Sampling as a source of uncertainty in metallurgical test results: Assessment and consequences

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While sampling error is increasingly considered when evaluating a deposit to quantify the uncertainty over its value, it is much less frequently considered when analysing the relevance of metallurgical test results, which are nevertheless included in the economic evaluation of the deposit. And yet, the uncertainty over the results of metallurgical tests is greater than that of deposit evaluation, since it incorporates not only errors linked to the drilling strategy and core or cuttings sampling, but also all the errors of subsampling or composite homogenisation, not to mention measurement errors and intrinsic variabilities linked to the treatment process being tested. This paper aims to review the main ore characterisation procedures and metallurgical tests most often used in preliminary, pre-feasibility and feasibility studies, focusing on the various sources of uncertainty, and the means of estimating and controlling their variance. All these errors in intermediate measurements contribute to the overall uncertainty in test results, whether for typical parameters of the ore (such as Bond work index) or for the performance of the envisaged processing stages (such as recovery). This general review is illustrated by examples in the fields of grinding and gravimetric concentration.

INTRODUCTION

During the development of a mining project, samples are taken for metallurgical tests. The sample can be a composite sample made of material from a selection of various drilling products or a sample from a mining test. It can be also a sample from an existing plant with the objective to investigate process enhancement. This sample can represent several tonnes of material which are analysed and split for use in various tests. To consider the ore variability in terms of mineral contents and processability, it is recommended to perform variability studies (Dominy *et al.*, 2018a, 2019; Rossi, 2023) using samples representing various horizons or years of production (Morales *et al.*, 2019). But for a given ore type, it can be necessary to perform many tests with different operating conditions. In that case, the variability of the samples is not due to the heterogeneity of distribution of the deposit, but only on the heterogeneity of constitution of the primary sample. The level of confidence associated with the estimated performance parameters is directly linked to the overall error (including sampling error) of the measurements performed during the tests.

If the real variability of the deposit is increasingly considered for the metallurgical tests through geometallurgy (Dominy *et al.*, 2018b; Rossi, 2023), and if sampling error is also increasingly considered to quantify the uncertainty of the estimated value of the deposit and its impact on the process variability, the measurement error is much less frequently considered when analysing the relevance of metallurgical test results, which are nevertheless included in the economic evaluation of the deposit.

And yet, the uncertainty over the results of metallurgical tests is greater than that of deposit evaluation, since it incorporates not only errors linked to the drilling strategy and core or cuttings sampling to obtain processed material, but also all the errors of subsampling or composite sample homogenisation, not to mention measurement errors and intrinsic variabilities linked to the treatment process being tested. The standardised reports, following the JORC code or the NI 43-101 standard, rarely indicate the accuracy and precision of the results of the tests, or it is limited to comparison between replicated tests.

Such a quantification of uncertainty about the process performances may be a plus for estimating the quality of the results and its subsequent risk level (technical and financial), as part of Quality Assurance and Quality Control (QAQC) strategy.

After presenting which kinds of uncertainty appear at the level of the metallurgical tests, the ones due to measurement and sampling errors are discussed, and their impact on the process performance parameters. This approach is then illustrated with two case studies for ore grinding and gravity concentration, while flotation is briefly discussed. General guidelines are then proposed to incorporate, into the feasibility studies, the uncertainties coming from the measurement errors associated with the process performance parameters. The guidelines set out the approach for quantifying these uncertainties, but also indicate how to use the detailed results of metallurgical tests to improve knowledge of ore heterogeneity and thus obtain better estimates of the sampling errors.

UNCERTAINTY AND METALLURGICAL TESTS

Metallurgical tests are widely used all along the life of a mining project, from the preliminary studies about the deposit, to the mining closure and site remediation. These tests can be performed at laboratory scale or pilot plant scale, sometimes at full plant scale. The expected level of confidence for their results depends on the status of the project and the objective of the study for which these tests have been conducted (Bullock, 2018). Consequently, the accuracy of the test results must be in accordance with this aim. In other words, the error, which is the difference between what is observed during the test and what is observed in normal industrial operation, must be limited to an acceptable range in terms of financial risk. This error is due to uncertainty, in its broadest sense, resulting from a combination of numerous sources of uncertainty.

Sources of Uncertainty

There are various reasons why the results of a metallurgical test may be uncertain. They are of different types:

- Uncertainty from the processed material itself, which is a sample trying to represent a lot such as the entire deposit, a part of it, a block of the geological block model, or a bench. It is mainly due to the true variability of the material characteristics inside the lot and is generally referred to as the 'resource uncertainty' (Dominy *et al.*, 2018b). This uncertainty can be mitigated using geostatistics. But, in that case, it also includes the uncertainty due to the imperfection of the characterisation of the material collected during the drilling campaign.
- Uncertainty from the characterisation of the metallurgical sample used to perform the test. Indeed, in most cases, the primary sample used for the test is specifically characterised as it is generally a composite sample. This characterisation is carried out on a subsample, subject to the sampling uncertainty during taking and preparation, to which is added the uncertainty due to the analysis. This uncertainty will contribute to the difficulty in determining a clear relationship between the performances of the investigated process and the characteristics of the feed.
- Uncertainty due to the variability of the feed of the different test runs. Several metallurgical test runs are generally conducted for the same process stage, with different operating conditions to find a relationship between the observed performance and these conditions. The main objective being to find the optimal conditions maximising the economic performances. Because the metallurgical tests modify the processed material, the different runs cannot be performed on the same feed sample. The primary sample must then be

divided into as many subsamples as the number of test runs. As for the previous uncertainty type, this division will generate a variability of the characteristics of the fed material, leading to the same difficulties as above. This also includes duplicate test runs performed with the same operating conditions. The observed differences are partially due to this type of uncertainty.

