

# ***SELF-DEFENSE FOR MINERAL ANALYSTS***

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## ***Abstract***

*The responsibility to procure unbiased test results with an acceptable and affordable degree of precision ranks high amongst the duties of mineral analysts. Measurement procedures in mining and metallurgy typically consists of a sampling stage, a sample preparation stage and an analytical stage. Mineral analysts are invariably responsible for the analytical stage, and use Certified Reference Materials (CRMs) to test analytical methods for bias and interlaboratory tests and internal standards to measure and monitor analytical precision and the absence or presence of bias. Mineral analysts are often responsible for the sample preparation stage but seldom for the sample selection stage.*

*Such a dichotomy of responsibilities may lead to argument and debate in which the mineral analyst who is unfamiliar with the fundamentals of sampling theory and practice is at a distinct disadvantage. This paper examines how to quantify uncertainties at the sample selection stage, at the sample preparation stage and at the analytical stage, and how to optimize the measurement procedure. Given the critical importance of measurement technology in mining and metallurgy, it follows that the mineral analyst should be familiar with the potential for bias and all possible sources of variability in measurement procedures if only in self-defense.*

## **Introduction**

Whenever the validity of a measurement procedure at a mine or a smelter is questioned a mineral analyst is required to examine the matter. Is the test result biased? Is it too imprecise? Is the analytical method suspect? Is the sample preparation procedure flawed? Is a systematic error at the sample selection stage perhaps to blame?

medium grade, and more than 2.5 g/t as high grade, the probability that a measured grade of 1.84 g/t will fall in a certain category is simple to calculate. For example, dividing the difference of 0.66 g/t between the measured grade of 1.84 g/t and 2.5 g/t for the high grade ore by the standard deviation of 0.277 give a t-value of  $0.66/0.277=2.38$  which exceeds  $t_{0.95;39}=2.02$  but is below  $t_{0.98;39}=2.42$ . Thus the probability is somewhere between 95% and 98% that the measure grade of 1.84 g/t is indeed medium grade.

Dividing the difference of 1.34 g/t between the measured grade of 1.84 g/t and the cut-off grade of 0.5 g/t by the standard deviation of 0.277 gives a t-value of  $1.34/0.277=4.84$  which exceeds  $t_{0.999;39}=3.56$ . In this case, the probability is much less than 0.1% that the measured grade of 1.84 g/t is waste, and the probability exceeds 99.9% that it is ore grade material.

The tabulated value of  $t_{0.95;39}=2.02$  is close to  $t_{0.95;\infty}\approx z_{0.95}=1.96$  so that the Gaussian or normal distribution can be used if the number of blasthole grades is large enough. This property of the t-distribution allows tabulated values of the z-distribution to be used to calculate the probability in percent that the measured grade of 1.84 g/t belongs in either of these four categories. Such calculations are slightly more complex and require more space than this paper affords.

## Summary

The objective of assaying test portions is not to determine the grades of blasthole cuttings but to obtain unbiased point estimates for the grades of all elementary units in the set into which a blasthole grid divides a block of *in-situ* ore. Since the measurement variance is extraneous to the intrinsic variability of gold, it can be deducted from the first variance term for the ordered set of blasthole grades before precision estimates for block grades are calculated. Several bulk sampling programs have indicated that the measurement variance is indeed extraneous to the intrinsic variability.

Even though the measurement variance is extraneous, it is erroneous to assume it need not be optimized. If the measurement variance is too high, *in-situ* blocks cannot be divided into ore and waste but if it is too low, the cost for sample preparation is unnecessarily high. Therefore, a program should be implemented to routinely measure and monitor the total variance, the analytical variance and the intrinsic variability of gold.

Applied statistics has found wide application in science and engineering. In mineral exploration and mining, however, geostatistics is extolled as a viable alternative. Ironically, it is the only variant that violates the requirement of functional independence and is unencumbered with degrees of freedom. In applied statistics, every distance weighted average in the infinite set that two or more measured values define in n-dimensional sample spaces has its own variance. Moreover, the variance of any subset of an infinite set is a mathematical aberration. In geostatistics, however, kriging variances of sets of kriged estimates are deemed valid.

Even more troubling is that each kriged estimate defaults to the arithmetic mean of a set of measured values as the distance between its position and

## Precision Estimates for Grade

The objective of measuring blasthole grades is to divide a block into ore and waste, and to determine the grade of ore in an unbiased manner and with an acceptable and affordable degree of precision. Unbiased grade estimates and unbiased precision estimates for ore grade material is the essence of quality and grade control at gold mines.

If the gold grade of a subset of forty (40) blastholes turned out to be 1.84 g/t, and if the intrinsic variability of gold in terms of a CV were 95%, the variance of this set of grades would be  $(95 \cdot 1.84/100)^2 = 3.06 \text{ (g/t)}^2$ . The Central Limit Theorem teaches that the variance of the mean of a set of forty (40) point estimates in a block of *in-situ* ore is equal to  $3.06/40 = 0.0765 \text{ (g/t)}^2$  (Merks 1992).

