Part 2: Experimental calibration of sampling parameters *K* and alpha for Gy's formula by the sampling tree method

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Transaction Paper

Synopsis

Application of Gy's formula is appropriate for calculation of the Fundamental Error (FE) prior to samples being collected at any stage of the minerals process, provided that sampling parameters *K* and α (alpha) have been determined by calibration of the ores. Two types of calibration procedure are well established in the industry, the Hetrogeneity test and the 32-piece sampling tree experiment. The sampling tree experiment uses the variance and average grade of metal analyses from four series of samples at different nominal fragment size, as the basic input data. Pierre Gy suggested the cube as an appropriate exponent of the nominal fragment size for use in his equation. More recent work suggests that this exponent is variable and characteristic of specific ores and therefore requires calibration. The method described here explains the experimental procedure of the calibration. It is then possible to calculate a sampling nomogram using the sampling parameters *K* and α. This allows the sampler to determine a suitable protocol for the sampling event that will ensure that the FE does not exceed a predetermined precision at any point in the sampling procedure.

Introduction

The pioneering work of Pierre Gy (1973, 1979, 1982, 1992, 1995 and 1998) in the area of sampling of broken ores has provided the minerals industry with the theory and methods for identifying and quantifying a variety of different errors that arise during the sampling procedures and was described in Part 1 of this work. His work was initially directed to evaluating errors associated with the sampling of broken ores that are handled at various stages in the minerals industry. One of these errors, referred to as the Fundamental Error, arises because of both the constitutional error and distributional error that are inherent in any broken ore, and is given by Gy's formula as:

$$\sigma^2_{FE} = \frac{Kd^3_N}{M_S}$$

where K is a constant specific to a given ore-type at a given grade, d_N is the nominal top-size of fragments in the sample in cm, measured as 95% passing a given screen size, and M_S is the mass of the sample in grams. Gy suggested the use of the cube of the nominal top size was appropriate, but empirical work around this exponent has led to the suggestion that its value is a function of the ore type, rather than having a constant value of three. The work undertaken by François-Bongarçon (1993 and 1995) opened a new understanding and approach to the interpretation of paired samples and the way in which the Fundamental Error could be evaluated. Furthermore, both the exponent and the value for K can be calibrated, as demonstrated in this paper.

Application of the formula provided by Pierre Gy to the construction of the sampling nomogram for specific ores requires that two parameters, K and alpha, that are unique to every ore type, be determined. There are at least two methods for determining these parameters, namely the heterogeneity test, a method proposed by Pitard (1993, 2004 and 2005), and the sampling tree method proposed by François-Bongarçon (1995 and 1998). The aim of this paper is to describe the sampling tree experiment, a method for deriving values for the constants K and alpha, to be substituted into Gy's formula so that a sampling nomogram for a specific ore type can be compiled. The method proposed by François-Bongarçon (1995) followed a detailed analysis of the application of Gy's formula to the determination of the Fundamental Error. This phase of research led to a number of significant confirmations and clarifications of

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concepts that were already known in the theory of sampling; firstly, that paired assay data can be used to determine the sampling parameters *K* and alpha and hence to derive the Fundamental Error (FE) for any given mass at any given nominal fragment size; secondly, that the liberation size could be determined using the sampling parameters *K* and alpha derived by the proposed experimental procedure; and thirdly that the FE is a function of the grade of the ores being sampled (François-Bongarçon, 1999).

This paper explores the experimental procedure proposed by François-Bongarçon (1995) and attempts to lay the processes out in such a way that they can be used to establish the sampling parameters. Application of the sampling parameters to the construction of sampling nomograms will provide those responsible for the sampling protocols at mining operations with a means of reducing the FE to a minimum.

