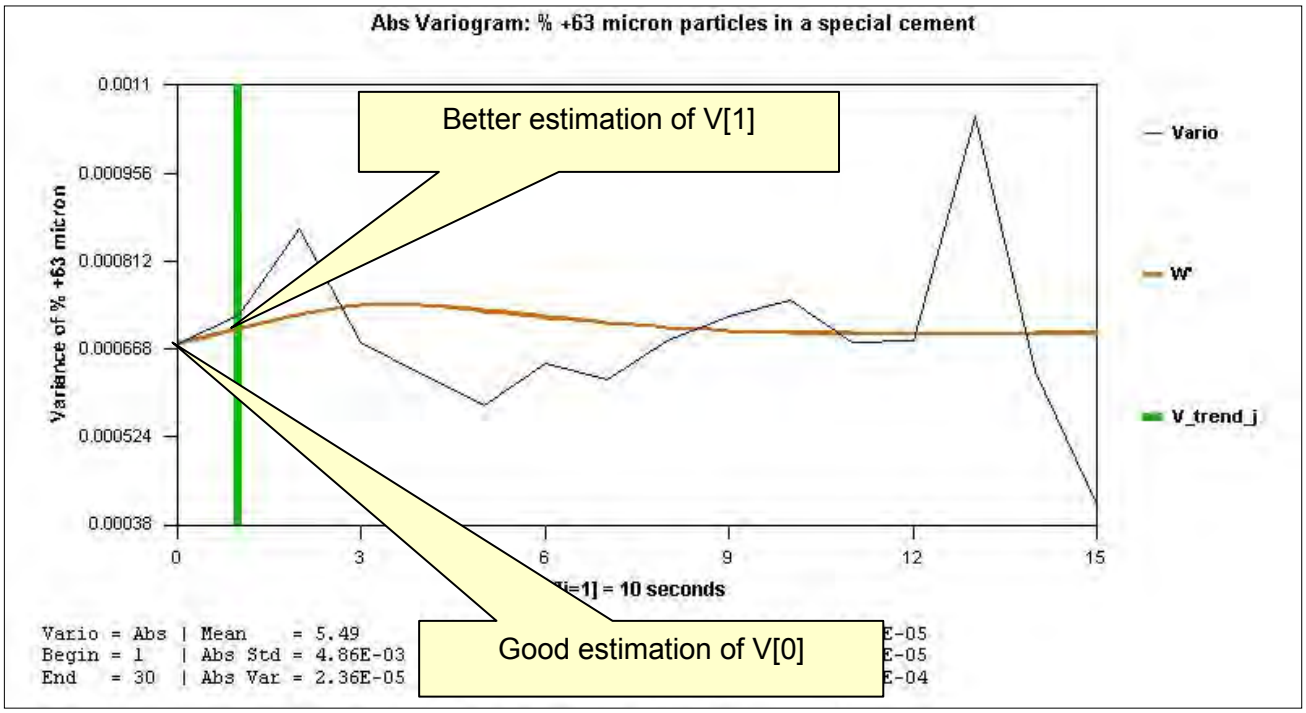


Figure 19.8: Values for V[0] and V[1] can be easily determined from the second order integral.

6. Definition of contractual specifications

The following data is derived from the variogram: Mean = 5.49, V[0] = 0.000676 (%+63 micron)^2 and V[1] = 0.000724 (%+63 micron)^2 The challenge now is how to apply this information. The way the information is

used and applied will, of course, depend on DQO. Without DQO there can be no determination of specifications or targets. Specification limits are guidelines to satisfy a contract with a client, or to ensure the optimum performance of a process. They have nothing to do with the actual variability of a process. Specification limits are nothing more than a wish list. Firstly, define:

- **US:** The Upper Specification
- **TA:** The Targeted Average
- **LS:** The Lower Specification

Control limits are not to be confused with specification limits. Process variability must be understood first and thereafter set reasonable and realistic control limits which allow one to investigate the process correct it if necessary and maintain it within contractual specifications. If the process is well understood, contractual specifications often become self-imposed specifications for a given process parameter.

a. Definition of process control limits

Process control limits were defined in the section of variography (Chapter 19). They are tools which enable the process to give feedback and minimise the negative effect of all sources of variability which cannot be controlled.

Calculate values for the following limits (***Table 24.2***) and superimpose them onto the variability diagram:

UCL and LCL: Upper and Lower Control Limits quantifying the variability component 1, 2, or 3 $\sqrt{V[0]}$

i. UCL and LCL = $5.49 \pm 3 \times 0.026 = 5.49 \pm 0.078 = 5.568$ or 5.412
ii. UCL' and LCL': Quantifying the variability component {1, 2, or 3 $\sqrt{V[0]}$} + $\sqrt{V_2}$ [j=1]
iii. UCL" and LCL": Quantifying the variability component {1, 2, or 3 $\sqrt{V[0]}$} + $\sqrt{V_2}$ [j=1] + $\sqrt{V_3}$

These variability components can be changed as follows:

iv. UCL and LCL: Improve the sampling/measurement protocol.
v. UCL' and LCL': The above and taking samples/measurements more often.
vi. UCL" and LCL": The above and eliminating or minimising the process cycle. This may require engineering modifications.

Table 19.3: Calculation of variability limits for V[0], V[1] and V[cyclic]

Targeted average (TA)	5.50	3 sigma			
Mean	5.51				
US		5.80	LS		5.20
UCL		5.58	LCL		5.44
UCL'		5.60	LCL'		5.42
UCL''		5.66	LCL''		5.36

b. Select a realistic wish list:

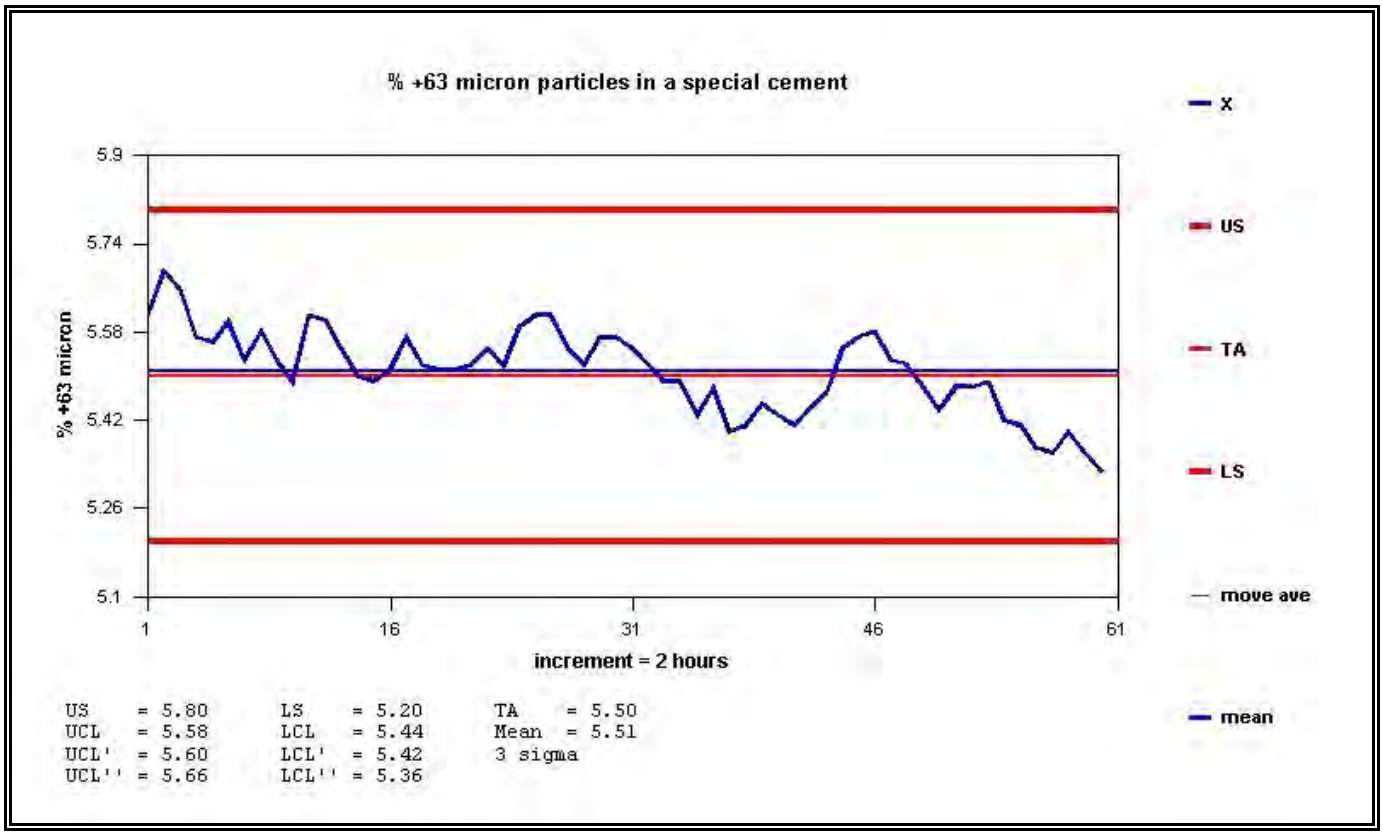


Figure 24.9: Particle size is within specification limits ([40] FF Pitard)

c. Check the capability of the sampling/measurement protocol

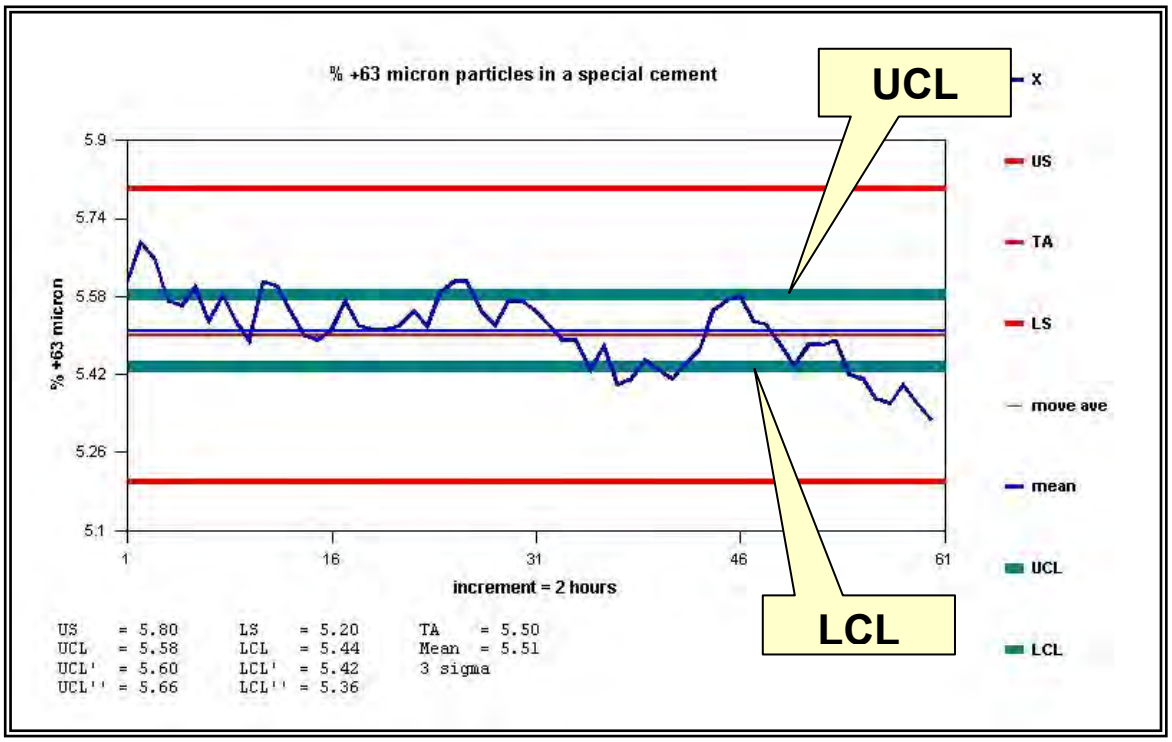


Figure 19.10: Several points lie outside the limits set by the upper and lower control limits ([40] FF Pitard)

d. Check the capability of the sampling/measurement interval

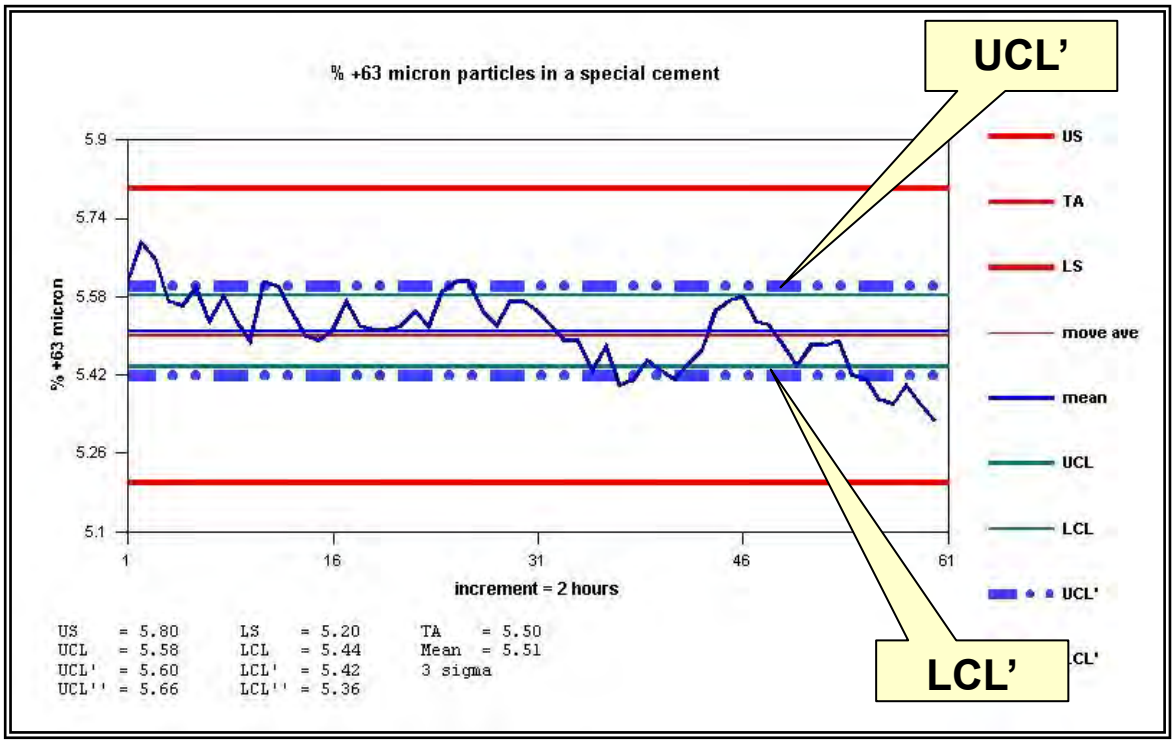


Figure 19.11: Limits on variability as set by the sampling interval are now superimposed; very little difference between these and the upper and lower control limits ([40] FF Pitard)

e. Check the impact of the time-stable cycle

This really is the area with the biggest problem even though the process has a lot of room to maneuver inside the limits set by the time stable cycle. Provided the variability in the process remains between the two limits defined by the time stable cycles, all will go well and the process can be left alone to manage itself. Once the process approaches the time stable limits the operator tries to adjust the process and over-corrects. This type of variability was introduced by a poorly designed feeding silo which, when corrected, resulted in much better process stability. It took several days before the source of the variability was identified, but once identified and corrected, the process was fine.

