

# Quality and sampling error quantification for gold mineral resource estimation



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Sampling is a vital component during all stages of the mine value chain. It includes the sampling of *in situ* material and broken rock for geological, metallurgical and geoenvironmental purposes. Sampling errors are defined in the context of the Theory of Sampling (TOS), where incorrect actions may lead to uncertainty and create a significant overall sampling + measurement error.<sup>1-3</sup> The TOS breaks down this error into a series of contributions along the full value chain (the planning to assay-measurement process). Errors are additive throughout this pathway, unavoidably exacerbating risk.<sup>2,4-6</sup> After collection, sampling errors also occur throughout all subsequent downstream processes contributing to uncertainty in test work and any decisions made thereon. Across the full mine value chain, the sum of these errors generate both financial and intangible losses. In essence, poor-quality, non-representative sampling increases project risk and may consequently often lead to incorrect project valuation. There is hardly any other application field where this is as critically important than for Gold mineral resource estimation, because of the very low grades and the extremely irregular mineralisation heterogeneities encountered (Figure 1).

## Sampling—the first critical success factor in the mine value chain

The data produced must be fit-for-purpose to contribute to mineral resources/ore reserves reported in accordance with the 2017 PERC<sup>7</sup> or other international codes. Quality assurance/quality control (QA/QC) is critical to maintaining data integrity through

documented procedures, sample security, and monitoring of precision, accuracy and contamination. Samples and their associated assays are key inputs into important decisions throughout the mine value chain.

The TOS was first developed in the 1950s by Dr Pierre Gy to deal with sampling challenges in the mining industry, though it has far wider applications



**Figure 1.** For optimal sampling error quantification for Gold mineral resource estimation no efforts are spared: Reverse Circulation grade control drilling at the Novo Resources Corporation Beatons Creek project in Western Australia.

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today.<sup>1–3</sup> The TOS provides critical guidelines for reducing sampling errors, Table 1.

## Quality assurance and quality control

Quality assurance and quality control are the key components of a quality management system.<sup>8–10</sup> Quality assurance is the collation of all actions *necessary* to provide adequate *confidence* that a process (e.g. sampling, test work and assaying) will satisfy the pertinent quality requirements. While QA deals with prevention of problems, QC aims to detect these—in time. Quality control procedures monitor both precision and accuracy of samples and data, as well as possible sample contamination during

preparation and assaying. Throughout any mineral resource sampling programme, QA/QC is a key activity to determine the imperative of fit-for-purpose samples.

Protocols should be set up to cover: field collection, laboratory preparation and analysis. During grade control, QA/QC should include field duplicates and certified reference material (CRM) submission, e.g. a minimum of three CRMs at a range of grades, including blanks. Laboratory QA/QC shall include internal CRMs, pulp duplicates, umpire sample submission, pulp screen tests and contamination tests. In particular, duplicate field samples provide a measure of variability of the entire sampling and analysis process. Best

practice QA/QC is a very comprehensive framework, Table 2.

Documentation of sample collection and laboratory activities is an important part of QA/QC, as is appropriate staff training and monitoring. It is the opinion of the present authors that quality samples only follow from well-trained and experienced personnel. Companies should ensure that all staff involved in sampling activities are appropriately trained in sampling and, during their first few months, have adequate mentoring (sampling QA). This will be additional to other standard operational and safety training. Proper training shall be facilitated by well-written and illustrated documentation, see examples in Reference 3.

**Table 1.** Definition of TOS sampling errors.

Sampling error	Acronym	Error type	Effect on sampling	Source of error	Error definition
Fundamental	FSE	Correct Sampling Error (CSE)	Random errors—Precision generator	Characteristics of the ore type. Relates to constitution and distribution heterogeneity	Results from grade heterogeneity of the broken lot. Of all sampling errors, the FSE does not cancel out and remains even if a sampling operation is “correct”. Experience shows that the total nugget effect in variographic modelling can also be artificially high because sample masses are not optimal.
Grouping and Segregation	GSE				Relates to the error due to the combination of grouping and segregation of rock fragments in the lot. Once rock is broken, there will be segregation of particles at all scales, e.g. surface stockpile or laboratory pulp.
Delimitation	IDE	Incorrect Sampling Error (ISE)	Uncontrollable inconstant errors—Bias generators	Sampling equipment and materials handling	Results from an incorrect shape of the volume delineation of an increment or a sample.
Extraction	IEE				Results from the incorrect extraction of a sample. Extraction is only “correct” when all fragments within the delineated volume are fully extracted.
Weighting	IWE				Samples should represent a consistent mass per unit (e.g. kg m <sup>-1</sup> ).
Preparation	IPE				Refers to issues during sample transport and storage, e.g. mix-up, damage, loss and alteration, preparation (contamination and/or losses), and intentional actions (sabotage and salting), as well as unintentional actions (carelessness and non-adherence to protocols).
Analytical	TAE	—		Analytical process	Relates to all errors during assay and analytical processes, including issues related to rock matrix effects and debilitating analytical equipment maintenance, faulty calibration etc.