Calibration curve

In simplified form, Gy's formula relates the variance of the Fundamental Error directly to the size of the fragments comprising the sample and inversely to the mass of the sample, and is given by:

$$\sigma_R^2 = \frac{Kd_N^\alpha}{M_S} \quad [1]$$

where K is a constant factor being the product of the mineralogical factor (c), the shape factor (f), the granulometric factor (g) and the liberation factor (l). The individual components of *K* have been described in detail by Pitard (1993, 2004) and are not dealt with further in this paper. The cube of the nominal top-size of the fragments in the sample has been replaced here by alpha, α, a variable that can be determined by calibration. The form of Gy's equation can be changed by taking the logarithms of both sides of Equation [1] after transposing to get:

$$\ln\left(\sigma_R^2 \times M_S\right) = \alpha \ln d_N + \ln(K) \quad [2]$$

This is a straight equation line of the form $y = mx + c$ that relates the product of the sample mass and the variance of the Fundamental Error ($\sigma_{FE}^2 \times M_S$) to the nominal top-size of the fragments d_N comprising the sample. Estimates of the error variance are derived from four sets of analyses of the grade of the samples at different nominal top-sizes and a linear calibration curve is derived by plotting $\ln(\sigma_R^2\ M_S)$ against $\ln(d_N)$. The best-fit line through the data points allows the sampling parameters to be estimated:

- The slope of the line is α
- The intercept of the line on the y-axis is ln (K)

Experimental procedure

The following procedure tends to be specific to the gold mining industry, but modified versions are applicable to all types of mineral ores. The procedure for creating the four points on a calibration curve is described as follows. Firstly depending on the ore itself, a 30 kg to 60 kg lot of run-of-mine ore, or reef material for the ore zone of a mine, can be selected, depending on the reasons for undertaking the calibration exercise. The primary crushing stage is through a jaw crusher that reduces the nominal size of the ore to a uniform 95% passing -1.9 cm. One-quarter of the total lot at 95%–1.9 cm is split out and forms the first nominal size fraction. The remainder of the ore is then crushed to 95% passing -0.95 cm and the lot is then split into three equal portions. One of the three portions is set aside and constitutes the second nominal size fraction. The remaining two fractions are recombined, crushed to 95% passing -0.1 and split into two portions of equal mass. One portion is set aside as the third nominal size fraction and the remaining portion, the fourth in the series, is crushed to 95% passing -150 microns. Thus we have four portions of more or less equal mass, about 10 to 15 kg per portion, of ore at four different nominal sizes namely, -1.9 cm, -0.95 cm, -0.1 cm and -150 microns.

Using a riffle splitter, each of these portions is now split into 32 sub-samples, the binary sampling tree used to construct the experiment being shown in Figure 1. Each nominal size fraction will be split into 32 samples, from which two samples are chosen at random for granulometric analysis, leaving 30 samples per size fraction. Thus the whole lot is split into 120 samples of more or less equal mass, say 250 g to

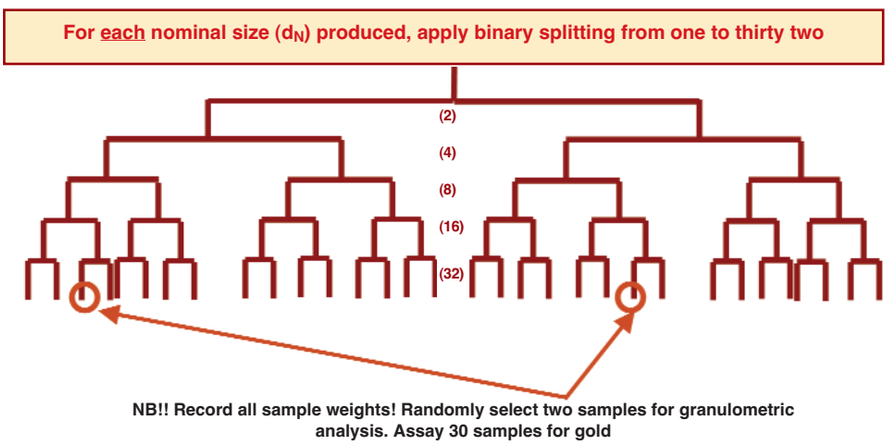

Figure 1—The binary sampling tree applied to each of the four nominal size fractions

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500 g each depending on the mass of the original lot. All sample masses must be recorded and the samples are submitted for assay according to standard procedure.