This variance of  $0.0765 \text{ (g/t)}^2$  is equivalent to a standard deviation of  $\sqrt{0.0765} = 0.277 \text{ g/t}$ , a 95% confidence interval (95% CI) of  $sd(\bar{x}) \cdot t_{0.95;39} = 0.277 \cdot 2.019 = \pm 0.56 \text{ g/t}$ , or  $0.56 \cdot 100/1.84 = \pm 30\%$ , and a 95% confidence range with a lower limit of 95% CRL =  $1.84 - 0.56 = 1.28 \text{ g/t}$ , and an upper limit of 95% CRU =  $1.84 + 0.56 = 2.40 \text{ g/t}$ .

The value of  $t_{0.95;39} = 2.019$  can be found in the t-distribution at 95% probability and reflects that a set of 40 grades has 39 degrees of freedom. Multiplying the standard deviation with a tabulated value of  $t_{0.90;39} = 1.683$  and deducting the product from the gold grade of 1.84 g/t gives an asymmetric 95% lower limit of 95% ACRL =  $1.84 - 0.277 \cdot 1.683 = 1.37 \text{ g/t}$ .

Thus it appears that 95 out of 100 times the unknown true grade of this set is expected to fall within the symmetric 95% confidence range from 1.3 up to 2.4 g/t. Similarly, 95 out of 100 times the unknown true grade is expected to exceed the asymmetric 95% confidence limit of 1.4 g/t.

Table 3 summarizes the most relevant precision estimates for the gold grade of a block defined by a set of forty (40) blasthole grades:

TABLE 3  
PRECISION ESTIMATES FOR BLOCK GRADE

PARAMETER	SYMBOL	VALUE
GOLD GRADE in g/t	$\bar{x}$	1.84
95% CONFIDENCE INTERVAL in g/t	95% CI	$\pm 0.56$
95% CONFIDENCE INTERVAL in %	95% CI	$\pm 30.0$
95% CONFIDENCE RANGE in g/t	95% CR	
LOWER LIMIT	95% CRL	1.3
UPPER LIMIT	95% CRU	2.4
ASYMMETRIC LOWER LIMIT	95% ACRL	1.4

Low, medium and high grade stockpiles are sometimes maintained to ensure a reasonably constant feed grade to the processing plant. When less than 0.5 g/t is classified as waste, 0.5 to 1 g/t as low grade, 1 to 2.5 g/t as

ANOVA determines in probabilistic terms whether mineralization between measured grades is likely to be continuous. In this case, the probability is less than 95% but more than 90%. The  $\chi^2$ -distribution can be applied to determine the probability at the 50% level (even odds to encounter either ore or waste), or any other probability level listed in that fundamental distribution. However, ANOVA can only be applied if mathematically valid variance estimates are available and degrees of freedom are known.

The gold grade of 2.5 g/t and the variance of 9.18 (g/t)<sup>2</sup> for the randomly distributed set of blasthole grades gives a CV of  $\sqrt{9.18 \cdot 100 / 2.5} = 121\%$  while the variance of 6.80 (g/t)<sup>2</sup> for the ordered set gives  $\sqrt{6.80 \cdot 100 / 2.5} = 104\%$ . The variance of 6.80 (g/t)<sup>2</sup> is the most reliable estimate for the intrinsic variability of gold within the block under examination. CVs give intrinsic variability a convenient relative dimension.

If the F-ratio between the first variance term for the ordered set and the total variance is statistically significant, their difference is a valid estimate for the intrinsic variability. For example, the F-ratio of  $9.86 / 3.06 = 3.22$  exceeds  $F_{0.95;198;10} = 2.56$  but is still below  $F_{0.99;198;10} = 3.96$ . These tabulated F-values reflect that the first variance term has  $2(n-1) = 198$  degrees of freedom and that the total variance has 10 degrees of freedom. However, if only 5 pairs of secondary samples were selected of 100 primary samples, the F-value of 3.96 would be below  $F_{0.95;198;5} = 4.38$ . Hence, sufficient pairs of secondary samples should be selected, prepared and assayed to ensure that an unbiased estimate of the intrinsic variability of gold in each block is obtained.

Even if it were possible to select small secondary samples in an unbiased manner, the variance of the selection stage would add so much to the total variance that a valid estimate for the intrinsic variability can no longer be obtained. For example, if the total variance were to increase from 3.06 to 5.25 (g/t)<sup>2</sup>, the first variance term would become  $9.86 + 5.25 - 3.06 \approx 12.05$  for a calculated F-ratio of  $12.05 / 5.25 = 2.29$ , which does not exceed  $F_{0.95;198;10} = 2.56$ . Apparently, the same difference of 6.80 (g/t)<sup>2</sup> is no longer a valid estimate for the intrinsic variability of gold. Therefore, the total variance of the measurement chain should be optimized to ensure that *in-situ* blocks can be divided into ore and waste, and that reliable precision estimates for the gold grade of ore can be obtained.

If the measurement variance and the intrinsic variability were statistically identical, blasthole grades would be randomly distributed within blocks. By implication, *in-situ* blocks can no longer be divided into ore and waste, measurement chains degenerate into expensive random number generators, and mineral analysts are in trouble.