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**Table 2.** Best practice QA/QC for a Gold grade control sampling programme for sound resource estimation.

QA/QC action	Rate <sup>k</sup>	Instigator	Key Performance Indicators (KPI)	
			Fine Gold/[Coarse Gold]	
Field duplicates <sup>a</sup>	1 in 20	Operator	90% ± 10–25% HARD <sup>j</sup> [90% ± 25–50% HARD]	90% ± 13–35% RSV [90% ± 35–70% RSV]
Sample quality index <sup>b</sup>	All	Operator	Depends upon sample type; for saw-cut channels >80%; for diamond drill core >85% recovery	
Coarse reject duplicates <sup>c</sup>	1 in 20	Laboratory	90% ± 10–20% HARD [90% ± 20–50% HARD]	90% ± 13–28% RSV [90% ± 26–70% RSV]
Pulp duplicates <sup>d</sup>	1 in 20	Laboratory	90% ± 10% HARD [90% ± 10–20% HARD]	90% ± 13% RSV [90% ± 13–28% RSV]
Certified Reference Materials <sup>e</sup>	1 in 20	Operator and laboratory	<2δ ("safe zone") no action required 2δ–3δ ("warning") investigate (re-assay 25% of batch if required) >3δ ("action") re-assay 100% of batch	
Blanks <sup>f</sup>	1 in 20	Operator and laboratory	Less than 0.05 g/t Au	
Pulp quality <sup>g</sup>	1 in 20	Laboratory	95% to be P95 –75µm	
Barren flush <sup>h</sup>	1 in 20–50	Laboratory	<<0.5% gold loss	
Umpire assays <sup>i</sup>	1 in 20	Operator	90% ± 10% HARD	90% ± 13% RSV
Laboratory audit	Quarterly	Operator	Full adherence to agreed practices and performance levels	
QA/QC review	Monthly	Operator and laboratory	Compliance across all metrics	

<sup>a</sup>Applies to any sample type collected.

<sup>b</sup>Applied to linear and drill samples; KPIs are based on sample type and expected mass.

<sup>c</sup>Laboratory crusher or reverse circulation (RC) rig rejects.

<sup>d</sup>Dependent upon nature of ore and assay method. For samples assayed via screen fire assay (SFA), a high precision would be expected for undersize fraction.

<sup>e</sup>Recommendation to have a minimum of three CRMs at grades ranging from cut-off, ROM and high-grade. For any batch of (say) 20–30 samples, three key CRMs should be added. Note that by their very need to be homogeneous, CRMs do not bear coarse "nuggety" gold, but they can be matrix matched by being quartz-dominated, sulphide-bearing a.o. The laboratory will also insert its own CRMs. CRMs used for SFA process will just be fire assayed. Action is required if 3δ breached, usually re-assay of the entire batch if possible.

<sup>f</sup>Blanks provide a measure of contamination. They should be inserted after expected high-grade and/or visible gold-bearing samples. If substantial visible gold is present, two separate blanks should be placed after the sample. One blank should be added together with the three CRMs per batch. Laboratory will also place blanks into the sample stream.

<sup>g</sup>Test involves screening or use of an autosizer of the pulp to ensure 95% passing. All samples should pass or the entire batch should be reground.

<sup>h</sup>Barren flush may be inserted after each and every sample for coarse gold samples. Assaying of the barren flush; for fine gold

ores, a rate of 1 in 50 is appropriate increasing to 1 in 20 for coarse gold ores. Careful management of coarse gold ores is required. It is suggested that laboratories include a "wash" after visibly high-grade (e.g. visible gold-bearing) samples. However, if the ore bears notable coarse gold, then cleaning is best after each sample given that even low-grade samples can bear coarse gold particles.

