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Evaluation of sampling error sources in a multiple cutter metallurgical sampler

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The head loss caused by metallurgical sampling for slurry streams can be significantly reduced by appropriate sampler design. When the process flow is sampled by vertical static cutters before an equal number of moving cutters, the installation requires less installation head space than other sampling arrangements and is easy to accommodate at suitable process locations. Low head loss reduces building costs for the processing plant and operational costs during the life time of the plant.

The presence of a possible systematic bias in the particle size distribution or the chemical composition between the vertical static cutters caused by segregation in the metallurgical sampler can be estimated by a designed sampling campaign where sub-samples are cut from each of the moving cutter sample streams simultaneously. The sub-sample assay results can be evaluated by an F-test to reveal if there exists significant variance between the cutter assays.

The Minimum Possible Error (MPE) caused by the sampling and analysis system can be estimated in another sampling campaign where spot samples are collected at equal intervals to perform a variographic experiment to characterise process heterogeneity and MPE by estimating the V(0) intercept. The V(0) is the variability of a single measurement and furthers an indication of the minimum sampling variance that can be expected in practice. MPE includes the Fundamental Sampling Error (FSE), the Grouping and Segregation Error (GSE), the Total Analysis Error (TAE) as well as preparation errors and the possible Incorrect Sampling Errors (ISE) perhaps not fully eliminated. In this paper we present an approach to evaluate the various sampling error sources and magnitudes in a multiple cutter metallurgical sampler.

Introduction

Outotec metallurgical sampler MSA 2/50 has a low head loss structure. A structural benefit is that installation is easier and operational costs are lower than with high head loss metallurgical sampler structures. Figure 1 shows the design of the sampler. The metallurgical sampler is composed of several parts. The first part consist of mixing tank where a flow gate regulates slurry mixing and the speed of the slurry to the second part, which is characterised by three static cutters. The third part houses three moving cutters.

The metallurgical sampler has been installed in a flotation feed process line in hydrocyclone overflow. The process flow rate was close to the maximum recommended level, indeed sometimes even higher, yet it was found to be able to work well under these conditions. In the first part of the study sub-samples were collected immediately behind the moving cutters, with a purpose to reveal if

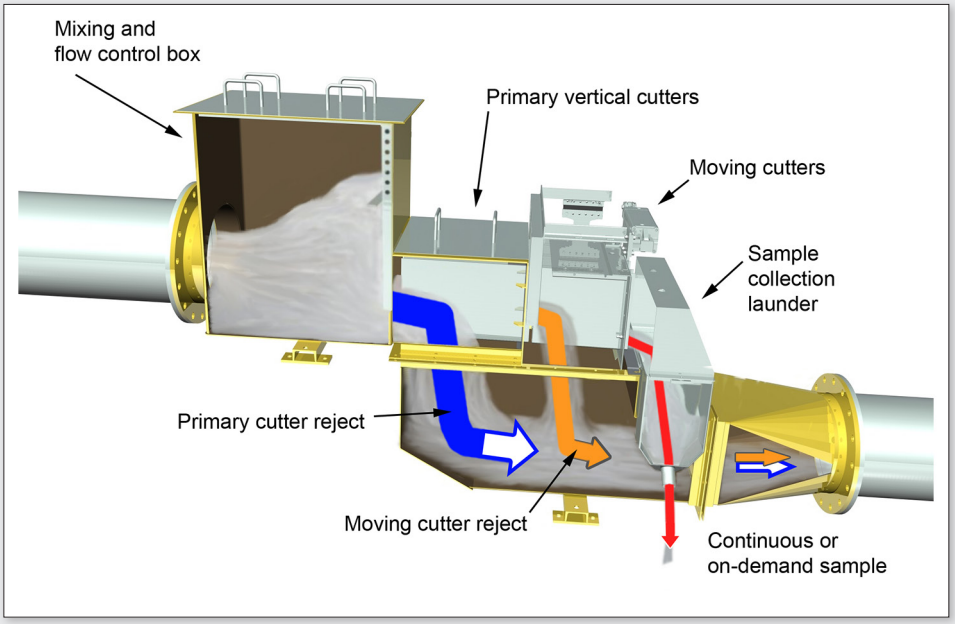

Figure 1. Metallurgical slurry sampler MSA 2/50. The cover of the sample collection launder was removed and three specially made boxes were placed in the launder for the taking the sub-samples used in this study. See Figure 10 for details of the moving cutters.

these show systematic differences with respect to chemical composition. If so, this would reflect an extraction error (IEE). Secondly, spot sample data were collected from a Courier on-line analyser and used in a variographic experiment to study process heterogeneity and to assess the Minimum Possible Error (MPE) by estimating the V_0 intercept. V_0 represents the short-range error variance of a single measurement.