The term *spatial dependence* implies that ordered sets of measured values in n-dimensional sample spaces display a significant degree of associative dependence at some unspecified probability level. Under such conditions, the variance terms for an ordered set of blasthole grades, when plotted as a function of the spacing between blastholes, together with the variance of the randomly distributed set and its asymmetric lower confidence limits at 95% and 99% probability, display a sampling variogram. Hence, sampling variograms are graphic interpretations of Fisher's F-test.

2.41). The same F-value would be significant at 99% probability if both the numerator and the denominator have more than 30 degrees of freedom ( $F_{0.99;30;30} = 2.38$ ), or if the numerator has about 20 degrees of freedom and the denominator close to 40 ( $F_{0.99;20;40} = 2.37$ ).

The question whether or not  $var(s_2)$ , the variance of the secondary selection stage, is a significant part of  $var(t)$ , the total variance, may seem academic. After all, the total variance is extraneous to the intrinsic variability of gold within a block and deducted before precision estimates for its gold grade are calculated (Merks and Merks 1991). This premise, which is based on the additive property of variances, becomes intuitive upon recognizing that small secondary samples give higher total variances than large secondary samples do, but that the intrinsic variability of a static stochastic system such as a block of *in-situ* ore is independent of the measurement procedure applied to interrogate it. It does not imply, however, that the mass of secondary samples can be reduced with impunity. This matter is examined in more detail in the next sections.

### Intrinsic Variability

The basic measure for variability and precision is the variance. The intrinsic variability of gold in a block can be calculated from the set of blasthole grades by deducting the measurement variance from the first variance term for the ordered set. The variance of the randomly distributed set of blasthole grades is required to verify whether the degree of associative dependence between ordered blasthole grades is statistically significant.

The variance of the randomly distributed set is obtained by summing the squares of the differences between each measured value in the set and its central tendency (the arithmetic mean or the volume or mass weighted averages), and dividing the sum by  $df = n - 1$ , the degrees of freedom of the set. In formula,  $var(x) = \sum (x_i - \bar{x})^2 / (n - 1)$ .

The first variance term for the ordered set is obtained by summing the squared differences between grades of adjacent blastholes, and dividing the sum by  $df = 2(n - 1)$ , the degrees of freedom for the first variance term. In formula,  $var_1(x) = \sum (x_i - x_{i+j})^2 / [2(n - 1)]$ , in which  $j = 1$  for the first term.

ANOVA is applied to the variance of the randomly distributed set and the first variance term for the ordered set to test whether their F-ratio is statistically significant, and thus whether the degree of associative dependence between blasthole grades is statistically significant. For example, a calculated F-ratio of  $12.24/9.86 = 1.24$  between a variance of  $12.24 \text{ (g/t)}^2$  for a set of one-hundred (100) randomly distributed blasthole grades and a first variance term of  $9.86 \text{ (g/t)}^2$  for the ordered set does not exceed  $F_{0.95;99;198} = 1.32$  at 95% probability. Hence, ordered blasthole grades do not display spatial dependence when tested at 95% probability.

Deducting the measurement variance of  $3.06 \text{ (g/t)}^2$  gives a calculated F-ratio of  $(12.24 - 3.06) / (9.86 - 3.06) = 9.18 / 6.80 = 1.35$  which exceeds the tabulated value of  $F_{0.95;99;198} = 1.32$  at 95% probability. Evidently, the power of Fisher's F-test to detect spatial dependence increases if the total variance is deducted before the test is applied.

used in reference to the measurement variance and  $var(s_2pa)$ , the sum of its components. This section describes how to estimate  $var(s_2)$ , the variance of the secondary selection stage, and how to test its validity on the basis of the F-ratio between  $var(t)$ , the total variance, and  $var(pa)$ , the sum of the preparation variance and the analytical variance.

A simple test program to estimate the total variance is to select a pair of secondary samples (at opposite positions of the primary sample), to prepare a test sample of each secondary sample, and to assay a single test portion of each test sample. Because a single pair has only one degree of freedom, several pairs are required to obtain reliable and realistic confidence limits for the measurement variance.

A solid case can be made that the total variance and the analytical variance be routinely measured and monitored as part of a statistical quality control (SQC) program. A cost-effective program would be to select a pair of secondary samples of 5% up to 10% of all blastholes in a block, to prepare a test sample of each secondary sample, and to select and assay duplicate test portions of each test sample. The selected blastholes should preferably located in sections where ore grade was encountered at higher levels because they give much more useful information than blastholes in waste do. Each pair of secondary samples gives one degree of freedom for the total variance, and two for the analytical variance.

The most complete and expensive test program would be to select a pair of secondary samples of a primary sample, to prepare a pair of test samples of each secondary sample, and to select and assay duplicate test portions of each test sample (see Figure B). For a single blasthole,  $var(t)$ , the total variance, has one degree of freedom,  $var(pa)$ , the sum of the preparation variance and the analytical variance, has two, and  $var(a)$ , the analytical variance, has four. Thus the lowest class in the measurement hierarchy (selecting and assaying test portions) has the most degrees of freedom and gives the most precise variance estimate, and the highest class (selecting secondary samples) has the least degrees of freedom and gives the least precise variance estimate. Yet the highest class gives the most valuable information.