- Uncertainty of the control of the operating conditions, which are subject to variability inherent to the control system. In addition, these operating conditions are determined and maintained during the test, using measurements subject to error.
- Uncertainty associated with the determination of the performances of the test run. This includes the errors of the different measurements performed during the test and to characterise the product of the operation. Once again, this characterisation is generally carried out on a sample taken from the product. The performance key parameters inherit the uncertainty of these measurements from which they are calculated.
- Uncertainty due to the lack of knowledge. All the types of uncertainty described above are well-known and well-identified physical parameters. These random uncertainties can generally be expressed as 'measurement errors' and managed. What about the unidentified parameters influencing the metallurgical test? What about the misinterpretation of the results? What about the differences between what we think is being measured and what we are actually measuring? This is the epistemic uncertainty inherent in all scientific and technical activities, which can only be reduced by increasing knowledge (Dominy *et al.*, 2018b).

In this paper, we will focus on the metallurgical test, not considering the first type of uncertainty, which has been thoroughly discussed by many authors, neither the last type.

Uncertainty due to Measurement Error

The terms 'error' and 'uncertainty' are the subject of much debate as to their meaning. For Pierre Gy (1979), error is the random variable describing the difference between the measurement result, subject to uncertainty, and the true value of a physical parameter. Lyman (2019) considers the term error as what is sometimes named 'systematic error', but mostly referred to as bias, reserving the term 'uncertainty' for what is sometimes named 'random error', referring to the natural random variations of the measurement process. He was considering the error as the result of incorrect sampling, which can be reduced or even eliminated by good practice trying to reach correct sampling. Uncertainty being the result of natural variability that cannot be avoided. The only opposition between these two definitions certainly comes from the mother tongues of the authors. What is important is to clearly define the terms to avoid misunderstanding.

The terms 'systematic error' and 'random error' must surely be banished because they are not two different kinds of error, but two aspects of the same random variable, generally represented by the first moments of its probability distribution, the mathematical expectation, and the variance respectively.

Having said that, the imperfection of the measurement protocols and devices generates uncertainty (in the sense of doubt), which can be mathematically described by the measurement error defined as the difference between the measurement result and the unknown true value of the measured parameter for the object to be characterised. The true value being a constant and the measurement result being aleatory, the difference, named absolute measurement error, is a random variable, which can be entirely described by its probability distribution. This difference divided by the true value, named relative measurement error, is also a random variable. The mathematical expectation of this random variable is the bias which must be as close to zero as possible to have an accurate measurement. The standard deviation, square root of the variance, gives the level of precision. By abuse of language, the term 'error' also refers to the 95 % confidence interval and therefore corresponds to 1.96 times the standard deviation.

A measurement can be performed on the object to be characterised itself, or on a representative of that object, namely a sample. The typical measurements which are commonly performed for analysing metallurgical tests are listed in Table I. A large part is done on a sample.

In the frame of metallurgical tests, in order to control and manage the accuracy of their results, the measurement errors must be estimated a priori. In other words, it is preferable to estimate these errors before performing the test and adjust the operating conditions and the measurement protocols to reach the desired level of precision.

Table I. The most common types of measurement carried out during a metallurgical test

Operation	Measurements	Direct / sample	Method
Characterisation of primary metallurgical sample	Moisture content	Subsample	Gravimetric
	Bulk density	Direct / subsample	Mass and volume
	Particle size distribution	Subsample	Sieving + mass / laser
	Particle density distribution	Subsample	Dense liquors / gravity concentrators
	Chemical composition	Subsamples	Spectrometry / chemistry
	Mineralogical composition	Subsamples	Quantitative mineralogy
Characterisation of process feed	Dry solids mass / flowrate	Direct	Scales / belt weigher
	Slurry volume / flowrate	Direct	Glassware / flowmeter
	%-solids or slurry density	Sample	Mass and volume
Characterisation of products	Dry solids mass / flowrate	Direct	Scales or belt weigher
	Slurry volume / flowrate	Direct	Glassware / flowmeter
	%-solids and slurry density	Sample	Mass and volume
	Particle size distribution	Sample	Sieving + mass / laser
	Particle density distribution	Sample	Dense liquors / gravity concentrators
	Chemical composition	Sample	Spectrometry / chemistry
	Mineralogical composition	Sample	Quantitative mineralogy
Process water characterisation	Volume or flowrate	Direct	Glassware / flowmeter
	Chemical composition	Sample	Spectrometry / chemistry
	Electrochemical parameters	Direct / sample	pH, Eh, conductivity…
Characterisation of reagents	Quantity and concentration	Direct	Mass or volume
	Chemical composition	Sample	Spectrometry / chemistry
Operating conditions	Energy consumption	Direct	Electric meter, steam flowmeter
	Temperature and pressure	Direct	Probes

Sampling Error as Part of Measurement Error

In the case of a measurement of a parameter necessitating sampling, the associated sampling error is defined as the difference between the unknown true value of that parameter for the sample and the unknown true value of that parameter for the lot to be characterised. The true value for the lot being a constant and the true value for the sample being aleatory (due to the random selection of the sample), the difference, named absolute sampling error, is a random variable, which can be entirely described by its probability distribution. This difference divided by the true value, named relative sampling error, is also a random variable. As well known, the difference between the observed parameter values from different samples drawn from the same lot is due to the heterogeneity of the material of the lot regarding that parameter, heterogeneity in constitution and in distribution (Gy, 1988; Pittard, 2009; Lyman, 2019; Esbensen, 2020).

