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Towards Representative Metallurgical Sampling and Gold Recovery Testwork Programmes

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Abstract: When developing a process flowsheet, the risks in achieving positive financial outcomes are minimised by ensuring representative metallurgical samples and high quality testwork. The quality and type of samples used are as important as the testwork itself. The key characteristic required of any set of samples is that they represent a given domain and quantify its variability. There are those who think that stating a sample(s) is representative makes it representative without justification. There is a need to consider both (1) in-situ and (2) testwork sub-sample representativity. Early ore/waste characterisation and domain definition are required, so that sampling and testwork protocols can be designed to suit the style of mineralisation in question. The Theory of Sampling (TOS) provides an insight into the causes and magnitude of errors that may occur during the sampling of particulate materials (e.g., broken rock) and is wholly applicable to metallurgical sampling. Quality assurance/quality control (QAQC) is critical throughout all programmes. Metallurgical sampling and testwork should be fully integrated into geometallurgical studies. Traditional metallurgical testwork is critical for plant design and is an inherent part of geometallurgy. In a geometallurgical study, multiple spatially distributed small-scale tests are used as proxies for process parameters. These will be validated against traditional testwork results. This paper focusses on sampling and testwork for gold recovery determination. It aims to provide the reader with the background to move towards the design, implementation and reporting of representative and fit-for-purpose sampling and testwork programmes. While the paper does not intend to provide a definitive commentary, it critically assesses the hard-rock sampling methods used and their optimal collection and preparation. The need for representative sampling and quality testwork to avoid financial and intangible losses is emphasised.

Keywords: metallurgical sampling; metallurgical testwork; geometallurgy; gold mineralisation; representative sampling; Theory of Sampling; quality assurance/quality control

1. Introduction

1.1. Sampling along the Mine Value Chain

Sampling is a vital component during all stages of the Mine Value Chain (MVC). It includes the sampling of in-situ material and broken rock for geological, metallurgical (including plant balances), geometallurgical and geoenvironmental purposes [1–5].

Sampling error is defined in the context of TOS, where actions may lead to uncertainty and create an overall measurement error [6,7]. TOS attempts to break down this error into a series of contributions along the sampling value chain (e.g., the planning to assay-measurement process: Table 1).

Table 1. Sampling value chain. Sampling errors are defined in Table 3.

Location	Site/Field			Laboratory		
Stage	Planning	Collection	Transport	Preparation	Testwork	Assaying
	1	2	3	4	5	6
Activity	Scope Develop Execute	Observe Collect Bag and tag QAQC Integrity/security Chain of custody	Integrity/security Chain of custody	Equipment operation Equipment clean QAQC Integrity/security	Equipment operation Equipment clean QAQC Integrity/security	Equipment operation Equipment clean QAQC Integrity/security
Sampling errors		In-situ nugget effect FSE, GSE DE, EE, WE	PE	FSE, GSE DE, EE, WE, PE	FSE, GSE DE, EE, WE, PE	PE AE
Dominant effect on results		Precision Bias	Bias	Precision (if splitting) Bias	Precision (if splitting) Bias	Bias
Material risk assuming average practice		High	Low	Moderate	Moderate	Low
Material risk assuming optimised practice		Moderate	Low	Low	Low	Low

FSE: fundamental sampling error; GSE: grouping and segregation error; DE: delimitation error; EE: extraction error; PE: preparation error; WE: weighting error; AE: analytical error.

Errors are additive throughout the sampling value chain and generate both monetary and intangible losses [8–10]. The aim is to collect representative samples to accurately describe the material in question. Sample collection is followed by reduction in both mass and fragment size to provide a sub-sample for testwork or assay. An assay is the quantitative measurement of the concentration (e.g., mass fraction such as g/t gold) of a metal by a given methodology, for example a 30 g fire assay followed by measurement of gold using an instrumental method (e.g., atomic absorption spectroscopy). This entire process can be particularly challenging in the gold environment and may require special protocols [11–14].

1.2. Metallurgical Sampling and Testwork

Metallurgical testwork is the laboratory-based bench-scale physical (e.g., gravity) or hydrometallurgical (e.g., cyanide leach) extraction of gold (or other metal/mineral) via a defined methodology [15–19]. Large-scale testwork includes pilot plant and ultimately demonstration or trial plant processing. The results from metallurgical testwork are used to support design studies for new plants and expansions, in short- to long-term mine plans, and to make decisions in an operating plant [20]. Key testwork outcomes relate to the definition of: recovery and comminution domains, domain variability and identification of problem ore types (e.g., deleterious elements and refractory ores).

After collection, sampling errors propagate throughout all subsequent processes contributing to uncertainty in testwork (e.g., measurements) and any decisions made thereon [21]. Across the MVC, these errors generate financial and intangible losses. In essence, poor-quality unrepresentative metallurgical samples increase project risk [22] and may lead to incorrect project valuation [23]. McCarthy [9] reports that metallurgical sampling and testwork issues are responsible for 15% of plant commissioning and operational under-performance. The consequences of poor metallurgical samples can be significant and relate to: incorrect recovery factors applied to Ore Reserve estimates, poor project development decisions, incorrectly designed process plants, poor mine-to-mill reconciliation, halted projects and/or reduced mine life and incorrect financial models and project valuations.

The difference between geological and metallurgical sampling programmes, and the definition of a representative sample, is illustrated by the number of samples collected. It is not unusual that >5000 samples are collected for resource estimation, but only 25–50 samples collected for metallurgical testing on the same deposit. The ratio of reserve definition samples to metallurgical samples often remains much the same for complex or heterogeneous mineralisation, despite a shift in risk from the reserve head grade to the characteristics that control plant performance (e.g., recovery). In reality, hundreds of metallurgical samples are likely to be required depending on mineralisation complexity and size. The modern geometallurgical approach aims to model variability based on correlating metallurgical testwork with rapid small-scale tests and by calibrating metallurgical properties with other features (e.g., proxies) that can be realised from resource drilling [11,24,25]. In the context of geometallurgical programmes, metallurgical sampling and testwork are a critical input.

1.3. Current Practice and Focus of This Contribution

Current mining industry practice frequently fails to place proper consideration on representative metallurgical sampling and quality testwork programmes to produce fit for purpose results. Despite its general acceptance for resource grade sampling, TOS is rarely applied during metallurgical programmes. In addition, few metallurgical campaigns apply QAQC. These deficiencies are in deference to the requirements of international reporting codes, where for example both the Australian JORC Code [26] and Canadian NI43-101 [27] require specific comment on the representativity of metallurgical samples and applicability of testwork.

This paper focusses on sampling and testwork (Stages 1 to 5 of the sampling value chain: Table 1) for gold metallurgical recovery determination (e.g., Leach, GRG and flotation methods). It should be noted that metallurgical testwork also includes comminution and geoenvironmental parameters, though these are not specifically discussed in this contribution. Whilst it focusses on gold, the conclusions are applicable to other commodities. It aims to provide the reader with the background to move towards improved design, implementation and reporting of representative and fit for purpose metallurgical testwork programmes. The paper does not intend to provide a definitive commentary, but rather be a first foray for improvement. It critically assesses the sampling methods used, and their optimal collection and preparation through the application of TOS. A case study illustrates the need for better designed sampling and testwork to avoid substantial financial and intangible losses. It demonstrates many of the issues discussed throughout this contribution.

2. Mineralisation Characteristics

2.1. Geological Characteristics

The physical and spatial characteristics of gold mineralisation have a strong influence on any sampling programme. Mineralisation may comprise (i) an individual vein up to 5 m wide or (ii) numerous individual veins forming a larger composite lode up to 20 m wide. Additionally, mineralised systems may comprise large complex structures ranging from 20 to 100 m in width such as igneous intrusives, skarns, alteration/replacement zones, and networks of cross-cutting (e.g., stockworks) or sub-parallel (e.g., sheeted vein systems) veins.

The nature and style of mineralisation and gangue material should be considered to determine their impact on recovery. Key characteristics that influence sampling include, but are not limited to: mineral species and composition, grain size and morphology, texture and mineral associations. The sampling process can be particularly challenging in the presence of coarse gold particles, which are considered to be those >100 µm in size. Where it constitutes more than 10% of all gold present, problems are likely to be encountered during sampling, testwork and assaying.

There is evidence to suggest that relationships exist between increased gold grade and larger, potentially clustered gold particles [28]. In some cases, fine- and coarse-gold particles may be part of overprinted paragenetic stages [29]. Fine gold particles are more likely to be disseminated throughout the mineralisation and responsible for a "background" grade, whereas coarse particles may be more clustered and related to high-grade zones that are difficult to sample effectively [28,29].

2.2. Metallurgical Characteristics

Gold ores are commonly classified as either free-milling or refractory [30]. Free-milling ores are defined as those where over 90% of gold can be recovered by conventional cyanide leaching. Refractory ores are those that give a low recovery from conventional cyanide leaching. Mineralogy has a significant impact on gold recovery, where the key factors are (Table 2): mineralogy (including particle size and deportment), geochemistry (e.g., carbon, silver and copper content, and surface chemistry), grade (grade-recovery relationships) and texture (e.g., liberation).

Table 2. General recovery methods based on gold liberation size and host mineralogy.

<table>
<tr><th>Liberation Size</th><th>Quartz</th><th>Pyrite</th><th>Arsenopyrite</th><th>High Silver</th></tr>
<tr><td>Very coarse: >1000 µm</td><td>Jigs, tables</td><td>Jigs, tables</td><td>Jigs, tables</td><td>Jigs, tables</td></tr>
<tr><td>Coarse: 100–1000 µm</td><td>Gravity, CIL/CIP</td><td>Gravity, CIL/CIP</td><td>Gravity, CIL/CIP</td><td rowspan="2">Gravity, CIL
Merrill Crowe
Flotation</td></tr>
<tr><td>Fine: 50–100 µm</td><td>CIL/CIP</td><td>CIL/CIP, Flotation</td><td>CIL/CIP, Flotation</td></tr>
<tr><td>Very fine: 10–50 µm</td><td>CIL/CIP</td><td>Flotation</td><td>Flotation</td><td>-</td></tr>
<tr><td>Sub-microscopic: <10 µm</td><td>-</td><td>POX, BIOX</td><td>POX, BIOX</td><td>-</td></tr>
</table>

CIL: carbon-in-leach; CIP: carbon-in-pulp; POX: pressure oxidation; BIOX: bio-oxidation; Gravity: gravity recoverable gold (GRG) is a recovery parameter based on liberated gold or high-grade composite gold particles that can be recovered by physical separation methods.