Because sub-sample collection and variographic data collection was conducted as two different events, the ore fed to the process had changed in the particle size distribution and elemental content altering the scale of the fundamental sampling and, consequently, all analysis results of the studies are not directly comparable.

Sub-sample study

When sub-samples are taken simultaneously by the three moving cutters, the sampling error is caused by three error sources: long-range fluctuation in the process stream during the study, between cutters variation and short range variation consisting of the fundamental sampling error (FSE). If the error source variances are significantly larger than nil, their magnitude can be estimated from the experimental design shown in Figure 2 by using analysis of variance (ANOVA). In the following calculations σ^2 denotes theoretical variances and s^2 denotes estimated variances.

Due to the sample collection and preparation design used in this study the analytical error variance σ^2_a cannot be separated from the short term variance σ^2_{sh}. Instead, their sum can be estimated:

$$\sigma_0^2 = \sigma_{sh}^2 + \sigma_a^2 \tag{1}$$

Two other sampling variances, the between-cutter variance (σ^2_{bc}) revealing the local segregation, and the long-range variance (σ^2_{lr}) which includes all process changes during the experimental study, can be resolved from the four experimental variance estimates (s_1^2, s_2^2, s_3^2 and s_4^2) shown in Figure 2 (ANOVA). These are linear combinations of the three contributing error sources; they can therefore be used to calculate estimates for the individual error source variances (Equations 2–12). Here n = 10 is the number of primary samples from each cutter, j = 3 is the number of parallel cutters and df is the number of degrees of freedom for variance.

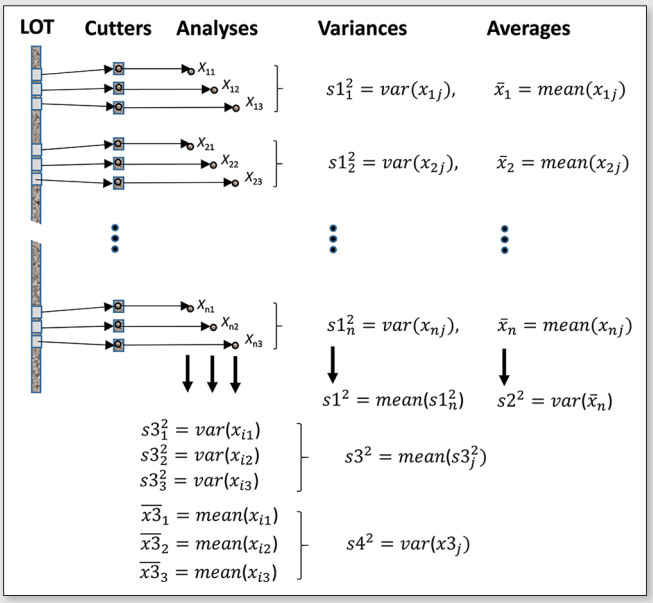

Figure 2. Experimental setup for ANOVA and calculation of four variances s_1^2–s_4^2 needed to estimate variances generating the total sampling error.

$$s_1^2 = \frac{\sum s_{1i}^2}{n} \approx \sigma_{bc}^2 + \sigma_0^2 , \qquad df = n(j-1) \tag{2}$$

$$s_2^2 \approx \sigma_{lr}^2 + \frac{\sigma_1^2}{j} = \sigma_{lr}^2 + \frac{\sigma_{bc}^2}{j} + \frac{\sigma_0^2}{j} , \quad df = n-1 \tag{3}$$

$$s_3^2 \approx \sigma_{lr}^2 + \sigma_0^2, \qquad df = j(n-1) \tag{4}$$

$$s_4^2 \approx \sigma_{bc}^2 + \frac{\sigma_3^2}{n} = \sigma_{bc}^2 + \frac{\sigma_{lr}^2}{n} + \frac{\sigma_0^2}{n} , \quad df = j-1 \tag{5}$$

The significance of the experimental variances was analysed by using an F-test with the ratio of the sample variance estimates as the test statistic. The significance of the long-range variance is tested with Eq. 6 and the between-cutters with Eq. 8.