From their definitions, it is clear that the sampling error is a part of the total measurement error in the characterisation process of the considered lot. The remaining part being the analytical error defined as the difference between the measurement result and the unknown true value of the parameter for the

sample. Any estimation of the measurement error must include sampling error and analytical error. Theory of Sampling (TOS) allows a priori estimation of the sampling error (Gy, 1979; Lyman, 2019). The Guide to the Expression of Uncertainty in Measurement (GUM) approach allows a priori estimation of the analytical error (JCGM; 2008).

Error Propagation to Performance Parameters

Several metallurgical test runs being performed with different operating conditions for the same processing operation; the comparison of their results is done using performance parameters which are calculated from a set of measurement results. The performance parameters are then random variables as they are functions of measurement random variables.

The random variable Y, corresponding to a calculated parameter, is a function of a set of independent random variables $\{X_i\}$, corresponding to the measurements: $Y = g(\{X_i\})$. If the full probability distributions of the measurements are known, the full probability distribution of the calculated parameter can be determined using different technics (Lyman, 2019). Unfortunately, in most of cases, only an estimate of these probability distributions is accessible. It is generally supposed to be a Normal distribution characterised by its mathematical expectation and its variance. Under this assumption, and if the dispersion of the probability distribution of the measurements is sufficiently low, corresponding to a small variance, approximated values of the mathematical expectation (Equation 1) and of the variance (Equation 2) of the random variable Y can be calculated.

$$E(Y) \approx g(\{E(X_i)\}) \quad [1]$$

$$\sigma^2(Y) \approx \sum_{j=1}^{n} \left[\frac{\partial g}{\partial X_j}(\{E(X_i)\})\right]^2 \sigma^2(X_j) \quad [2]$$

This calculation method is also used to determine basic mineral processing parameters such as moisture content, particle size distribution, density and density distribution, or %-solids.

A Priori Measurement Error Versus Statistical Control

It is a common practice to replicate metallurgical tests by carrying out two or more runs in the same operating conditions and comparing the results. It is equivalent to using sample duplicates for analysis in the frame of quality control procedures. But it is not a way to have an estimate of the variability of the test runs as such experimental variance calculation necessitates at least 30 runs to be called a statistical approach. Such replicate practice is only a way to control the reproducibility of the studied process. Depending on the observed difference between results, many questions arise:

- If the difference is small, is this proof of good accuracy and precision of the test? The answer is no.
    - It will never constitute proof of accuracy because a bias cannot be detected by such statistical control, even if numerous test runs are carried out.
    - This is not proof of precision as there is always a non-negligible probability of obtaining close results.
- If the difference is significant, what could be the reason?
    - Is it due to a mistake made during one of the test runs? But for which one? Which one is valid? Knowing that both can be wrong.
    - Is it due to epistemic uncertainly?
    - Or is it simply due to the natural variability described above?
    - Can an additional replicate help to answer these questions?

Only an a priori calculation of the measurement errors can help to answer these questions. Indeed, if the difference observed between the values of a given parameter is less than the measurement error associated with that parameter, the difference can be considered normal. If the observed difference is significantly greater than the measurement error, the hypotheses of a mistake during a test run or epistemic uncertainty can be retained. Investigations are needed to find the source of this uncertainty

and improve the test protocol to reduce it. Consequently, before carrying out any metallurgical test, some actions must be done to effectively control the validity of its results, as described in the General Guidelines section below.

CASE STUDIES

To illustrate this talk, some case studies concerning the most common metallurgical tests are proposed. Some of them have already been published.

Grinding

The first case concerning grinding has already been published (Brochot, 2012a). The metallurgical tests have been performed on a lateritic ore which is a blend of limonite, earthy saprolite and rocky saprolite facies. Limonite and earthy saprolite are characterised by a large proportion of -100 µm, earthy saprolite having a larger proportion of millimetric particles. Rocky saprolite is characterised by a large proportion of +10 mm.

The metallurgical test campaign is focused on the size reduction stage by semi-autogenous grinding (SAG). A pilot plant scale SAG mill was used for several batch tests in various operating conditions. A primary sample of 5615 kg (wet mass) coming from the deposit was used to perform these tests. The representativeness of this sample relatively to the deposit is not considered at this stage. The primary sample is then considered as the lot and only the sub-sampling for characterisation and the feed variability between the tests are studied here.