In addition, the proportion of sub-microscopic (<10 µm) gold is important. Sub-microscopic gold refers to gold contained in the structure of other minerals in a minor or trace quantity. Gold particle size issues during metallurgical processes relate to liberated gold after grinding. Generally, gold ores are milled to 80% passing (P_{80}) 75 µm and 300 µm. As such the true in-situ particle size does not control metallurgical recovery per se, though it has a marked influence given that coarser gold particles will require more grinding to reduce particle size. For coarse-gold dominated mineralisation, the focus should be on liberation and the early removal of coarse particles from the mill circuit by gravity concentration or gold traps. Finer gold particles will require more grinding to liberate prior to flotation or cyanide extraction.

3. Theory of Sampling

3.1. Overview

TOS was developed in the 1950s by Dr. Pierre Gy to improve sampling within the mining industry [6]. It defines and provides guidelines for the reduction of sampling errors throughout the MVC [6,10], though its application to metallurgical sampling has been minimal [31]. Some resistance to its application relates to the use of the Fundamental Sampling Error (FSE) equation and confusion as to its calibration and application [6,10]. TOS has a wider usage than simply the FSE equation and includes a number of errors that must be considered to achieve representative samples (Table 3). It is often forgotten that the application of TOS includes the mandatory use of QAQC.

3.2. Nugget Effect

The heterogeneity (variability) of mineralisation can be quantified by the nugget effect and has a direct link to TOS [32–34]. The nugget effect is a quantitative geostatistical term describing the inherent variability between samples at very small separation distances; though in reality has a wider remit than just differences between contiguous samples [13,34,35]. It is effectively a random component of variability that is superimposed on the regionalised variable, and is defined in a variogram as the percentage ratio of nugget variance to total variance (the sill). Deposits that possess a nugget effect above 50% and particularly above 75% are the most challenging to evaluate. The magnitude of the total nugget effect relates to:

- Geological (geological or in-situ nugget effect: GNE) heterogeneity of the mineralisation.
    - Distribution of single grains or clusters of gold or sulphide-hosting gold particles distributed through the ore to larger continuous zones.
    - Continuity of structures such as high-grade gold carriers within the main structure or vein-lets within wall rocks.
- Sampling induced error variability (sampling nugget effect: SNE).
    - Sample support (sample size—volume-variance).
    - Sample density (number of samples at a given spacing—information effect).
    - Sample collection, preparation, testwork and assay procedures.

A clear indication of the GNE is where two halves of a drill core (e.g., on the cm-scale) are assayed and show order of magnitude or more difference in assay grades, or where two closely spaced face samples for example also show an order of magnitude or more difference. A high GNE leads to high data variability, particularly where samples are too small and protocols not optimised. The presence of visible gold in mineralisation is often an early sign of grade variability and a flag for variability in metallurgical recovery testwork results.

The SNE component is related to errors induced by inadequate sample size, sample collection, preparation methods and analytical procedures. In some instances, the SNE is the dominant part of the total nugget effect and reflects non-optimal protocols. Throughout the MVC, optimised sampling protocols aim to reduce the SNE thereby also reducing: total nugget variance, skewness of the data distribution, and number of extreme data values. All sampling errors contribute to the SNE.

3.3. Sampling Errors

TOS provides an insight into the causes and magnitude of errors that may occur during the sampling of particulate materials (e.g., broken rock). It does not strictly include sampling of in-situ material such as drill core or linear samples, as the sample does not have an equiprobable chance of being collected. However, its application is relevant and highlights some of the challenges of field sampling through analysis of the so-called incorrect sampling errors (ISE; Table 3) during the sample collection process [36–39].

The correct sampling errors are considered unavoidable because they cannot be removed by perfect sampling (CSE; Table 3). They relate to the inherent heterogeneity of the material being sampled and control precision. Precision specifically relates to the constitution heterogeneity of the material in question and leads to the fundamental sampling error (FSE). Poor precision in samples generates ore/waste misclassification. The FSE can be estimated via the FSE equation [6,10]; it is controlled via the optimisation of sample mass and size reduction process (Table 4).

In practice, the grouping and segregation error (GSE) cannot be measured but may have a material effect on the total sample error. It is controlled by accumulating many small increments to form a composite sample. Although segregation can theoretically be reduced by homogenisation, this is a futile exercise in the presence of liberated gold particles where it promotes further segregation.

Table 3. Definition of key TOS 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 after a sampling operation is perfect. Experience shows that the total nugget effect can 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 any scale
Delimitation	DE	Incorrect Sampling Error (ISE)	Systematic Errors-Bias Generator	Sampling equipment and materials handling	Results from an incorrect shape of the volume delimiting a sample
Extraction	EE				Results from the incorrect extraction of a sample. Extraction is only correct when all fragments within the delimited volume are taken into the sample
Weighting	WE				Relates to collecting samples that are not of a comparable support. Samples should represent a consistent mass per unit
Preparation	PE				Refers to issues during sample transport and storage (e.g., mix-up, damage, etc.), preparation (e.g., contamination and/or losses), and intentional (e.g., sabotage and salting) and unintentional (e.g., careless work practices) actions
Analytical	AE	-		Analytical process	Relates to errors during the assay and analytical process, including issues related to rock matrix effects, careless work practices, and analytical machine maintenance and calibration. In the testwork context also refers to test machine operation parameters and maintenance

Table 4. Cause, effect and solutions to TOS errors.

Sample Type	Error Type	Cause	Error	Effect	Nugget Effect Component	Solution
In-situ (e.g., linear samples and core)	Random (CSE)	In-situ heterogeneity (nugget effect)	Local representativity	Poor precision	Geological	Larger samples More samples
Broken rock (from rock chips to blasted material and laboratory pulps)		Constitution Heterogeneity	FSE	Poor precision	Sampling	Optimised protocols Larger samples More samples
		Constitution and Distribution Heterogeneity	GSE	Poor precision	Sampling	Optimised protocols More sample increments
	Systematic (ISE)	Poor quality sampling protocols—sampling errors	DE, EE, WE, PE and AE	High bias	Sampling	Optimised protocols Efficient training Strict QAQC

Preparation errors (PE) in the sampling context are non-selective operations without change of mass, such as crushing, grinding, mixing, sample transfer, drying, etc. These issues are often ignored during metallurgical sample preparation and testwork, though their effect can be marked. Typical errors include sample contamination, sample loss (e.g., due to sample spillage), moisture loss, and operator mistakes, such as mixing up sample labels. These errors can be eliminated using correct sampling equipment and practices. For example, care also needs to be taken to ensure that the finer particles are not lost in crushers or mills or during sample division due to excessive air flow from dust extraction systems installed in sample preparation laboratories.

Table 4 shows the cause and effect of the sampling errors and provides some solutions. The ISE arise as a consequence of the physical interaction between the material being sampled and the technology employed to extract the sample. They result in bias, which can be reduced by the correct application of sampling methods, equipment and procedures.

3.4. Fundamental Sampling Error (FSE)

3.4.1. Background

The FSE results from grade heterogeneity of the broken lot [6,10]. Of all the sampling errors, the FSE does not cancel out and remains even after a sampling operation is perfect. Experience shows that the total nugget effect can be high because sample masses are not optimal (e.g., the FSE is too high). The FSE can be estimated before material is sampled, provided certain characteristics are determined [6,10]. The FSE equation addresses key questions in respect of broken rock sample mass and degree of crushing and grinding required.

3.4.2. Calibration of FSE Equation Inputs

The key parameter that requires determination is the gold liberation diameter—a particle size parameter [6,10,40]. For gold mineralisation, it can be defined as the screen size that allows 95% of gold given a theoretical liberated lot to pass (d_ℓ or d_{95Au}—[34,40]. If gold particles cluster, then d_{95Au} should be redefined as the cluster diameter—d_{AuClus} [28,34]. Approaches to d_{95Au} determination range from guesswork to the implementation of Heterogeneity Tests (HT) or Duplicate Series Analysis (DSA). The results of both HT and DSA can be used to calibrate the FSE equation—effectively defining d_{95Au} through estimating the sampling constant K [6,10,41].

The value of K is dependent on the microscopic geostatistical properties of the minerals, and varies with gold grade and liberation diameter. The higher the K value, the more challenging an ore type is to sample effectively. K values between 1000 and 5000 indicate some major sampling challenges that are likely to require specialised protocols. Values >5000 indicate the need for specialised protocols and potentially bulk sampling.

The HT is most commonly applied calibration method in the mining industry. The DSA approach is both complex and time consuming to apply, so relatively rare compared to the HT. The HT is prone to severe precision problems, particularly when coarse gold is present [12]. A further problem is that it provides values for K only at the fragment size at which the calibration exercise is carried out (often 10 mm). It has been shown that in such cases the values for d_ℓ are far too low. In the presence of coarse gold, the HT approach may require samples of hundreds of kilogrammes in size. The method may not be a standard industry approach, since it is likely only to provide a correct value when mineralisation is disseminated.

The authors recommend a holistic approach to characterisation which provides a range of outputs including [42–44]:

- Realisation of gold deportment, in particular the partitioning of gold as free gold, gold in sulphides and refractory gold.
- Gold particle size curve(s), including effects of clustering and relationship between gold particle size and grade (e.g., high grade versus background grade).

- Definition of key FSE equation inputs (e.g., d_{95Au} versus d_{AuClus}) and the sampling constant K.
- Recommendations as to optimum in-situ sample mass requirements.

A direct approach to d_{95Au} is recommended, which can include a combination of particle size determination via detailed core logging and underground/surface rock observation, optical and automated microscopy, X-ray tomography, and crush-screen-concentration [1,42,44–46]. Characterisation studies allow the practitioner to set the sampling expectations across a number of d_{95Au}-grade scenarios. Where this requires specialist and potentially costly protocols, then it will be possible to determine the level of risk involved in using more practical methods. In essence, a gap analysis between the theoretical need and practical reality of sampling can be undertaken.