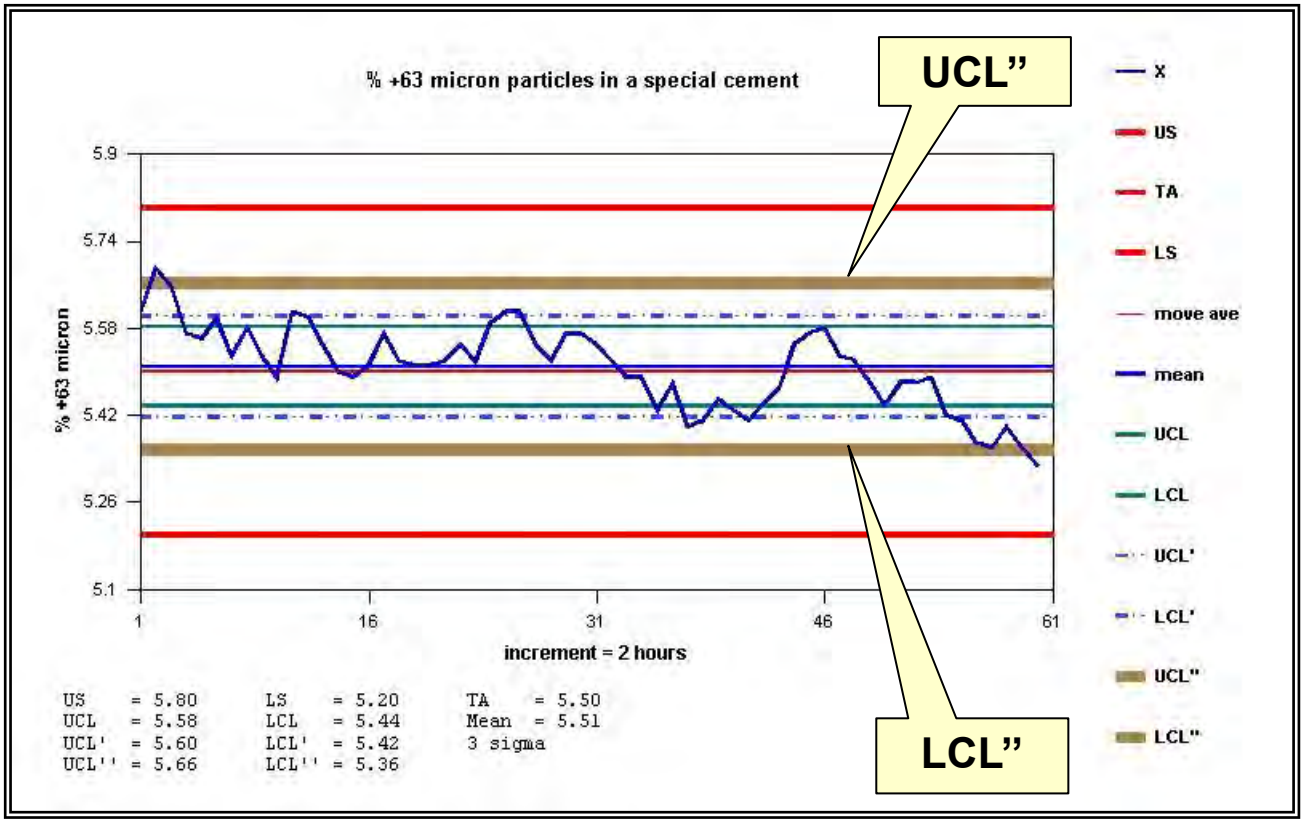


Figure 19.12: Superimposed time stable cycle (^{40} FF Pitard)

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CHAPTER 20 - SUMMARY: PITARD'S 18-POINT STRATEGY

1. Sources of Ore Grade Reconciliation Problems

a. Geological model: Continuity and interpolation of geological boundaries and domains can be major sources of errors and uncertainties associated with a model. These aspects depend mainly on the information obtained primarily from drill holes. The density of drill holes will, to a large extent, be determined by the range of influence gleaned from a semi-variogram of available data. Continuity of the ore body and uncertainty may be severe when contacts or other geological information is extrapolated to the point that lines intersect other known contacts.

Geological, mineralogical and economic boundaries; fault lines, trends and transition zones must be fully understood before geostatistical estimations can be undertaken. The nature of boundaries (hard, soft or gradational) must also be understood. The extent, accuracy and ability to select which blocks of ground should go to the mill and which to the waste dump is a major area of concern and only a correct sampling protocol and implementation thereof can constitute good practice. Confidence attributed to the selection of mining blocks is largely wishful thinking. Selection can be verified only through the use of duplicate samples and by plotting a precision ellipse.

Perhaps one important tool would be to establish a low-grade stock pile which could be sampled on a regular basis. Regular surveys of geological boundaries with consistent updates of the geological map of an open pit is essential if the knowledge that comes from sound geological insights is to benefit any mining operation. Another area which requires ongoing attention and consideration is the mineralogy of an ore body with respect to weathering and changes in sulphide composition with depth and lateral extent.

b. Logistics: More and more mining operations rely on the outsourcing of drilling, blasting and earthmoving activities. The activities of drilling contractors require particular monitoring with regard to drill-rig positioning and drill penetration rates. Inventory stockpiles should be evaluated as they are being built and when they are reclaimed. The temptation to purchase equipment which is inadequate in the hope of being ultra-selective should be avoided. Shovels, in particular, and trucks and other equipment must be compatible with the thickness of the mining bench. Water accumulation in an open pit operation can result in major losses if the level is not carefully monitored. Loss of metal in the form of dust can be severe and the extent of such losses must not be underestimated.

c. Sampling protocols: The first errors encountered are INE's. Comments on the coarsest minerals, indicator minerals and clusters of minerals should be made wherever these occur and should be related to the basic volume of observation. FSE should be determined and verified by the means suggested. Potential problems arising from GSE should be identified and evaluated. Sampling intervals should be optimised at the plant by identifying the presence and influence of cycles. Appropriate sampling modes will ensure that drilling is done at the appropriate intervals.

d. Implementation of a sampling protocol: Once the sampling protocol has been optimised it should be implemented. This should be accompanied by detailed attention to and minimisation of IDE, IEE, IPE and IWE. Equiprobable selection of sample material should be *the* main concern. Misplaced weightometers and sampling stations should be identified and appropriate steps taken to correct any problems.

e. Understanding variability: Time series analysis, variography and variability diagrams must be plotted and interpreted in order to identify random, non-random and cyclical patterns of behaviour in the plant. Run-of-mine variability must be determined through regular and duplicate sampling of the run-of-mine feed. The behaviour of high- grade material and the rates of recovery of metal in the plant from such high-grade ores should be monitored. High-grade and low-grade ores should be separated, wherever possible, rather than being sent to the plant without appropriate blending. If high-grade base-metal ores are fed to the mill there is and almost immediate decline in the rate of recovery. Cut-off grades should be accompanied by a statement regarding the expected levels of uncertainty associated with them. The application of correction factors should be interrogated and queried before being accepted. Finally, the variability associated with the heave of material during blasting in an open-pit operation should be monitored and accommodated for when doing reconciliations.

f. Common measurement variability: Moisture content, density and AE require regular verification as they may change as the pit depth increases and the mine deepens.

g. Training: Ongoing training of geologists and miners in fields of sampling theory and geostatistics is essential. Metallurgists and chemists should be made aware of and be kept up to date with developments in chronostatistics and sampling theory. High-level accountants should be made aware of the dangers of being so preoccupied with visible costs that no cognizance is given to the staggering implications of reducing budgets on invisible costs.

2. Use Standards

a. Using ISO Standards as a framework/guideline: ISO standards can be used as guidelines to improve performance and efficiency in many areas. However, ISO standards are not adequate for sampling, geostatistics or chronostatistics. They are only logical, organisational guidelines. It is up to a company's senior management team to create standards in various areas, appropriate for the short-term and long-term interests of the company. These standards should include:

- Heterogeneity Tests to optimise sampling protocols
- Correct sampling equipment
- Mandatory check-lists for new sampling equipment proposed by engineering firms or manufacturers
- Preparation of standard reference materials
- Variability studies using existing data

b. Use ISO standards as a framework for implementing preventative maintenance: Trucks and shovels at mines, mills and floatation cells at the plant need preventive maintenance along with:

- Weighing systems, such as balances and weightometers
- Sampling stations
- Sampling systems on drilling machines
- Sampling equipment at the laboratory
- Sampling equipment for process control
- Analytical equipment at the plant and the laboratory
- Training of supervisors and managers

c. Training Courses: Typical training courses at a mine site, given up to three times a year, may include:

- Communication, presentation, assertiveness skills
- Report writing
- Time management basics
- Effective meetings
- Supervision
- Negotiation skills
- First aid
- Safety
- 6 sigma, SPC and TQM

The following courses are rarely offered but should be, at least once a year:

- Statistics, geostatistics, chronostatistics
- Sampling theory and practice
- Identification of invisible costs

7. Do Not Misuse Standards

A strong feature of ISO and other standards is their flexibility in being able to include additional elements into a standards manual. Additional elements, such as correct sampling, SPC, TQM, geostatistics and chronostatistics should ensure that standards are not misused. The symptoms of misuse are:

- Status quo is the rule
- Employees loose their creativity
- Employees use standards as a shield to justify their actions or of the lack thereof
- The certification agency runs the company,
- Management pursues only certification,

- Too much emphasis on metallurgical accounting.

4. The Three-Legged Table

It is good to pursue standards' certification, as long as it helps the company organise in very important areas. This leads to the concept of the three-legged table:

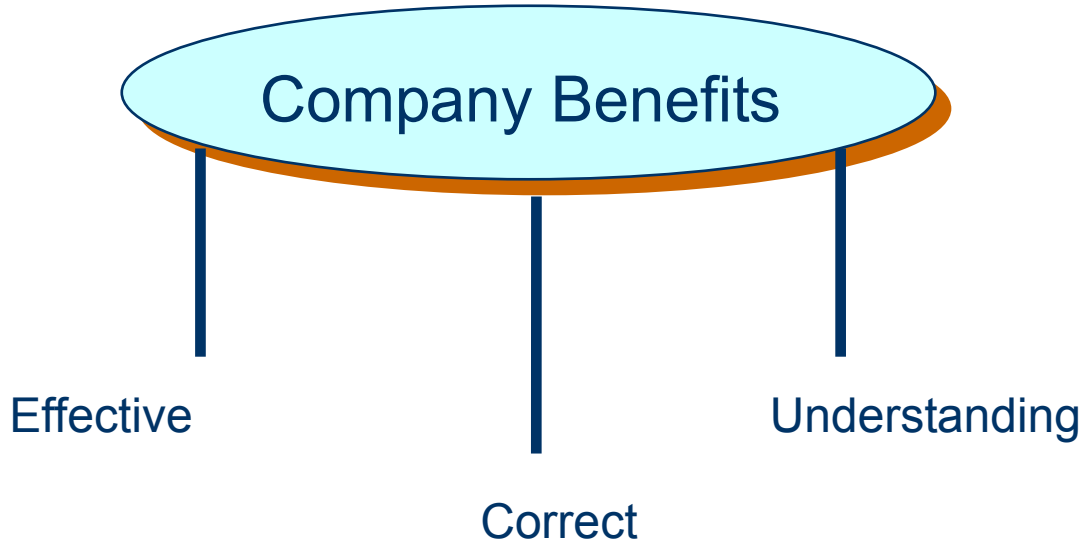


Figure 20.1: Three-legged table([40] FF Pitard)

a. Leg 1- Effective management:

- Identify structural problems
- Invest in solutions to structural problems
- Identify circumstantial problems (time and money can be saved)
- Understand and act on all sources of variability
- Be proactive instead of reactive

b. Leg 2 - Correct sampling & measurement:

- In-Situ INE
- Fundamental Sampling Error (FSE)
- Grouping & Segregation Error (GSE)
- Increment Delimitation Error (IDE)
- Increment Extraction Error (IEE)
- Increment Preparation Error (IPE)
- Process Integration Error (PIE_2)
- Periodic Integration Error (PIE_3)
- Increment Weighting Error (IWE)

- Analytical Error (AE)

c. Leg 3 - Understanding variability:

- Variability generates both visible and invisible costs
- Variability is both a problem and an opportunity
- There are many types of variability
- All sampling errors magnify variability
- Geostatistics analyses variability at the mine
- Chronostatistics analyses variability at the plant
- Minimise variability with a constantly improving strategy

d. **Integrate good sampling, SPC and TQM into a single program:** Sampling, SPC and TQM must be carefully integrated into one common program yet, more often than not, they are erroneously kept as separate programs, handled by different departments which rarely communicate with each other. If one leg of the table is broken the table is useless. It is common for companies to be supported on one leg only – reactive management – making it impossible to optimise the recovery of natural resources.

d. **Implement a Philosophy of Equiprobalistic Sampling**

Ore grade control and metallurgical accounting must implement equiprobabilistic sampling, giving an equal chance of selection for all material in a lot to be sampled. There are four relatively unknown sampling errors which are notorious for introducing the largest biases during exploration, at a mine, mill, smelter and laboratories. These are:

- Increment Delimitation Errors
- Increment Extraction Errors
- Increment Preparation Errors
- Increment Weighting Errors

Engineering firms and manufacturers of sampling equipment know very little about these errors and for many years they have built and installed sampling equipment which transgresses the most elementary rules of sampling correctness. Furthermore, standards committees don't understand these important sampling errors and this does not help the situation.

a. The Increment Delimitation Error:

- Example: during exploration
- Example: at the mine
- Recommended blast hole sampling
- Example: at laboratories
- Selecting the analytical sub-sample

- Examples: at the plant
- Correct sampling at the plant

b. The Increment Extraction Error:

- Good recovery is critical
- Recover all fines
- Example: at the plant
- Example: at laboratories

c. The Increment Preparation Error:

- Example: during exploration
- Example: at laboratories
- Example: of new technology

d. The Increment Weighting Error:

- Example: at the mine and mill

Furthermore weightometers are notorious sources of reconciliation problems. The accuracy of many weightometers is doubtful because of the locations in which they are installed and the manner in which they are cleaned, maintained and calibrated. Field experience proves them to be far less optimal than manufacturers' guarantee. Nevertheless, if a few precautions are taken weighing accurately with weightometers is possible. Four vital elements are necessary in order to implement a successful program:

***i. The initial installation of the weightometer needs to be correct so that*:**

- The weightometer and the conveyor do not exist independently of each other: It is necessary for engineering firms to design them together as an integrated system.
- The location selected to install the weightometer is critical, as it must be placed where the tension of the belt is least.
- The weightometer must be installed where the conveyor is horizontal.
- Run-out of idler rolls must be minimised.
- The angle of troughing idlers must be very accurate.
- Deflection of conveyor stringers must be minimised.
- The ambient temperature must be kept constant; weightometers do not belong outdoors.

ii. A logical test program needs to be implemented whereby:

- Every six months, three runs with weighed material are performed
- Each run needs to last about 30 minutes

- The three tests must be done with different weights in order to find out if the tarring zero is correct
- Weighed material can be calibrated in trucks.(This test is costly and disturbs production).
- Every two weeks, the standard test can be performed.

iii. Monitor deviations on relative difference plots: In order to determine if, after a few weeks, deviations are random or non-random variables, calibrate W1 and W2 in the morning. By the afternoon of the same day they often are already out of alignment.

iv. Effects of stockpiles: Compare W1 and W2. Over a long period of time these two weightometers should show exactly the same tonnage, but there is a large stockpile between the two. Assuming the two weightometers are well calibrated, the difference between W1–W2, should be a random variable, regardless of the stockpile.