The primary sample has been divided and a 352 kg subsample has been dried for moisture content measurement (279 kg dry subsample, 20.7 % moisture content). After division, a 137 kg subsample of dry ore was sieved at 100 mm, 50 mm, 11.2 mm, 5 mm, 2,8 mm, 1,4 mm and 0.1 mm to obtain eight particle size classes which have been weighed for particle size distribution determination. Considering only the sampling step drawing the subsample of mass M_S for particle size distribution analysis from the primary sample of mass M_L, it generates a fundamental sampling error (FSE) for the measurement of the proportion t_c of the size class c, the relative variance of which is given by the formula of simple particles (Equation 3).

$$\sigma_c^2(FSE) = \left(\frac{1}{M_S} - \frac{1}{M_L}\right)\left[m_c \frac{1-2t_c}{t_c} + \sum_{i=1}^{N_s} t_i m_i\right] \quad [3]$$

For the N_s size classes with index i ordered from the coarsest to the finest, the mean particle mass m_i is estimated using the shape factor, the mean size, and the mean density of the particles of this class. The proportions t_i of all the size classes also influences this sampling error. In the case of the cumulative passing size distribution, the passing proportion is given by Equation 4 and the variance of its associated FSE is given by Equation 5. The Figure 1 gives the measured feed size distribution and its uncertainty envelope corresponding to the 95 % confidence interval (plus or minus two standard deviations).

$$T_c = \sum_{i=c}^{N_s} t_i \quad [4]$$

$$\sigma_c^2(FSE) = \left(\frac{1}{M_S} - \frac{1}{M_L}\right)\left[\sum_{i=c}^{N_s} t_i m_i \frac{1-2T_c}{T_c^2} + \sum_{i=1}^{N_s} t_i m_i\right] \quad [5]$$

The large FSE for the measurement of the 100-200 mm size proportion is due to the small number of particles in the subsample size fraction (3 or 4) in this class. In that case, the approximation done to obtain the Equation 3 is not valid. It is necessary to return to the primary formulae based on Poisson

distribution of particle numbers (Gy, 1979; Lyman *et al*, 2010). Nevertheless, we will keep these variances as they indicate a huge uncertainty even though it can appear outside the physical range.

Considering the other components of the sampling error, mainly the Grouping and Segregation Error (GSE) in that case, the variance of the total sampling error can be as large as twice the one of FSE. The analytical error, coming from the imperfection of the scales used for weighing the size fractions, and, with a negligible impact, the uncertainty due to sieving efficiency, becomes negligible compared with the sampling error. This is the first source of uncertainty coming from the characterisation of the primary sample (considered as the lot).

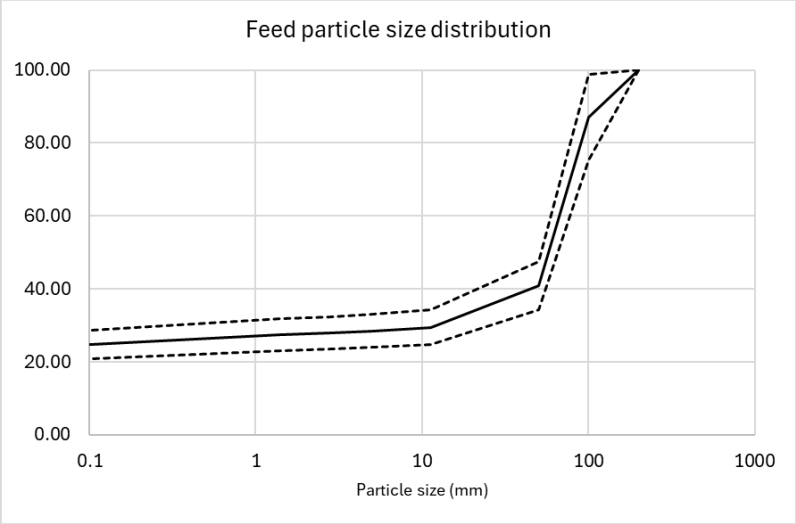

Figure 1. Particle size distribution of SAG mill feed (solid line) and its uncertainty envelope corresponding to the 95% confidence interval (dash lines).

But the different batch tests are performed on different subsamples taken from the primary sample. The characteristics of these subsamples are slightly different, and their variability can be high if the sample is small. In the published case, the masses of material fed in the SAG mill vary from 58 kg to 146 kg. The Figure 2 gives the uncertainty envelop (with 95% confidence) of the feed size distribution for these two extremes of sample mass when considering the variance of FSE given by Equation 5. As the different subsamples are taken from a relatively small lot without replacement, the sampling operations are not totally independent and the formula for the variance is slightly different (Lyman, 2019). The number of particles in the lot being sufficiently large, the difference is negligible.

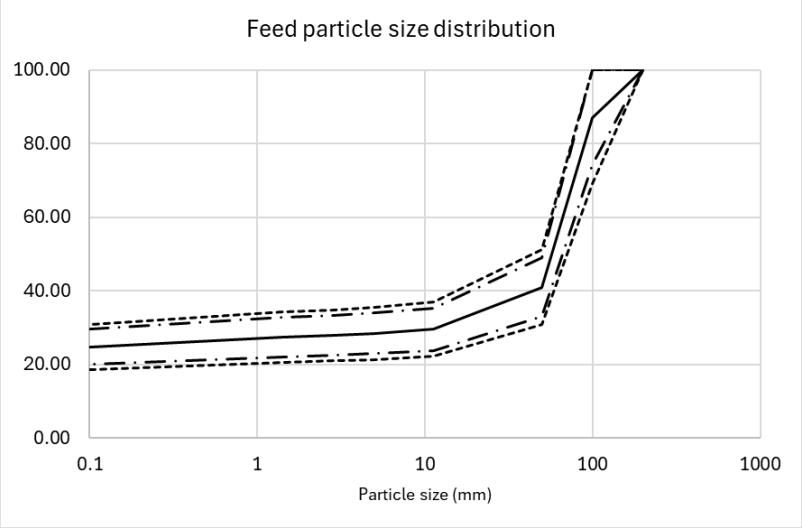

Figure 2. Particle size distribution of SAG mill feed (solid line) and its uncertainty envelope corresponding to the 95% confidence interval for 58 kg (dash lines) and for 146 kg (dash-dot line).