Table I shows the assay results for the four series of analyses from the sample tree experiment. The basic statistics have also been calculated and are shown in Table II. For this particular exercise we assume an analytical variance of 4% which is just below the 5% that is normally assumed for this type of exercise.

Details of the standardization procedure necessary to derive values of $\ln(\sigma_R^2\ M_S)$ and In d_N from the experimental data are provided in Table II. This procedure yields the relevant data, namely the $\log_e$ value of the sample variance times the sample mass, $Ln((\sigma_r)^2 \times M_{sample})$ and the $\log_e$ value of the nominal top size, $Ln(d_N)$, in the right units at the right scales. Gy's formula

$$\sigma_{FE}^2 = \frac{Kd_N^{\alpha}}{M_S}$$

can be re-arranged to give a liner equation in $\log_e$ as follows:

$$Ln((\sigma_R)^2\ M_{sample}) = Ln(K) + \alpha Ln(d_N)\ t$$

On this basis plot $Ln((\sigma_R)^2\ M_{sample})$ against $Ln(d_N)$ to obtain K (the Y-intercept) and α (the slope). It is also important to remember that at liberation

($d_N < d_l$ and $\alpha = 2.5$; thus $K_{lib} = K_{nlib}/d_l^{2.5\ -\ \alpha}$).

The data for the calibration curves for determining K and alpha are provided in Table III and are plotted in Figure 2.

Using the technical data provided in Table III and an appropriate preparation protocol it is possible to determine the sampling parameters and compile a sampling nomogram for this ore. This raises two questions about the sampling protocol: 'What does the sampling nomogram tell you about the sampling protocol?' and 'How can the protocol be improved?'

This procedure describes:

- The sampling tree experiment that is used to create the sample series to be used for analysis
- The analysis and reduction of the analytical results
- The determination of the main sampling parameters
- The compilation of the sampling nomogram
- The error associated with the analytical procedure.

Protocol for the sampling tree experiment

The position in the ore flow, from stope face to mill, at which the broken ore is selected for analysis, depends on the aim and objects of the exercise. In the particular experiment described here the ore was collected from the crushed ore conveyor belt immediately after the primary crusher that reduced the blasted run-of-mine (RoM) ore to about 95%–40cm. This experiment was designed specifically to establish the sampling parameters for these ores in order to compile an appropriate sampling nomogram. Approximately 35 kg of dried RoM ore was collected from a conveyor belt after the primary crush and then comminuted to 95%–2.5cm. The 35 kg sample was split into four equal sub-samples (A, B, C and D) using a riffle splitter, each sub-sample weighing about 8.75 kg. Sub-samples B, C and D were then recombined.

Treatment of sub-sample A

Using a riffle splitter, split sub-sample A into 32 fractions each weighing about 273 g, according to the sampling tree pattern shown in Figure 1.