$$F_1 = \frac{s_1^2}{s_2^2} = \frac{\frac{\sum s_{1i}^2}{n}}{s_2^2} = \frac{3s_2^2}{s_1^2} \approx \frac{3\sigma_{lr}^2 + \sigma_1^2}{\sigma_1^2} \tag{6}$$

In cases where the F test (6) is insignificant the long-range process variance does not differ significantly from zero and σ^2_{lr} can be assumed to be close to zero. In cases where the test results are significant, the estimate of long-range variance caused by long-range process fluctuation is:

$$s_{lr}^2 = s_2^2 - \frac{s_1^2}{j} \tag{7}$$

Similarly, the significance of the between-cutters variance can also be tested Eq. 8.

$$F_2 = \frac{ns_4^2}{s_3^2} \approx \frac{n\sigma_{bc}^2 + \sigma_3^2}{\sigma_3^2} \tag{8}$$

ANOVA showed that the between–cutter variance was insignificant, and consequently, the between-cutters variance can be assumed to be nil ($s^2_{bc} \approx 0$). The sum of the analytical error and short term process variance, s^2_0, and total variance of a single sample, s^2_{tot}, can also be estimated (Eqs. 10 and 11). The total variance is the sum of all error generating variances.

$$s_0^2 = s_1^2 - s_{bc}^2 \tag{9}$$

The total variance of a single measurement is the sum of all variances

$$\sigma_{tot}^2 = \sigma_{lr}^2 + \sigma_{bc}^2 + \sigma_{sh}^2 + \sigma_a^2 \approx s_{tot}^2 = s_{lr}^2 + s_{bc}^2 + s_0^2 = s_{lr}^2 + s_1^2 \tag{10}$$

If each j parallel cuts from n primary cuts are analysed the variance of the mean from the test period, excluding the possible autocorrelation discussed in the next chapter, is

$$s_{average}^2 = \frac{s_{lr}^2}{n} + \frac{s_{bc}^2}{n \cdot j} + \frac{s_0^2}{n \cdot j} \tag{11}$$

Because the sampling variance between cutters was not significant according this study (see above), the total observed variance is simply the sum of the long and short range process variances and the analytical variance. The design of feed box eliminates the horizontal segregation in the process stream and differences between the points of vertical cross cuts of the process stream were

Table 1. Anova variance components. Three replicate sub-samples were taken and analysed from ten primary process increments.

Measurement	Variance	Absolute standard deviation (%)	Relative standard deviation (RSD%)
A (%)	$s^2_{lr} = 0.00989$	0.099	0.88
	$s^2_{bc} \approx 0$	0	0
	$s^2_0 = 0.00872$	0.094	0.83
	$s^2_{tot} = 0.01867$	0.137	1.21
B (%)	$s^2_{lr} = 0.00586$	0.0024	2.30
	$s^2_{bc} \approx 0$	0	0
	$s_0^2 = 0.00024$	0.016	1.48
	$s^2_{tot} = 0.00083$	0.0040	2.74

insignificant, no systematic bias caused by the MSA 2/50 cutters could be observed in this study. The results of ANOVA are presented in Table 1. Because the data are confidential it was decided to denote the "analytes" as A and B without loss of generality.

Variographic experiment

The most complete theory on sampling for chemical analysis of particulate and solid matter in mineral processing industry that takes into account both the technical and statistical aspects of sampling, has been developed by Pierre Gy's and presented in two fundamental books[1,2] and in many later developments. Pitard[3] has also published a book based on Gy's sampling theory explaining variography in detail. A generic variogram is shown in Figure 3.

Gy has shown that the long-range Point Selection Errors, (PSE_1 and PSE_2), and the short term Point Selection Error (sum of Fundamental sampling error, FSE and Grouping and Segregation Error, GSE) can be estimated with a variographic experiment, in which N, a sufficient number of samples (minimum of 30 preferably more than 100) are collected systematically most often with equal time intervals. According to Gy's definitions the heterogeneity contribution is a structural property of the material. The heterogeneity contribution of every sample can be estimated,

$$h_i = \frac{a_i - a_L}{a_L}\frac{M_i}{M}, i = 1,2,\ldots,N \qquad (12)$$

in which i is the sample or increment number, a_i is the analysis result of sample i, a_L average of the process sequence, M_i is the weight of sample i and M is the average sample mass. If the sample size is proportional to the process flow or the flow rate correlates with the analysis result, the heterogeneity contributions and the average concentrations must be statistically weighted.

$$a_L = \frac{\sum M_i \cdot a_i}{\sum M_i} \qquad (13)$$

The heterogeneity contributions are the most often used format used as the basis for the variogram, V_j (as a function of sample lag interval j)

$$V_j = \frac{1}{2(N-j)}\sum_{i=1}^{N-j}\left(h_{i+j} - h_i\right)^2, j = 1,2,\ldots,\frac{N}{2} \qquad (14)$$

From the variogram variance and standard deviation estimates can be solved for the three principal sampling modes (systematic, random or stratified) thus providing useful information for optimising specific sampling plans[4,5].