When the degree of associative dependence between CVs and grades is not significant at the analytical stage, it is even less likely to be significant at the secondary selection stage. Under such conditions, CVs can be used to calculate total variances. For example, a CV of 70% for a gold grade of 2.5 g/t gives  $var(t) = (70 * 2.5 / 100)^2 = 3.06 (g/t)^2$ . The question whether  $var(s_2)$ , the variance of the secondary selection stage, adds significantly more to  $var(t)$ , the total variance, than does  $var(pa)$ , the sum of the preparation and analytical variances, can be solved by comparing the calculated F-value of  $3.06 / 1.27 = 2.41$  with tabulated values of F-distributions at 95% and 99% probability and applicable degrees of freedom.

The probability level determines which F-distribution to select, the degrees of freedom for the numerator and the denominator determine in which cell the required F-value can be found. For example, the F-value of 2.41 would be significant at 95% probability if  $var(t)$ , the total variance (the numerator in Fisher's F-test), and  $var(pa)$ , the sum of the preparation and analytical variances (its denominator), have 15 degrees of freedom ( $F_{0.95;15;15} =$

The flowchart in Figure B illustrates that a blasthole pattern divides a block of *in-situ* ore into a set of elementary units, that the primary sample is a mass of cuttings around a blasthole in the center of an elementary volume, that a secondary sample is a part of the primary sample, that a test sample is a part of the secondary sample, that a test portion is a part of the test sample, and that the analysis of a test portion gives the test result (a point estimate for the gold grade of an elementary unit).

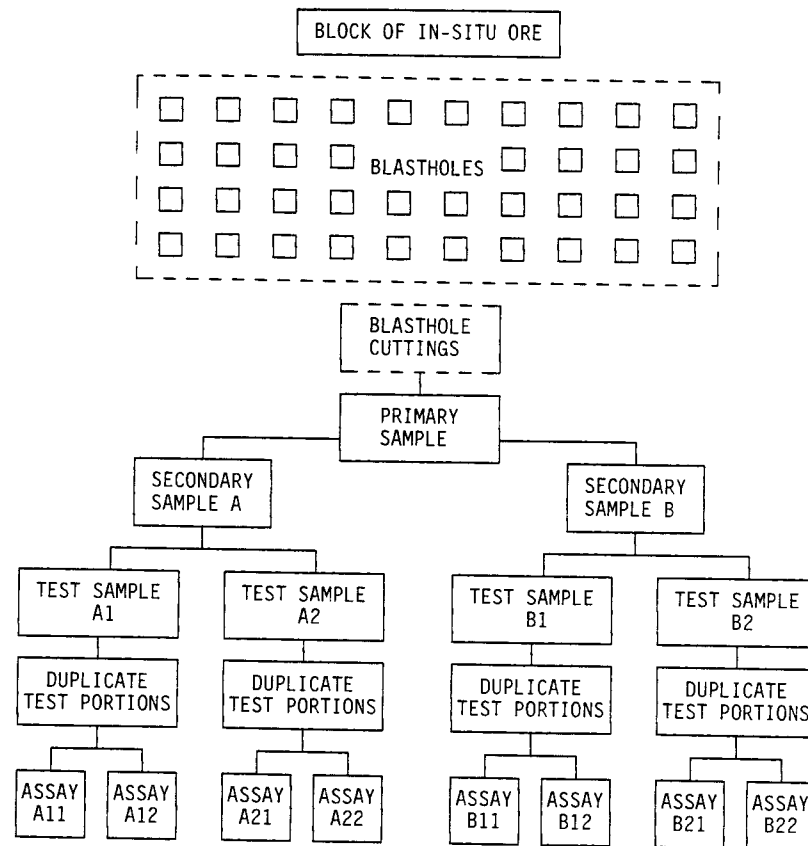


FIGURE B—COMPLETE TEST PROGRAM FOR BLAST HOLE GRADES

The intrinsic variability of gold within a block of *in-situ* ore depends on several factors, not the least of which is the distance between blastholes. In fact, the variance terms for the ordered set become smaller with the distance between blastholes while the variance of the randomly distributed set stays the same, if not numerically than at least statistically. Therefore, the intrinsic variability is almost invariably the difference between the first variance term for the ordered set of blasthole grades and the total variance of the measurement chain.

The measurement variance for each point estimate is the sum of  $var(s_2)$ , the variance for selecting a secondary sample,  $var(p)$ , the variance for selecting a test sample, and  $var(a)$ , the variance for selecting and assaying a test portion. For simplicity, the term *total variance* and the symbol  $var(t)$  are

distribution. In sampling practice, only carefully homogenized masses of particulate material tends towards the perfect degree of homogeneity, and degrees of freedom are only awarded to two or more measured values.