3.4.3. Applicability to Gold Ores

Some authors question the applicability of the FSE equation applied to gold; as with any model-based approach ultimate validation with reality is required. Controlling the FSE during metallurgical sampling and testwork is critical, since any sample splitting/size process will result in the generation of an FSE. François-Bongarçon and Gy addressed key issues and proposed a modified equation [40]. The modified equation has been applied successfully by practitioners to optimise sampling campaigns. The use of the FSE equation represents an idealised expectation that may or may not be attained in practice, but it provides a starting point from which protocols can be optimised. The standard approach optimising the sampling of gold ores is to apply the FSE equation to gold grade. If there is a strong correlation between gold and sulphides (e.g., pyrite), then the pyrite FSE can be modelled. Sulphides will require sulphur assays, potentially plus base metals depending upon mineralogy, to estimate their abundance.

3.5. Sample Representativity

3.5.1. Representative Sampling

A sample is representative when the analytical or testwork results are within acceptable levels of bias and precision. For a primary sample to be representative, it should provide a realistic estimate of the parameter in question (e.g., grade, GRG, etc.). Precision (reproducibility) can be determined from duplicate samples and resolved into the magnitude of sampling, preparation and analytical error components [14,47,48]. Sampler bias can be accessed via sampling proficiency testing [49] and analytical bias can be determined from certified reference material results analysis [50,51].

In order for a sample to be deemed representative of the population from which it was extracted, it must contain similar relative proportions of all original constituent elements present in the population. In the case of gold, this implies that the sample should contain an identical grade to the original material, not only overall grade, but also on a size-by-size basis. Given gold's relatively low abundance in most mineralisation, this can be a difficult criterion to meet [1,34,52].

The 2013 Danish Horizontal Standard (DS3077) uses the relative sampling variability (RSV) metric to measure total sampling variability: the percentage coefficient of variation for repeat sample values [53]. The higher the RSV, the poorer the precision indicating that the sampling procedure requires improvement. RSV measures the total empirical sampling variance influenced by the heterogeneity of the lot sampled under the current sampling procedure [53]. The RSV comprises all stages of the sampling protocol and includes errors that are incurred by mass reduction as well as the total analytical error. DS3077 applies the RSV at the 68% reliability, though it can be calculated at the 90% and 95% reliabilities if required [53].

The accepted value of RSV is up to the practitioner and based upon the nature of the mineralisation in question, the data quality objectives and what is cost-effective and practical. The RSV for a given sample support can be reduced through TOS and QAQC application [54]. In reality, the truly representative field sample probably does not exist, unless a bulk sample is collected and processed. Sub-samples taken from the original field sample should be representative and can be optimised using the FSE equation [6,10]. Assays (measurements) undertaken in the laboratory to support testwork can also be validated [13,50,55].

3.5.2. Fit for Purpose Samples

A sampling, testwork and assaying programme must produce data that are fit-for-purpose. In this context, fit-for-purpose refers to the production of data that enables the practitioner to make technically correct decisions [56]. In the mining context, results must be able to contribute to a Mineral Resource and/or Ore Reserve and can be reported in accordance with The 2012 JORC Code [26] or other codes.

Development of sampling protocols in the context of TOS must be based on the specific mineralisation. This will need to consider sample density and number of samples, where for example, clustered samples or samples located from solely high-grade zones are unlikely to be fit-for-purpose for plant design. Sampling and sub-sampling should result in representative samples. A critical input is that of QAQC to maintain data quality through documented procedures, sample security, and monitoring of precision and bias. If a batch of samples is deemed to be representative and assaying complies with QA documentation and QC metrics, then it is fit-for-purpose.

3.5.3. Data Quality Objectives

The data quality objective (DQO) is the level of total error that the sampling value chain is designed to achieve (Table 1). It is quoted as a precision value reported at a given confidence limit, usually the 68%, 90% and 95% limits. The confidence limits can be defined as the "reliability", where for example 90% means that the testwork results will lie within the give precision 90 out of 100 times [57].

Pitard [34,58] states that the total relative error for resource and grade control sampling should not be more than ±32%, with the FSE component not more than ±16%. In the mining context, FSE is generally reported at the 68% reliability. It is up to the practitioner as to which relative error and reliability to accept, but for metallurgical samples to achieve a minimum FSE of ±15% at 68% is optimal. If mineralisation is highly heterogeneous, then ±20% at 68% may be a more practical target. For validation work (e.g., bulk sample or trial mining programmes), then a reliability of 68% or 90% and relative error ±5–15% can be applied.

All sampling errors are cumulative and contribute to the total, which in turn contributes to the sampling nugget effect. In reality, the FSE and GSE may contribute up to 90%, with DE, EE and AE up to 25% of the total [10]. Analytical (measurement) error generally accounts for between 1% and 13% of the relative error [48]. It is generally recognised that the total sampling value chain error is dominated by the sampling process, which can be in the range of 15% to 60% [37,48,51].

3.5.4. Approaches to Sampling Optimisation

Early in the MVC (Table 5; Exploration-early evaluation) when minimal information is available on the ore type(s) present, the application of the model-based FSE equation to evaluate sampling protocols is required [6,10]. As a project develops, then duplicate samples permit an empirical approach to the estimation of uncertainty through the analysis of field, coarse and laboratory duplicates [21,47,48,59,60].

In practical terms, duplicate sampling during metallurgical programmes is rarely undertaken and often difficult at the field stage. Many metallurgical samples are composites from remaining half-core, therefore field duplicates are impossible. Coarse duplicates after crushing may be available, though in many cases the paucity of material available for testwork may preclude their use. Laboratory test duplicates are the most likely to be available, particularly during assay of concentrates and tails samples. Duplicates are an important part of the QAQC process (see Section 9) and can be planned for in a dedicated metallurgical programme.

Table 5. General MVC showing broad metallurgical-geometallurgical activities, inputs and outputs (after [11]).

Stage	Strategic Geometallurgy				Tactical Geometallurgy
	Exploration-Early Evaluation	**Resource Definition Drilling**	**Reserve Definition Drilling**	**Feasibility**	**Mining**
Study	Scoping (SS)	Pre-feasibility (PFS)	-	Feasibility (FS)	(Grade/ore control) (Expansion studies)
Resources/Reserves	Inferred Mineral Resources	Inferred and Indicated Mineral Resources	Mineral Resources and Ore Reserves	Mineral Resources and Ore Reserves	Mineral Resources and Ore Reserves
Key activity	Develop orebody knowledge; Drilling and sampling	Develop orebody knowledge; Drilling and sampling; Data analysis and modelling	Develop orebody knowledge; Drilling and sampling; Data analysis and modelling	Develop orebody knowledge; Drilling and sampling; Data analysis and modelling	Develop orebody knowledge; Drilling and sampling; Data analysis and modelling
Inputs	Core logging; Develop proxy tests; Mineralogy; Geochemistry; Met. testwork; Physical testing	Core logging; Proxy tests; Mineralogy; Geochemistry; Met. testwork; Physical testing	Core logging; Proxy tests; Mineralogy; Geochemistry; Met. testwork; Physical testing	Core logging; Proxy tests; Mineralogy; Geochemistry; Met. testwork, incl. pilot or trial plant testing; Physical testing	Core logging; Proxy tests; Mineralogy; Geochemistry; Met. testwork; Physical testing
Outputs	Establish database Prelim.; characteristics of mineralisation; Geological model; Geoenvironmental	Expanded database; Geomet. domains; Block model; Prelim. mine plan; Geomet. models; Prelim. process design; Geoenvironmental	Expanded database	Expanded database; Geomet. domains; Block model; Mine plan; Geomet. models; Flow sheet; Scenario analysis; Economic analysis	Expanded database; Geomet. domains; Block model; Mine plan; Geomet. models; Forecasts; Reconciliation

4. Geometallurgy

A critical development over the past ten years is that of geometallurgy, essentially a life-of-mine optimisation process [11,25]. Geometallurgy seeks to resolve grade, metallurgical and mining variability based on information such as geochemistry, mineralogy, grade and lithology obtained from spatially distributed samples or measurement points. Multiple spatially distributed small-scale tests are used as proxies for grade, mineralogy, process parameter and rock mass variability.

Geometallurgy can be conveniently sub-divided into two approaches: strategic and tactical [25]. Strategic and tactical geometallurgy programmes can be described in terms of a logical process flow that begins with planning and, importantly, ends with reconciliation to the plan. Key stages in the process flow are: planning, timeframe, team, drilling, testwork, data management, modelling, and in the case of tactical geometallurgy, mining and processing [25]. The two clearly overlap, though differ in timeframe. This two-fold approach to geometallurgy forms a systematic process that needs to be planned and implemented in an integrated manner. Both strategic and tactical geometallurgy can be worked on by the same professionals, who can share data, methodologies and potentially models [25].

The pre-geometallurgical approach focussed on plant design through the testing of a number of composite samples that are reported to be "representative" of the ore body [61]. Testwork is carried out to determine factors such as grindability, floatability, leach recovery and/or other parameters. Subsequently, a process plant is constructed and commissioned and at some point, often within the first year of operation, found to be not performing to design. The common reason for this relates to insufficient and unrepresentative samples and potentially inappropriate testwork. The traditional approach generally fails to represent the orebody and likely variability within [31,61]. Geometallurgy aims to resolve such variability, but still requires high-quality metallurgical testwork based on representative samples.

Traditional metallurgical sampling and testwork are critical for plant design and are an inherent part of geometallurgy [11,31,62]. In a geometallurgical study, multiple spatially distributed small-scale tests are used as proxies for grade, mineralogy, process parameter, etc. These will generally be validated against traditional testwork results [63]. In the context of geometallurgical programmes, metallurgical sampling and testwork are a critical input. Traditional testwork programmes yield hundreds of results at feasibility level, where a strategic geometallurgical programme will result in thousands of spatially distributed data points [64–66].

5. Project Studies

Three types of project studies are recognised within the mining industry: from a scoping study through pre-feasibility to a feasibility study. These studies describe investigations of increasing detail, which is supported by greater volumes of metallurgical sampling and testwork (Table 5). The level and focus of metallurgical sampling and testwork changes from laboratory-dominated to pilot-scale testwork (Table 6). Scoping testwork serves to support order-of-magnitude assessment. Pre-feasibility testwork should be sufficient to develop project design criteria, performance predictions and product analyses. The feasibility testwork should be confirmatory in nature and should be carried out using samples representative of the final mine plan, with emphasis on the first five years of production.