Table I

Assay results (g/t Au) for the four series of sub-samples

Series	Series 1	Series 2	Series 3	Series 4
Sample No	**2.5cm**	**0.3cm**	**0.1cm**	**0.05cm**
1	12.96	12.08	12.76	11.42
2	14.80	11.82	13.38	13.96
3	11.86	12.08	14.24	11.08
4	11.94	12.16	13.40	12.34
5	9.56	12.88	13.14	12.98
6	10.38	12.42	12.54	11.86
7	15.76	11.76	12.72	12.32
8	17.94	12.78	12.28	12.40
9	9.88	11.32	14.28	13.04
10	5.44	11.80	12.52	13.22
11	9.54	12.74	12.78	12.28
12	14.10	12.90	13.90	12.16
13	10.28	10.86	12.40	11.62
14	12.78	12.34	13.04	12.04
15	10.78	12.24	11.36	13.24
16	14.12	12	12.74	12.76
17	9.92	12.18	13.20	12.18
18	10.30	14.04	14.18	12.04
19	24.26	12.76	12.50	12.86
20	8.78	12.56	12.98	12.78
21	6.66	14.90	13.16	12.66
22	9.98	13.40	13.94	12.50
23	13.30	12.94	13.10	12.22
24	16.48	11.28	14.44	12.40
25	29.40	12.10	11.60	12.18
26	11.68	12.96	12.7	12.44
27	9.52	14.40	13.20	11.92
28	13.46	15.70	12.94	12.58
29	20.14	12.98	12.66	12.72
30	12.64	13.97	13.26	12
Average	12.95	12.68	13.04	12.41
Variance	24.38	1.15	0.53	0.34

Treatment of recombined sub-sample B, C and D

Crush the recombined sample B, C and D to 95%–0.3cm and split the sample into three more-or-less equal portions (B, C and D), each weighing 8.75 kg, using a rotary splitter. Recombine sub-samples C and D.

Treatment of sub-sample B

Using a riffle splitter split sub-sample B into 32 fractions each weighing about 273g, according to the sampling tree pattern shown in Figure 1.

Treatment of recombined sub-sample C and D

Crush the recombined sample C and D to 95%–0.1cm and split the sample into two more-or-less equal portions (C and D), each weighing about 8.75 kg, using a rotary splitter.

Treatment of sub-sample C

Using a riffle splitter split sub-sample C at 95% –0.1 cm, into 32 fractions each weighing about 273 g, according to the sampling tree pattern shown in Figure 1.

Table II

Statistical results of assay data in Table I

Statistic	Series 1	Series 2	Series 3	Series 4
Total mass of sample (35 kg)	340.07	385.47	385.47	316.10
Nominal size	2.50	0.30	0.10	0.05
Average grade	12.95	12.68	13.04	12.41
Variance	24.38	1.15	0.53	0.34
Relative variance	0.1453	0.0071	0.0031	0.0022
Analytical variance	0.0016	0.0016	0.0016	0.0016
Residual variance	0.1437	0.0055	0.0015	0.0006
Standardised rel variance	1.882	0.091	0.041	0.027
$Ln(d_N)$	0.9163	-1.2040	-2.3026	-2.9957
$Ln(\sigma^2 \times M_S)$	6.4615	3.5522	2.7562	2.1428

Table III

Data from Table II for compiling a linear scale calibration curve

	Series 1	Series 2	Series 3	Series 4
$Ln(\sigma^2 M_S)$	6.4615	3.5522	2.7562	2.1428
$Ln(dN)$	0.9163	-1.2040	-2.3026	-2.9957

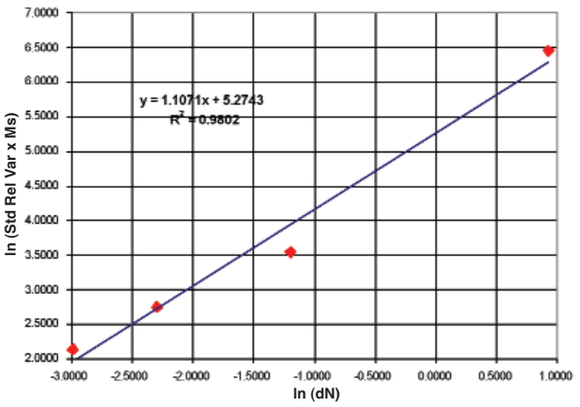

Figure 2—Calibration curve for *K* and α for a gold ore with an average grade of 12 g/t Au

Treatment of sub-samples D

Crush the sub-sample D to 95%–0.05 cm. Then using a riffle splitter split sub-sample D at 95%–0.05 cm, into 32 fractions each weighing about 273 g, according to the sampling tree pattern shown in Figure 1.