<sup>i</sup>Monthly submission of samples (typically pulps), including standards and duplicates is sufficient to provide a check of primary laboratory results. This is especially important where an on-site laboratory is being used as it provides independent confirmation of the results. Where SFA, LW or PAL is used, there may be no pulp residues to submit. In this case, coarse rejects can be used. Umpire samples (e.g. pulps or coarse duplicates) should be supplied to the mine and submitted by mine staff to the umpire laboratory. In some cases, the laboratory (mine or off-site) may submit umpire samples as part of their internal QA/QC.

<sup>j</sup>HARD is half the absolute difference of the pair divided by the pair mean; HARD value for fine versus coarse gold; HARD can be expressed as RSV, where  $HARD = \sqrt{2} \cdot RSV$ , e.g. ±10% HARD is ±13% RSV.

<sup>k</sup>It is important to ensure that enough QA data is collected, particularly during a small sampling programme. The rate of insertion of CRMs, blanks etc. may need to be increased beyond the nominal 1 in 20 to achieve a minimum of 10 results.

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Training and mentoring should be linked to continuous quality improvement programmes, where protocols are internally and externally audited at least annually. On-going supervision and periodic re-training are strongly recommended, and should always in part be based on practice at the rock face/in the core shed, not only in the classroom.

## Quantifying errors along the full sampling value chain

The results of *duplicate sampling* programmes document the magnitude of errors across the *full* sampling value chain, Table 3. These generally show that a large component of the total error is introduced during sample collection, especially during primary sampling. As a result, undertaking excessive efforts to reduce errors during preparation and analysis will not necessarily result in a substantive uncertainty reduction. In contrast, the collection of larger, high-quality field samples (for examples using a higher number of increments in composite samples) will result in significant error reduction provided that other protocols are optimised appropriately.

Test work from a Gold vein deposit exemplifies the impact of sampling error through comparison of chip vs channel samples, Table 4.<sup>13</sup>

Seventy-five sample *triplicates* (chip, hand-cut channel and saw-cut channel) were collected from around a 40 m × 20 m stope block (Figure 2). The mineralisation was known to have a moderate variability, containing visible gold up to 1.5 mm in size. The test block was sampled from faces located every 1.5 m along its upper and lower drives and two raises. After cleaning, a reference line was drawn across each face centre and the different types of samples were collected systematically from the bottom up: chip sample, hand-cut and saw-cut channels. The sample delimitation dimensions were estimated and designed to achieve a theoretical sample support of 3 kg m<sup>-1</sup>. All samples were subsequently prepared and assayed in identical fashion, via a total sample preparation and screen fire assay route. The FSE for this highly optimised protocol, was effectively zero. A QA/QC programme was applied, with all CRMs and blanks within expectation.

These results show a marked reduction in RSV and nugget effect between the three sample sets. The rigorous

laboratory protocol and QA/QC indicate that errors within the laboratory were at a minimum. Therefore, the remaining variability relates to the *in situ* nugget effect and sample collection. The dominant error for the channel samples relates to the *in situ* nugget effect, given that sampling error was minimal. The dominant difference between the chip and saw-cut channel samples relates to sampling error. These results corroborate many previous findings, showing that saw-cut channel samples provide the best sample quality. Most importantly, this experiment substantiates the critical role of *empirical* total sampling/preparation/analysis error quantification.

## Stage-wise error evaluation

More detailed error evaluations can be undertaken including each key stage along the sampling value chain. Thus Table 5 shows the results of such an analysis for two contrasting Gold ore types (termed mesothermal and epithermal). In both cases the highest stage error again turned out to be the field RSV, at 42% and 34% respectively.

For the epithermal system (no coarse gold), all stage errors were found to be

**Table 3.** Distribution of errors across stages of a gold sampling programme.

Error	Stage	Sample type/activity	TOS errors	Duplicate component error range <sup>a</sup>	FSE <sup>b</sup>	Other TOS errors <sup>c</sup>
Sampling	Collect and transport	<i>In situ</i> sampling (e.g. core and linear samples)	INE, IDE, IEE, IWE, IPE	±20–70%	±16%	INE
		Broken rock sampling (e.g. core and RC samples etc.)	FSE, GSE, IDE, IEE, IWE, IPE			
Preparation	Preparation	Drying	IPE	±5–20%	±11%	±23%
		Crushing/grinding	IPE			
		Splitting	FSE, GSE, IDE, IEE, IPE			
Analytical	Assay	Splitting	FSE, GSE, IDE, IEE, IPE	±1–15%	±8%	
		Analysis	TAE			
Total error				±20–70%	±21%	±23%
Target error (fine-gold)/coarse-gold				(±20%) ±40%		±32%

<sup>a</sup>Potential component error range as determined from duplicate sample (pair) analysis;<sup>11</sup> <sup>b</sup>Maximum recommended FSE distribution across the sampling stages;<sup>11</sup> <sup>c</sup>Maximum recommended *other* TOS error proportions across the sampling stages;<sup>12</sup> RC: Reverse Circulation.