Table 6. Recommended scale of metallurgical testwork for engineering studies (after [17]).

Type of Testwork	Scoping	Pre-Feasibility	Feasibility
Laboratory/bench scale	Yes	Yes	Maybe
Small/mini-pilot scale	Maybe	Yes	Yes
Pilot scale	No	Maybe	Yes
Demo/trial or full plant	No	Maybe	Maybe

The need for, and type of, pilot scale testwork is a project by project decision. It will be affected by factors such as the nature of the mineralisation (e.g., mineralogical complexity and/or heterogeneity), project size, availability of suitable sample material, process complexity and/or novelty, and requirements of finance providers.

6. Sampling for Metallurgical Testwork

6.1. Introduction

The context of sampling merits attention because metallurgical parameters are a function of geological factors such as grade, lithology, alteration, mineralogy, texture, spatial relationships and specific gravity. It is essential to use this information in order to optimise the sampling strategy [17,31]. Although limited sample sets were previously considered appropriate for metallurgical testwork, there is a realisation that as mineralogy and geology change throughout an orebody/domain, so do metallurgical characteristics. Samples should be both geologically and metallurgically representative. Mineralogical studies must be integrated with metallurgical testwork, as this is crucial in supporting process flow sheet design [67,68].

Metallurgical samples are required at all stages of the MVC, ranging from exploration, scoping, pre-feasibility and feasibility studies, and production (Table 5). The number of samples collected and tested can vary widely across different projects and stages. There is an expected increase in the number of samples at the feasibility stage, plus the use of bulk sampling/trial mining programmes. Based on the review of some 65 technical study reports, the number of samples is still relatively small, ranging from 0.07 to 2.8 samples per 100,000 t of resource. It is interesting to note that studies on relatively small deposits (e.g., <1 Mt) often bear a greater number of samples, ranging from 1.6 to 87 samples per 100,000 t. It should be noted that composite samples are predominantly used, compared to single source variability samples.

Early stage integrated geological and metallurgical sampling and testing are required to check for issues such as refractory ores, deleterious elements, complex textures/mineralogy, etc. Samples should cover different geological domains within the orebody and their full spatial extent. As a project advances, geometallurgical domains will be identified. At the early evaluation stage, at least two samples should be collected from each domain identified and where possible, samples should be collected in different grade zones within each domain. The total number of samples will continue to increase with increasing spatial extent and ore variability, which depends upon the mineralisation type and heterogeneity (nugget effect) within domain(s).

6.2. "Sampling for Sampling"—Characterisation for Sampling Programme Scoping and Development

At an early project stage, it is important to review mineralisation characteristics prior to metallurgical sampling programme design. In an existing project, enough information may be available to make this process relatively simple. Collaboration between geologists, geometallurgists, metallurgists, minerals engineers, mining engineers and environmental geoscientists is critical for effective execution. At this stage and throughout the MVC, there is much information that can be shared between disciplines across grade distribution and ore/waste properties including alteration, mineralogy, texture, geochemistry and physical rock properties. Review of resource grade drilling is most likely to provide a measure of mineralogical and textural variability. The training of geologists to think metallurgy when logging core is critical [69].

In a new or relatively unknown project, a sampling-for-sampling programme is required to evaluate the nature of the mineralisation. This can initially be in the form of simple observation followed by testwork [70]. It is critical, even if visible gold is not observed in exposure or drill core, that a number of samples are collected and tested to check for coarse gold. Low-grade gold ores dominated by fine-coarse gold, particularly if it clusters, can be particularly problematic to sample [71]. Simple observation may not always pick up the presence of or potential impact of coarse gold.

An important part of the sampling for sampling stage is the preliminary definition of geometallurgical recovery domains. This may require specialised tests to determine the presence of refractory gold [19,72] and preg-robbing potential [73]. This will be a long-term on-going process, but an early start in the MVC is advantageous. It is likely that different domains will have different sampling characteristics and may require a unique protocol based on a stratified approach [74,75].

6.3. Metallurgical Sample Mass Requirements

6.3.1. Testwork Mass Requirements

From a metallurgical recovery perspective, sample sizes are principally dictated by the testwork requirements [15,17]. Most fall into the broad categories of physical and hydrometallurgical treatment (Table 7). An individual sample is rarely collected for one test; rather a composite will be subjected to a number of tests, both for recovery and comminution.

Table 7. Typical metallurgical recovery testwork and test mass required for project studies (modified from [17]). X: expected application; (X): potential application.

Type of Test	Indicative Mass	Type of Study		
		Scoping	Pre-Feasibility	Feasibility
Flotation				
Rougher	1–2 kg	X	X	X
Cleaner test (grind-grade recovery)	15 kg	X	X	X
Locked-cycle	15–25 kg	-	X	X
Circuit design (optimisation/variability)	100–500 kg	-	X	X
Mini-pilot plant (Andrade et al., 2005)	200 kg	(X)	(X)	(X)
Pilot plant	>10 t	-	(X)	X
Physical Separation				
Gravity (gravity recoverable gold: GRG)	25–150 kg	X	X	X
Gravity (continuous gravity recovery: CGR)	25–100 kg	X	X	X
Heavy liquid	0.5 kg	X	X	X
Magnetic or electrostatic	50–100 kg	X	X	X
Pilot plant	1–20 t	-	(X)	X
Leaching				
Bottle roll	Up to 5 kg	X	X	X
Diagnostic leaching	1–2 kg	X	(X)	(X)
Batch agitation (CIL/CIP)	2–5 kg	-	(X)	X
Semi-continuous (CIL/CIP)	30–50 kg	-	-	X
Small diameter columns	9 kg	X	X	X
Intermediate diameter columns	80 kg	-	X	X
Large diameter columns	60 t	-	-	X

6.3.2. Theoretical In-Situ Sample Mass Evaluation

The theoretical in-situ sample mass requirement for grade samples can be estimated by knowledge of mineral particle size models. Metallurgical recovery functions such as GRG and leaching are primarily controlled by gold particles which reflect grade. Thus a gold particle driven approach is a reasonable tool for GRG or leaching sample mass estimation [76]. With discrete gold particles, use of a Poisson-based method provides an indication of the sample mass that may be required to yield a given precision [33,34,77,78]. Poisson distributions are suitable for modelling low-grade deposits containing discrete particles with the property of interest. For gold deposits with discrete gold, there is a high probability of drawing non-gold particles. Hence, if a sample is too small, it will contain no gold.

The Poisson model is a limiting case of the Binomial model where the proportion (p) of the rare component (e.g., gold) is very small, while the proportion of the material surrounding the gold (q) is practically unity (where $q = 1 - p$). It can be applied to both liberated and non-liberated (i.e., in-situ) cases, since on the sample scale gold particles are rare events and can be considered as being discrete.

The method assumes a model (the equant grain model—[79]) where: (1) particles are free with no composite particles; (2) particles are either nuggets or gangue minerals; (3) nuggets have the same size, shape and composition; (4) the gold occurs only in the nuggets; (5) a small number of nuggets are present in samples; and (6) a large number of gangue grains are present in the samples. The critical assumptions are (3) and (5) above. Assumption (3) is unlikely to be true since the population will have variable particle sizes and shapes. For Assumption (5), the larger nuggets will be rare, but the fine background gold is likely to be more abundant and disseminated. The Poisson approach defines a maximum theoretical sample mass based on the largest gold nuggets present.

Sample mass is estimated at a given precision and generally reported at the 90% reliability. A precision of ±15–20% is acceptable, though this can be increased or reduced as appropriate. The precision defined at this stage is the target precision of the field sample; it does not include the TOS errors. It is the optimum mass to overcome the in-situ nugget effect (Table 3).

The reliability can be reduced for highly heterogeneous mineralisation. For example, gold mineralisation at a mean grade of 3.5 g/t Au and a sampling liberation diameter of 1 mm, requires a 250 kg theoretical sample mass to yield a precision of ±20% at 90% reliability. If the mineralisation is evaluated by PQ diamond core (wireline diamond drilling that yields approximately 15 kg/m of core based on a 122.6 mm drill bit), then with a mean width of 2.5 m each intersection yields nominally 37.5 kg of whole core sample. In this case, seven intersections will yield a total mass of 263 kg. Whilst each individual sample may be locally unrepresentative, the seven spatially distributed samples will provide a composite that is representative of that domain.

The theoretical sample mass required for GRG testwork in a low-grade (4–6 g/t Au) coarse-gold mineralisation was up to 3 t [71]. Actual sample masses of 50 kg undercalled the measured GRG, essentially negating the use of gravity recovery despite its proven applicability. Improved representativity was gained with larger samples, where those over 250 kg provided a strong indication for GRG potential, but still understated reality. Only 3 t pilot and 16 t bulk samples provided the best GRG evaluation. Samples of 50 kg yielded an RSV of 155% for head grade. This reduced to 75% respectively for 250 kg samples, and 30% for the 3 t samples [71].

In another case study, moderate- to high-grade mineralisation required a theoretical sample mass of 200 kg for high-grade core zone (21 g/t Au) mineralisation [37,80]. Whole diamond core (17 kg) and development bulk (140 t each) sampling was used to determine gold grade, and GRG and leach recovery parameters. Grade RSV values were 138% and 27% respectively for the core and bulk samples. Whilst the resource 17 kg 2 m whole-core composites were not individually representative of the mineralisation, they permitted construction of grade and recovery block models, which were subsequently validated by bulk sampling, trial mining and production.

6.3.3. Sampling Domains

The practitioner needs to review the optimum sample mass in the light of geometallurgical domains. In the simplest case oxide, transition and primary domains are likely to have different sample mass requirements. More complex local domains may also exist, where different theoretical sample masses are also required (Tables 8 and 9).

The example given in Table 8 shows a relative consistency of theoretical sample mass requirements across the three domains. The sample mass requirement per domain needs to take into account the width of the domain, and can be presented as the mass per unit width. In this case the mass per unit width varies from 52 to 150 kg/m.

In the second example (Table 9), both the theoretical mass and mass per unit width vary considerably from 5 to 620 kg and 3 kg/m to 1.2 t/m respectively. This marked difference reflects the narrow coarse gold-bearing high-grade laminated vein domain which requires a large sample mass.