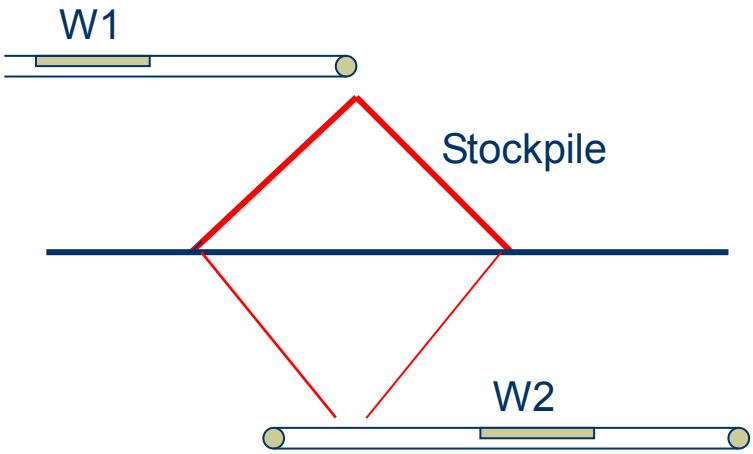


Figure 20.2: ***([40] FF Pitard)***

If calibration is deteriorating on any of the weightometers, the difference between W1 – W2 will no longer be a random variable. This suggests that plotting relative and dimensionless differences expressed as % between W1 and W2 three times a day on a control chart and following a 20-point moving average, should indicate when the relative difference [w1-w2]/ [(w1+w2)/2] is no longer a random variable.

e. **Do Not Confuse Quantity of Work with Real Productivity**

Geologists, miners, metallurgists and chemists must learn to do the job right, first time. Quality and productivity are not incompatible. Both can be achieved at the same time if the following requirements are carefully implemented during the life-time of a project:

- Study the heterogeneity carried by constituents of interest as early as possible.
- Optimise sampling protocols according to heterogeneity facts.
- Implement sampling protocols using correctly designed and maintained sampling systems.
- Quantify the accuracy and precision of sampling and sub-sampling protocols and analytical measurements.

- Identify and quantify the variability components carried by generated data.

Unless this sequence is thoroughly implemented, quantity of work will be unnecessarily inflated and productivity will be poor.

7 Establish a Realistic Wish-List for Every Process Parameter

Mine and mill reconciliation problems can be minimised if every process-controlling parameter is carefully kept under control. For every key parameter, establish a wish- list consisting of three key values:

- An Upper Specification (US), above which the process no longer works well.
- A Targeted Average (TA), at which the process performs best.
- A Lower Specification (LS), under which the process no longer works well.

For any given parameter many managers and engineers are unclear regarding what the wish list is. Such behavior shows a poor understanding of the different sources of variability inherent to the process and to sampling/measurement systems.

a. No wish-list means no meaningful variability analysis is possible: A nickel mine has several pits working simultaneously. One major concern is the level of arsenic that should be kept under 30ppm. The following data covers a six-month period.

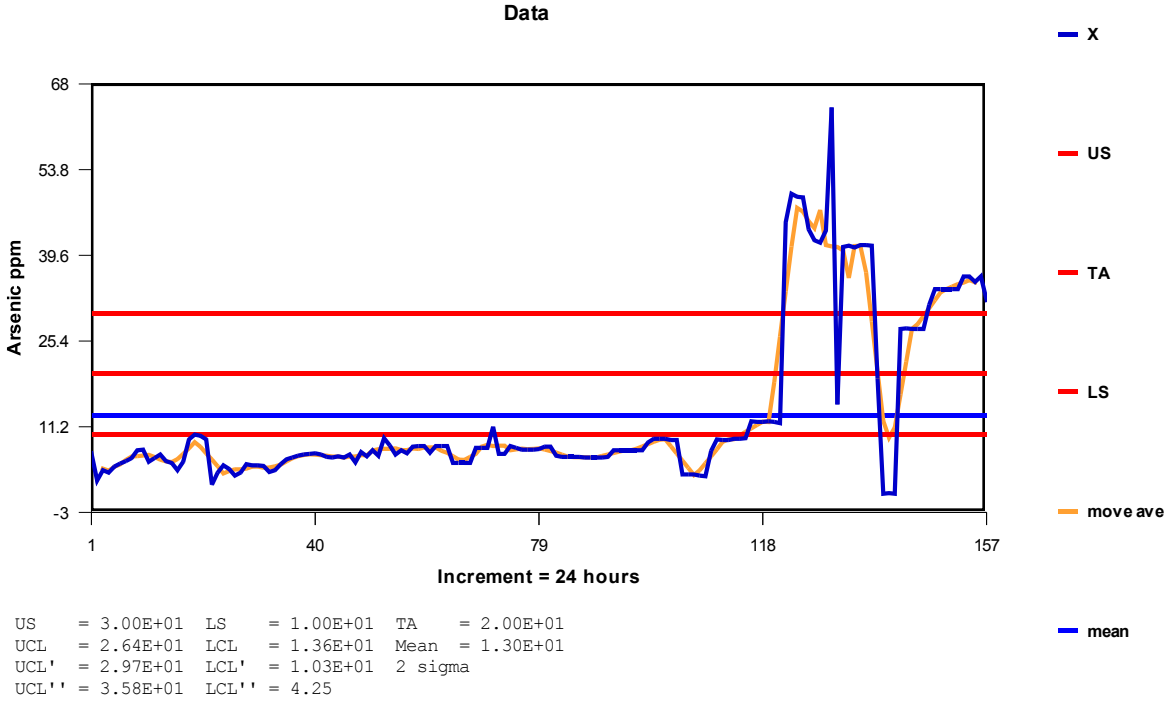


Figure: 20.3: Data Quality Objectives (Wish List) for the content of arsenic in a ROM nickel mine

b. A balanced wish-list is the result of a careful variability analysis: A wish-list may have dynamic values that change with time. Nevertheless, the concept of the wish-list remains the same and it is up to a manager to decide when and how the list should change. It takes careful planning to coordinate a wish-list

with other important parameters. A logical wish-list cannot be improvised and should be the result of a careful variability analysis. Therefore, a wish list is necessarily an iterative decision-making process.

f. **Work Smarter, Not Harder**

A good understanding of the various kinds of heterogeneity and the variability they generate in the process helps to implement a successful statistical process-control program. In the following example the cyclic variability affecting the tons/hour processed through a SAG mill forces the targeted average (TA) to be set at 540 tons/hour in order for the mill to rarely go above a critical point set at 600 tons/hour (US). Above US the performance of the mill may totally collapse.

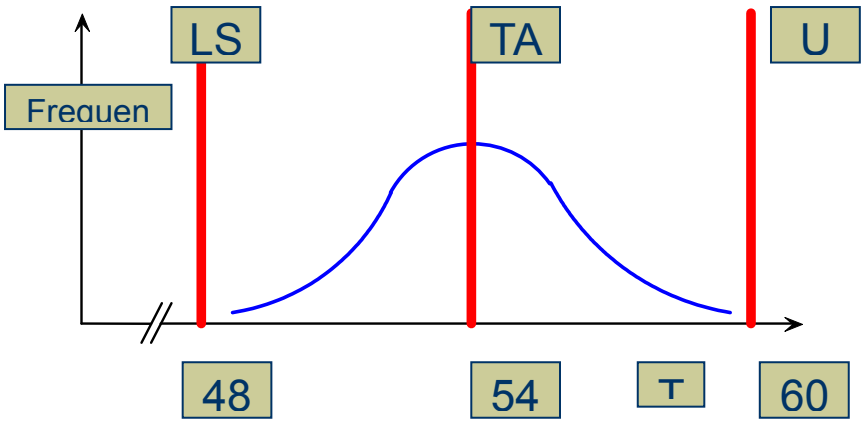


Figure 20.4: Data Quality Objectives (Wish List) for the productivity of a SAG mill *([40] FF Pitard)*

a. Minimising stockpile segregation:

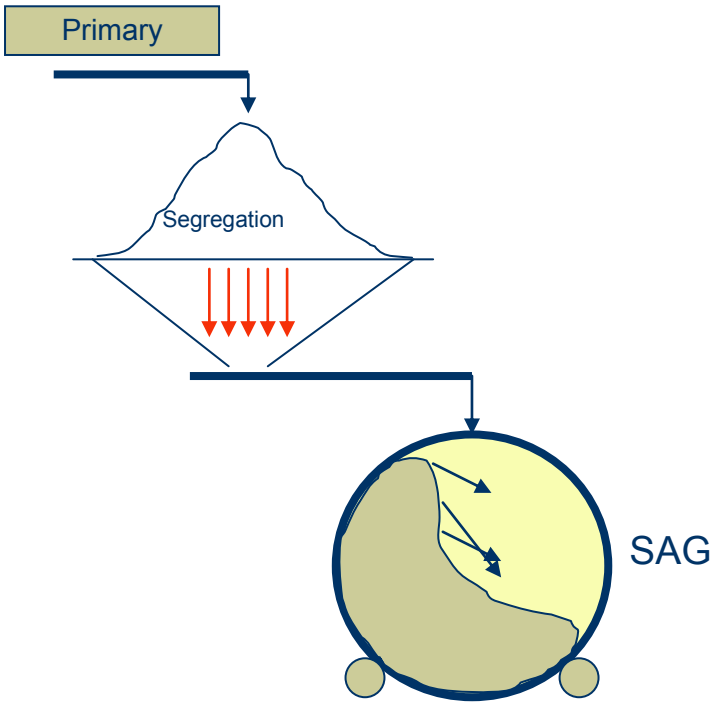


Figure 20.5: Segregation affecting the performance of a SAG mill ([40] FF Pitard)

b. Minimising the amplitude of the cycle:

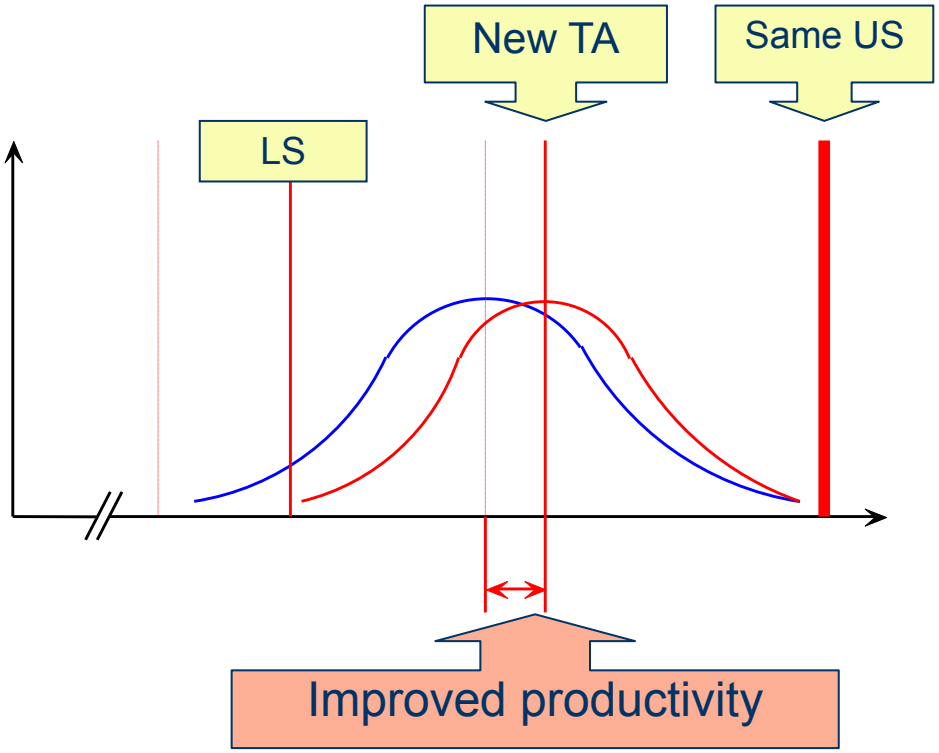


Figure 20.6:* Minimizing the amplitude of the cycle can improve productivity *([40] FF Pitard)

c. Example 2: Optimising blast breakage:

Drilling and blasting at the mine is often viewed as a task for which the cost must be minimised. However, the cost of milling is about 20 times the cost of blasting. Therefore, carefully optimising breakage with blast holes should have a tremendous downstream economic effect as follows:

- Minimising ore dilution
- Increasing shovel and truck productivity
- Minimising crushing costs
- Minimising milling costs
- Increasing milling productivity
- Increasing metal recovery
- Decreasing impurity levels in concentrate, etc.

d. **Example 3: Mining/milling more tons per day:** "Mining and milling more tons increases company benefits." The cultural appeal of the above statement is created by the refusal to admit there are invisible costs. "If I cannot put a cost in the accounting books, it does not exist." The main objective of drilling and

blasting should be to maximise fragmentation and minimise dilution. Mining/milling more tons per day can be a terrible paradox.

g. Beware Of Economic Units within a Company

Today, in many companies, the mine, mill, smelter, refinery and sometimes even the laboratory may work as independent economic units within the same company. Management knows that independent economic units perform better as they are responsible for their own cost-effective performance. It is a sound principle but not without danger. Indeed, all these units work in a logical, inescapable chronology, where the performance of one greatly affects the performance of the other. Therefore, the by-product of economic units is the exportation of a problem to the next unit since there is no longer an incentive to correct some important details. The philosophy becomes “the smelter will take care of this; the refinery will take care of that”. To become cost-effective in this way, one may well create a nightmare for smelters and refineries

a. Feedback between units:

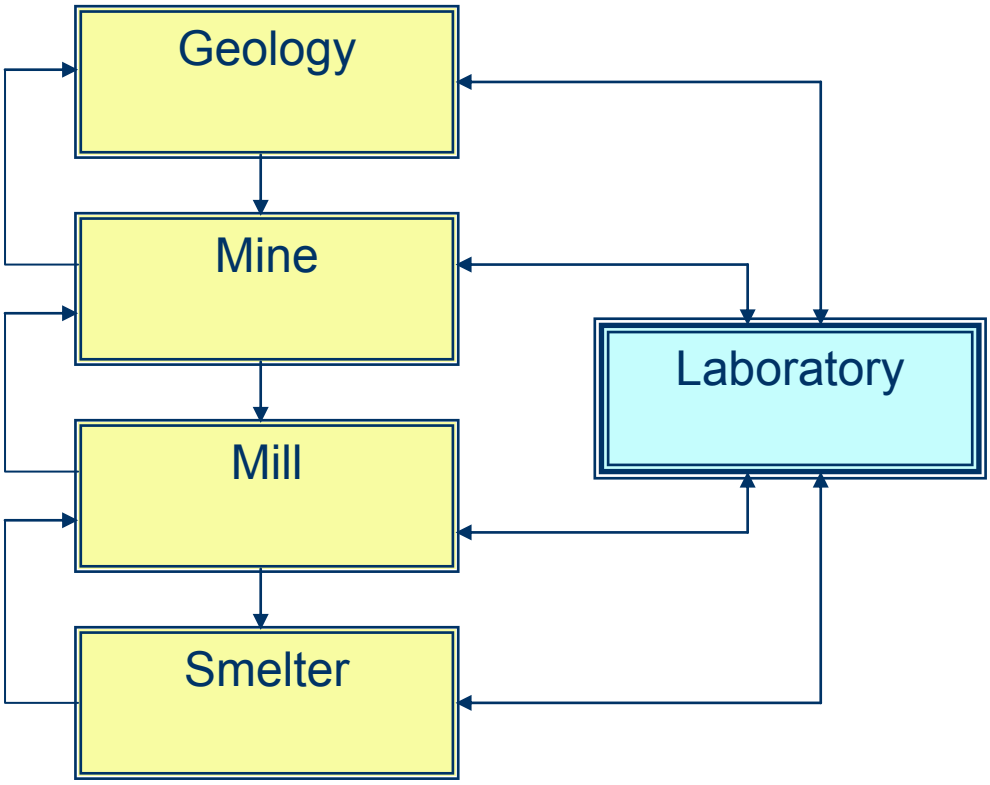


Figure 20.7: Feedback between economical units and good communications with the laboratory are essential

b. **Direction by objectives: Yes, but…!:** Data Quality Objectives (DQO) should always come first.