The variance of the total sampling error for the feed sample drawing (twice the one of FSE and no analytical error) must be added to the variance of the total sampling error for the characterisation sample. This results to large errors for the coarsest size classes, but also for the finest ones (32% for 146 kg and 42% for 58 kg). Due to this large dispersion of the probability distribution, Equation 2 for error propagation cannot be rigorously applied. Nevertheless, it will be used to calculate the measurement error associated to the calculation of the d_{80} of the distribution (Equation 6 with i chosen to verify $T_{i+1} \leq 0.8 \leq T_i$ and Equation 7) and then the operating Bond work index (Equations 8 and 9).

$$\ln(d_{80}) = \ln(x_{i+1}) + \frac{0.8 - T_{i+1}}{T_i - T_{i+1}}(\ln(x_i) - \ln(x_{i+1})) \quad [6]$$

$$\sigma^2(d_{80}) = \frac{(\ln(x_i) - \ln(x_{i+1}))^2}{(T_i - T_{i+1})^4}[(0.8 - T_{i+1})^2 T_i^2 \sigma^2(T_i) + (T_i - 0.8)^2 T_{i+1}^2 \sigma^2(T_{i+1})] \quad [7]$$

$$W_i = \frac{W}{\frac{10}{\sqrt{d_{P80}}} - \frac{10}{\sqrt{d_{F80}}}} \quad [8]$$

$$\sigma^2(W_i) = \frac{d_{F80}\sigma^2(d_{P80}) + d_{P80}\sigma^2(d_{F80})}{4\left(\sqrt{d_{F80}} - \sqrt{d_{P80}}\right)^2} \quad [9]$$

The feed fineness d_{F80} is calculated by log-linear interpolation from the size distribution measured on the sample for characterisation, and its sampling error variance with the calculated variance of the sampling error as presented above. The product fineness d_{P80} is similarly calculated from the size distribution of the mill product that is measured by sieving of the entire product material. The sampling error is then null, and the analytical error is estimated to 7%, including the interpolation uncertainty. The Figure 3 gives the values of the reduction ratio and the operating work index for the four test runs corresponding to four different values of the %-solids, 25 %, 30 %, 40 % and 50 %, the other operating conditions being constant. The error bars are only considering the FSE and analytical error. It clearly shows the difficulty to conclude about the influence of the %-solids on these performance parameters. If one can say the operating work index and the reduction ratio are respectively increasing and decreasing with the %-solids, it is not possible to conclude about an apparent extremum around 30% solids.

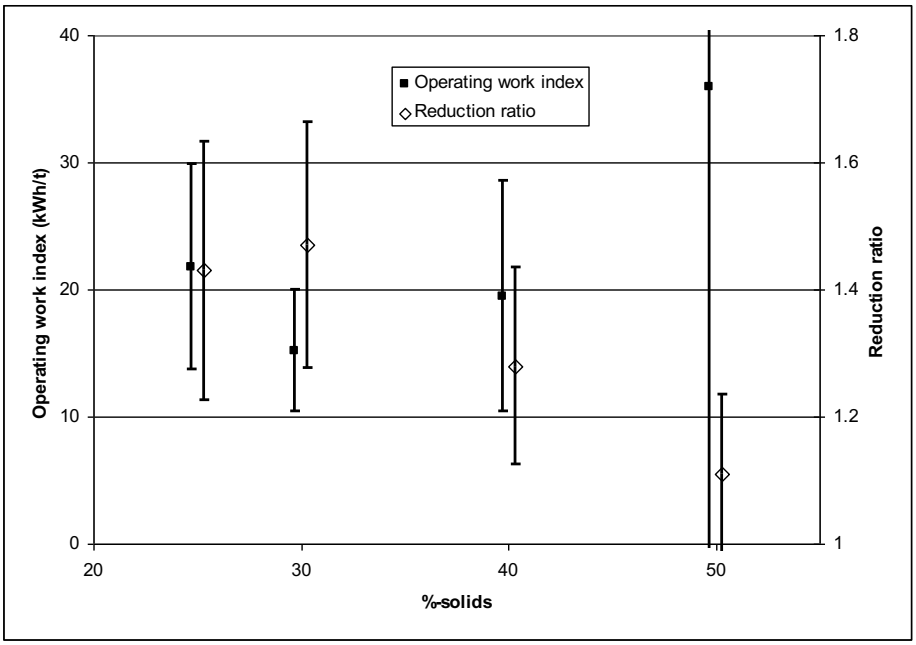

Figure 3. Variation of reduction ratio and operating work index with %-solids.