In previous examples (Tables 8 and 9), the mineralisation and local domain widths are relatively narrow. Wider mineralisation domains may also require different theoretical sample masses (Table 10).

In this case, the vein-bearing skarn theoretical sample mass can be applied to both zones, which is a practical outcome particularly as the domains intermingle.

It is impractical to continuously change the sample mass collected across each domain, particularly as they may vary in spatial location from being separated to intermingled. The sampling and characterisation of each domain would be a favourable outcome, though it may not be practical. Optimisation is best achieved using the expected underground stope width or open cut bench height. Early in a project this may not be known, so a pragmatic approach is required. Within any domain, there is also likely to be a d_{95Au}-grade relationship indicating local variations in sample mass requirements [29]. Characterisation studies will allow sampling expectations to be set across a number of d_{95Au}-grade scenarios, thus allowing the worst case scenario to be applied via an achievable protocol across the mineable width.

In the narrow vein examples (Tables 8 and 9), a diluted stope width variability sample mass of 300 kg and 1 t was extracted from underground locations. Thirty-two and 16 samples were collected respectively from each example. In the massive skarn example (Table 10), samples were composited across 5 m wide zones. Whole PQ-core samples yielding 75–80 kg per 5 m were extracted via a dedicated metallurgical variability drilling programme. Ten mineralisation width samples were collected for each domain (e.g., vein-bearing and massive skarns). In the three cases (Tables 8–10), the testwork head grade RSV values are 45%, 52% and 49% respectively. These results are not unreasonable given the optimum mass estimate at ±20% plus all other errors constitute the RSV value.

The definition of geometallurgical sampling domains is an iterative process. Early in the MVC there will be less information, and therefore less resolution of domains. As a project progresses through pre-feasibility to feasibility, resolution will improve. The authors reiterate the importance of early stage characterisation and liaison with other technical disciplines.

6.4. Number of Samples

A commonly asked question early in a programme is how many (metallurgical) samples are required? The best response will be as many as are required to describe the variability. Once the project has reached the scoping stage, sample number can potentially be investigated by variography and TOS, with the aim of understanding the continuity of recovery response. Once the key drivers (e.g., sulphide mineral distribution) are understood, then additional samples and testwork can be used to further resolve continuity for spatial modelling.

Too few metallurgical samples are typically related to poor liaison between geologists and minerals engineers. Early planning is required to ensure that sufficient samples of the correct mass and spatial distribution are available. The greater the budget allocated to early characterisation, the better the representativity will be and the less uncertainty later on.

Recommendations on the number of samples required are not simple. It is inappropriate to design a plant on one or two samples. At an early stage (e.g., Scoping), a given domain probably requires at least two to three composites and variability samples, although further down the project development chain twenty or more may be required (Table 11).

Table 8. Example of sampling characteristics for mesothermal lode/sheeted vein mineralisation across local domains (after [54]).

Local Domain	Type	Characteristic	Average Domain Width (m)	Grade (g/t Au)	d_{95Au} (μm)	Sampling Constant (K) (g/cm$^{1.5}$)	Theoretical Mass (90 ± 20%) (kg)	Theoretical Mass per unit Length (kg/m)
HW	Hangingwall	Wallrock alteration with veinlets with visible gold	5	5.5	1100	12,800	340	68
CZ	Core (lode) zone	Composite laminated vein with visible gold	2	19	1600	6500	300	150
FW	Footwall	Wallrock alteration with veinlets with visible gold	5	4	900	13,000	260	52
All	-	-	11.5	9.5	1400	10,600	400	35
DSW	Diluted mine stope width		2.3	17.2	1500	6500	280	122

Table 9. Example of sampling characteristics for epithermal vein mineralisation across local domains.

Local Domain	Type	Characteristic	Average Domain Width (m)	Grade (g/t Au)	d_{95Au} (μm)	Sampling Constant (K) (g/cm$^{1.5}$)	Theoretical Mass (90 ± 20%)	Theoretical Mass per unit Length (kg/m)
HW	Hangingwall	Wallrock alteration with minor veining	1.5	4	50	170	10 kg	7
LV	Laminated vein	Composite laminated vein with abundant visible gold	0.5	30	2500	8000	620 kg	1200
BV	Brecciated vein	Matrix-supported breccia vein with sulphides	1.5	10	250	760	10 kg	7
FW	Footwall	Wallrock alteration with minor veining	1.5	4	50	170	5 kg	3
All	-	-	5	8.5	2000	20,300	1.1 t	220
DSW	Diluted mine stope width		2.3	13.6	2200	14,600	930 kg	404

Table 10. Example of sampling characteristics for massive skarn mineralisation across local domains.

Local Domain	Type	Characteristic	Average Domain Width (m)	Grade (g/t Au)	d_{95Au} (μm)	Sampling Constant (K) (g/cm$^{1.5}$)	Theoretical Mass (90 ± 20%) (kg)	Theoretical Mass per unit Length (kg/m)
VSK	Vein skarn	MSK with multiple veins of quartz, calcite, epidote and chlorite ±sulphides	15	5	1000	12,200	300	20
MSK	Massive skarn	Garnet, pyroxenite and wollastonite skarn	20	6.5	250	1200	10	0.5

Table 11. General recommendation for the number of metallurgical recovery samples (composite, variability and pilot/trial) for different project stages based on gold deposit heterogeneity. Numbers of samples per metallurgical type/domain. Any one composite may yield a number of tests depending on sample mass and project need.

Project Stage	Low Heterogeneity			High Heterogeneity		
	Composite	Variability	Pilot	Composite	Variability	Pilot
Scoping	2–5	2–5	No	5–15	5–10	No
Pre-feasibility	10	10	No	20	20	Maybe
Feasibility	10	10	Maybe	20	20	Yes
Total at FS	>25	>25	Maybe	>50	>50	Yes

Heterogeneity plays a major part in determining the number of samples required. Where heterogeneity (nugget effect) is high, a large number of samples will be required (Table 11). In many cases, a bulk sampling/trial mining programme to support pilot or test processing is required [54,77,81].

There is a need for spatial distribution through a deposit, where samples should avoid being based solely on high-grade areas, specific mineralised zones (e.g., oxide versus sulphide) or lithologies (e.g., where alteration and/or rock composition may affect comminution properties) (Figure 1). At the SS stage, composites are likely to be spatially restricted based on ease of mineralisation access. As the project develops into the PFS stage, then a regular grid pattern of samples should emerge. It is important that composites from a given domain reflect the grade population of that domain. The resource-grade frequency-curve linked to the domain 3D model and drill database can be used to track core samples at a given grade to provide a composite, thus honouring the grade and spatial distribution.

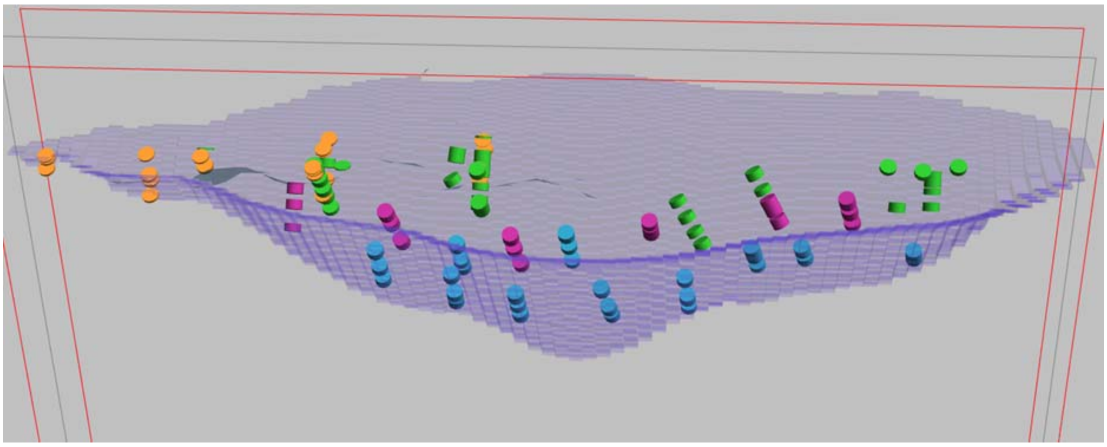

Figure 1. Spatial distribution of metallurgical composite samples. Orange: scoping stage phase 1; Green: scoping stage phase 2; Blue: pre-feasibility; and Pink: feasibility stage. A total of 87 composites were collected to support 295 recovery and 160 comminution tests. Some of the larger contiguous composites formed variability testwork samples. Total project reserve base 90 Mt. Pit strike length 1.8 km.

Metallurgical samples and testwork should focus on the life-of-mine extents of the mineralisation. It is not unusual in a large body to have more samples/testwork in the portion of the resource that will be mined during years 1 to 5. A general recommendation is a 50%, 25% and 25% split across the mine life. For example, in a deposit with a 15 year mine life, 50% samples/testwork may represent years 1–5, 25% years 5–10, and 25% years 10–15.

6.5. Sample Type and Collection

6.5.1. Composites versus Variability Samples

Metallurgical samples may be either: (i) composite; (ii) variability or (iii) variability composite samples:

- Composite samples should represent a given domain and comprise samples from different intersections (e.g., drill holes) or locations (e.g., other sample types) in that domain.
- Variability samples will be individual samples taken across a zone and submitted separately to investigate variability within the domain. Where possible, variability samples are preferable as they provide a measure of likely variability.
- Variability composite samples represent composites that are combined from samples with minimal spatial separation. As such they are composites, but reflect variability between localised areas.

Composites smooth variability and may hide the true picture of the orebody and potential plant feed. There are warnings as to the compositing of so-called "average ore" composites, which can be misleading and nothing like the orebody or domain in question [17]. It is critical to ensure that composites take account of: deposit limits, mining sequence, ore type (geometallurgical domains) and dilution.

Sample lengths should represent the mineralisation as the mineable width. For wide orebodies (>5 m) with variable mineralogy, etc., composite or variability samples taken over the entire width may be misleading if the orebody will be selectively mined across strike, for example if the footwall zone only is extracted. Similarly in a large orebody that will be extracted by an open pit, there is little point basing a composite on a 100 m intersection where the bench height will be 5 m. In this case a series of intersections along the same horizon will best form the composite. One strategy is to collect variability or variability composites on a domain basis, subject them to the required testwork, and then average the results to form larger composites [82].