At this stage the RoM ores have been equally subdivided into four series each at different fragment sizes namely 2.5 cm, 0.3 cm, 0.1 cm and 0.05 cm. Each series has then been split into 32 sub-samples. A random selection of two samples from each series will provided sufficient material for petrological studies. The remaining 30 samples from each series will be pulverized and submitted for fire assay.

Analytical data for the sampling tree experiment

The analytical values for the four series of gold samples are listed in Table I and the appropriate descriptive statistics for each series are shown in Table II.

Plotting the calibration curve

The residual variance is the total variance minus the analytical variance of 0.04 g/t². The calibration curve can now be determined from the data given in Table II. It is possible to calculate $Ln(\sigma^2 \times M_S)$ and $\ln(d_N)$ and plot the one against the other on a linear scale. The value of $Ln(d_N)$ lies along the x-axis and the y-axis is $Ln(\sigma^2 \times M_S)$. The line is plotted in the graph of Figure 2 and is a straight line.

According to Gy the equation relating mass to nominal size is given as:

$$Ln\left(\sigma^2 \times M_S\right) = \alpha Ln\left(d_N\right) + Ln\left(K\right)$$

which is the form of a straight line with the equation $y = mx + c$. From the graph the intercept on the y-axis = $Ln(K)$ and the slope $m = \alpha$. The slope of the line provides a value for α, while the constant is the intercept on the y-axis and provides an estimation of a value for *K*.

Calculating a value for the slope (α) from the curve

Again using the four experimental points construct a regression line through the points. Using this straight line, estimate values for intercepts at the end of the lines as shown in Figure 2. The data used to compile the calibration curve is shown in Table III.

$$\text{Slope, } \alpha = \frac{y}{x} = \frac{\ln\left(\sigma_r^2 \times M_S\right)}{\ln\left(d_N\right)}$$

$$\frac{\ln\left(\sigma_r^2 \times M_S\right)}{\ln\left(d_N\right)} = \left[\frac{6.4615 - 2.1428}{0.9163 - \left(-2.9957\right)}\right] =$$

$$\left[\frac{4.3187}{3.912}\right] \text{ so } \alpha = 1.104$$

Calculating a value for the intercept K from the curve

At the point where $\text{Ln}(d_N) = 0$, *K* the intercept ($\sigma_R^2 \times M_S$) is 5.25; and $Ln(K) = 5.25$ so that $K = e^{5.25} = 190.56$. It is important to use the point where $Ln(d_N) = 0$.

So α and *K* can be 'calibrated', thus providing estimates of these parameters to a particular ore (rather than using Gy's approximated liberation factor). So the best estimates are:

$\alpha = 1.1$, $K = 190.6$

Plotting the calibration curve using log-log paper

It is possible to plot exactly the same calibration using log-log paper, a slightly more tiresome exercise, but the calibration of α and *K* is possible. The relevant values for compilation of such a plot are listed in Table IV.

An example of the plot is shown in Figure 3 for interest sake.

Calculation of the liberation size d_ℓ

Having obtained the estimate for alpha and *K* we can now calculate values for d_l the liberation size using the equation:

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Table IV

Data from Table II for compiling a log-log scale calibration curve

Statistic	Series 1	Series 2	Series 3	Series 4
Mass*stand.rel.var.	639.99	34.89	15.74	8.52
Nominal size (cm)	2.5	0.30	0.10	0.05