The main result of this sampling approach is the large uncertainty at the level of the primary sample characterisation as well as the variability of the feed of the different test runs. And this uncertainty does not account the uncertainty in the primary sample representativeness. Even though this observation seems pessimistic it has not to question the results of the conducted metallurgical tests. Indeed, the specificity of the ore are well considered. The results of this first campaign have been confirmed by another one, at least concerning the behaviour of the ore during grinding. But all the results must be read at the light of the mentioned uncertainty. The following improvements can be mentioned for recommendations:

- Perform the size distribution analysis on a larger sample.
- As the main component of the uncertainty is coming from the variability of the test feeds, each feed can be entirely analysed for the size distribution before performing the test and then homogenised after the blending of the size fractions. This has the advantage of avoiding the sampling error of the characterisation stage and of using the 'true' feed size distribution to calculate the grinding test performances.

Gravity Concentration

The second case concerning gravity concentration of gold ore has already been published (Brochot, 2016). The standard Gravity Recoverable Gold (GRG) test, using the Laboratory Knelson Concentrator (LKC) or a Laboratory Falcon Concentrator, has the advantage of giving information not only about the ability of ore to be concentrated by gravity separation, but also about the particle size distribution after each grinding stage, the liberation of the minerals of interest (in this case native gold or gold bearing minerals) and the distribution of these minerals (Wardell-Johnson, Bax, Staunton, McGrath, & Eksteen, 2013). Such results give a first level of description of the heterogeneity of the ore regarding the metals or minerals of interest. Additional analyses performed on the concentrates and tailings (such as enhanced separation between free gold and heavy minerals, cyanidation, or quantitative mineralogy) allow the increase of the level of details to refine the heterogeneity description.

The standard GRG test as initially proposed (Laplante, Woodcock, & Huang, 2000) is performed in three stages where the tailings of one stage are reground to feed the next stage. From a primary sample of mass M_{G1} crushed and milled to 100 % minus 850 µm, a secondary sample of mass M_{S1} is taken to feed the stage 1. The typical mass for this sample is 50 kg; but it can be adjusted (from 25 kg to 140 kg) depending on the expected amount of GRG and the size distribution. Generally, the size distribution

and the Au content per size class of the feed is back calculated from the analysis of concentrate and tailing. Nevertheless, a subsample of mass M_{S1A} can be taken from ground primary sample for size distribution analysis. The entire mass M_{C1} of concentrate is analysed for size distribution. A subsample of mass M_{T1A} is taken from the tailings (mass M_{T1}) for size distribution analysis. Another sample of mass M_{G2} is taken and ground to have between 45 and 60 % minus 75 µm. If the entire remaining tailings are ground, this mass is $M_{G2} = M_{T1} - M_{T1A}$. A sample of mass M_{S2} is taken from the ground material to feed the second stage. Generally, only the feed is ground ($M_{S2} = M_{G2}$). Optionally, a subsample of mass M_{S2A} can be taken from second stage feed for analysis. As for stage 1, the entire mass M_{C2} of concentrate is analysed and a subsample of mass M_{T2A} is taken from the tailings (mass M_{T2}) for analysis. Similarly, a mass M_{G3} is ground, from which a subsample of mass M_{S3A} can be taken for analysis and a mass M_{S3} feeds the third stage to generate a mass M_{C3} of concentrate and a mass M_{T3} of tailings, from which a mass M_{T3A} is taken for analysis. Figure 4 summarises this procedure.

The analyses of the concentrates and the samples of tailings consist in the determination of their size distribution by sieving. If M_{xi} is the mass of material in size class i of sample x (x being S1A, C1, T1A, S2A, C2, T2A, S3A, C3, or T3A), the size fraction (in g/g) is given by $f_{xi} = M_{xi} / M_x$. Each size fraction can be subject to additional separation. When coarse free gold is observed, panning can be used to recover the gold flakes and analyse the reject separately. This procedure has the advantage of having a better characterisation of the free gold particles in terms of size distribution and shape factor. If M_{xia} and M_{xib} are respectively the masses of material in the flakes and reject products from size class i of sample x (x being C1, C2, or C3), the density class fraction (in g/g) is given by $f_{xia/b} = M_{xia/b} / M_{xi}$. Each size fraction (or each density class of each size fraction) of the concentrates is fire-assayed (FA) up to extinction, giving the gold content a_{xi} or $a_{xia/b}$. The tailing fractions coarser than 105 µm are pulverised to -105 µm. Each size fraction of the sample of the tailings are divided to obtain a sample of mass M_{xiFA} (x being T1A, T2A, or T3A) which is fire-assayed to give gold assay a_{xi}. The same procedure is applied for analysis of the feed samples, except that screen fire assay can be necessary for the coarsest size classes in the presence of coarse gold (Brochot, 2012b). If samples of the feeds are taken and analysed, the results can be compared to the values back calculated from the results of the product analyses. This redundancy of measurements, associated with their measurement errors, can be valorised using data reconciliation by material balance to improve the global accuracy of the test.