Variographic analysis provides an estimate of the intercept V_0, also known as nugget effect, at zero lag. The intercept V_0 is a sum of several components, FSE, GSE and the variance of all the other components of the incorrect sampling errors, ISE, e.g. delimitation error and extraction error that potentially were not partially or fully eliminated. Because all the random errors of the sampling process are included in the intercept V_0, it provides an estimate of the precision of online sampling and analysis system. N.B. A proper variographic analysis must be made on accurate (unbiased) data.

The intercept V_0 is related to the zero point variability; it is the variance that would occur if the same sample could have been taken twice. It is the best obtainable estimate of the minimum sampling variance (minimum possible error, MPE) expected in process sampling in a one-dimensional lot such as process stream. The theory of sampling identifies this value as minimum practical error[6].

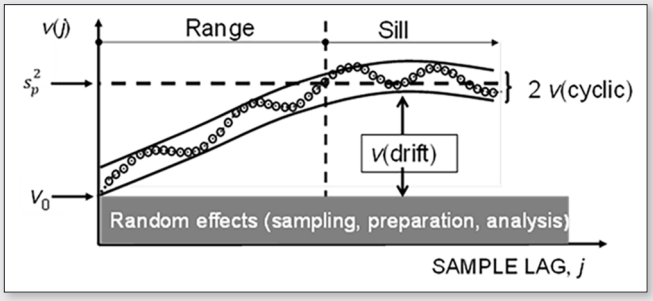

Figure 3. Generic variogram and its components. The variogram delineates the individual components of random and periodic process variances as well as the variance of a zero lag sampling point (plus the analysis error); s_p^2 is the long-range process variance, aka the sill, of a stationary process.

The variographic analysis of metallurgical sampler

The MSA 2/50 variograms were studied using two time series, each based on 10 or 15 minute intervals. The data used in the study were from a Courier XRF on-line elemental analyser data. Courier analyses the multiplexed sample streams at regular programmed intervals and provides elemental analysis data suitable for variographic study. Variograms calculated from the Courier data were used to extrapolate the V_0 intercept. Estimates of the relative standard deviations as a function of a sampling interval of systematic and stratified sampling modes were calculated by using Gy's method explained in detail in references 1 - 3. Figures 4 and 5 show the time

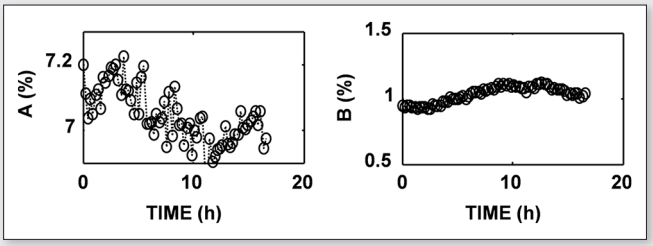

Figure 4. Time series plot of elements A and B, measurements at 15 minute intervals.

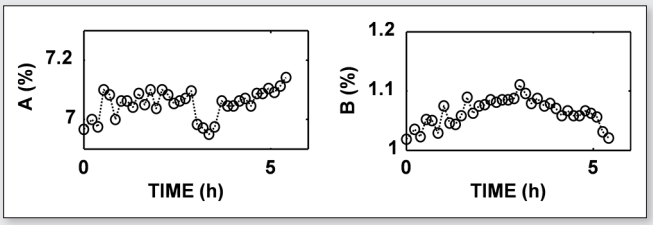

Figure 5. Time series plot of elements A and B, measurements at 10 minute intervals.

series of the elements A and B. Figures 6 and 7 show the corresponding variograms calculated together with the relative standard deviation graphs as function of a sampling interval. These values can be used to calculate standard deviations of, and confidence intervals for, point estimates and the average of several samples for the different sampling modes[5]. If n samples are taken with intervals j from a lot (systematic sampling), or one random sample from every consecutive substrata of length j (stratified sampling), the relative variance of the mean of the lot, a_L is

$$s^2_{a_L} = \frac{s^2_{sy/st}}{n} \quad (15)$$

where is $s^2_{sv/st}$ is the variance estimate of systematic or stratified sampling mode at lag j obtained from the variographic analysis.