Fixed testwork masses may be generally appropriate for comminution testing, though not necessarily for metallurgical recovery testwork which require the presence of the critical ore minerals (e.g., gold or gold-bearing sulphides in this discussion). Non-representative testwork samples (even if the collection and splitting process was optimal) may affect the results of all recovery methods across flotation, physical separation and leaching. The effect can be particularly marked for coarse gold-bearing (high-nugget) GRG testwork results [71]. In such cases, a preliminary characterisation stage is required to evaluate sampling needs. Representativity issues are also relevant to sulphide-hosted gold particles where the sulphides are patchy.

Where composites are collected, it is critical that (1) individual composites or (2) master composites honour the grade frequency distribution. In the case of individual composites, these may be collected from a given domain as individual samples to represent grades across, for example: cut-off, low, run-of-mine (ROM) and high. These are generally formed by identifying core (whole or half) that have assays close to the required grade. A number of different intersections will then be composited to yield a given mass at the required grade. For master composites, these will generally aim to achieve a ROM grade and will comprise different intersections across the grade frequency distribution to achieve this aim. It is important to interrogate the grade frequency distribution from the original core or block model to ensure that each grade range (e.g., decile) is proportionally represented within the master composite. Given that the definition of composites is not always universal, it is critical that the practitioner communicates exactly how composites have been formed and from where.

6.5.2. Sample Type

A number of different sample types are available to the minerals engineer for testwork, across diamond core and reverse circulation drilling, linear samples and bulk samples (Tables 12 and 13).

Table 12. Characteristics of sample types (modified from [17]).

Sample Characteristics	Type of Sample					
	Grab	Linear	Reverse Circulation (RC) Cuttings	Small Core	Large Core	Bulk
Spatial coverage	Poor	Good	Good	Good	Varies	Poor
Mass of sample	Low-good	Low-good	Low	Moderate-high	Good	Best
Particle size distribution	Poor	Fair-good	Poor	Fair-good	Good	Good
Cost	Low	Low	Moderate	Moderately high	High	High-very high
Mass	Up to 10 kg	Up to 10 kg 100 s kg for panel samples	Up to 60 kg/m	1.5–16 kg/m	49–90 kg/m	>1 t

Table 13. Summary of sampling errors and related risk rating for different sample types. Errors are best case scenarios, based on optimised sample collection; Red: high; Orange: moderate; Green: low (modified from [37]).

Sample Type	Summary	Incorrect Sampling Errors			Risk Rating for Metallurgical Use
		DE	EE	WE	
Linear (underground workings or surface pits and trenches)					
Chip/chip-channel (hand cut)	Relatively easy to collect and fast Moderately high number can be collected				**High**
Channel (hand cut)	Less easy to collect, requires effort Moderately high number can be collected				**Moderate**
Channel (saw cut)	Requires effort and specialist equipment Moderate number can be collected				**Low**
Panel	Medium-large sample size Less easy to collect Moderate number can be collected				**Low**
Broken rock (underground and surface rock piles)					
Grab	Relatively easy to collect Moderate number can be collected Prone to very high FSE and GSE				**High**
Drilling					
Diamond core	Well established method Good geological information				**Low**
Large diamond drill core	Well established method Good geological information Provides much larger sample mass per m drilled				**Low**
RC	FSE potentially moderate-high when sub-sample split from original Potential loss of fines Fines generation during drilling problematic for metallurgical purposes				**Moderate**
Sludge	FSE and GSE potentially very high when collecting and/or splitting at the rig Loss of fines and fines generation problematic for metallurgical purposes				**High**
Bulk (underground or surface)					
Bulk	Well established approach to gain large sample mass from 1 t to 1000 s t FSE and GSE potentially high if sub-sampling Careful planning required Excellent geological and geotechnical information				**Low**

The sample types best suited to metallurgical testing are either HQ (wireline diamond drilling that yields 63.5 mm diameter core to give approximately 8.7 kg/m of core) or PQ (wireline diamond drilling that yields 85 mm diameter core to give approximately 15.6 kg/m of core) diameter diamond drill core or underground/surface linear samples (Table 13). Large diamond drill core provides up to 90 kg/m of sample for testwork (Figure 2) [17].

Figure 2. Eight-inch (200 mm) large diameter diamond drill core (after [83]).

RC chips are rarely appropriate, given their relatively finely ground nature and strong risk of mineral liberation leading to segregation errors. If RC samples are used for recovery tests, then particular care should be taken when sub-samples are extracted, otherwise the effects of the FSE and GSE can be large. RC drilling generally produces between 30 and 50 kg/m of sample. In fine gold-dominated mineralisation, then a sub-sample split of between 3 and 8 kg may be appropriate to achieve an FSE of below ±20%. For coarse gold mineralisation the entire sample is likely to be required, effectively reducing the FSE to zero as no sample mass reduction takes place (e.g., whole core sampling and full sample assay by LeachWELL) [84]. If the rejects to be sampled have lain for a while, then GSE will be material. Sub-sampling must be via a riffle splitter, preferably a rotary sample divider. Scooping from the top of the bag would be grab sampling (Table 13).

Rejects from any source (e.g., RC drilling, and laboratory coarse or pulp rejects) should be used with care. Time may have led to deterioration such as oxidation, together with container integrity and/or labelling issues. As with any broken rock material, the effects of FSE and GSE must be considered during sample collection and sub-sampling.

Rock face samples (e.g., panel samples; Figure 3) should be collected within the framework of TOS to ensure minimal bias through reduced DE and EE. In particular fly rock from channel or panel samples can result in both sample loss and contamination. Where underground workings or surface trenches or pits are available, samples can be collected from walls and floors (Figure 3).

Figure 3. Collection of metallurgical sample via underground panel sampling.

6.5.3. Core Sample Collection

The best quality metallurgical samples generally come from recently drilled diamond-drill core related to resource development programmes. The traditional paradigm sees geologists take half-core and submit it to a laboratory for assay. The other half is retained for reference. It is this remaining half-core that is generally available for metallurgical studies. The industry norm is to cut the half-core into two quarters, retain one and use the other for metallurgical compositing (Figure 4).

An alternate solution is to leave a so-called fillet, which is a segment of core that is retained as a geological sample. The remaining core is then cut in half to provide an original sample and a duplicate. Designing a core-cutting template will automate this procedure in a safe way. It is recommended that at least 70% of the core, the original plus duplicate, is submitted for testwork and/or assaying leaving 30% as the reference filet. In the case of PQ core for example, the filet would represent approximately 4.5 kg/m of core.

Figure 4. (**Left**): Half NQ2 core prior to quarter cutting for testwork. NQ2 is a standard wireline diamond drill type, where the core diameter is 50.6 mm to yield approximately 5.5 kg/m for whole core; (**Right**): Quarter cut core bagged prior to transportation to the laboratory. This intersection was combined with another to form a 50 kg composite sample for GRG testwork.

Core should be re-logged, with emphasis on ore and waste mineralogy, and detail of mineral grain size and shape, associations and textures. The usual geological features such as rock type, alteration and structure should be recorded or re-checked from the original log. It is good practice to liaise with geologists to embed geometallurgical observations into routine logging.

The sample collection process is of concern, given that this stage can impart substantial errors into the sampling programme. A major issue with core is that of total core recovery and quality [85]. From a sampling perspective, perfect core drilling shows low DE and EE. However, due to poor ground conditions and/or poor drilling control, EE may be high as seen in poor core recovery (e.g., core fragmentation and/or material loss) [85]. In such a case, metallurgical test work, either comminution or recovery tests must be interpreted with caution or preferably avoided. If a sample must be taken from a poor recovery zone—this is not recommended—the only option is to take all core for testing. As an alternate option, all the core can be collected, crushed to around $P_{90} > 2$–4 mm and split in half, with one half retained and the other taken for testwork. In this case, the crush/split error must be reviewed, as the FSE value could range from acceptable (below ±15%) to above ±150%.

Additional bias during diamond drilling is related to the plucking of gold particles from the core surface as a result of the drilling process. This is not a consistent issue, but is known to occur and leads to a negative grade bias—in effect EE. Core sawing can also lead to gold loss in the cuttings [7,84]. A metre of NQ core can yield up to 200 g of cuttings, assuming a 3 mm blade. NQ is a standard wireline diamond drill type, where the core diameter is 48 mm to yield approximately 5 kg/m for whole core. Whilst not necessarily a consistent issue, it can lead to gold loss and sometimes loss of the only gold in the core (i.e., cuttings return a gold grade, whereas the two halves return none). To achieve minimal sampling error and reduce the nugget effect, the taking of whole core samples is an option.

An alternate approach to the half or quarter-core paradigm is to integrate metallurgical testwork with resource grade determination. This option involves taking half or whole core samples and processing the entire sample by, for example, the single-stage GRG test [18], large-scale screen fire assay and/or bulk leaching.

Dominy et al. [37,80] applied whole HQ 2-m composites (approximately 17 kg samples) to a coarse gold orebody. A single-stage GRG test with tails leach (and some flotation testwork) was used to provide data for GRG and tails leach recovery, and head grade. The data was applied to a resource model reported in accordance with The 2012 JORC Code.

6.5.4. Sample Integrity and Chain of Custody

For sulphide-rich mineralisation such as some lode-gold styles and massive sulphide deposits, oxidation can be a major issue. Samples will need to be purged with nitrogen and sealed in plastic bags, or stored in freezers. Hydrocarbon contaminants (e.g., oil, grease, drilling muds and surfactants) can also be an issue that leads to depressed flotation responses or preg-robbing during leaching. This can be minimised by good drilling practice, including effective housekeeping to remove oil/grease and washing the core immediately to remove any drilling chemicals.

Chain of custody procedures must provide a documented, legally defensible record of the custody of samples from collection through analysis [86]. A sample is considered to be in custody if it meets at least one of the following conditions, where the sample is: in someone's physical possession or view, secured to prevent tampering or secured in an area restricted to authorised personnel.

Chain of custody may be defined as "an unbroken trail of accountability that ensures the physical security of samples, data, and records". This definition relates to secure traceability. Compliance implies that it should be known and documented who has custody of a sample(s) at any moment. As long as this principal is followed, the chain of custody concept is respected.