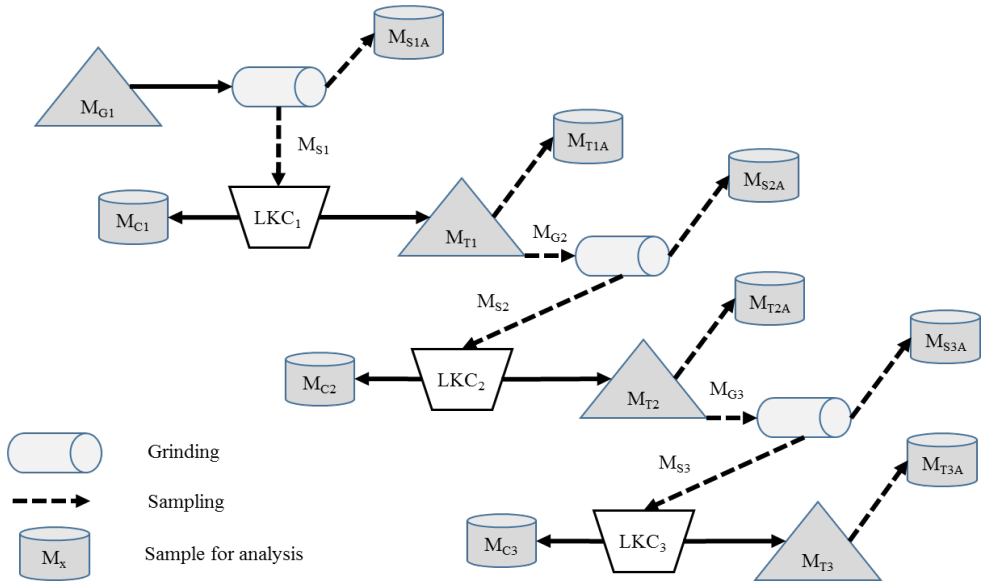

Figure 4. GRG 3 stages procedure using Laboratory Knelson Concentrator.

The base formulae of the theory of sampling (Gy, 1979) are considering particles individually with their key parameters: unit mass and critical content. As it is impossible to have such a fine description, particles are classified in numerous families in which they are supposed similar. Each family is then

characterised by three parameters: the mean unit mass and the mean critical content of its particles, and the mass proportion of that family in the lot. These parameters are obtained by measurement through specific experiments, such as the present GRG test. As long as the number of particles in the different families of the heterogeneity model is sufficiently high for a given sample size, the probability distribution is approximately a Normal distribution which can be characterised by its variance. Gy (1979) proposed the formula for varied particles giving the relative variance of the FSE for the measurement of the content a_L of the critical component in the lot (Equation 10).

$$\sigma^2(FSE) = \left(\frac{1}{M_S} - \frac{1}{M_L}\right)\sum_{i=1}^{N_F} t_i m_i \left(\frac{a_i - a_L}{a_L}\right)^2 \quad [10]$$

The N_F families, numbered with index i, should be as homogenous as possible, meaning that all the particles in one family have slightly the same unit mass, m_i, and the same critical content, a_i. t_i is the mass proportion of the family in the lot. Here, the family groups the particles reporting to a sub-class of a size class of one of the concentration products and its index i is composed of the letter C (concentrate) or T (tailings) to designate the product, the index of the size class, and the letter A (bearing) or B (barren) to designate the sub-class. For example, t_{C4B} is the proportion of barren particles in the size class 4 of the concentrate relatively to the total amount of the feed.

The same formula (Equation 10) with the same set of families (the same heterogeneity model) can be used to estimate the variance of the FSE of the different performed measurements, only the critical component and its content per family are changing. The Table II lists some measurements that are performed during a GRG test with the critical contents which are used to estimate their FSE variance. In addition to these examples, the FSE can be calculated for all the measured parameters including all size distributions, all gold contents, the yield and the proportion of bearing mineral in each stage, but also performance parameters such as the gold recovery.

Table II. Measurements performed during a GRG test. Values of the critical content a_i per family and as calculated for the lot in the case of the first concentration stage

Measurement	**Family critical content a_i**	**Lot critical content a_L**
Feed size distribution: proportion of size class j	$a_{CjA} = a_{CjB} = a_{TjA} = a_{TjB} = 1$ $a_{CiA} = a_{CiB} = a_{TiA} = a_{TiB} = 0$ for $i \neq j$	$a_L = \frac{M_{S1Aj}}{M_{S1A}}$
Proportion of concentrate in size class j relative to feed	$a_{CjA} = a_{CjB} = 1$ $\quad a_{TjA} = a_{TjB} = 0$ $a_{CiA} = a_{CiB} = a_{TiA} = a_{TiB} = 0$ for $i \neq j$	$a_L = \frac{M_{C1j}}{M_{S1}}$
Proportion reporting to concentrate (yield)	$a_{CiA} = a_{CiB} = 1 \quad \forall i$ $a_{TiA} = a_{TiB} = 0 \quad \forall i$	$a_L = \frac{M_{C1}}{M_{S1}}$
Feed gold content	a_i = assayed gold contents	$a_L = \sum_i t_i a_i$

This case clearly shows that a GRG test is both a characterisation procedure and a metallurgical test, enabling the recovery process to be properly designed. But the set of obtained detailed data can be used to build a heterogeneity model able to calculate the sampling error associated with all the performed measurements during the test, and then estimate its validity and the expected level of variability. Going further, this heterogeneity model gives more information about the sources of heterogeneity of the ore from which the primary sample has been taken. The sampling error occurring at the primary sample drawing can be estimated and consequently the variability of the processing plant feed.