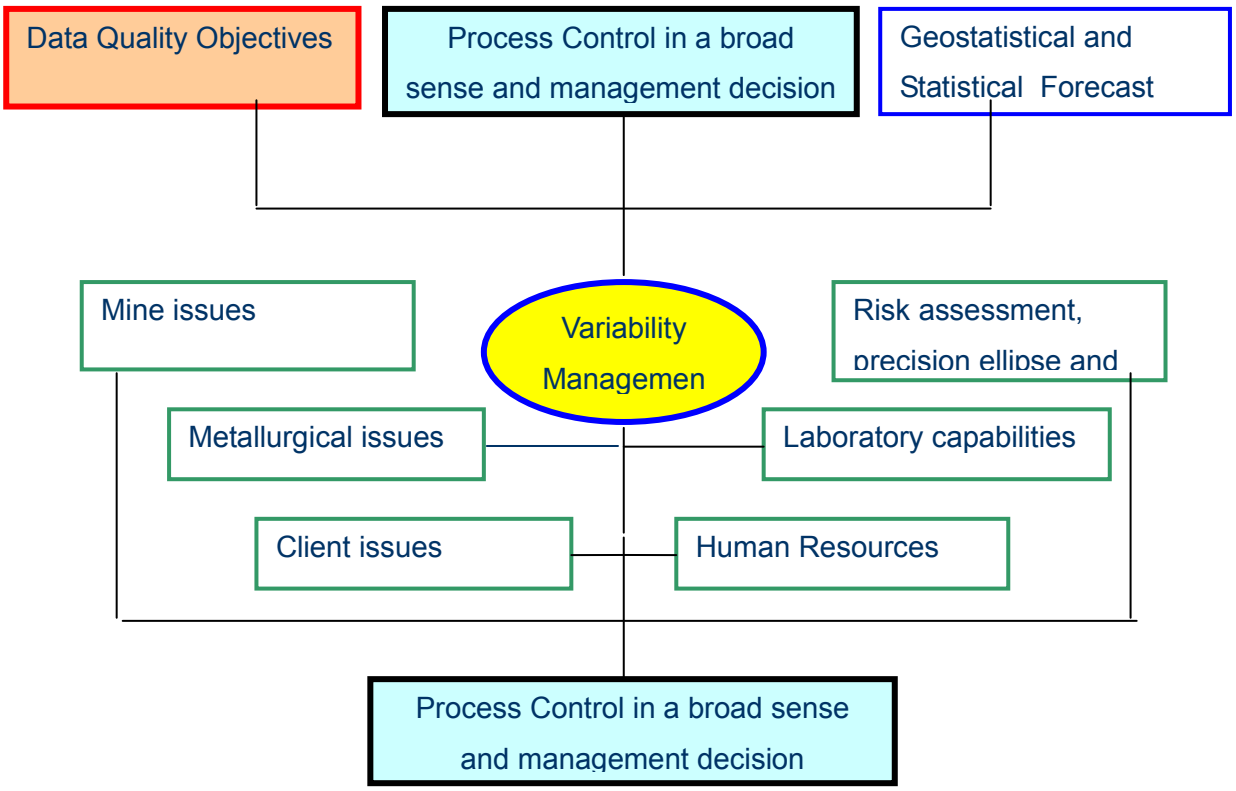


Figure 25.8: Data Quality Objectives and variability management are strategic points for continuous improvement ([40] FF Pitard)

c. Cut-off grade selection - beware of misclassification: Geology, mine and mill feedback must be the object of careful statistical analysis. An example is the cost of precision affecting blast hole samples. Precision, which may generate huge ore grade misclassification, is not taken seriously enough in most mining operations. Its effects are compounded by the desire to become over-selective through the use of unrealistic ore grade cut-offs, based on economic facts alone. The next sketch illustrates a case whereby management selects an economical gold grade cut-off to feed to the mill with a theoretical average grade predetermined by a feasibility study. Random variability affecting the data base results in ore grade misclassification. If the expected grade is not obtained, management may be tempted to increase the gold grade cut-off. This is a reactive decision, does not address the causes of the problem and drastically cuts in on the ore reserves.

d. The precision ellipse - a vision of reality:

Table 25.1: Blast hole duplicate samples in a gold mine

Sample	Sample A	Sample B	Sample	Sample A	Sample B	Sample	Sample A	Sample B
1	4.62	3.92	34	1.37	0.9	67	3.83	3.63
2	0.83	3.62	35	1	1.09	68	3.78	3.39
3	0.49	1.1	36	1.76	1.35	69	4.08	4.05
4	1.4	0.86	37	4.47	2.51	70	4.23	2.12
5	2.07	1.47	38	4.81	3.01	71	1.55	1.27
6	1.4	1.59	39	1.72	2.22	72	1.43	1.39
7	1.34	1.63	40	1.45	1.41	73	1.33	1.05
8	1.78	2	41	1.21	1.55	74	0.41	0.36
9	1.39	1.03	42	3.4	1.33	75	0.29	0.31
10	1.67	1.14	43	5.58	3.46	76	1.51	0.93
11	2.33	2.39	44	3.56	3.5	77	0.72	0.82
12	5.57	3.91	45	1.4	1.11	78	2.88	1.13
13	4.13	4.48	46	0.47	0.56	79	2.84	3.09
14	3.97	4.09	47	0.51	0.59	80	0.32	0.35
15	3.8	3.75	48	1.24	0.81	81	0.29	0.26
16	2.65	3.45	49	0.89	1.01	82	1.01	0.83
17	4.63	5.6	50	1.3	1.11	83	0.36	0.37
18	3.81	3.76	51	2.54	2.18	84	1.21	1.61
19	7.3	7.21	52	3.69	3.42	85	2.42	1.92
20	5.41	5.73	53	4.32	3.91	86	2.09	0.65
21	5.12	4.84	54	4.81	4.43	87	4.06	4.14
22	3.93	4.74	55	7.79	8.04	88	2.26	3.14
23	4.65	4.74	56	5.61	5.76	89	5.69	5.33
24	7.71	6.8	57	5.67	8.15	90	4.11	4.12
25	1.64	3.3	58	7.93	6.74	91	7.41	7.88
26	4.28	4.49	59	6.29	6.05	92	8.72	8.86
27	2.86	2.96	60	5.59	4.53	93	7.24	12.9
28	4.37	4.43	61	9.48	13.5	94	9.45	9.36
29	3.73	3.53	62	14.1	13.4	95	14.1	6.24
30	2.98	3.49	63	9.45	14.2	96	9.48	13.5
31	2.37	2.71	64	7.22	9.45	97	8.79	10.7
32	1.31	1.48	65	7.62	6.29	98	8.4	10.3
33	2.08	1.11	66	6.79	6.89	99	8.28	3.41

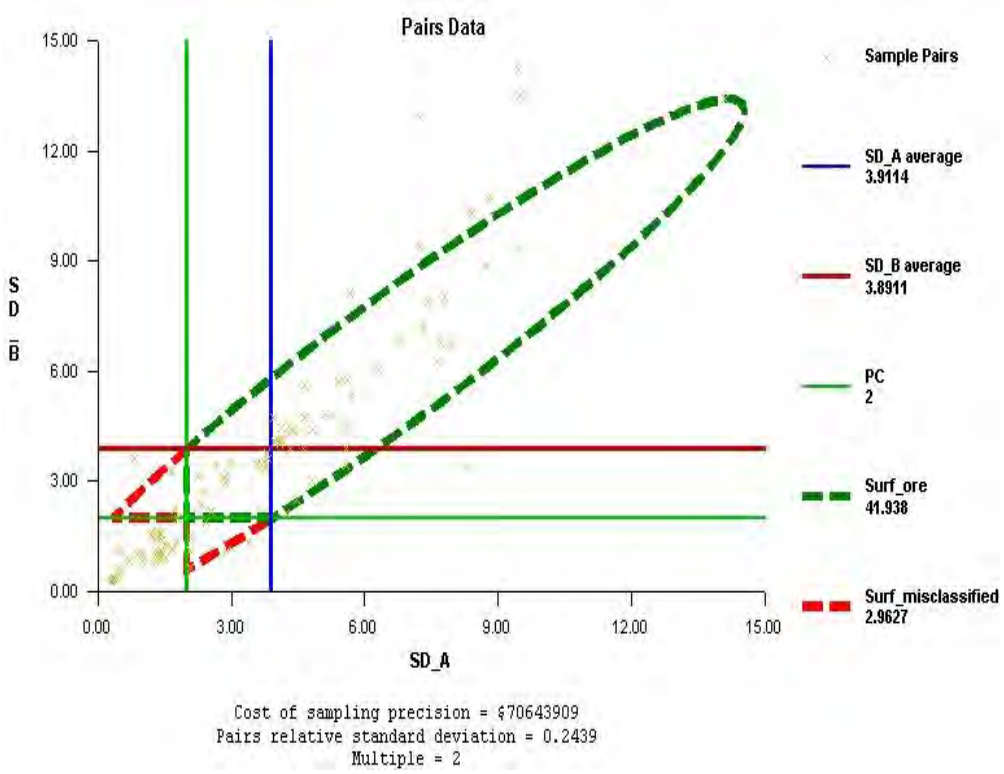


Figure 20.9: The precision ellipse: Detection of an invisible cost *([40] FF Pitard)*

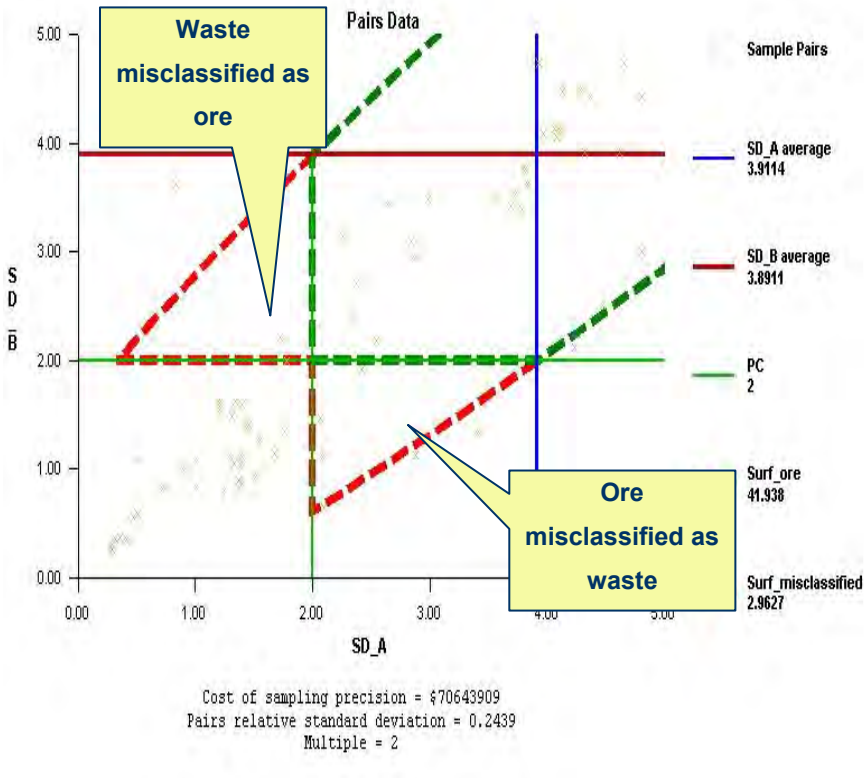


Figure 20.10: The precision ellipse: Misclassified ore ***([40] FF Pitard)***

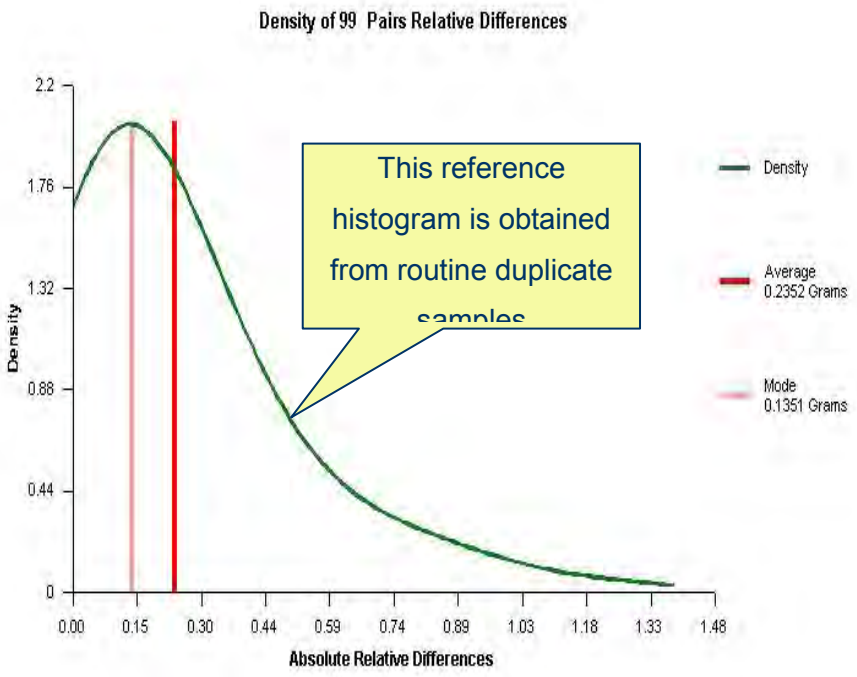
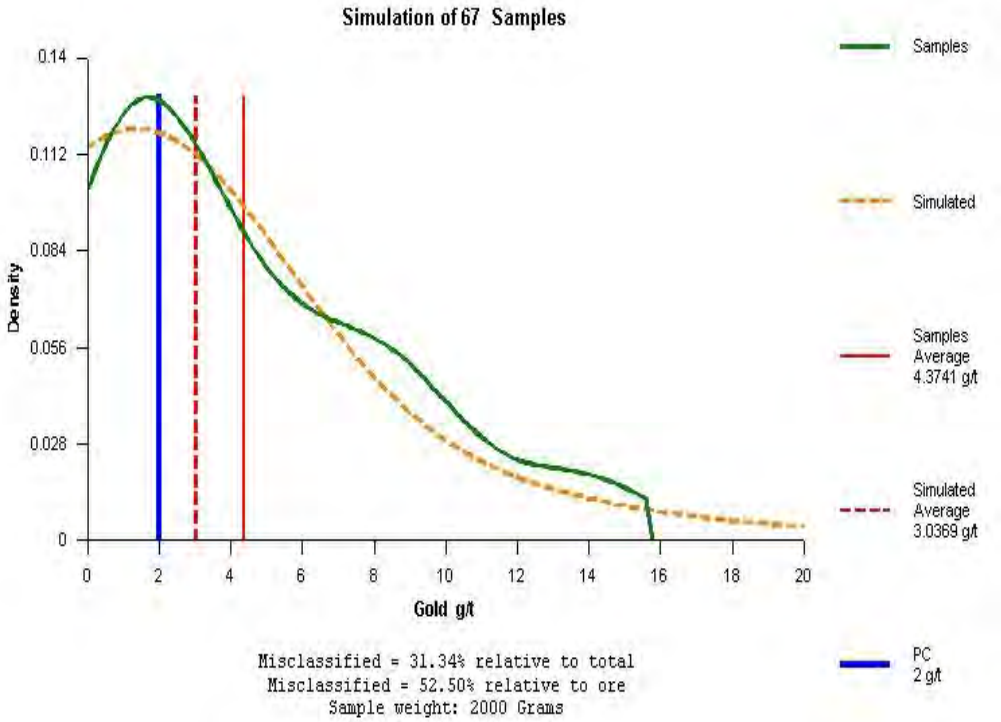


Figure 20.14: The precision histogram: Relative differences (^{40} FF Pitard)

The new sample histogram and the simulated histogram



e. The Four Enemies of Precision

i. In-situ Nugget Effect (INE): The variance of in-situ INE is inversely proportional to the volume of the sample.