The greatest risk of sample tampering is during collection and bagging, prior to sealing of the sample bag(s). Similarly, at the laboratory once a bag seal is broken there is some risk. At collection, samples should bagged, tagged and sealed as soon as is possible and preferably in the presence of an independent observer (Figure 5). Once sealed, samples must be stored in a restricted access and secure compound prior to transportation.

(**a**) (**b**)

Figure 5. *Cont.*

(c) (d)

Figure 5. (**a**) Plastic buckets (25 kg capacity), (**b**) metal core trays, (**c**) 205 litre drums (250–350 kg capacity) and (**d**) plastic sample bag sealed to prevent spillage and tampering.

The secure handling of samples during transport to the laboratory is important and can be aided by the use of sealable containers (Figures 5 and 6).

Figure 6. Bagged and sealed metallurgical samples packed for road transport to prevent spillage and tampering.

An important consideration during testwork is that of security, which includes samples, rejects and concentrates, most importantly the security of any liberated gold, where accidental or intentional loss constitutes a PE. Concentrate streams should be monitored 24/7 and concentrates stored in a secure and restricted area.

7. Sample Preparation, Testwork and Assaying

7.1. Overview

Beyond the actual testwork itself, a frequently forgotten aspect of metallurgical testwork is the associated preparation, sub-sampling and assaying of feed, head, concentrate and tails samples. Laboratory activities include, weighing, drying, fragment size reduction (crushing and grinding), sample mass reduction, sample blending and assaying.

7.2. Drying and Weighing

The dry and weighing stages are relatively simple operations, though can impart sampling errors. The optimal drying temperature is 100 °C ± 10 °C, where it is important to check whether some sulphide-rich ores are reactive and heating results in oxidation. Care should be taken to avoid dust and contamination during drying. As a sample(s) progresses to dryness, opening the dryer door or handling the sample may cause dust release. All weighing devices must be calibrated on a regular basis as recommended by the unit manufacturer. It is important to ensure that all samples in the dryer are properly labelled to ensure trays or vessels are not mixed-up.

7.3. Sample Mass and Size Reduction

Mass reduction entails making the sample mass smaller, but should preserve the size distribution of the sample. Size reduction entails preserving the mass of the sample, but reducing the size distribution in order to generate more particles. Mass reduction is necessary for assaying purposes since the laboratory is unlikely to be able to assay many kilogrammes of sample. Size reduction is necessary to preserve the assayed grade: by reducing the size distribution, more gold particles become liberated from each other, which in theory increases the probability of representative sub-sampling [8]. This becomes a significant problem, as coarse gold becomes progressively liberated it does not comminute effectively because of its malleability. As noted previously, in this case then whole sample assaying is likely to be required.

Core or broken rock samples can be submitted directly to the metallurgical laboratory. Assuming that an appropriate mass is available, after drying they can pass directly to crushing and testwork. In this case it is assumed that no sample splitting is required.

If a sample requires crushing and sub-sampling for other or duplicate tests (e.g., a head grade assay) then care is required. Crushing and particularly splitting invokes a number of sampling errors [13]. Both the FSE and GSE will have a material impact on splitting unless it is done effectively. The entire sample should be crushed to a size that minimises the liberation of gold, potentially between a P_{80} of 0.5 cm to 1.5 cm. If gold is coarse and the grade relatively low, splitting at a coarse crush may lead to a high FSE. In this case, FSE calculations will indicate the need for a finer crush (e.g., a few mm), thus potentially liberating gold and/or sulphides prior to testing. Compromise will be required with respect of sample mass and crush size. If gold is liberated during crushing and/or grinding, then a rotary sample splitter should be used (Figure 7).

Figure 7. Laboratory rotary sample divider.

In some cases a reference sample will be split from the primary sample to facilitate additional or check testwork. However, it is important to ensure that the reference sub-sample is representative or it becomes a pointless exercise. Similarly a head sample is often taken to facilitate a head assay determination. Again, it is important to ensure that the head sample is representative or it also becomes a pointless exercise. When the sub-sample is taken, the FSE of the split can be calculated via the FSE equation, but also the FSE of the primary sample less the remaining sample (e.g., mass of primary less the head/reference sample).

Sample splitting methods such as fractional shovelling, alternate shovelling and coning and quartering are considered outdated and prone to high bias [13]. Both may impart RSV values of 20% or more (key errors: FSE, GSE, DE, EE and PE). However, if the actual sub-sample is selected at random then the often overwhelming effect of the DE is reduced [10]. The rotary sample splitter has been demonstrated to show relative error values down to 0.5% and riffle splitters around 3% [13].

7.4. Sample Blending

Samples may be delivered to the laboratory as separate parts of a single composite. In such a case the various parts need to be blended together. If the entire composite is taken for a given test and not split, then blending of the individual samples is not an issue. However, if the composite is subsequently going to be sub-sampled, then a blending process is required. A traditional approach may be to feed alternate core pieces from each sample through the crusher, thus providing a blended product. A better option is to crush each sample and then feed them alternately through a rotary sample splitter (Figure 8). It should be remembered that all sample handling in the laboratory poses risk of both loss (EE) and contamination (PE).

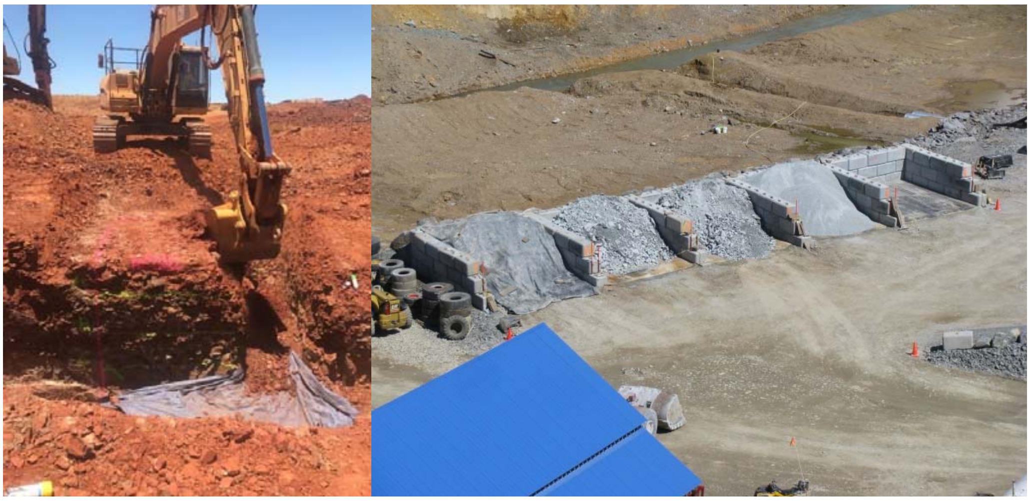

Figure 8. The collection of surface (**Left**) or underground bulk samples must be undertaken with care to ensure full sample extraction, transport and storage (**Right**) prior to processing.

Samples can be fed individually into the rotary sample splitter or in an interleaved fashion thereby enabling blending. To maximise the blending effect of the rotary sample splitter, the composite sample should be fed at least twice through the rotary sample splitter, before forming the two or more required splits. This process effects blending and minimises the GSE.

The sample blending process is time consuming and labour intensive. A more efficient option is reported in Whittaker [87], where the time to blend 200 kg of broken core is reduced from five days to around three hours. The process involves two-stage crushing (jaw and Boyd crushers) of broken core to −1.7 mm. The product is then screened, with the oversize re-crushed and the fines stored in barrels. On completion of crushing, the material is fed to a 150 kg or 1 t capacity conical screw mixer for blending, and then passed to a rotary sample splitter for splitting.

7.5. Testwork

Testwork imparts sampling errors, particularly through loss (EE) and contamination (PE), poor equipment operation (AE) and careless work practices (PE and AE). Key needs relate to: ensuring equipment maintenance and calibration, optimal equipment operating parameters to ensure reproducibility between samples, cleaning of all equipment and associated pipework, pumps, etc. between samples and avoidance of careless work practice covering equipment usage, sample mix-ups and general bad and/or unsafe and inefficient practice (Table 1).

7.6. Tailings Sampling

Metallurgical recovery tests generally produce tailings (residues) which require an assay(s) to permit head grade and recovery reconciliation. In most cases, tails are fine-grained (generally, 1 mm) and of a low grade that allow a sample to be taken easily. Best practice should see the use of a riffle splitter or preferably a rotary sample splitter to ensure an effective sub-sample for assay. Scooping or grab sampling from bags or buckets should be avoided. For any ore type, consideration should be given to the tailings based on ore characteristics. For example, in some coarse gold ores, relatively-rare but coarse influential particles may get into the tails stream and require larger samples to be representative.

7.7. Assay Sub-Sample Preparation and Assaying

Once a head, concentrate or tails sub-sample has been delivered to the assay laboratory, it is important that it be prepared and assayed correctly. Particular care is required in the presence of coarse gold, with possibilities for gold loss and/or contamination [88]. Large pulverisers such as the LM5 (maximum capacity approximately 3 kg) are useful, however removal of the pulp needs to be maximised (EE) and contamination controlled (PE). It is recommended that laboratories include a barren flush after coarse gold-bearing samples or concentrates. Assaying of the pulveriser wash (e.g., sand) is recommended at a rate of between 1 in 10 and 1 in 20.

The traditional paradigm of crushing and pulverising the entire sample (e.g., testwork head or tails sample) and taking a 30–60 g sub-sample for fire assay is potentially flawed [1,7,54]. The approach is prone to high correct and incorrect sampling errors (Table 3), particularly when the assay charge is scooped from the pulp [1]. The propensity of gold not to pulverise efficiently (exacerbated in the presence of coarse gold, which may still be present in a tails sample for example), promotes FSE and GSE, and potentially loss or contamination (e.g., PE) during sub-sampling for the fire assay charge [88,89]. The use of whole samples is a potential option. Whole samples followed by full sample assay effectively yields FSE and GSE values of zero. With good laboratory practice, the PE and AE can be minimised.

There are a number of techniques employed to measure gold concentration, these include traditional fire assay and large charge methods such as screen fire assay (SFA), LeachWELL (LW) and pulverise and leach (PAL). The Chrysos Photon Assay method provides potential for fast assays, based on 0.5 kg crushed samples [90]. Sample assay protocols should not be left solely to the laboratory to decide, but established by the project team based on material characteristics.

