Design of primary samplers for slurries in concentrators and statistical methods for measuring components of variance in sampling

by H.E. Bartlett*

Synopsis

In the operation of some flotation plants treating platinum ores it was found that there were imbalances between the inputs and outputs. The inputs and outputs were sampled as slurries and it was determined that the imbalances arose from biases in the sampling, despite the samplers having been designed according to well-established rules.

The launder feeding the sample cutters, and the sample cutters themselves were redesigned using finite element analysis to model the flows. A new set of design rules were formulated.

The application of a Components of Variance Analysis, to determine the variance of primary and secondary sampling and of analysis in the lab for a concentrate is presented to illustrate the need for frequent sampling of process streams.

Introduction

In the assessment of the metallurgical balance and for control around a metallurgical plant it is necessary to have unbiased measurements of the metal contents of the main input and output streams and to know the confidence that can be placed on the results.

The determination of content involves measurement of the dry mass of the process streams, the taking of accurate, or unbiased samples, and accurate analysis in the laboratory of the samples that are taken. The measurement of dry mass is a subject on its own and involves the maintenance and calibration of belt mass-meters or weighbridges and procedures for determining the moisture content of ore feed and concentrates. These aspects are not considered in this presentation. Similarly, the obtaining of accurate chemical analyses of the samples is also a subject on its own and this is also not considered.

This paper focuses on the sampling of the slurry feeds, tailings and concentrate from a typical flotation plant and describes:

➤ Work done mainly on platinum metal concentrators but also on gold plants to obtain unbiased or accurate samples

➤ Statistical techniques that have been applied to sampling data to measure the overall variance of sampling and to quantify the components of variance of the primary and secondary sampling, and of analysis that make up the overall variance. Knowing where the major variances are enables one to optimize the sampling frequency, the number of sub-samples and assay aliquots that are taken.

Basic requirements

The samples that are taken must be unbiased or accurate

It is vital that a clear distinction is made between the concepts of accuracy and precision, terms that are very commonly confused. Accuracy implies an absence of bias. The meaning of bias is best illustrated by way of an example. Assume that the gold content of a sample is 6.00 g/t. This value is not known but let us suppose that by repeated analytical determinations an average result of 6.10 (plus/minus 0.02 g/t) is obtained. The figures within the brackets are those within two standard deviations of the mean. With more measurements these limits can be reduced but the mean result will be higher than the unknown true value because the sample is biased.

The cause of bias in sampling can be from a number of reasons. For example, in flotation plants, samples of pulp feed, concentrate or tailings can often be biased because the sampler only takes part of the process stream. In the sampling of run-of-mine ore biases can occur because the larger rocks are often relatively barren and the samplers, because of their design, tend to miss these larger rocks and the samples are therefore biased high.

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Biases cannot be eliminated by statistical methods. They must be minimized by good design of the samplers.

- Having designed accurate sampling systems, statistical tests are needed to measure the precision of the sampling systems to optimize the number of samples that are taken, the frequency and size of the samples, the protocols to be followed for sample reduction and the number of assays that are needed, within practical constraints.

**Design of primary samplers for slurries**

At a typical concentrator plant, run-of-mine ore is fed through a crushing plant and mills or to autogenous mills to produce an ore feed slurry. It is generally accepted that the best sample of the head grade is the sample of the ore feed slurry because the ore is well mixed and the particle size is reduced. Sampling of the run-of-mine ore or of a crushed product is used in some cases, for example if a gravity concentrate is removed during milling or if ore from different sources is to be separately evaluated.

In a typical plant, the slurry is treated by flotation and appropriate regrinds to produce a final concentrate and final tailings. Numerous control samples would be taken but the only samples required for metallurgical accounting are the feed, final concentrate and tailings samples.

For these samples it is required that the samples are accurate. Therefore, in accordance with sampling theory, every particle in the process stream must have an equal probability of being included in the sample. This requirement precludes the use of devices that do not sample the whole process stream. Examples of such devices are popette samplers that have a plunger used to insert an extractor cup through the side of a pipeline into the process stream, or various models of pipe samplers which have a fixed pipe or slot to extract a fraction from a process stream. These samplers are cheap and are easy to install in existing plant. Often they give results that are in agreement with the results from a well-designed cross-stream sampler but on other occasions the results do not agree. And it is not possible to predict whether the sampler will be representative in any particular situation.