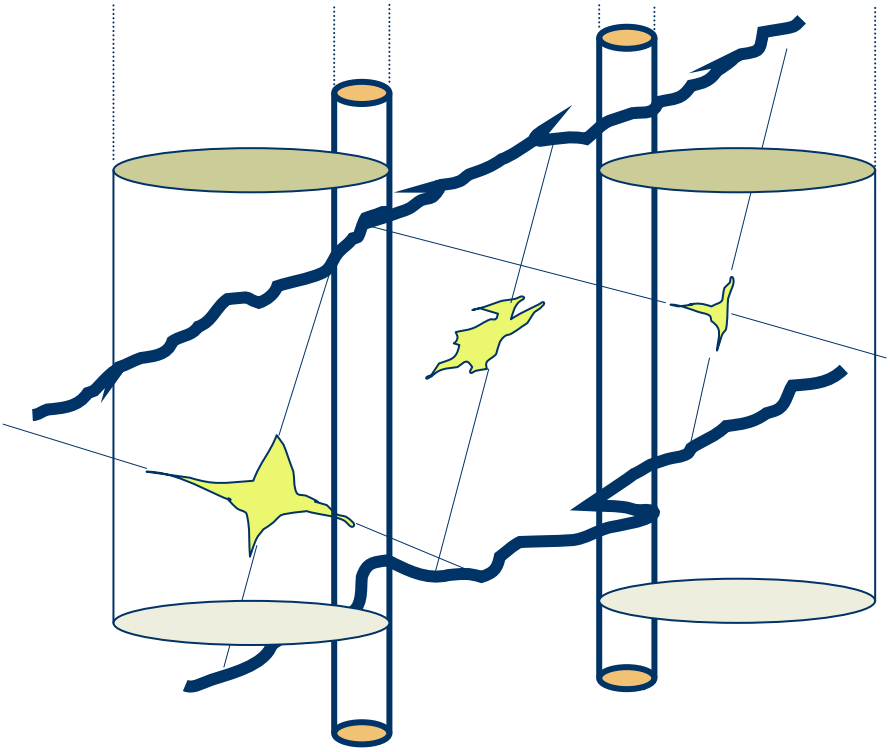


Figure 20.12: In-situ Nugget Effect (INE) ***([40] FF Pitard)***

ii. The Fundamental Sampling Error: FSE is the direct effect of the Constitution Heterogeneity of a lot. The Constant Factor of Constitution Heterogeneity IHL can be divided into the following components: It is appropriate to recall the definition of the liberation size *do*, as it is often misused in sampling literature. The liberation size of gold *do* is defined as the size below which 95% of the material must be ground in order to completely liberate at least 85% of the gold. Therefore, it should not come as a surprise that *do* may become extremely small when a substantial amount of gold is in solution with the rock. This problem will be analysed further in a later section of this course. Knowing IHL and all its other components can lead to a calculation of do. The product K = fgc√do is relatively constant from one state of comminution to another (not to be confused with one size fraction to another where *c* can substantially change). Then, the sampling nomograph can be calculated using the following formula:

$$s^2_{FSE} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] Kd^{2.5}$$

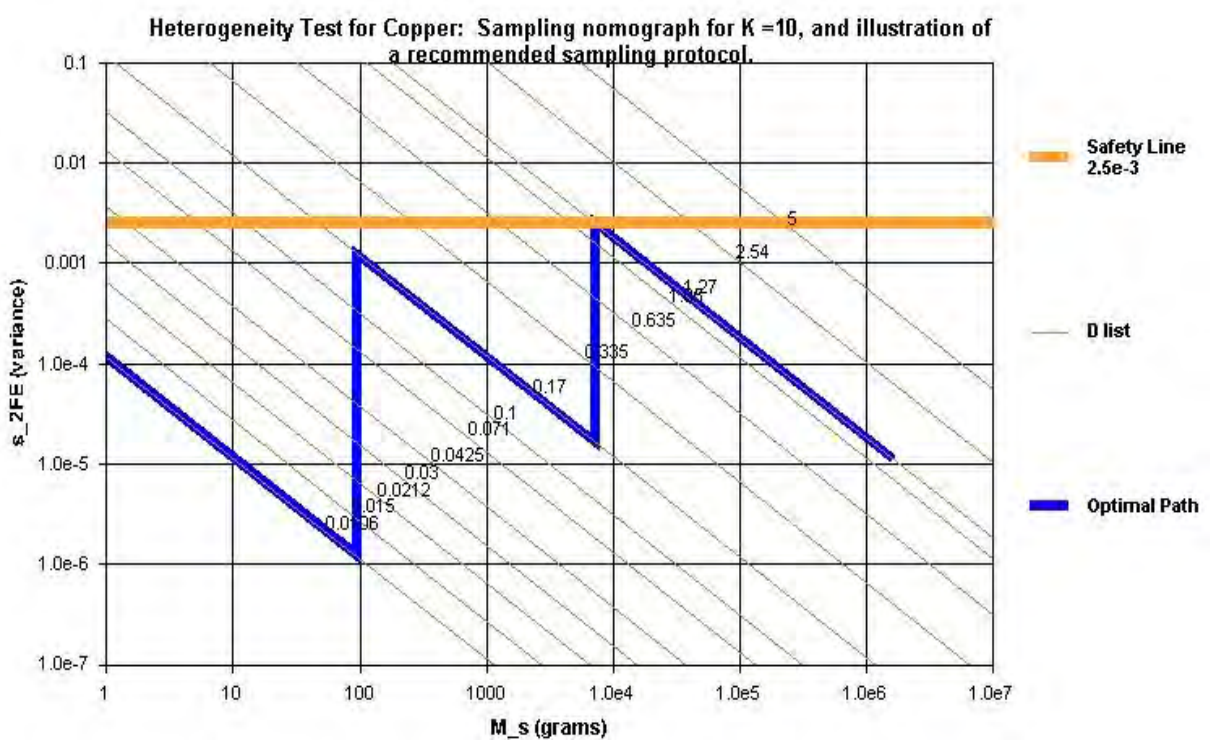


Figure 20.13: The sampling nomograph: A recommended sampling protocol ***([40] FF Pitard)***

iii. The Grouping and Segregation Error: GSE is the direct effect of the Distribution Heterogeneity of a lot (DHL), which is expressed as follows:

$$DH_L = (1 + YZ) N_G \sum_i \frac{a_i - a_L{}^2 M_i^2}{a_L^2 M_L^2}$$

Where Y is a grouping factor, Z is a segregation factor and NG is the number of groups of fragments in a lot. NG is set by the size of the increment collected to make sample. Thus, in sampling theory the variance of GSE is expressed as follows:

$$s^2_{GSE} = YZ\, s^2_{FSE}$$

This new variance is impossible to calculate accurately because the segregation factor Z is set by the amount of segregation taking place in a lot, which is a transient phenomenon depending on gravity and other factors. The calculation of this new variance is an exercise in futility. However, one should discuss the likely domain of that variance.

It is known that DH_L varies between a maximum, CH_L and a minimum, $(DH_L)min$. Because of the omnipresence of gravity, severe differences in mineral densities and in fragments sizes, DH_L is never zero. $(DH_L)min$ is a random variable with a mean that can be calculated as follows:

$$\text{mean } DH_{L\ min} = \frac{N_G}{N_F} CH_L$$

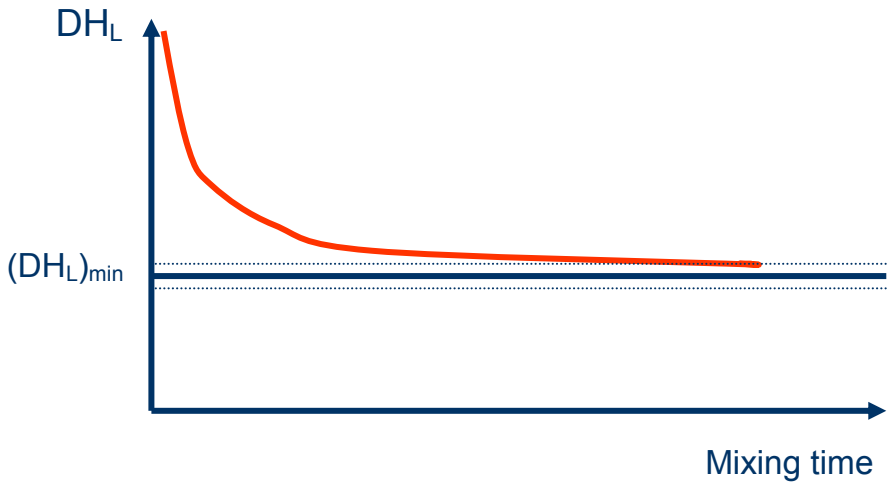


Figure 20.14: Homogenizing cannot improve beyond $(DH_L)_{min}$ *([40] FF Pitard)*

iv. The Analytical Error:

An analytical procedure is likely to increase precision and accuracy variability due to:

- Scope versus principle
- Additive interferences
- Proportional interferences
- Drying temperature baselines
- Dissolution techniques
- Composition of dissolution residues
- Contamination and losses

11. Difference Between Random and Non-Random Variability

A process may be forced to drift unless the operator is properly trained in how to react to sampling/measurement variability. An operator could inadvertently double the total variability of a process, when the best thing would have been to leave it alone.

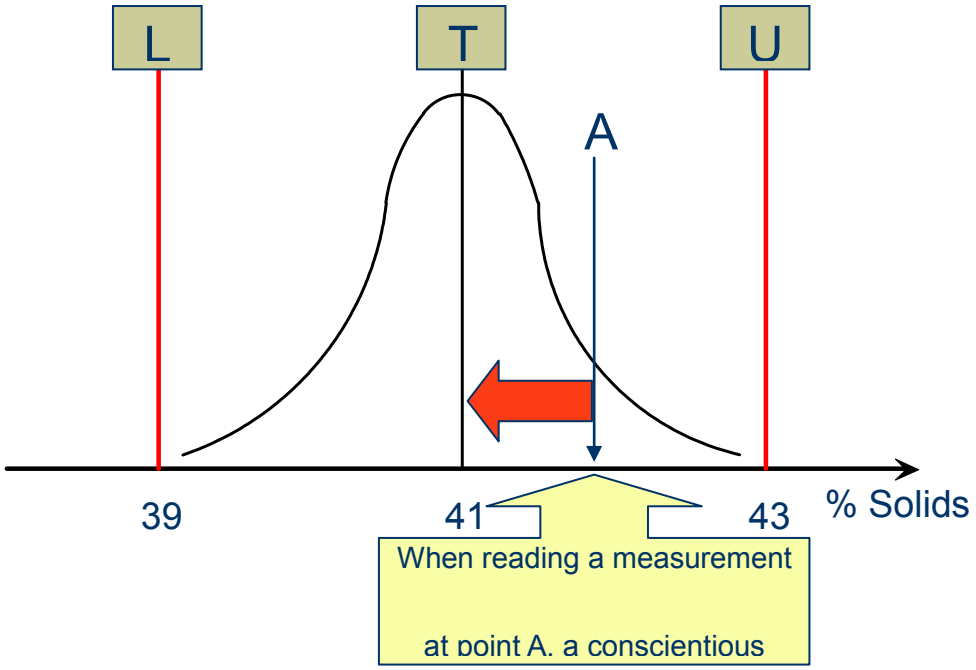


Figure 20.14: Reacting on random variability ([40] FF Pitard)

a. Forcing the process to drift:

The operator did not realise the random variability had nothing to do with the process

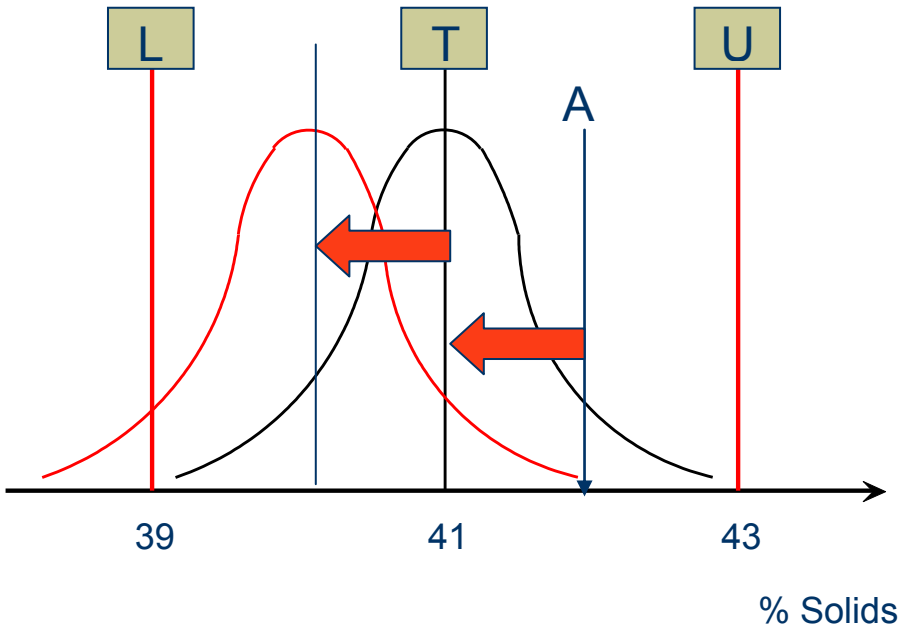


Figure 25.15: Increasing process variability by reacting on random variability ([40] FF Pitard)

b. The danger of over-control: Panic time!: Surprised about a reading at point B, the operator corrects B for TA, repeating his mistake a second time.