Flotation

Flotation being the most frequently used process in the world, a huge number of flotation tests are conducted each year. Unfortunately, the uncertainty of the results is rarely questioned, generally limited to research works by statistics over replicated tests (Napier-Munn, 2012). The uncertainty due to the ore body variability and to the primary sampling error is more frequent (Dominy, 2018a), rarely in standardised reports.

The flotation tests are comparable to the GRG tests. Indeed, the batch flotation test runs are generally a succession of concentration stages (roughing, scavenging, several cleaning) with detailed analysis of the products (Runge, 2010). A similar approach is used to build a heterogeneity model. Due to the fineness of the processed material, the sampling error is generally small compared to the analytical error. It is the case when measuring the size distribution and the chemical content. It is less the case for the size-by-size chemical or mineralogical contents, and the sampling error can be high when considering quantitative mineralogy with the different types of particles and the liberation data.

GENERAL GUIDELINES

In the frame of a feasibility study for a mining project, whatever it is for a greenfield or a brownfield project, the main objective of the metallurgical tests is to deliver a lot of information about the technical and financial feasibility of the selected processing route of the ore. Associating the results of these tests to the geological model of the ore deposit allows to manage the variability in terms of the ore characteristics and the expected processing performances, the role of geometallurgy (Dominy, 2018b). But another source of variability comes from the uncertainty of the metallurgical test results themselves, consequence of the errors of measurements (in their random meaning), including the sampling error. Knowing these errors, through their variance, is a way to control and manage this uncertainty. The variance can be estimated relatively accurately by defining the heterogeneity model for the sampling error components and the uncertainty budget for the analytical part. Additionally, the metallurgical tests provide a lot of information allowing to define a sufficiently detailed heterogeneity model (as for the example of the GRG test).

From the experience of these case studies and the observation of many industrial or research projects, the following general guidelines can be proposed. Reporting their application in the frame of a code (such as JORC or NI 43-101) may be a plus for estimating the quality of the results and its subsequent risk level (technical and financial), as part of QAQC strategy.

Before carrying out any metallurgical test, the following works are recommended:

- Clearly define the level of necessary precision in accordance with the objective of the study for which the metallurgical tests are performed. This level of confidence is linked with the acceptable technical and financial risks at the stage of the study (McCarthy, 2003).
- Draw up the list of all the performance parameters required by the technical and economic objectives of the study and define all the necessary measurements needed to obtain these parameters.
- Characterise the material to be processed, in accordance with the test objectives, by performing all necessary measurements and calculating their associated error. Based on these results, build a provisional heterogeneity model of this material and of the expected processing products, with some assumptions if necessary.
- Calculate the errors associated with the measurements to be done during the test, including the sampling errors based on the provisional heterogeneity models, considering adjustable variables such as quantity of processed material, measurement technics and protocols, equipment sizes, test procedures. Calculate the errors associated with the performance parameters.
- Design the metallurgical test and all measurement procedures to distribute the acceptable total measurement error between its different components by adjusting the variables.
- Track all the potential risks of incorrect sampling, source of bias, and use procedures and devices avoiding them.

During the metallurgical test, in addition to carrying it out in the best conditions and following the rules of good practice, it is necessary to pay particular attention to:

- The way the samples for feeding the test are taken and divided, respecting the equiprobability of each particle to be selected.
- Same attention concerning sampling of the products.

- The measurement conditions and frequency.
- The quality of preparation of collected samples and their analysis.

After the test, when all measurement results are available, in addition to calculate all the performance parameters and compare the different test runs, the following steps must be done to estimate the level of uncertainty of conclusions of the metallurgical test campaign:

- From the collected information, improve the provisional heterogeneity models adding some details and using values coming from measurement in place of assumed values.
- Calculate the sampling error for all sampling stages on all sampled materials. Including this sampling error, calculate the full measurement error for all measured basis parameters.
- Improve the accuracy and precision of the measurement results using data reconciliation by material balance if there are enough redundancies.
- Calculate the error associated with each performance parameter using the error propagation rule.
- Consider the calculated errors and their associated confidence intervals for the conclusions, concerning the behaviour of the tested processes, by discussing their relevance.
- From these confidence intervals, estimate the technical, and then the financial, risk.
- To go further, use the detailed heterogeneity model to estimate the primary sampling error and to design the future sampling campaigns, such as for core drilling campaign or for processing plant survey.

CONCLUSION

Twenty years ago, McCarthy (2003) pointed out "inadequate metallurgical testing" as one of the key drivers of mining underperformance, and one of the reasons for such mistake comes from "inadequate sampling for grindability and liberation studies to determine recovery". Admittedly, progress has been made since then to remedy these underperformances, by the promotion of geometallurgy, but the quantification of the risk associated with the treatment process remains unclear and often overoptimistic, leading to delays, additional costs and undercapacity plants.

Quantifying measurement errors before carrying out the tests enables them to be better designed in terms of the number of runs, the quantity of material to be processed, the sampling and analysis procedures, and the means of measurement. After the tests, these measurement errors can be adjusted by including the collected information in the calculation process. Finaly, the risk is reduced, by selecting and designing adequate and more accurate metallurgical test runs, and accurately estimated, using the calculated confidence intervals of the key parameters. The guidelines above attempt to indicate the approach to achieving this objective.

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