Estimation of the Fundamental Sampling Error FSE

The fundamental sampling error gives an estimate of the relative sampling variance after all other error sources have been eliminated (if/when possible). FSE is caused only by the properties and of the sampled material.

$$\sigma^2_{FSE} = fgcld^3\left(\frac{1}{M_S} - \frac{1}{M_L}\right) \quad (16)$$

in which f is the particle shape factor, g is granulometric factor, c is mineralogical composition factor, $l = (d_l/d)^{1.5}$ is liberation factor (d_l is liberation size), d nominal top size of the sample, M_S mass of the sample and M_L mass of the lot, respectively. If the material is ground below the liberation size, as was done here, the liberation factor should be set as = 1. If the sampled lot is much larger than the sample, equation 17 simplifies to

$$\sigma^2_{FSE} = fgcld^3\left(\frac{1}{M_S}\right) \quad (17)$$

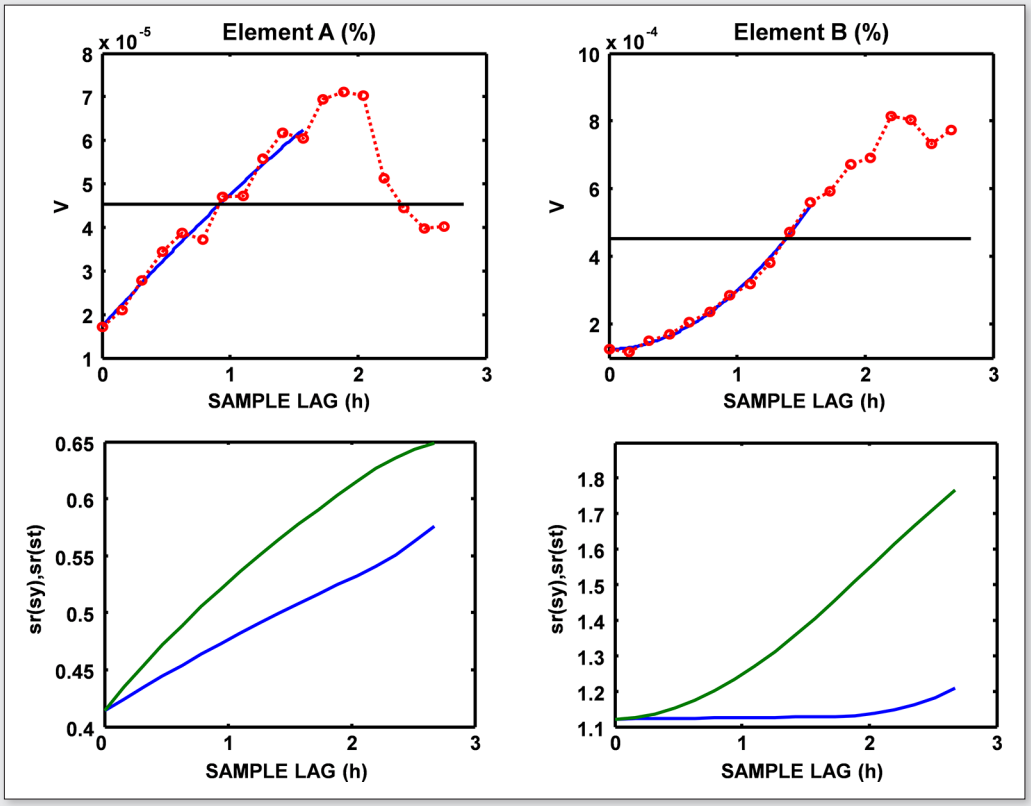

Figure 6. Flotation feed variograms of elements A and B instrumental analysis (Courier) in 10 min intervals (upper panels). Lower panels show estimated relative standard deviations for stratified (green) and systematic (blue) sampling modes.