Generally, the preparation variance can be estimated by selecting a pair of subsamples at the highest level in the measurement hierarchy, by preparing a test sample of each, and by selecting and assaying a test portion of each test sample. For example, a test program based on ten (10) pairs of test samples showed that the sum of the preparation variance and the analytical variance is equivalent to a CV of 45%, and that this CV is independent of grade within the range from 1 up to 5 g/t. For a gold grade of 2.5 g/t a CV of 45% gives  $var(pa) = (45 * 2.5 / 100)^2 = 1.27 (g/t)^2$ , in which  $var(pa)$  is the sum of the preparation variance for a single test sample and the analytical variance for a single test portion.

The previous section showed that a CV of 20% for a grade of 2.5 g/t gives an analytical variance of  $var(a) = (20 * 2.5 / 100)^2 = 0.25 (g/t)^2$  for a single test portion. ANOVA indicates that the calculated F-value of  $var(pa) / var(a) = 1.27 / 0.25 = 5.08$  exceeds not only  $F_{0.95;10;10} = 2.97$  at 95% probability but also  $F_{0.99;10;10} = 4.87$  at 99% probability. Hence, the probability that variance estimates of 1.27 and 0.25 (g/t)<sup>2</sup> are compatible (statistically identical) is less than 1%. By implication, their difference of  $var(p) = var(pa) - var(a) = 1.27 - 0.25 = 1.02 (g/t)^2$  is a valid estimate for the variance components associated with the preparation of a single test sample of a secondary sample.

If twenty (20) duplicates were used to calculate the analytical variance (duplicate test portions of each test sample), the calculated F-value would be  $var(pa) / [var(a) / 2]$ . In this case, however, the sum of the preparation variance for single test samples and the analytical variance for the means of duplicates is somewhat less (close to 0.125 to be exact) than 1.27 (g/t)<sup>2</sup>. Logically, the F-value of  $(1.27 - 0.125) / 0.125 = 9.16$  for duplicates is higher than  $1.27 / 0.25 = 5.08$  for single test results. The most reliable estimate for the preparation variance remains  $(1.27 - 0.125) - 0.125 = 1.02 (g/t)^2$ . After all, the additive property of variances implies that  $var(p)$ , the preparation variance, is independent of  $var(a)$ , the analytical variance, irrespective of how many test portions of the same test sample are assayed.

Since the preparation variance is significantly higher than the analytical variance it is tempting to suggest that it be reduced by increasing the mass of test samples, by pulverizing test samples to a finer particle size, or by a combination of both. Whether lower variances and higher costs for sample preparation improve the precision for gold grades of blocks of *in-situ* ore is an important subject. It will be explored in more detail in the next sections.

### Secondary Selection Stage

The secondary selection stage is the crucial link in the measurement chain applied to generate a point estimate for the grade of each elementary unit in the set into which the blasthole pattern divides a block of *in-situ* ore, and in the process of classifying one or more subsets as ore grade material, and calculating the gold grade of each subset and its precision estimates in terms of confidence limits.

## Sample Preparation Stage

Mineral analysts who are responsible for the preparation of test samples of all types of materials are aware that the potential for bias at this stage is high. Loss of native metals or fine dust are common causes for systematic errors but more insidious systematic errors occur. For example, exchange samples of sulfide concentrates should be prepared of test portions in which mass loss on drying is determined. Drying test samples for metal grades and test portions for mass loss under different conditions often causes systematic errors in metal grades of exchange samples. Exchange samples should be packed in air and moisture impervious envelopes within kraft envelopes to ensure their integrity during shipping and storage.

Whenever test results are at variance with expectations and the presence of analytical bias has been ruled out, the search for a plausible explanation invariably turns to the sample preparation stage. This section deals with the preparation of test samples selected of secondary samples, which, in turn, were selected of primary samples (cuttings around blastholes), and in particular with the variance associated with this stage of the measurement procedure. It does not deal with the preparation of test samples of primary samples such as sections of whole drill core, or of secondary samples such as sections of halve drill core or masses of crushed and divided drill core or cuttings.

Because Gy (1979) is often quoted when sample selection and preparation stages are discussed, a synopsis of his theory is presented. Gy's sampling formula implies that  $\sigma^2_{(FE)}$ , the variance of his fundamental error, is a function of  $C$ , his sampling constant, and of  $d$ , the top size of the particulate matter. In probability theory, the symbol  $\sigma^2$  refers to the population variance, an elusive constant for which measurement procedures provide estimates only. The passage from mathematical probability and its constant population variances to applied statistics with variances of samples of populations, to which confidence limits apply, necessitated the concept of degrees of freedom.

Gy's sampling constant  $C$  is a function of  $c$ , the mineralogical composition factor, of  $l$ , the liberation factor, of  $f$ , the particle shape factor, and of  $g$ , the size range factor. Given that each of these factors is a stochastic variable, it follows that the variance of Gy's sampling constant is  $var(C) = C^2 * [var(c)/c^2 + var(l)/l^2 + var(f)/f^2 + var(g)/g^2]$ . This formula implies that Gy's sampling constant is only a constant if the variances of his mineralogical factors are infinitesimally small. In the real world, the only constant is the International Unit of Mass, whose mass is known with absolute certainty if only by definition. Unlike Gy's sampling constant  $C$ , which is a function of stochastic variables whose variances can only be estimated with a finite degree of precision.