**Rules for representative samples with cross-stream cutters**

Rules for the design of cross-stream cutters are given by Holmes. The headings for these rules are:

- The sample cutter must be non-restrictive and self-cleaning, discharge completely each increment without any rebounding, overflowing or hang-up in the cutter
- The geometry of the cutter opening must be such that the cutting time at each point in the stream is equal. For linear-path cutters, the cutter edges (called lips) must be parallel, while for cutters travelling in an arc or circle (e.g. Vezin samplers) the cutter lips must be radial
- No materials other than the sample must be introduced into the cutter, e.g. dust or slurry must be prevented from accumulating in the cutter when in the parked position
- The cutter should intersect the stream in a plane normal to the mean trajectory of the stream

- The cutter must travel through the stream at a uniform speed. Electric drives are best in this respect
- The cutter aperture must be not less than three times the nominal top size, with a minimum size of 10 mm for slurries
- The cutter must have sufficient capacity to accommodate the increment mass at the maximum flowrate of the stream
- For slurries, loss of sample material due to dribbles must be avoided
- The maximum cutter speed should not exceed 0.6 m/s. This rule is taken from Gy2 who conducted experiments to quantify the extent to which particles of known particle size bounce from the cutting edges of a horizontal cutter, either into or out of, the cutter with a measured gap and travelling at set speeds. These measurements do not necessarily apply to slurries.

**Application of the rules to samplers for platinum metal concentrators**

These rules were applied to the sampler designs for the sampling systems for feeds and tailings at concentrators of Rustenburg Platinum Mines. The concentrates were evaluated by weighing and sampling the filter cakes that were sent to the smelter. From the sampling and mass measurement of the inputs and outputs for the concentrators, mass balances were constructed. Over a period of some months to allow for time lags and changes in thickener stocks, there should have been a balance between input and output for the various metals. However, at a number of concentrators there were consistent imbalances.

After exhaustive investigation into the accuracy of mass measurement and chemical analysis, it was reasoned that a reason for these imbalances was bias in the sampling of pulps, particularly of the feeds. These pulps have a range of particle sizes, with varying densities and grades of platinum and other elements in these different particle sizes and it was postulated that in the sampling process segregation between different sizes and densities occurred. The segregation is theoretically not by itself sufficient to cause a sampling bias as long as the sample cutters are correct and there is no splashing or differential behaviour of particles in the turbulence around the cutter.

The segregation was postulated to occur in two areas:

- While the cutters were traversing the process stream. This was investigated by finite element analysis with cutters of old designs where considerable turbulence occurred and with redesigned streamlined cutters.
- In the launders feeding the cutters. It was observed that coarse particles, particularly chromite which occurs in UG2 ores and which is low in platinum grade accumulates as a slow moving layer at the bottom of the launders. This was resolved by designing the launders to operate with a pulp depth of about 100 mm and having a slope of 8° to obtain a pulp velocity of more than 2 m/s to prevent sedimentation of large particles.

In addition to segregation, there was also a problem with the secondary samplers used to reduce the bulk of the primary sample only took one or two increments. This was
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In Figure 2, a photo of the existing cutter traversing a pulp stream is compared to the finite element analysis. A high velocity tongue of pulp can be seen entering the cutter. This corresponds to the region of high velocity into the cutter gap that can be seen on the finite element analysis.

Figure 3 gives the results of finite element analyses for the streamlined cutter for slurry velocities of 1 m/sec and 2 m/sec, for cutter speeds of 300 mm/sec and 600 mm/sec and for cutter gaps of 10 mm and 20 mm.

On the basis of these analyses it was concluded that the least interference (turbulence) to the streams was obtained at the slurry velocity of 2 m/sec, a cutter gap of 20 mm and a cutter speed of 300 mm/sec. Accordingly, these are the parameters recommended for cutter design.