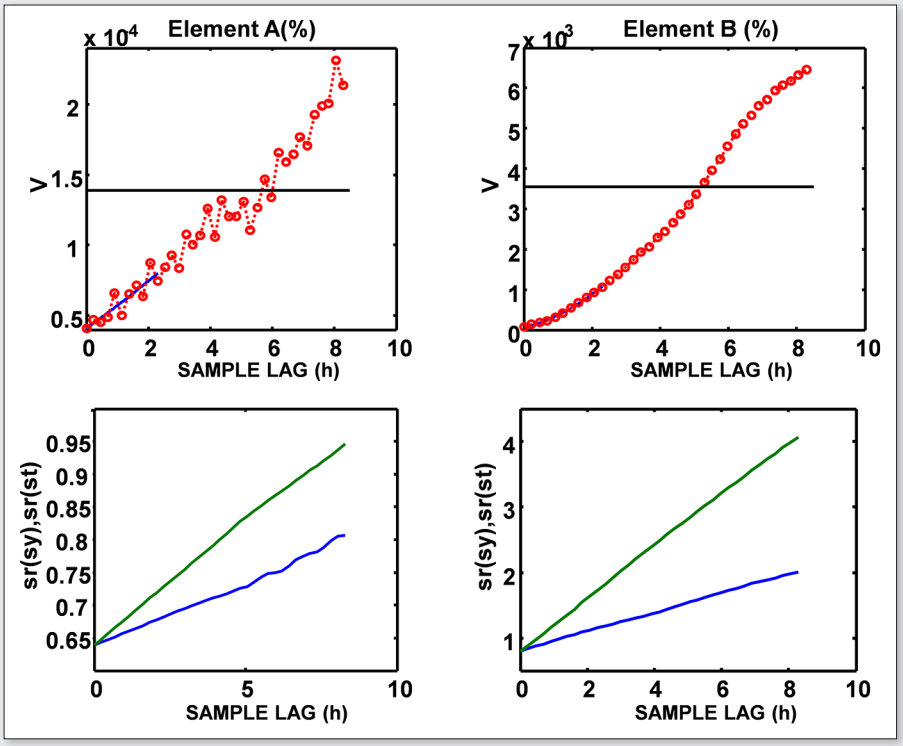

Figure 7. Flotation feed variograms of elements A and B instrumental analysis (Courier) in 15 min intervals (upper panels). Lower panels show estimated relative standard deviations for stratified (green) and systematic (blue) sampling modes.

The composition factor is estimated as

$$c = \frac{\left(1-\frac{a_L}{\alpha}\right)^2}{\frac{a_L}{\alpha}}\rho_c + \left(1-\frac{a_L}{\alpha}\right)\rho_m \qquad (18)$$

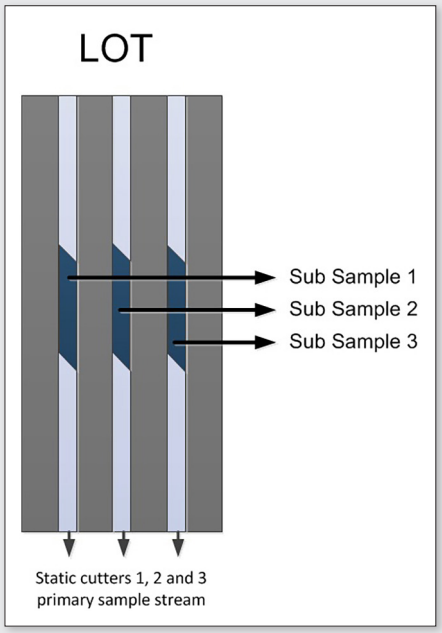

Figure 8. Schematic illustration of the experimental setup of the sub-sample study.

where a_L and α are the average concentration in the lot of and in the mineral that contains the element to be analysed, respectively and ρ_c and ρ_m are the densities of the mineral containing the analyte and the gangue (matrix).

Figure 8 shows schematically how the samples were cut with the moving cutters. In MSA 2/50 each of the three static cutters with equal width cut 8.3% from the process stream as the primary sample; in total the static cutters take one fourth of the process stream, in this case 600 m^3/h. The total flow-rate at the moving cutters is 150 l/min. The volume of the increments taken by cutters is 1.9 litres (5.6 l total sample). During the study the solids content of the slurry was 52% (m/m). Thus the individual sample mass was approximately 1.3 kg.