Whether Gy's factors and their variances, and the top size of the mass of particulate matter and its variance, can be obtained at lower cost than the variable of interest and its variance can be estimated is an intriguing question. Not only because Gy's sampling constant seems to defy the constraints of applied statistics with its finite variances and degrees of freedom but even more so because Gy's sampling formula is derived of the formula for the variance of a perfectly homogeneous binomial probability

applied to test whether the CV is constant, and can be used to calculate the analytical variance of other grades within that range. For example, a CV of 20% for a test portion with a gold grade of 2.5 g/t gives an analytical variance of  $(20 * 2.5 / 100)^2 = 0.25 \text{ (g/t)}^2$ . The same CV gives an analytical variance of  $(20 * 5.0 / 100)^2 = 1.00 \text{ (g/t)}^2$  for a test portion with a gold grade of 5.0 g/t.

The calculation of confidence limits for variables and variances requires the use of probability distributions in which values are tabulated as a function of degrees of freedom. The mineral analyst should be aware that degrees of freedom are essential in applied statistics but irrelevant in geostatistics (Merks 1993). In applied statistics, a pair of test results (duplicate test portions of a test sample) has one degree of freedom but a single test result does not have any. Generally, a set of  $n$  test results, determined in replicate test portions of a test sample, has  $n - 1$  degrees of freedom.

Analysis of variance (ANOVA) can be used to calculate confidence limits for variance estimates (Volk 1980). For example, the lower limit of the 90% confidence range for a variance estimate of  $0.25 \text{ (g/t)}^2$  is  $0.25 / F_{0.95; df; \infty}$ , and its upper limit is  $0.25 * F_{0.95; \infty; df}$ . The values of  $F_{0.95; df; \infty}$  and  $F_{0.95; \infty; df}$  are listed in the F-distribution at 95% probability as a function of degrees of freedom. The symbol  $df$  refers to the degrees of freedom.

If this variance estimate of  $0.25 \text{ (g/t)}^2$  were based on a pair of test results with a single degree of freedom, dividing it by  $F_{0.95; 1; \infty} = 3.84$  would give a lower 90% confidence limit of  $0.25 / 3.84 = 0.065 \text{ (g/t)}^2$ , and multiplying it with  $F_{0.95; \infty; 1} = 254$  would give an upper 90% confidence limit of  $0.25 * 254 = 63.5 \text{ (g/t)}^2$ . Table 2 summarizes the lower and upper limits of 90% confidence ranges for this variance estimate of  $0.25 \text{ (g/t)}^2$  when measured with increasing numbers of degrees of freedom:

TABLE 2  
90% CONFIDENCE RANGES FOR A VARIANCE ESTIMATE OF  $0.25 \text{ (g/t)}^2$

DEGREES OF FREEDOM	$F_{0.95; df; \infty}$	90% CRL	$F_{0.95; \infty; df}$	90% CRU
ONE	3.84	0.07	254	63.5
TWO	2.99	0.08	19.5	4.9
FOUR	2.37	0.11	5.63	1.41
NINE	1.88	0.13	2.71	0.68
SIXTEEN	1.64	0.15	2.01	0.50
TWENTY-FIVE	1.51	0.17	1.71	0.43
INFINITE	1.00	0.25	1.00	0.25

These confidence limits suggest that variances of small sets of test results are extremely imprecise, and should therefore be interpreted with caution. Table 2 also displays the astounding precision of confidence limits when the variance is determined with infinite degrees of freedom. Not surprisingly, any variant of applied statistics unencumbered with degrees of freedom should be viewed with suspicion. How to estimate the analytical variance and its confidence limits in an unbiased manner and at affordable costs is only the first step in effective self-defense for mineral analysts.

Variances are amenable to mathematical analysis. The properties of variances play a crucial role in measurement technology. The additive property of variances of volumes, wet masses, dry masses and contained metals is useful to geologists, mining engineers and metallurgists. The additive property of variances of variables within measurement hierarchies is of particular concern to mineral analysts. The additive property is not necessarily commutative because the difference between two variances is only a valid variance estimate if they are incompatible (statistically different). Therefore, analysis of variance (ANOVA) should be applied to verify that the F-ratio between two variances is statistically significant before mathematical analysis is applied to their difference.

### **Analytical Stage**

Mineral analysts are invariably responsible for the analytical stage, the last stage in the measurement hierarchy. How to test for analytical bias, how to estimate analytical variances, how to evaluate analytical methods, and how to calibrate analytical instruments, are familiar subjects. Mineral analysts should also know how to estimate the total variance, how to partition the total variance into its components, and how to optimize the measurement chain. Knowledge of those subjects is a key element of self-defense.