# Introduction to the Theory and Practice of Sampling

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with contributions from Claas Wagner, Pentti Minkkinen, Claudia Paoletti, Karin Engström, Martin Lischka and Jørgen Riis Pedersen

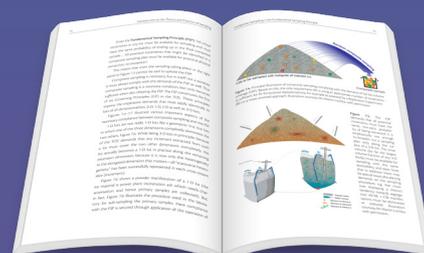
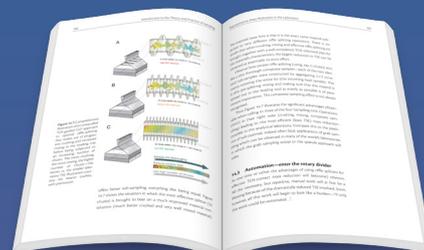
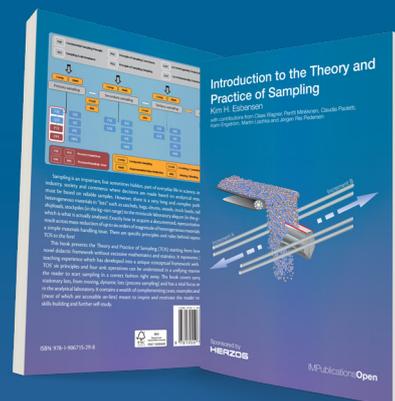
“Sampling is not gambling”. Analytical results forming the basis for decision making in science, technology, industry and society must be relevant, valid and reliable. However, analytical results cannot be detached from the specific conditions under which they originated. Sampling comes to the fore as a critical success factor before analysis, which should only be made on documented representative samples. There is a complex and challenging pathway from heterogeneous materials in “lots” such as satchels, bags, drums, vessels, truck loads, railroad cars, shiploads, stockpiles (in the kg–ton range) to the miniscule laboratory aliquot (in the g– $\mu\text{g}$  range), which is what is actually analysed.

This book presents the Theory and Practice of Sampling (TOS) starting from level zero in a novel didactic framework without excessive mathematics and statistics. The book covers sampling from stationary lots, from moving, dynamic lots (process sampling) and has a vital focus on sampling in the analytical laboratory.

**“I recommend this book to all newcomers to TOS”**

**“This book may well end up being the standard introduction sourcebook for representative sampling.”**

**“One of the book’s major advantages is the lavish use of carefully designed didactic diagrams”**



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**Table 4.** Empirical example: comparison between chip and channel sample replicates. Mean grades cut and diluted to stope width. Reconciled stope head grade 13.7g/t Au.

Type	Mean support (kg m <sup>-1</sup> ) [range]	Target support (%) [within ±5 %]	Mean grade (g/t Au)	Percent of mined grade (%)	RSV (%)	Nugget effect (%)	Sample collection errors <sup>a</sup>	Preparation and assay errors <sup>b</sup>
Chip-channel	2.1 [1.4–3.4]	22	21.3	+55	198	68	High: IDE, IEE and IWE	Low: entire sample crushed and pulverised prior to total sample screen fire assay with triplicate fine-fraction fire assay. All equipment cleaned between samples.
Channel (hand cut)	2.6 [2.1–3.3]	63	16.7	+22	135	54	Low-moderate: IDE, IEE and IWE	
Channel (saw-cut)	2.8 [2.3–3.1]	77	16.3	+19	81	41	Low: IDE, IEE and IWE	

<sup>a</sup>Indicative sample collection error definition: Red: high (>±50%); Orange: moderate (±20–50%); Green: low (<±20%). <sup>b</sup>Indicative preparation and assay error: Red: high (>±20%); Orange: moderate (±10–20%); Green: low (<±10%).