For estimating FSE for element B, some assumptions have to be made. The shape factor *f* is assumed to be 0.5 (meaning that particles are assumed to be spherical), and the granulometric factor *g* is set as 0.5 assuming that the material is classified. Element B concentration in dry slurry was 1%, and the density of the mineral containing it, α = 6 g/cm^3 while the density of gangue is 2.7 g/cm^3. Based on these data the composition factor *c* = 270 g/cm^3 can be calculated; the liberation factor *l* = 1 was used. FSE error estimates are based on 0.2 litre (180g) final increment size (taken with a secondary sampler), from which a 20 g laboratory analysis sample was finally extracted. The estimates of the relative standard deviation of the FSE at different sub-sampling stages and sample preparation were calculated and are presented in Table 3. As expected, a 20 g sample displays the largest sampling variance, 0.32% as relative standard deviation, while the primary increments only contribute 0.0035%. The reason for these small FSE is the small particle top size, which is about 150 µm only. The largest FSE for metallurgical sampling of elements with approximately 1/10% content in the

Table 2. Results of the variographic experiment for flotation feed elements A and B and Solids-%.

Estimated quantity	Lag (min)	Element A	Element B	Solids%
V_0	15	4.09E-05	6.47E-05	3.90E-05
	10	1.71E-05	1.26E-04	5.89E-05
Average, a_L (%)	15	7.05	1.03	40.99
	10	7.06	1.06	40.63
Relative Standard Deviation s_0 (%)	15	0.64	0.80	0.62
	10	0.41	1.12	0.77
Relative Long Range Standard Deviation of the process, s_p (%)	15	1.78	5.96	1.56
	10	0.67	2.13	1.13

flotation feed can be expected to be of the magnitude of relative standard deviation less than 0.1%. Much larger errors are caused during preparation of the analytical sample from the composite sample.

Discussion

Figure 9 shows Gy´s classification of the full complement of sampling errors. According to results of this study it is estimated is that the sum of the short term variation and analysis errors is responsible for the largest contributions to the total error for the flotation feed, in case of element B the relative standard deviation was estimated to be s_0 = 1.5% (ANOVA) and 0.8–1.1% (variographic experiment). These errors are caused by the short-range heterogeneity within the 1-D material flow, by the manual preparation of the analysis sample and by the laboratory analysis. In the variographic study s_0 was obtained by extrapolating the variogram to lag zero and it

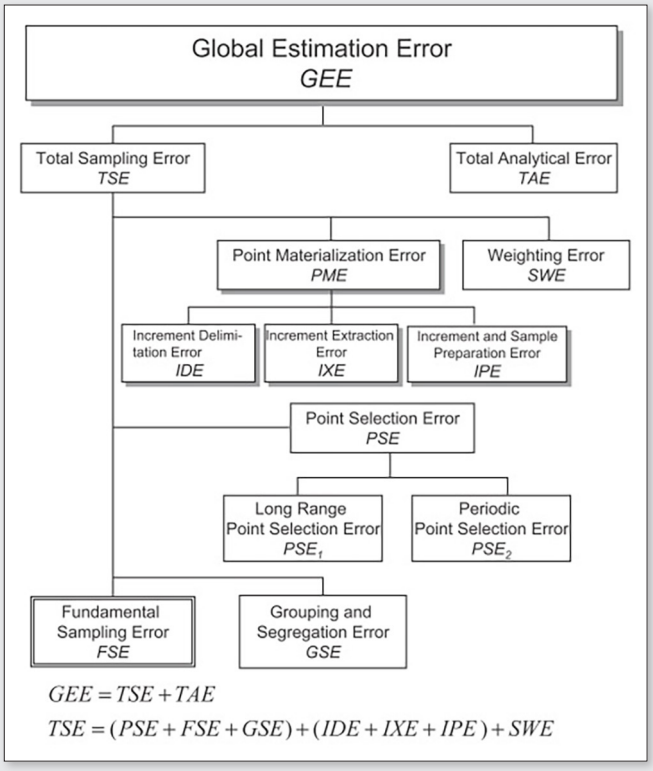

Figure 9. Gy's complete classification of sampling errors according to source[4].

includes the short-range and Courier analysis variations. The long range variation in the process caused by changes in the process feed in ANOVA study was s_{lr} = 2.3% and in the variographic study 6 and 2.1%, by comparison.

The main error sources in the sub-sampling study were likely operator errors during manual sub-sampling and manual sample preparation for laboratory analysis, and the actual analysis error, which could be estimated in this particular experiment. The FSE in the sub-sampling study is small due to small particle size S_{FSE} *(TOTAL)* = 0.34%.

According to the variographic experiment, the total element B measurement variance (15 min lag), V_0 = 6.47E-05, which includes the short term process variance and the variance caused by on-line XRF analyser. The intercept V_0 is the best possible estimate of the minimum practical (MPE) error for the present process sampling system[6].

MPE includes the fundamental sampling error, the grouping and segregation error, the analysis error and the point materialization errors. Therefore combined variance caused by fundamental sampling error, analysis error, grouping and segregation error and incorrect sampling error was during this study.