In Figure 4, photos of an existing cutter and the streamlined cutter that replaced the existing cutter going through a process stream are compared. It can be seen that there is considerable turbulence with the existing cutter with some sample splashing of extraneous material into the top section of the cutter. These pulps had a wide range of analyses of platinum in the different size fractions. It is assumed that there would be segregation of the different sizes and therefore of platinum during the splashing. This would cause the samples to be biased.

By contrast, the streamlined cutter causes little splashing. Even under extreme flow conditions the cutter is always within the stream as can be seen from the patterns on the side of the cutter.

Statistical methods for measuring components of variance

A generally applicable method of determining sampling precision is by the application of appropriately designed
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Figure 3—Finite element analyses for streamline cutter at different slurry velocities, cutter speeds and cutter widths

Figure 4—Existing and streamline cutters compared

Existing cutter. Note turbulence

Streamline cutter
statistical experiments to measure the variance with accurately taken samples. The experimental design would be adapted to the particular sampling situation. For example, for sampling of run-of-mine ore, the design would involve determining the variance of the primary and secondary sampling and the effect of reducing the particle size and mass of the ore from the primary belt samples down to the analytical portion submitted to the lab. The methods are applications of standard procedures for the statistical design of experiments and use standard statistical methods, such as Analysis of Variance and Components of Variance Analysis\textsuperscript{4,5}. For all designs the precision of sampling is measured by determining the variance associated with each of the sampling stages. Variance is the square of standard deviation and has the property that variances are additive for measurements that are not correlated.

The components of variance that need to be determined for the sampling of a slurry typically would be:

- **Primary sampling**—The variance of primary sampling is determined by taking twin interleaved shift composite samples. The twin interleaved samples are taken by having one 30-minute composite sample, composed by operating the sample cutter at a predetermined frequency, during a shift. The second composite is taken for the next 30 minutes. The two buckets collecting the samples are exchanged every 30 minutes. It is important to determine the precision of this primary sampling, separated from the precision introduced by the sub-sampling and analysis.

- **Secondary sampling stages**—Typically, primary samples are too large to be submitted to the lab and need to be reduced in mass. Typically, the primary samples are sub-divided using secondary Vezin samplers. There is often another stage of sample reduction where the samples from the secondary Vezin sampler on the plant are filtered and dried and then sub-divided using a Carousel divider.

- **Analytical variance**—Analytical variance is measured by analysis of replicate samples.

Table I is a ‘Sampling Tree’ presenting a situation of primary samples taken at different time intervals, each giving two secondary samples, produced after crushing and sub-division, and which are further pulverized, before splitting out the final samples for duplicate analysis.

The components of variance for each of the steps is calculated using a nested or hierarchical design for an analysis of variance.

For these statistical designs the error for the assays is nested within the secondary samples which are in turn nested within the primary samples.

The variance of the assay can be calculated from the differences between duplicate assays. The variance for the secondary samples is calculated from the differences between the means for the assays. The variance for the primary sampling can similarly be calculated from the differences between the means for the secondary samples. Methods for the manual calculations are given in Box, et al. However, Nested Analysis of Variance can also be done using appropriate statistical packages, such as Proc Nested in SAS.

**Typical results of sampling experiments**

Table II gives the results of sampling experiments for a platinum flotation concentrate following the sampling tree given in Table I but with different sampling frequencies for the primary increments.

The CV% is the coefficient of variation (sometimes called the relative standard deviation RSD) for the component of the error. The error contribution is a measure of the error associated with the component.

Table II shows:

- The CV% for assay is similar (2.80% versus 2.36%) for the two tests. The assay error is typical for repeated fire assay on platinum flotation concentrates
- The error contribution from the process itself is greater

<table>
<thead>
<tr>
<th>Table I</th>
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<tr>
<td><strong>Sampling components</strong></td>
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<table>
<thead>
<tr>
<th>Process</th>
<th>Shift 1</th>
<th>Shift 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Twin interleaved samples</td>
<td>A</td>
<td>B</td>
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<tr>
<td>Secondary sampling</td>
<td></td>
<td></td>
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<tr>
<td>Two secondary samples</td>
<td>A.1</td>
<td>A.2</td>
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<tr>
<td>Analysis. Duplicate analysis</td>
<td>A.1.1 A.1.2</td>
<td>A.2.1 A.2.2</td>
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<table>
<thead>
<tr>
<th>Table II</th>
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<tr>
<td><strong>Components of variance for sampling concentrate</strong></td>
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</table>