The analytical variance includes a component of variance associated with the selection of a test portion of a test sample (analysis sample or pulp sample). Therefore, the mass of a test portion, and the presence of coarse particles (the single particle effect) or of native gold (the nugget effect), impact on the analytical precision. As a result, some overlap between this section and the section on sample preparation could not be avoided. After all, comminuting test samples to a finer particle size, homogenizing test samples prior to analysis, and assaying larger test portions, reduces the variance for selecting test portions of test samples, which is an integral component of the analytical variance.

While such corrective actions do reduce the analytical variance, the cost for sample preparation soars which proves the adage that precision always has its price. The question is then whether lower analytical variances translate into higher degrees of precision for gold grades of *in-situ* ore.

Screening test samples and assaying coarse and fine fractions separately solves the nugget effect most effectively but the cost is too high for drill holes at the exploration stage and for blastholes at the mining stage. Gold grades of mill feed at mineral processing plants, and of shipments to other mines or to smelters, can be determined with a significantly higher degree of precision when the coarse and fine fractions of test samples are weighed and assayed separately and the mass weighted average grades are used for settlement. For a gold ore with an average grade of 15 g/t it reduced the analytical variance in terms of a CV from more than 50% to less than 5%.

The analytical variance is estimated either by assaying a set of test portions of the same test sample or by assaying a pair of test portions of each test sample in a set with different gold grades. The latter approach is preferable because the analytical variance and its standard deviation are frequently a function of grade. Therefore, correlation-regression analysis should be

stage is taking a part of the primary sample mass. The sample preparation stage is the process of preparing a test sample of the secondary sample, and the analytical stage is the process of selecting a test portion of the test sample and assaying it. Figure A is a flowchart outlining the various stages of this measurement hierarchy.

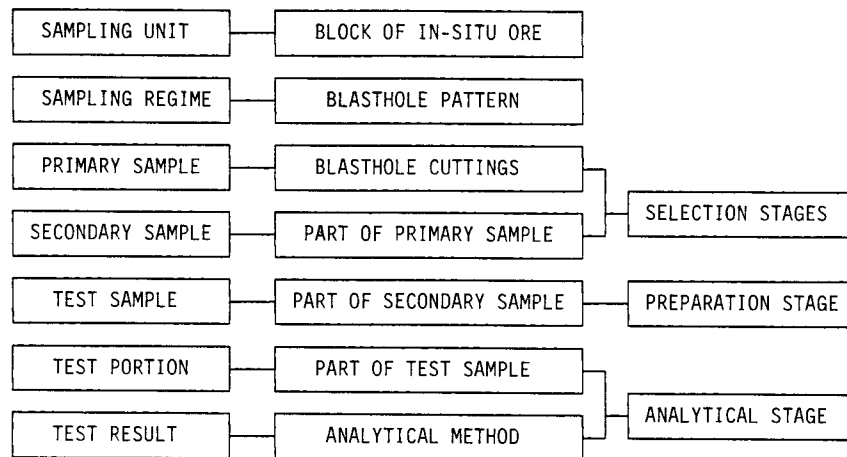


FIGURE A--STAGES OF THE MEASUREMENT HIERARCHY

Each stage of this measurement hierarchy adds a component of variance to the total variance of the entire chain, which, in turn, is added to the intrinsic variability of gold within the block. Given that the intrinsic variability determines the precision for grade, it follows that the total variance of the measurement chain should be deducted before precision estimates are calculated.

### Measures for Variability and Precision

The variance is the fundamental measure for precision. Because its dimension is the square of the measured value, the variance is not very user-friendly. Derived measures for precision such as the standard deviation, which is the square root of the variance and has the same dimension as the measured value, and the coefficient of variation (CV), which is the standard deviation as a percentage of the measured value, are readily-understood and user-friendly measures for precision.

Standard deviations of analytical methods are often linear functions of grades. Because the ratio between a standard deviation and a measured value reduces this effect, CVs are effective measures for the analytical precision of single test results. Confidence intervals and ranges, and asymmetric lower or upper limits at different probability levels, are commonly used to report precision estimates, either for the arithmetic mean of a set of measured values with identical weight factors or for the weighted average of a set of measured values with different weight factors.

A statistical evaluation indicated that the sampling variance was by far the largest component of the total variance. The preparation variance was low and indeterminable because the sum of the sample preparation variance and the analytical variance could not be partitioned into its components at affordable cost. The question was how to reduce the sampling variance such that the sample selection stage and the sample preparation and analytical stages would add comparable components to the total variance.

A test program indicated that the sampling variance could be reduced to an acceptable level if a 25 mm button were punched from every cathode after its halves were firmly pressed together following stripping of the feeder sheet. Sampling theory teaches that the position of each punch should be selected at random. Sampling practice suggests that, for a sampling unit of 640 cathodes, each position should be selected at random from a 25 by 25 matrix (a set of 625 positions).

Dividing the set into a pair of interpenetrating subsets such that one consists of all odd-numbered buttons and the other of all even-numbered buttons, and analyzing each subset for selenium in duplicate, generates estimates for the total variance and the analytical variance. This sampling protocol provides all the information required to implement statistical quality control for trace elements in copper cathode production.