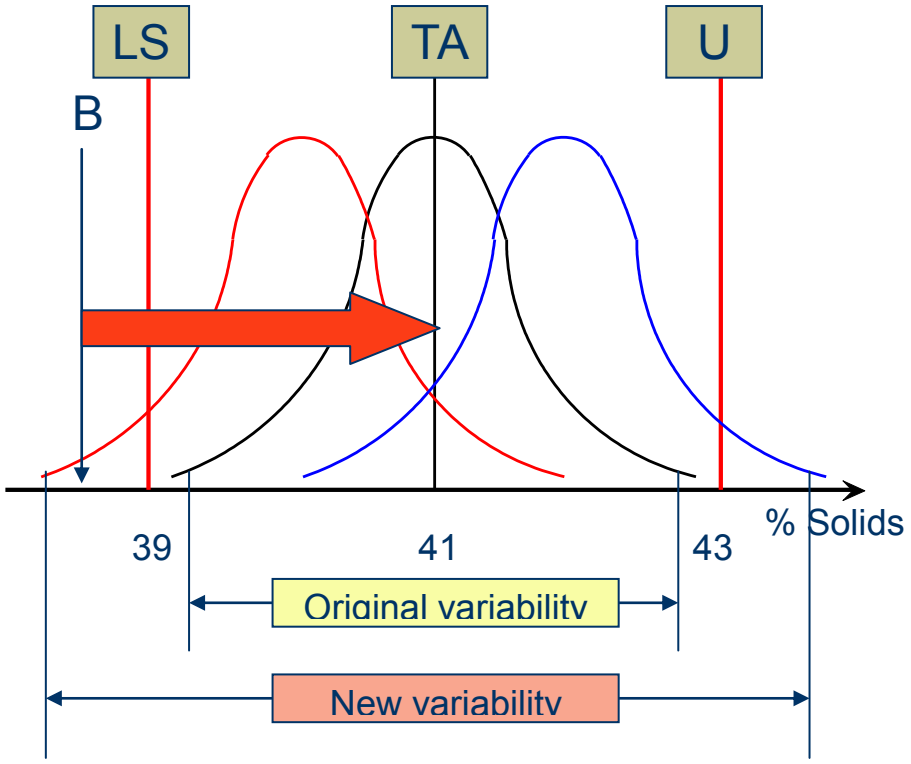


Figure 20.16: The operator who did not understand random variability drastically increased the process variability ([40] FF Pitard)

12. Constantly Minimise Invisible Cost

Meeting requirements provided by a wish list does not result in constant minimisation of invisible cost. Improvement is not a one-off effort. Using statistical thinking, it is important to balance visible and invisible costs. Many people believe that meeting specifications results in zero money losses, as suggested in the following:

Every time a cyclone overflow, feeding a floatation plant, does not meet % solids specifications, a drop in metal recovery and productivity equivalent to US$1000 takes place.

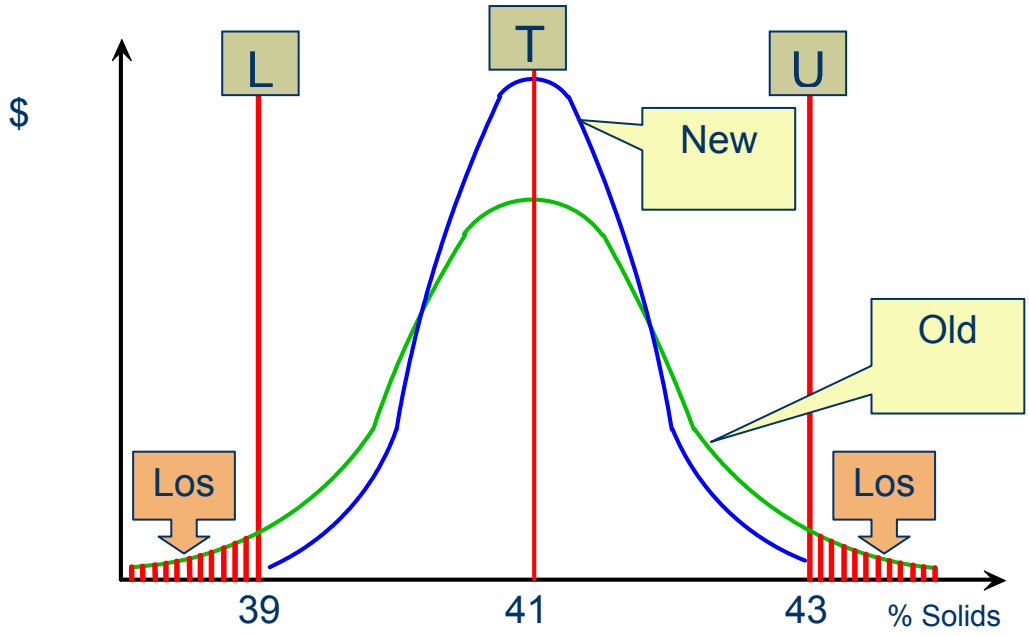


Figure 20.17: Illustration of a step-function cost: a naive view of a cost ([40] FF Pitard)

a. Money losses based on visible and invisible costs:

For ore grade control, any value of the (PRE) generated by sampling and measurement generates a money loss.

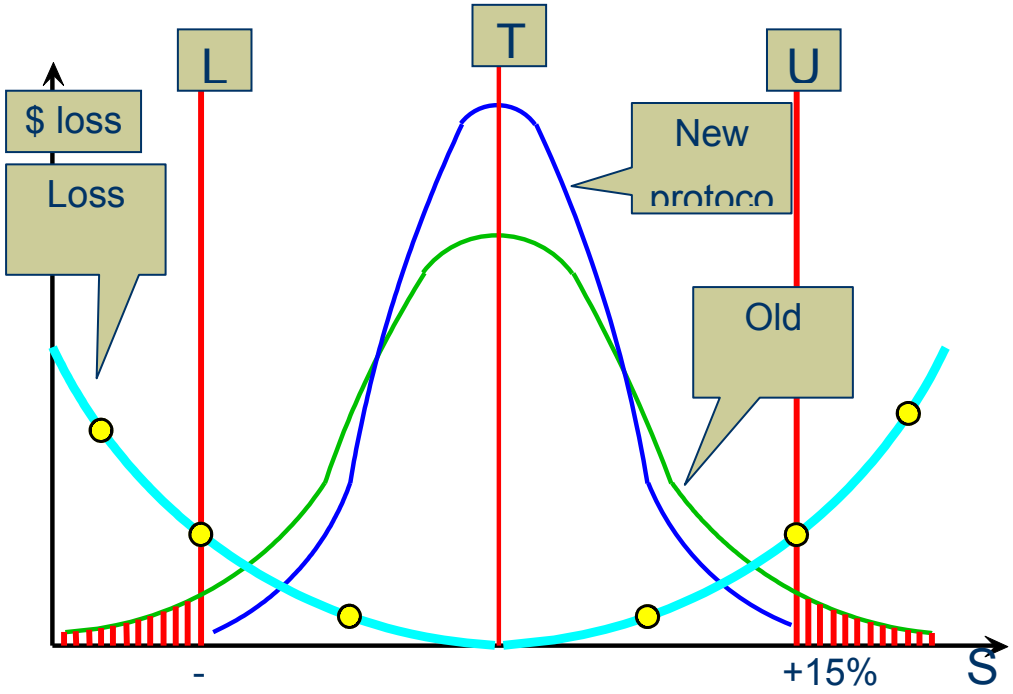


Figure 20.18: Illustration of a loss function: a more realistic model of a cost ([40] FF Pitard)

US and LS should be selected according to what is judged as a reasonable financial loss. However, it should be clearly understood that even between US and LS, there is a loss.

a. Minimise the surface of the precision ellipse for ore grade control:

The minimisation of the surface of the precision ellipse for ore grade control can be achieved by carefully monitoring the precision of every sampling, sub-sampling and analytical stage:

- Duplicate samples during exploration
- Duplicate samples from blast holes
- Duplicate samples from every sample weight reduction stage
- Duplicate assays

Every time the variance analysis of these duplicates detects a weak point, action must be taken, either by changing protocols, changing sampling equipment or training people.

13. Beware of Correcting Factors

Incorrect sampling and poor measurement may introduce biases in a data base. It is tempting to adjust biased data by using correcting factors based on observations and experiments. Unfortunately, there is no such thing as a constant bias in sampling. Therefore, filtered information, elimination of embarrassing figures and the use of correcting factors are symptoms of poor management, which only increase confusion further. Correcting factors give management a false sense of security.

b. Example of an analytical bias:

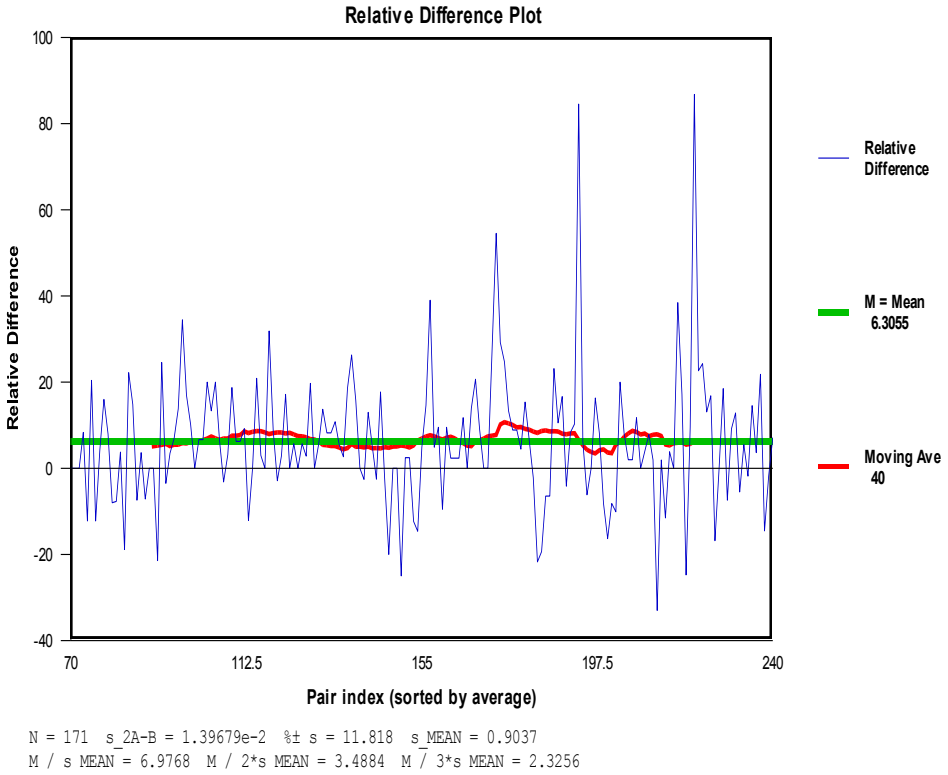


Figure 20.19: Analytical biases are relatively constant. ([40] FF Pitard)

b. Example of a sampling bias:

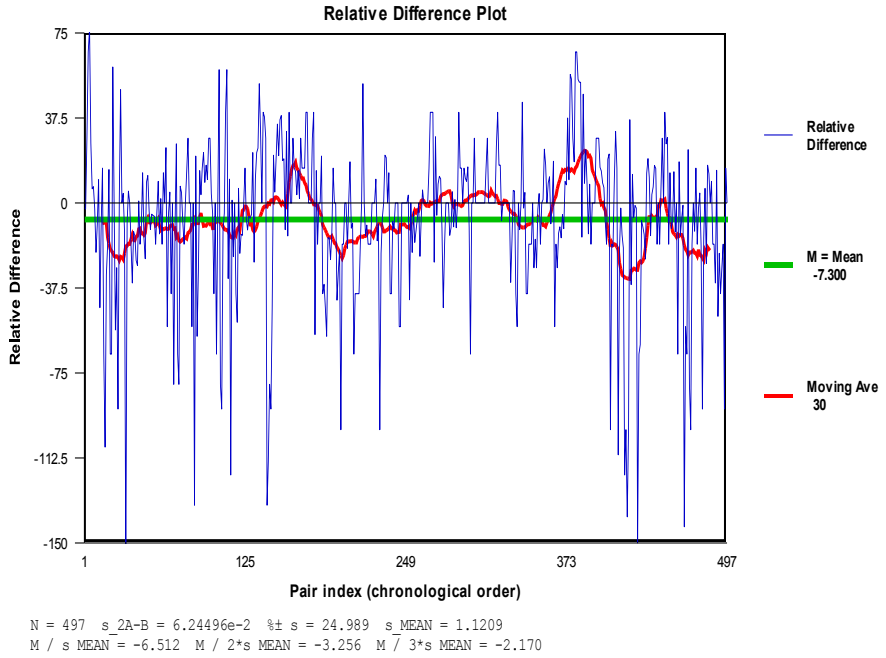


Figure 20.20: Sampling biases are never constant

14. Implement a Creative Thinking Cycle

Mine and plant personnel must work in an environment where they can talk freely about their problems. The following graphic depicts management failure:

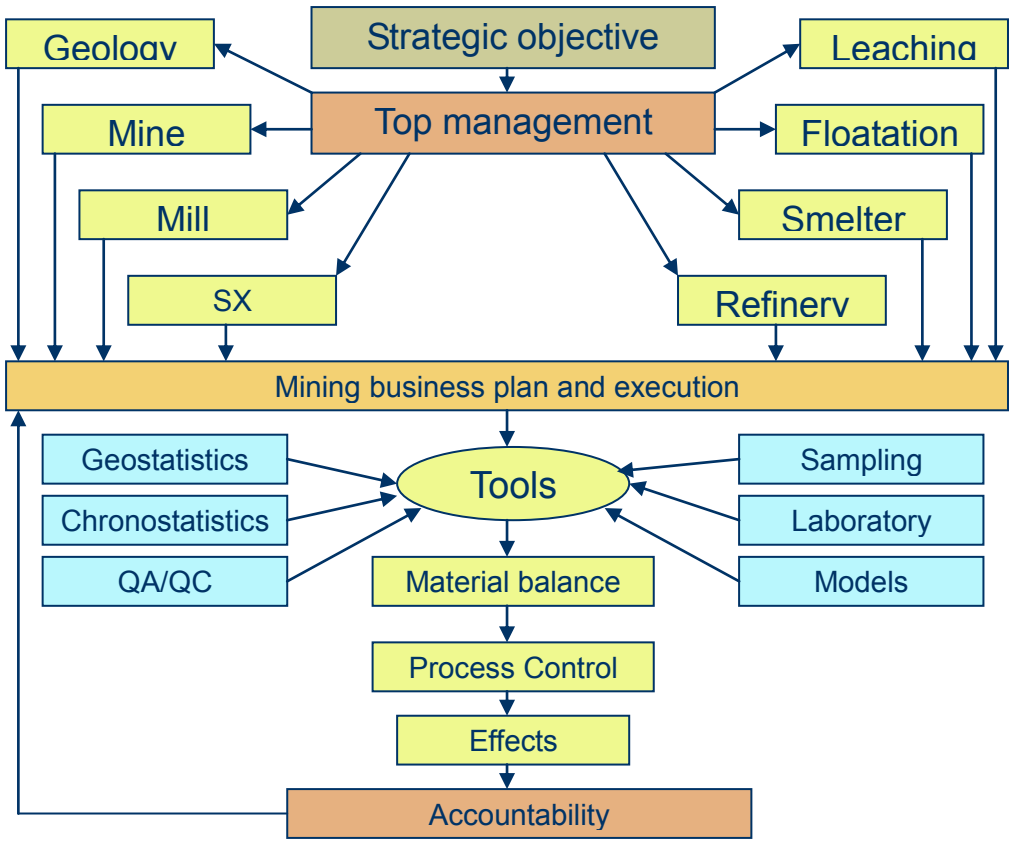
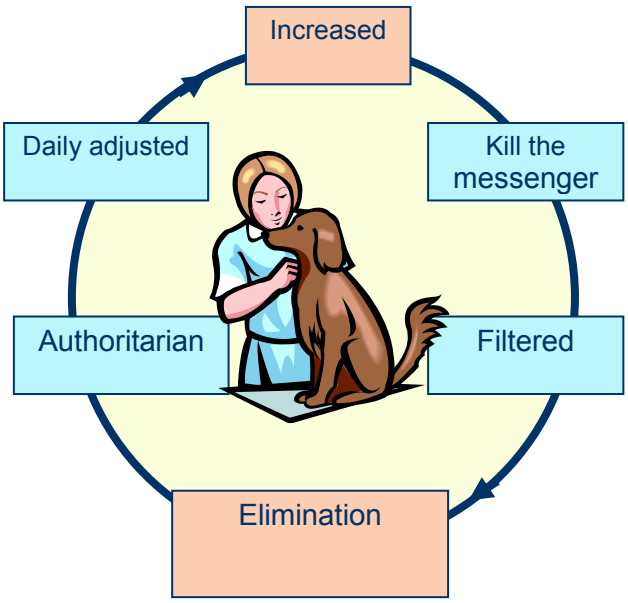