<table>
<thead>
<tr>
<th>Frequency of primary sample</th>
<th>Test 1</th>
<th>Test 2</th>
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<tbody>
<tr>
<td>190 seconds</td>
<td>26 seconds</td>
<td>190 seconds</td>
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</table>

<table>
<thead>
<tr>
<th>Components of variance</th>
<th>CV%</th>
<th>Error contribution, %</th>
<th>CV%</th>
<th>Error contribution, %</th>
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<td>Assay lab duplicates</td>
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<td>5.00</td>
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</tbody>
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(95%) when the cutter frequency is 26 seconds as opposed to 58% when the cutter frequency is 190 seconds. This means that the measuring process has a lesser contribution to the overall error when the sampling is more frequent. The sampling process is measuring real changes in the process when the frequency is higher.

➤ The CV% for the primary sampling is lower (3%) with the more frequent sampling as opposed to 6.1% with sampling at 190 seconds.
➤ The error contribution from primary sampling is 24% for the 190 second interval and only 2.5% for the 26 second interval.
➤ The CV% for secondary sampling is lowest of all the components indicating little error in the sub-sampling.

From these results it is clear that the grade of the flotation concentrate varies considerably over a short time span.

In terms of practical application it is important that the frequency of primary sampling is increased as far as possible consistent with the requirement of a particular sample size to be delivered to the lab.

**Calculation of overall sampling error from components of variance**

An important statistical property is that variances are additive for uncorrelated variables. Therefore the overall variance for sampling according to the sampling tree in Table I is:

\[ V_t = V_{ps} / N_{ps} + V_{ss} / N_{ss} + V_{assay} / N_{assay} \]

Where:
- \( V_t \) = Total variance
- \( V_{ps} \) = Variance of primary sampling as measured by experiment
- \( V_{ss} \) = Variance of secondary sampling as measured by experiment
- \( V_{assay} \) = Variance of assay as measured by experiment
- \( N_{ps} \) = Number of primary samples. Usually one per shift
- \( N_{ss} \) = Number of secondary samples. Usually 1 or 2 per shift
- \( N_{assay} \) = Number of replicate assays. Usually more than 2 replicates.

**Conclusions**

In the operation of some flotation plants it was found that there were imbalances between the inputs and outputs to the plants. These imbalances were ascribed to biases in sampling, which arose despite the construction of samplers according to well-known principles.

The biases in sampling occurred because of segregation in the launders feeding the primary samplers together with excessive turbulence and splashing caused by the cutters used for the primary samplers.

The flow in these launders and around newly designed streamlined cutters, to minimize splashing, has been investigated using Finite Element Analysis and a set of rules formulated for the design of launders and primary samplers.

A statistical procedure, using Components of Variance analysis, to determine the variance of primary and secondary sampling and of analysis in the lab is presented. The results of such an analysis, applied to the sampling of a concentrate slurry in a platinum metal concentrator, are given.

**References**


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**CSC in South Africa has appointed Mark Austin to manage its new Mining Vertical (Division)**

The decision to create a dedicated industry vertical for mining follows CSC in South Africa’s early successes in this industry. CSC in South Africa recently won a R3-million deal to project-manage the re-engineering of the Local Area Network (LAN) in the Johannesburg office of BHP Billiton and in July this year CSC launched a new web-enabled R1.5-million Mineral Rights Information System (MRIS) and Central Contracts Register (CCR) for managing BHP Billiton’s mineral rights information. CSC is currently negotiating with another leading mining house to provide it with a mineral rights information system.

Austen spent 17 years in the mining industry before changing careers—from a consulting geologist for a mining house to an account manager for an IT company. He joined CSC in November 2001. He regards the move from a technical career to a more service-orientated one as his most significant achievement and is excited by the challenges facing CSC as it takes advantage of the opportunities presented by the local mining industry.

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