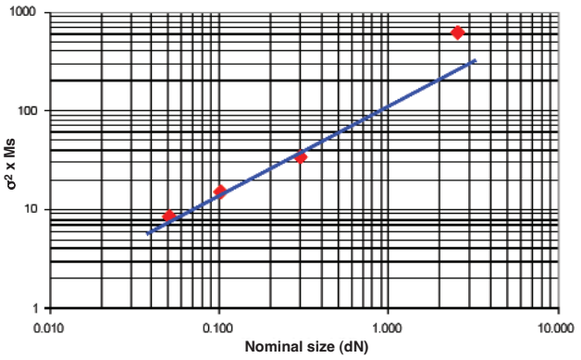

Figure 3—Calibration curve for *K* and α for a gold ore with an average grade of 12 g/t Au using log-log paper

$$Kd_N^3 = c.f.g.d_l^{3-\alpha} \times d_N^\alpha$$

$$d_l = \left[\frac{Kd_N^3}{c.f.g.d_N^\alpha}\right]^{\frac{1}{3-\alpha}} \quad [3]$$

The liberation size must be calculated at the nominal size of $d_N = 1$ because $d_N^3 = 1$ at this point, in which case d_N^α also equals 1, which allows us to calculate the liberation size.

$$Kd_N^3 = c.f.g.d_l^{3-\alpha} \times d_N^\alpha$$

$$d_l = \left[\frac{Kd_N^3}{c.f.g.d_N^\alpha}\right]^{\frac{1}{3-\alpha}}$$

Now substituting the relevant values we get

$$d_l = \left[\frac{190.6}{16000000 \times 0.5 \times 0.25}\right]^{\frac{1}{3-1.1}} \times 10000$$

$$d_l = 0.00288 \times 10000$$

$$d_l = 288 \text{ microns}$$

So the size of the gold particles is nearly 288 microns and this is the liberation size we calculate for d_ℓ.

Compilation of sampling nomograms using 'calibrated' constants for a particular ore

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,

$$\sigma_R^2 = \frac{Kd^\alpha}{M_S} \quad [4]$$

Taking logarithm of both sides gives:

$$\ln\left(\sigma_R^2\right) = \ln\left(\frac{1}{M_S}\right) + \ln\left(Kd^\alpha\right) \quad [5]$$

$$\ln\left(\sigma_R^2\right) = (-1)\ln\left(M_S\right) + \left[\alpha\ln(d) + \ln(K)\right] \quad [6]$$

Equation [3] 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 $\ln(K) + \alpha\ln(d_l)$ is a constant say $c(d)$ and $\ln(\sigma_R^2) = -\ln(M_S) + c(d)$. So for a given fragment size d, this is a line with a slope of -1.

Input for calculating σ_R^2

Using earlier estimates of $\alpha = 1.1$ and $K = 109.6$ and substituting these values into Equation [4], it is possible to calculate the data shown in Table V for use in compiling the nomogram.

Plotting the nomogram

On the basis of the data shown in Table V the sampling nomogram for a gold bearing ore with an average grade of 12 g/t Au is compiled, as shown in Figure 4 using log scales for both axes.

Table V

Data required for compilation of the sampling nomogram

Nominal size (cm)	Mass (g) relative	Standardized variance	Relative std dev	Position
5.0000	10,000	0.112	33.46%	A
2.0000	10,000	0.041	20.21%	B
2.0000	5,000	0.082	28.59%	C
0.5000	5,000	0.018	13.34%	D
0.5000	1,000	0.089	29.82%	E
0.1000	1,000	0.015	12.30%	F
0.1000	300	0.050	22.46%	G
0.0050	300	0.002	4.32%	H
0.0050	50	0.011	10.59%	I
Incremental variance		0.420	64.80%	

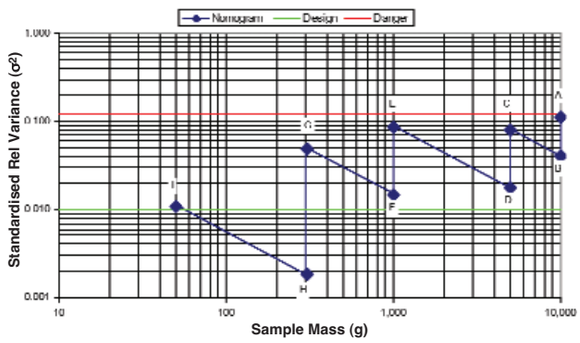

Figure 4—Sampling nomogram for a gold ore with an average grade of 12 g/t Au

Results achievable from the protocol

The incremental variance σ_I^2 is calculated from the data in column 3 of Table V using the formula:

$$\sigma_I^2 = \left(\sigma^2_{\text{Step A}} + \sigma^2_{\text{Step C}} + \sigma^2_{\text{Step E}} + \sigma^2_{\text{Step G}} + \sigma^2_{\text{Step I}} + \right) - \left(\sigma^2_{\text{Step B}} + \sigma^2_{\text{Step D}} + \sigma^2_{\text{Step F}} + \sigma^2_{\text{Step H}}\right)$$

$$\sigma_I^2 = \left(0.112 + 0.082 + 0.089 + 0.05 + 0.011\right) - \left(0.041 + 0.018 + 0.015 + 0.002\right)$$

$$\sigma_I^2 = 0.42 \text{ and } \sigma = 64.8\%$$

Values for the incremental variance and overall precision of the nomogram are presented in Table VI.

Importance of sampling nomograms

As a result of this process, any sampling operation (mass reduction at constant rock particle size d_N) at each stage can be plotted on the chart as a path along a straight line of slope -1 (there is one such sampling per comminution size). For example, in Figure 4, (D) to (E) is just such a path. The mass corresponding to Point D is the mass of crushed material, in this case 5 kg. The mass corresponding to Point E is the mass of material split out for the next stage of comminution, in this case 1kg. The difference between σ_R^2(D) and σ_R^2(E) represents the increase in segregation-free relative sampling variance for this particular stage of mass reduction. Crushing and grinding stages, such as (G) to (H) reduces the relative sampling variance by σ_R^2(G) minus σ_R^2(H), in this case 0.048, a movement of the vertical segment. The entire sampling preparation protocol can thus be visualized, with the contribution of each stage of the overall precision variance appearing clearly (see Figure 4). Such charts constitute valuable tools for the design, assessment and control/improvement of sampling processes.

Gy's recommended 10% relative error safety line is plotted on Figure 4. Above this 'safety line' the sampling operation involves an unacceptably skewed distribution of errors, which means the precision may get out of control, and possibly out of the region of validity of our model. The behaviour of the sampling nomogram suggests that these ores are fairly manageable in terms of the sampling protocol and that the sampling variance can be maintained below the safety line with careful attention. It also suggest that the nugget effect is fairly low and that the gold is fine grained as suggested by the calculation of the liberation size at about 228 microns.

Conclusions

The sampling parameters for ores at numerous different mines have been determined using the method suggested here. The method suggested is simple: determine the variances of 30 assay values for ores at a range of different calibrated comminution sizes and use regression to derive best fit values for *K* and alpha. This is only one of several methods for determining the sampling variance of the Fundamental Error. Others have been described by Pitard (1993) using the heterogeneity test, by Bartlett and Viljoen (2002) using an analysis of variances derived by crushing and splitting ore samples, by De Castilho *et al.* (2005) using variances from ores at different comminution sizes and a regression approach, and Minkkinen and Paakkunainen (2005), using a variographic approach. Each method has merit depending on the type and nature of the ores being investigated.

One of the principal objections to the use of the sampling tree method for determining the sampling parameters *K* and alpha, is the way that these results carry a bias introduced from the total error plus the analytical error that is not accounted for. As a result, the fundamental error will be overestimated. The relatively recent interest in the approach to sampling suggested by the theory of sampling means that a considerable amount of research remains to be undertaken, particularly in South Africa. A comparison of the different methods of determining the fundamental error variance and the sampling parameters *K* and alpha by different methods is an essential next step for researchers in this field.

Table VI

Values for the incremental variance and precision of the sampling nomogram

Description	Value
Incremental variance	0.42
Precision (% relative standard deviation)	64.8%

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