Experiments with the punch module indicated that the sampling protocol was optimized when a single button was punched of every other cathode. Precision estimates in terms of 95% confidence intervals and ranges for manual sampling (2 cathodes out of 640) and for mechanical sampling (320 buttons out of 640 cathodes) are listed below:

TABLE 1  
PRECISION FOR SELENIUM IN CATHODE COPPER

PARAMETER	SYMBOL	MANUAL	MECHANICAL
MEAN in ppm	$\bar{x}$	2.5	1.1
95% CONFIDENCE INTERVAL	95% CI	±6.7	±0.24
95% CONFIDENCE RANGE	95% CR		
LOWER LIMIT	95% CRL	0	0.9
UPPER LIMIT	95% CRU	9	1.3

In this case the mineral analyst recognized that the precision for selenium in cathode copper could be improved by reducing the sampling variance, and not by reducing the analytical variance. The design of a button punch module, and its installation between the strip and strap modules in the cathode handling system, presented an acceptable compromise between cost and precision, and added a key component to the quality management system at the copper refinery.

A typical measurement hierarchy at an open-pit mine is the determination of blasthole grades of blocks of *in-situ* ore. The primary selection stage is extracting a mass of drill cuttings from a block, and the secondary selection

The ability to examine, in a consistent and commonsensical manner, the integrity of measurement procedures is the essence of self-defense for mineral analysts. Almost invariably the suspected value is either a single measured value of a random variable (a stochastic variable), or the mean of a set of replicates. A single measured value does not give any information on the precision of the procedure but two or more do. How to estimate the precision for systems and procedures at affordable cost is an important element of measurement technology.

The term *random variable* or *stochastic variable* implies that two or more measured values of a variable are not likely to be numerically identical due to randomly distributed variations in the measurement chain. The sum of such randomly distributed variations (random variations) tends towards zero while the cumulative effect of a systematic error or bias tends towards a value significantly different from zero. How to partition such uncertainties into random variations and a bias is an important subject in metrology, the science of measurement.

Examples of random variables in mining and metallurgy are the mass of gold contained in a gold deposit, the copper grade of mill feed processed during a shift, the mass of silver contained in a shipment of lead concentrate, the selenium concentration in a shipment of cathode copper, and many others. Even though each of those random variables can be expressed as a single value, they are not equally precise nor are they necessarily unbiased. How to estimate precision and how to test for bias are important steps in the validation of measurement systems and procedures.

### Measurement Hierarchy

It is much more complex and expensive to estimate gold grades of deposits than it is to estimate selenium concentrations in cathode copper. Each of the applied measurement procedures has a sample selection stage, a sample preparation stage and an analytical stage, which form a *measurement hierarchy of nested classes*. Each stage adds a component of variance to the total variance, the sum of all variances within the measurement chain, and thus an element of uncertainty to the measured value of the variable.

The symbol  $var(s_i)$  refers to the variance of the  $i$ th sample selection stage,  $var(p)$  to the variance of the sample preparation stage,  $var(a)$  to the variance of the analytical stage, and  $var(t)$  to the total variance of the measurement chain. The total variance is the sum of the variances for the sample selection stages, the variances for the sample division stages during sample preparation, and the analytical variance (Merks 1985, Gy 1979).

The determination of selenium in cathode copper is a typical example of a measurement hierarchy that caused a severe problem to a mineral analyst. Whenever a client complained that the selenium concentration in a cathode copper shipment exceeded the maximum allowable limit, the mineral analyst would analyze the client's sample and confirm that selenium did not meet the specification. At the refinery trace elements in cathode copper production were determined in each of a pair cathodes randomly selected of the set of 640 in a single tank.

the measured values increases. Hence, kriging augments gold grades when applied to *in-situ* ore with higher than average grades. By implication, a similar volume or mass with lower than average grades should be kriged to ensure that the mass of contained gold remains constant. The evidence suggest that kriging ore with high grades is far more popular. Kriging also enhances spatial dependence and creates it where none exists.

In applied statistics, the probability to encounter mineralization between measured values is quantified by applying analysis of variance to the variance of randomly distributed grades and the first variance terms for ordered grades. The application of analysis of variance demands that the degrees of freedom of these variances be known. In geostatistics, the concept of degrees of freedom is trivialized and ignored.

If gold grades of closely spaced blastholes do not even display a significant degree of spatial dependence, the validity of any geostatistical model that is based on widely spaced exploration data is suspect. Yet discrepancies between such models and the grades of in-situ ore, mined ore or mill feed invariably cause more problems to mineral analysts and their analytical methods than to geostatistical modelers and their questionable techniques.

A case in point is the current trend to prepare large test samples, to assay large test portions, or to assay large test portions of large test samples. Neither course of action necessarily improves precision estimates for grades but it certainly raises costs for sample preparation and analyses. Mineral analysts who are familiar with probability and statistics are in an excellent position to optimize the measurement chain applied to estimate blasthole grades and to calculate precision estimates for grades. Indeed, knowledge of applied statistics is the key to effective self-defense for mineral analysts.

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