Figure 25.21: Illustration of a poor management strategy

15. Fear = Unsolvable Mine/Mill Reconciliation Problems: ***([40] FF Pitard)***



Talking freely about problems

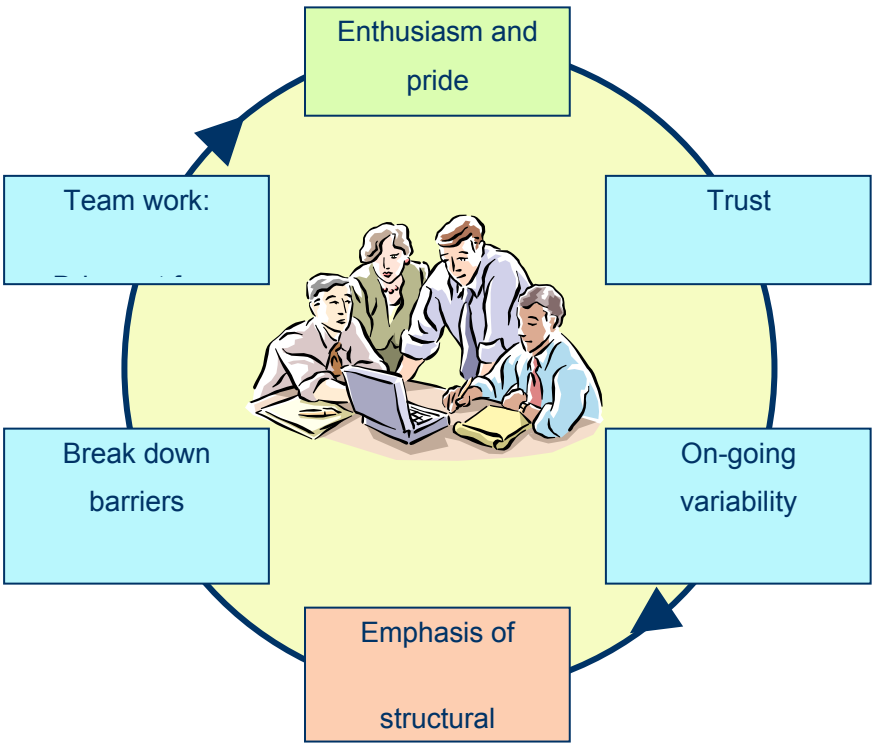


Figure 25.22: Establishing a communication strategy with no fear of talking about structural problems

16. **Set Priorities in an Economic Way**

Many people are discouraged by the complexity and the idiosyncrasy of mine/mill reconciliation. However, problems can be solved, or at least minimised, if a good strategy is implemented.

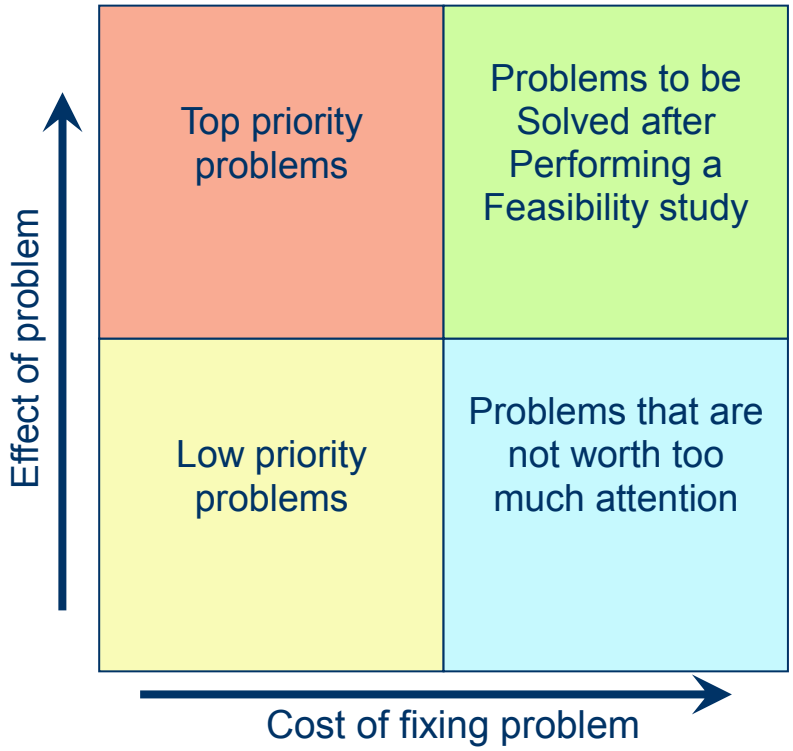


Figure 25.23: Giving priority to problems with large effects in an economic context ([40] FF Pitard)

a. Setting priorities – example for a gold mine:

On-going variability analyses of all existing chronological data can help with dealing with priorities in a logical way.

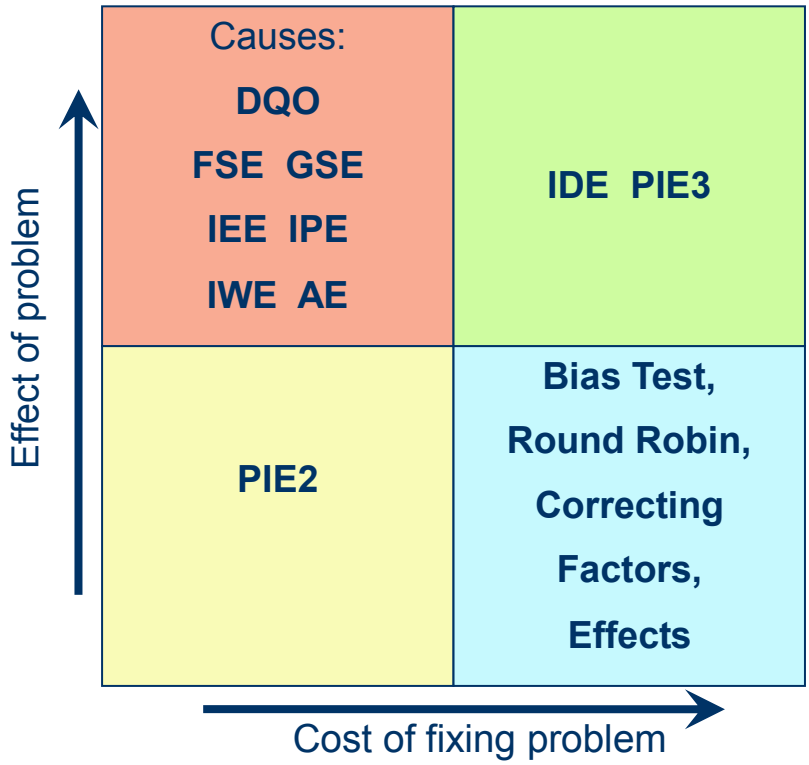


Figure 25.24: Sorting priorities in a gold mine ([40] FF Pitard)

17. Improved Variability is Improved Metal Recovery

It is the experience of Francis Pitard Sampling Consultants that tighter controls on the metal of interest grade-ranges delivered by the mine results in a substantial impact on metal recovery and quality of concentrates.

Every mine which had a management-endorsed ore-blending policy realised substantial improvement at the mill, smelter and refinery within one year.

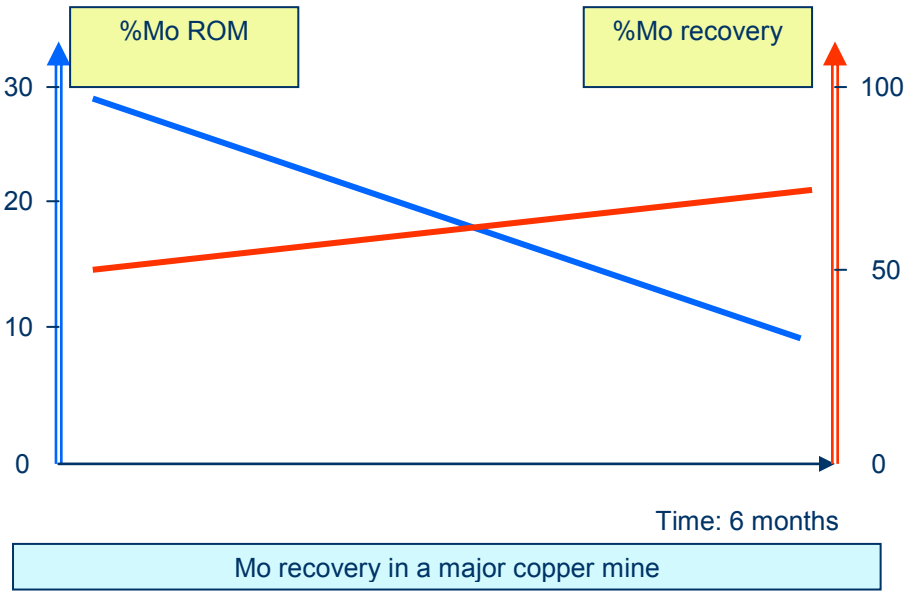


Figure 25.25: Illustration of the correlation of metal recovery and ROM variability ([40] FF Pitard)

18. To Improve, Use Pragmatic Statistical Tools Based on Graphic Observation

Complex statistical analyses often fall short of transmitting a clear message to users at the mine and the mill. Computers allow for graphic observations and this tool must be used so that management can graphically observe process variability happening and thus take effective action in due time. For example, correlation coefficient, significant bias tests and other sophisticated calculations can and should be performed. This is often not the case. A relative differences plot associated with a simple moving average, as shown below, can help a manager in a far more effective way. Not only can the magnitude of a bias be immediately quantified, but its evolution as a function of time or grade can also be followed. After all, in sampling, there is no such thing as a constant bias.

a. Example: Blast holes for gold fire assay – cyanide leach: Time

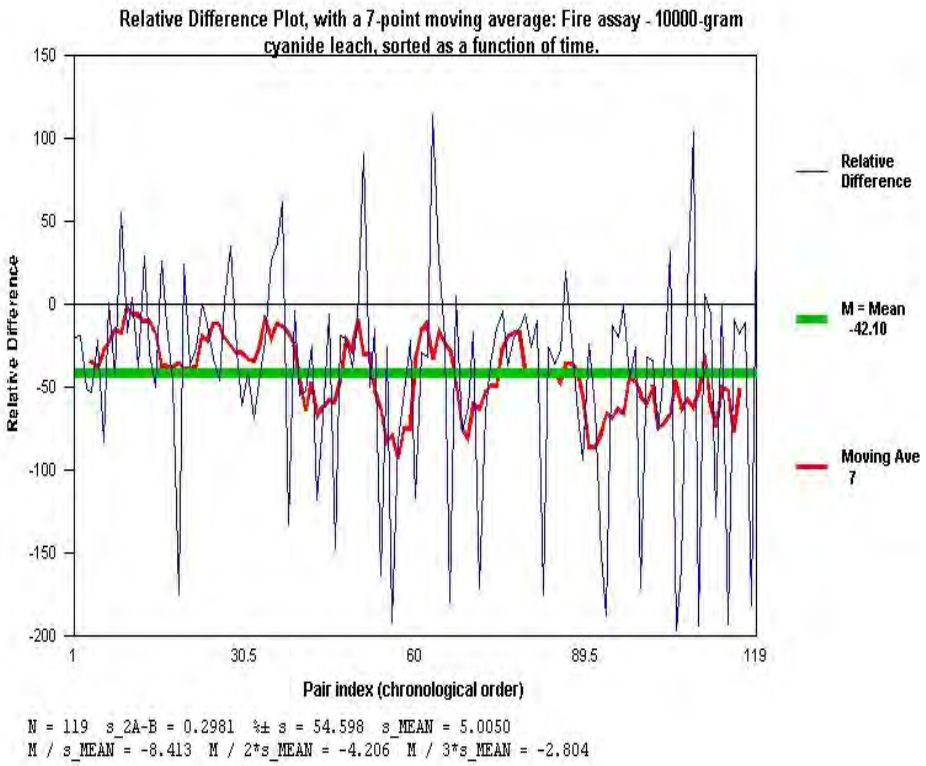


Figure 25.26: Effective illustration of a sampling/analytical bias for gold (^{40} FF Pitard)

b. Example: Blast holes for gold fire assay – cyanide leach: Grade

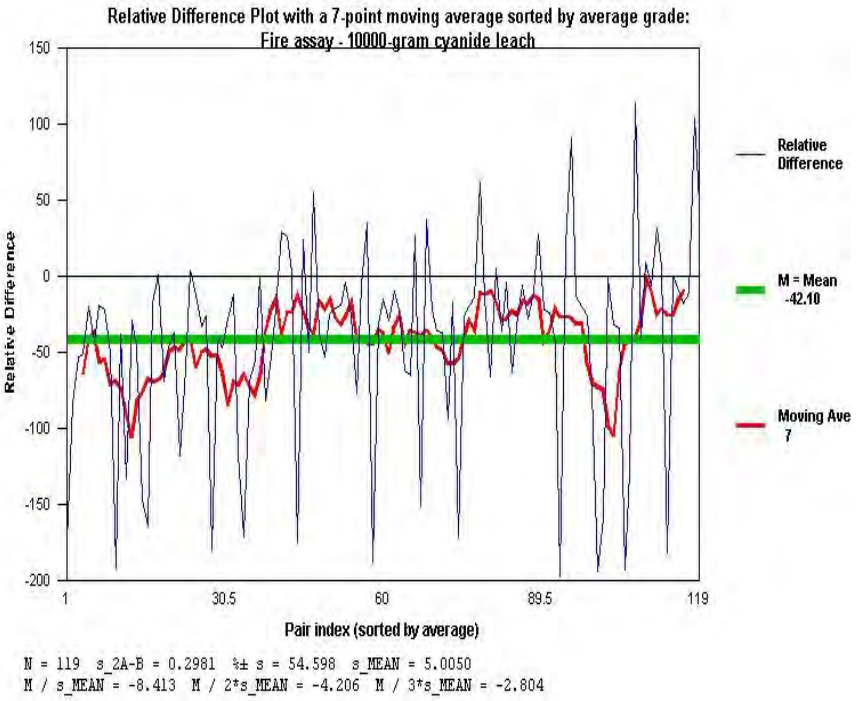


Figure 25.27: Effective illustration of a sampling/analytical bias for gold (^{40} FF Pitard)