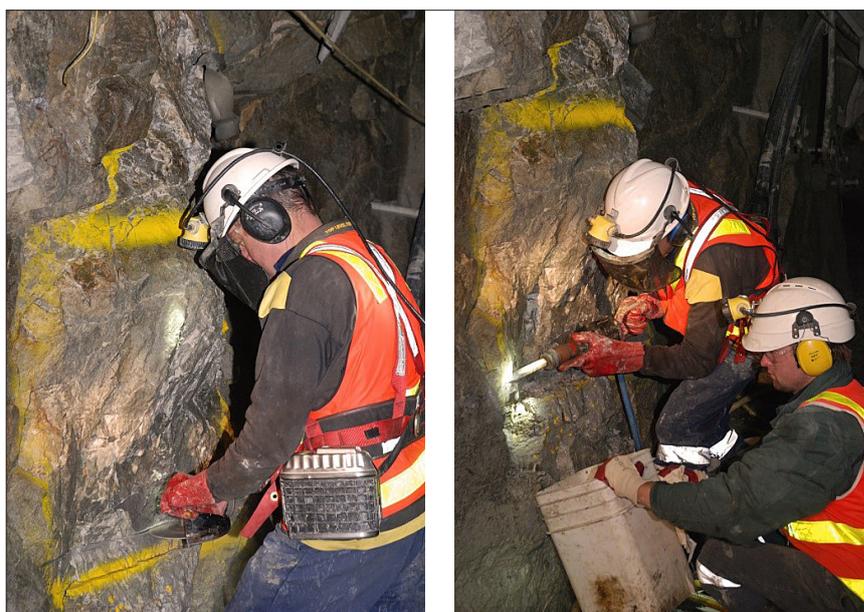
reasonable and did not require further action (Figure 3).

For the mesothermal system (coarse gold, i.e. “nugget” gold) both the field and analytical RSVs were deemed *high*. In order to improve on this situation, the field RSV was attempted to be reduced by taking a *larger* split at the rig (up from 2kg to 4kg) and assaying the entire 4kg by a more precise analytical method (LeachWELL). Based on initial duplicates from the revised protocol, the field RSV was now reduced to 36% and the analytical RSV 4%, now acceptable for a coarse gold mineralisation.

There is a need, and a clear advantage, in moving towards full quantification of errors for objective QC assessment, where a first step is the application of the RSV sampling + analysis variability characteristic as defined in DS3077.<sup>3,14</sup> Resolution of individual relative errors across the complete sampling, preparation and analysis stages can be gained from simple duplicate sample pairs, as evidenced by Table 5.

## Gold—always special

For Gold resource estimation, special issues are about, compared to many other materials and commodities. Thus deliberately strenuous practical measures are recommended to reduce the risk of *tampering* of samples. These could include: maintaining increased security between the sample site (e.g. mine face and drill rig) and sample transport and



**Figure 2.** Underground sampling. Collection of optimal saw-cut channel sample. Left: cutting channel “delimitation” slots; right: sampling (“extraction”) of the channel material.



**Figure 3.** Left: logging and marking diamond drilling cores for sampling (at former Castlemaine Goldfields Ltd Wattle Gully project in Victoria, Australia). Right: sample preparation: diamond drilling (DD) core ready for cutting.

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**Table 5.** Stage-wise error estimation for two contrasting Gold ore types (RC = Reverse Circulation drilling; DD = Diamond Drilling).

RC mesothermal deposit with minor coarse Gold			
	Field RSV <sup>a</sup>	Crush RSV	Analytical RSV
Duplicate RSV	47%	20%	13%
Stage RSV	42%	15%	13%
Relative proportion	83%	10%	7%
DD epithermal deposit with no coarse Gold			
	Field RSV <sup>b</sup>	Crush RSV	Analytical RSV
Duplicate RSV	35%	7%	2%
Stage RSV	34%	7%	2%
Relative proportion	95%	4%	1%

All figures rounded to the nearest whole %. <sup>a</sup>Rig duplicate; <sup>b</sup>Core half duplicate.

careful recording of who has access to samples between collection and shipping, and maintaining a copy of that record.

## Conclusions

Geologists and analytical chemists must acknowledge the systematic *rigour* of the TOS framework and should readily be able to appreciate the help from proper management of all associated errors.

Empirical error estimations of all stages involved in the complete “from-lot-to-aliquot” pathway demonstrated above and the value of the critical information gained has been laid out in no uncertain way. Where samples are analysed to support any resource estimate, a QA/QC programme must be introduced to ensure continuous quality information of both sampling and assaying. Written protocols and procedures, staff training, periodic auditing of protocols and people, and re-training are all required. DS3077<sup>14</sup> provides a framework on how to produce transparent protocols regarding the specific sampling pathway. There are many QA/QC frameworks that can be

applied—more on this latter issue in later Sampling Columns.

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