$\sigma^2_{MPE} = \sigma^2_{FSE} + \sigma^2_{TAE} + \sigma^2_{GSE} + \sigma^2_{PME} = 6.47 \times 10^{-5}$ for analyte B, which had on average 0.01052 mass fraction in a mineral form in the flotation feed. The relative error is thus: =1.12%.

The analysis of variance of sub-sampling study showed no significant bias between moving cutters, that is s^2_{bc} of elements A and B in Table 1 are nil. The difference between the dry solid material sampling from a conveyer belt and fine particle slurry sampling in flotation process is that even with a high solids fraction, e.g. 50% w/w, the volumetric solids fractions is about 25% v/v, which means that particle trajectories in slurries are also controlled by turbulent water flow and not only particle-particle interactions. Consequently, although the static cutters are not fully compatible with the theory of sampling in the sense that they do not cut a full cross-section from the process stream, the present results show that the mixing chamber before the static cutters is effective in randomising the slurry flow, essentially converting it into a fit-for-purpose 0-D sampling target at the time of cutting the primary increments before the moving cutters. For comparison, there has recently been developed an analogous full cross-section, vertical increment cutter sampler for pneumatically conveyed internally ducted two-phase (air/solid particles) aggregate sampling, Wagner & Esbensen[7]. In this solution the mixing is taken care of by the turbulent conducting transportation itself. Although addressing very different types of materials, both approaches share the prime objective of counteracting the

Table 3. Estimation of variance and relative standard deviation (%) of the fundamental sampling error (FSE) for element B assays at different stages of sampling and sub-sampling.

Source of variance	M_S (g)	σ^2_{FSE}	Relative Standard deviation (%)
σ^2_{FSE} static cutters	141120	1.20E-09	0.0035
σ^2_{FSE} moving cutters	3780	4.31E-08	0.021
σ^2_{FSE} sub sample	1260	8.93E-08	0.030
σ^2_{FSE} single increment	180	1.12E-06	0.11
σ^2_{FSE} laboratory analysis sample	20	1.00E-05	0.32
σ^2_{FSE} TOTAL		1.13E-5	0.34

bias-generation effects of vertical segregation in moving streams of matter.

PSE1 and PSE2, together with the analytical error, are the main contributors with the largest variation to the total measurement uncertainty. Increment and Sample Preparation Errors are largest sampling error sources, but it was impossible to estimate these individually for which reason they were included in the compound s^2_0 estimation. The fundamental error estimation shows how the total measurement uncertainty increases as sample mass is reduced; the only way to reduce the fundamental sampling error is to reduce the particle size and/or increase the composite sample mass. However the fundamental error is insignificant in the MSA 2/50 sample volumes and the Grouping and Segregation error (GSE) was also minimal according to the sub sample study.

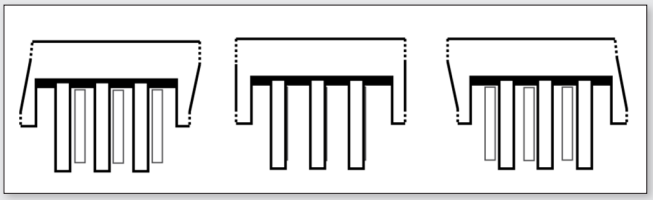

Figure 10. Operating principle of the MSA 2/50 Metallurgical sampler's moving cutters.

Conclusions

Based on this study the Outotec metallurgical sampler is able to represent the process variance reliably for elemental content and is able to substantiate timely process control actions. In the mineral beneficiation process the top particle size is very small, usually much less than 300 µm, and process flow velocities have to be sufficient effective to prevent the slurry particles from settling in the process piping, which reduces segregation in the sampled flow. The MSA sampler's feed box design controls the process stream flow velocity and randomizes the sample flow. According to the sub-sample study, when the process stream was cut by vertical static cutters no horizontal segregation could be observed.

If/when the total error budget estimates arrived at are acceptable for the operating company, this study has qualified the MSA 2/50 as a fit-for-purpose metallurgical sampler.

A complete cross section of the primary sample stream is cut by the moving cutter stage. The operating principle is shown in Figure 10. The moving cutters move at an adjustable velocity and frequency across the primary sample streams from the static cutters and thus provide the on-line analyser with a continuous sample by-pass flow that represents variations in the process validly and which can be sampled with a separate composite sampler for the metallurgical accounting purposes.

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