19. **Use Concrete Graphic Tools to Quantify Invisible Cost**

Existing Data								
Weight Units:	Grams							
Content Units:	g/t							
Ore:	Gold							
PC:	1.5							
Low Background Content Chart								
LBC Chart Title	Low Background Gold Content							
LBC X axis:	Samples ordered by increasing % Gold							
LBC Y axis:	g/t Gold							
Random Variability Chart								
VAR Chart Title	Random Variability Guidelines							
VAR X axis:	Weight of Gold Sample in Grams							
VAR Y axis:	S g/t Gold							
Composite Mode:	Veritcal							
Sample Weight:	8000							
Grid Data:							Horizontal Composites	
	0.5	0.4	1	1.8	2.3	0.7	1.12	
	0.9	1.3	0.1	2.7	4.5	0.1	1.6	
	0.1	1.9	2.1	0.6	3.7	6.5	2.48	
	0.4	1	3.2	39	2.1	0.6	7.72	
	0.5	0.7	2.3	0.5	5.6	0.2	1.63	
	1.7	2.3	2.9	4.6	1.2	0.5	2.2	
	0.6	1.5	2.7	3.1	0.7	0.2	1.47	
							Q_h:	6
Vertical Composites:	0.67	1.3	2.04	7.47	2.87	1.26	Q_v:	7
N samples:	42							
N missing:	0							
V ne:	34.08		5.96	S_2:	2.5	Q:	7	
A:	272675							
B:	1.4							

a. Use the sampling diagram to accumulate facts:

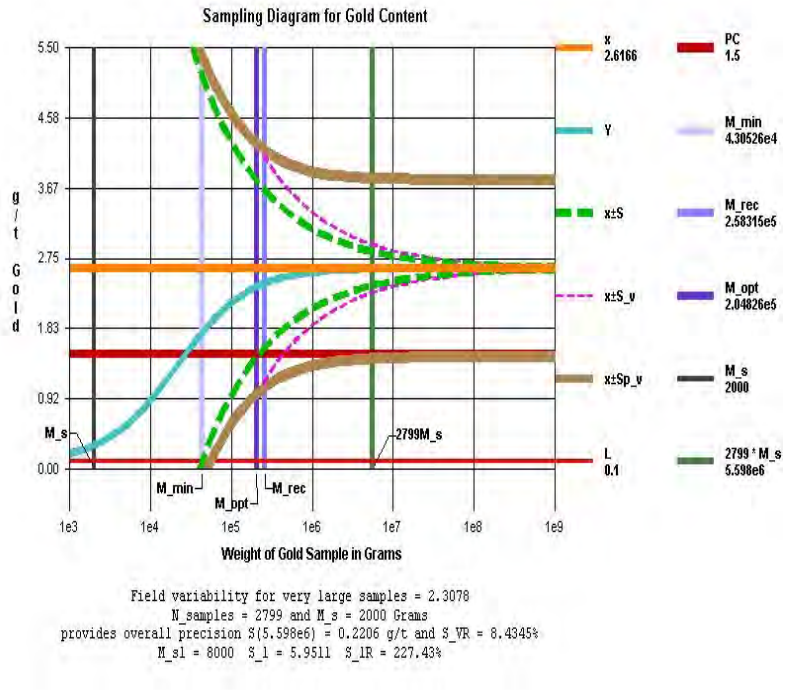


Figure 25.28: Illustration of the Ingamells sampling diagram: a good analysis of variability ([40] FF Pitard)

b. Minimise work and cost for maximum quality information:

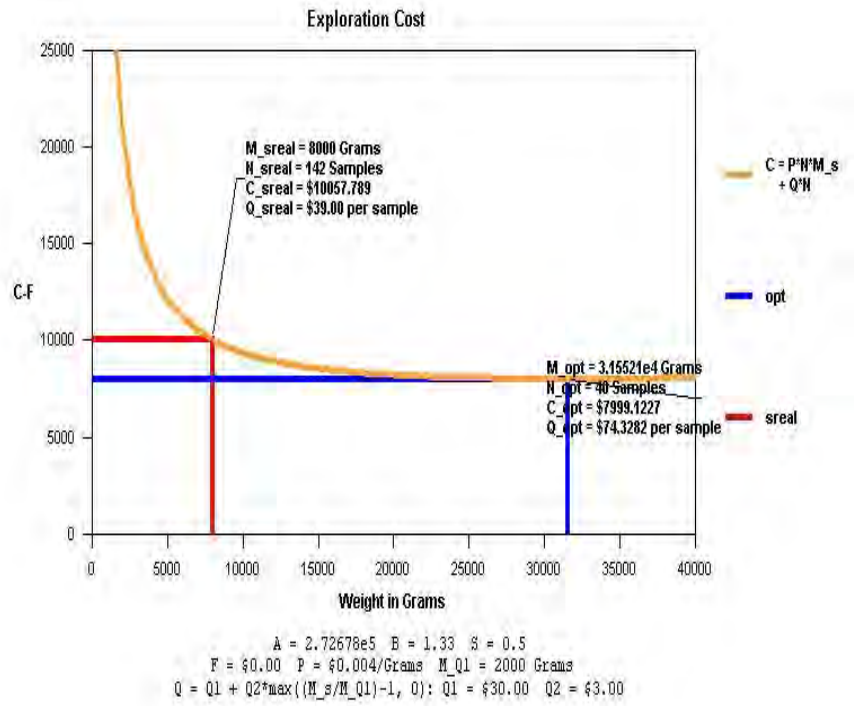


Figure 25.29: Illustration of the sample mass optimization for minimizing exploration cost ([40] FF Pitard)

APPENDIX 1: SIEVE SIZES AND UNIT CONVERSIONS

UNIT CONVERSIONS	
microns	**cm**
1	0.0001
10000	1
inch	**cm**
1	2.54
0.39	1
foot	**cm**
1	30.48
0.0328	1
(troy)ounce	**g**
1	31.103
0.032	1
ounce	**g**
1	28.35
0.035	1
pennyweight	**g**
1	1.555
0.643	1
pound	**g**
1	453.6
0.002	1
short ton (sh tn)	**g**
1	907185

SIEVE SIZES	
Mesh	**cm**
1.05inch	2.6900
0.883inch	2.2600
0.742inch	1.9000
0.624inch	1.6000
0.525inch	1.3500
0.441inch	1.1200
0.371inch	0.9510
2.5	0.8000
3	0.6730
3.5	0.5660
4	0.4760
5	0.4000
6	0.3360
7	0.2830
8	0.2380
9	0.2000
10	0.1680
12	0.1410
14	0.1190
16	0.1000
20	0.0841
24	0.0707
28	0.0595
32	0.0500
35	0.0420
42	0.0354
48	0.0297
60	0.0250
65	0.0210
80	0.0177
100	0.0149

Long ton (tn)	g
1	1016047
tonne	**g**
1	1000000
Assay ton	**g**
1	29.17
grain (gr)	**g**
1	0.0648
Hundredweight (cwt)	**g**
1	50800

115	0.0125
150	0.0105
170	0.0088
200	0.0074
250	0.0063
270	0.0053
325	0.0044
400	0.0037

SUMMARY FORMULAS

When the fragment size is above the liberation size: $d \geq d_\ell$

$$\sigma_R^2 = (1/M_s - 1/M_L) f.g.c.d_\ell^{3a}.d_N^{3(1-a)} \text{ where } b = 3a \text{ and } b = 3-\vartheta$$

$$\sigma_R^2 = (1/M_s - 1/M_L) f.g.c.d_\ell^{b}.d_N^{(3-b)} \text{ where } b = 3-\vartheta$$

$$\sigma_R^2 = (1/M_s - 1/M_L)\ f.g.c.\ (d_\ell/d_N)^b.d_N^3 \text{ where } b = 3-\vartheta$$

$$\sigma_R^2 = (1/M_s - 1/M_L)\ f.g.c.\ d_\ell.d_N^3$$

$$\sigma_R^2 = (1/M_s - 1/M_L)\ K.dN^\alpha$$

$$\sigma_R^2 = (1/M_s - 1/M_L) c.f.g.d_\ell^{3-\alpha} d_N^\alpha$$

$$\sigma_R^2 = (1/M_s - 1/M_L) c.f.g.)d_\ell/d_N)^{3-\alpha} d_N^3$$

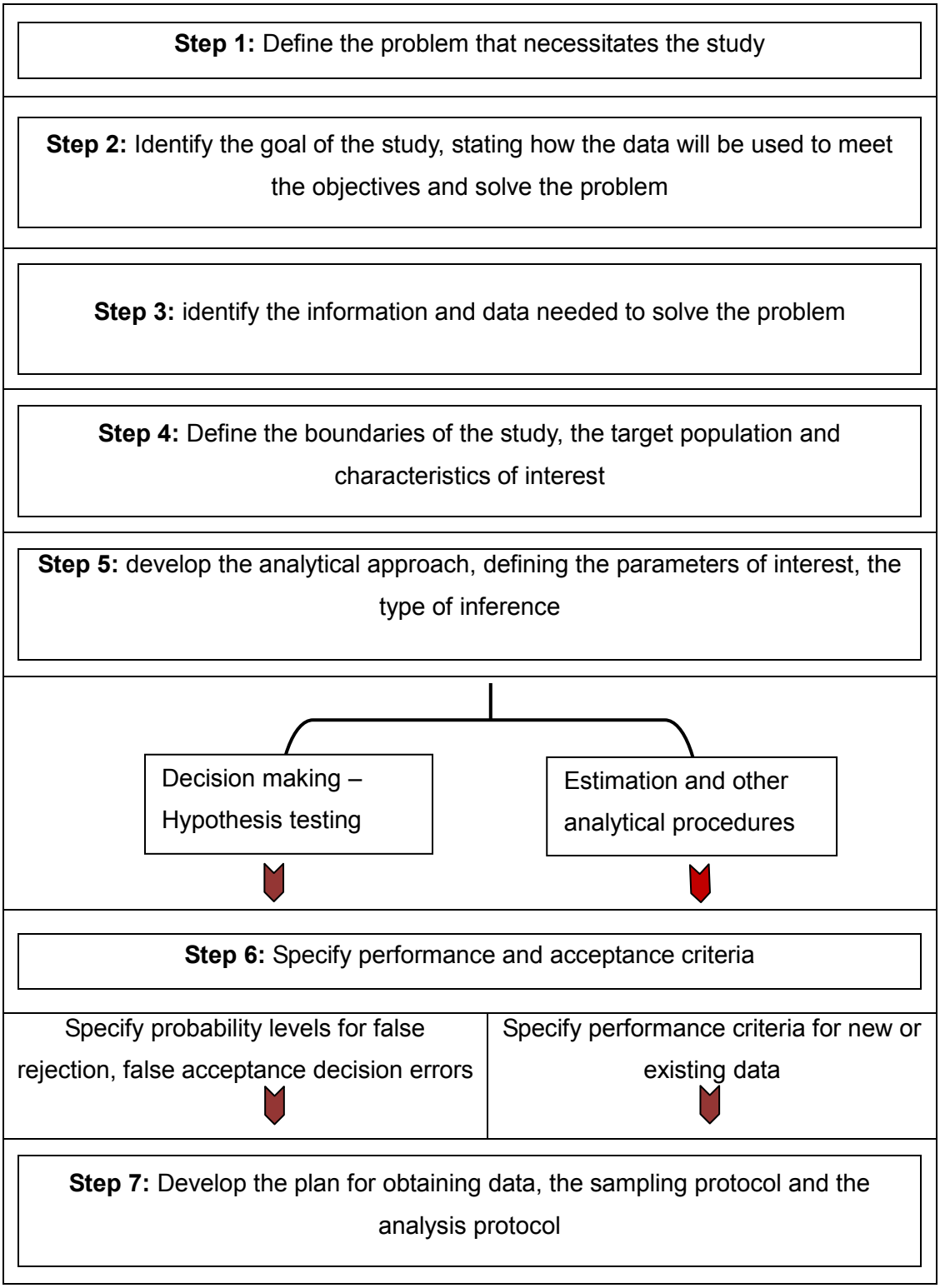
When the fragment size is below the liberation size: $d \leq d_\ell$

$$\sigma_R^2 = (1/M_s - 1/M_L)\ K'.\ d_N^3 = 1/X \text{ where } K' = c\ f\ g$$

Or the Binomial Law

Relative Sampling Variance = 1/X

X = average number of grains per sample.


Step 1: Define the problem that necessitates the study
Step 2: Identify the goal of the study, stating how the data will be used to meet the objectives and solve the problem
Step 3: identify the information and data needed to solve the problem
Step 4: Define the boundaries of the study, the target population and characteristics of interest
Step 5: develop the analytical approach, defining the parameters of interest, the type of inference
Decision making – Hypothesis testing
Estimation and other analytical procedures
Step 6: Specify performance and acceptance criteria
Specify probability levels for false rejection, false acceptance decision errors
Specify performance criteria for new or existing data
Step 7: Develop the plan for obtaining data, the sampling protocol and the analysis protocol

Dear Fred

Please find the quotation for Dick's book attached. I only included proofreading as I believe it would be sufficient as we are working with technical terms.

As discussed, the once off (pre-press) fee is usually not taken into consideration when the RRP is determined (this could for example be sponsored). Should the pre-press fee also be taken into consideration and the total costs have to be covered through sales via book stores (which takes 30% to 50% discount), the Recommended Retail Price (RRP) should be R1 600.00. This seems a bit high to me? Please note that through direct sales via yourself to Wits students etc the profit margin will me more than 100% should the book be sold for R852.00 upwards (cost price per book with pre-press included is R452.62) BUT sales via book stores with the discount and service fees in mind should also be taken into consideration.

I will explain all the details to you when we meet in December. The quotation to be approved is accurate and as soon as approval is given we can commence with the book.

I attached an example of a royalty agreement with the RRP at R1 450.00 (which I believe should rather be lowered) if the pre-press or part of the pre-press could be sponsored. Please note that sponsors' logos could be placed on the front and back cover and recognition to the sponsor on the imprint page (page with the book information at the start of the book). This is worth quite a lot and you could also limit this to a certain amount e.g. for the first 300 books printed, thereafter another sponsor could receive the opportunity to advertise. You can still start with printing 100 books and can reprint books up to a specified quantity of your choice with the same sponsor's logo on the book.

We can discuss all of this when we meet in December ☺.

Regards,

Jean Lung

Feather Communications

082 854 6697

012 991 4758 (W & F)

jean@feathercommunications.co.za