7.8. Reject and Residue Materials

Sampling and testwork programmes produce reject and residue materials. Rejects generally comprise primary sample material that is either purposively split-off as a reference sub-sample or not required for testwork. Residues comprise material that is left after testwork, generally tailings with the majority of the critical metal or mineral removed. In the case of rejects, it is critical to ensure that they are split from the primary lot with all errors minimised, particularly the FSE. If a later programme will use rejects for testwork, it must be possible to demonstrate that the rejects are representative. All rejects and residues must be contained and stored carefully for future usage.

8. Mineralogical Sampling and Analyses

8.1. Mineralogical Analysis

Mineralogical studies play an important role in optimising gold recovery processes. It is widely used to characterise gold in ores and mill products, and to determine potential problem(s) causing gold losses. The information acquired can be used as a basis for metallurgical testwork programme design or optimization. Ideally, a mineralogical study should be conducted prior to the start of, or at the early stage of a testwork programme. Mineralogical studies are utilised throughout the stages of a mine's operation to closure, adding essential information to the MVC.

Automated mineralogical analysis (AMA) is an analysis method which is deemed as routine and utilises scanning electron microscopy (e.g., Mineralogic, Mineral Liberation Analyser, QEMSCAN, etc.) [68]. These are specialised systems with applied software, exclusively for the characterisation of ores. Specific programmes are tailored for minerals of interest (e.g., gold), which present challenging textures, or rarity of abundance. Programmes may also be manipulated to capture the internal elemental differences through increasing X-ray measurement times. This is particularly useful when trying to understand stoichiometry/elemental substitutions. For example, auriferous pyrite or gold grains with substituted elements.

8.2. Sampling and Preparation

Sample selection at each stage of the MVC can be different, largely due to the nature of the materials being collected. Study media will be based on either: (1) rock samples, rock chips or drill cores or (2) granulated material (e.g., mill products or reject material). Granulated samples can be pre-concentrated and sized into fractions. Rock chips or drill cores can be crushed for sub-sampling. For complete analysis, each fraction will need be analysed. Samples for AMA are usually mounted in epoxy resin 25 or 30 mm diameter blocks or glass slides. Various methodologies for sample preparation and analysis are reported: [42,68,73,91–93].

Samples can be taken directly from drilling programmes (Tables 14 and 15). Size distributions and a size by size analysis of RC samples can provide preliminary details on mineral deportment. Diamond drill core provides the best material for AMA, where tiles can be cut, prepared and mounted in the SEM. Cores can also be halved, quartered, crushed and sized. It is recommended that crushed samples are analysed by on a size by size basis. Mixed sized samples are hard to measure on the SEM, textures can be "over measured" or conversely, missed. Replicate and duplicate samples must be factored into the sample collection.

AMA software provides modal mineralogy and has internal assay reconciliation. As part of the QAQC process, each size by size sample should have a further sample taken for X-ray diffraction (XRD) and assay (fire assay, etc.). If analyses are staged, then it is prudent to understand the deportments. Deportment should highlight the potential opportunity to pre-concentrate the samples before looking at analysis programmes such as liberation by free surface or particle composition.

Table 14. Metallurgical sampling and testwork validation actions across the MVC.

Validation Type	Project Stage	Activity	Comment	Validation Target
Duplicate samples	Advanced exploration to Feasibility	Field duplicates	Based on field duplicates (variability samples) taken proximal to each other, or duplicate composites taken from the same drill holes and intersections via half or quarter-core. In reality, rarely taken during metallurgical sampling	Total relative error: ±50% Sampling error: ±40%
		Coarse duplicates	Splits of laboratory crushed material	Total relative error: ±25% Preparation error: ±20%
		Laboratory (pulp) duplicates	Splits taken from final testwork sub-samples after grinding	Total relative error: ±15% Analytical error: ±15%
Pilot testing	Pre-feasibility and/or Feasibility	Processing of composite or variability samples through pilot plant	Tonnes-scale of material taken from core or bulk samples. Compares testwork prediction (model) with small process tonnage	Within ±20% of prediction for a given tonnage
Demo/full plant testing	Feasibility	Processing of composite or variability lots through process plant	Material taken from bulk sample or trial mining programmes. Compares testwork prediction (model) with large process tonnage	
Production plant	Production	Processing of ore through plant	Production mining. Compares testwork prediction (model) with actual mining	Within ±20% of prediction over a given time period (e.g., month)

Table 15. Bulk sample/trial mining material processing options.

Lot/Process Option	Laboratory Bench	Pilot Plant	Demo/Full Plant
Whole lot	-	(1)	(1)
Lot batch	-	(2)	(2)
Lot sub-sample	(3)	(3)	-

8.3. Optimal Sampling Plan

Understanding that a polished block requires approximately 5 g of materials to form a mono-layer of particles poses a challenge to create a representative sample. Liberation and pre-concentration is the best option [77,91,93–95]. Any sample for pre-concentration needs to be sub-sampled from an optimised field sample in the light of TOS. Henley [92] recommends a minimum sample mass of 5 kg for the pre-concentration approach, whereas [91] use a 10–50 kg sample and Goodall, Scales and Butcher [73] a 100 kg sample. Any sub-sampling of the field sample requires minimisation of the FSE.

Stages of sample mass reduction include rotary sample divider or micro-riffle splitting to an optimum size, usually in the 38–150 μm range. Jones and Cheung [96] emphasise the challenges, where a sample of 1 g/t Au at a grain size of 1 μm requires two polished sections to be theoretically representative. If the gold grain size was 100 μm, then some 20,000 polished sections would be required.

Lane and McComb [93] present a case study where sixteen 25 mm polished blocks of a 6.5 g/t Au ore were produced from drill core, and only three small gold particles were identified. They subsequently took a composite core sample of 5 kg, crushed it, used heavy liquid separation to remove the low density phases and produced a gold concentrate by Superpanner. The coarse, middling and tails products from the Superpanner were studied by AMA. Some 275 gold particles up to 2 mm in size were identified.

9. Quality Assurance/Control and Validation

9.1. Quality Assurance and Quality Control

Quality assurance (QA) and quality control (QC) are the key components of a quality management system [86,97]. QA is the collation of all actions necessary to provide adequate confidence that a process (e.g., sampling, testwork and assaying) will satisfy quality requirements. QC refers to the operational techniques and activities that are used to satisfy quality requirements. QC includes the system of activities to verify if the QC activities are effective. While QA deals with prevention of problems, QC aims to detect them. In practical terms, QC procedures monitor precision and bias of the measurement values, as well as possible sample contamination during preparation, testwork and assaying [86].

Resource drilling and assaying programmes will have a QAQC component; however this is generally ignored during metallurgical sampling and testwork [64]. The principles of QAQC must be applied to metallurgical programmes, although there are metallurgical specific considerations in both generating and analysing the data. Key metallurgical QAQC considerations are:

- Duplicate field samples that are spatially distributed within a given domain to honour the gold grade frequency distribution. Field duplicates are rarely collected during metallurgical programmes due to the relative lack of material available, quantity of material required and high cost of testwork.
- Sub-sampling must be controlled by the FSE equation to ensure that they are representative.
- Assays to be supported by certified reference materials (CRM) and blanks to quantify analytical bias and contamination.
- Blanks should be inserted after known high-grade samples.
- Duplicate tests (e.g., two separate tests from same primary sample) should be undertaken on a regular basis (target 5% of the time) to monitor precision.
- Second test in alternate (umpire) laboratory.
- Introduction of reference material for leach and/or flotation testwork.
- Written and audited laboratory procedures with appropriate staff supervision to ensure compliance.

QAQC of metallurgical programmes provides a number of benefits across:

- Quantitative assessment of laboratory performance, enabling review and investigation of any issues.
- Experimental error for key parameters.
- Data to support design of testwork programmes.
- Compliance for public reporting and use in resource/reserve estimates.

A key part of QAQC is documentation and training. Written protocols and procedures, staff training, periodic auditing of protocols and people, and re-training are required. DS3077 [53] provides a framework on which to produce transparent protocols. Control is through the examination of laboratory and audit procedures results, and adherence to standard or industry-accepted operating procedures. The quality of the laboratory should be verified by the project manager and, in the case of complex or novel processes, they should visit the facility performing the work.

9.2. Validation of Metallurgical Sampling and Testwork Programmes

Critical to any sampling and testwork/assaying programme is validation to indicate robustness of the results and in particular representativity of samples and quality of testwork. The level of validation is dependent upon project stage; the more advanced the project the better the validation. Approaches range from duplicate field and coarse crush samples through to trial mining lots through a process plant (Table 14). Duplicate sample data can be analysed to determine the sampling, preparation and analytical errors [14,48,97].

Duplicate samples measure variability across the sampling value chain (Table 1). For example, field duplicates provide the cumulative error across the GNE, sample collection, preparation activities (including comminution and splitting) and testwork/assaying. Whereas a laboratory (pulverised) duplicate, provides the pulp sub-sampling error and testwork/assaying error. Duplicate results are presented as pairwise precision values, where precision improves from the field to pulp duplicates. Pilot and demo/full plant testing provide a different level and scale of validation, through the reconciliation of modelled recovery based on testwork with processing at different scales.

10. Large Sample Pilot and Demo Plant Testing

10.1. Introduction

Validation and scale-up work generally involves the use of pilot plant testing of bulk samples or processing of trial mining lots [75,77,98]. Such large-scale activities are often a critical part of the feasibility stage, though require careful planning and management to ensure that they are providing appropriate validation of a given grade and/or metallurgical model [54,77,81].

The collection of large samples (e.g., >1 t to a few 1000 t) from surface and/or underground locations characterises the pilot to demo/full plant test stage. Bulk sampling (>1 t to 500 t) and trial mining (>500 t to 10,000 t or more) programmes are used to verify grade and metallurgical parameters in complex deposits. Bulk samples tend to be smaller (from a few tonnes to 250 t), more numerous samples from single locations or grouped together from underground development (e.g., 30,000 t at Nalunaq, Greenland [99]) or surface trenches. In relatively rare cases, bulk sampling may be the *only* way to evaluate gold grade. In some cases, a bulk composite of 1 t or more may be formed from drill core material. Trial mining activities tend to represent underground stope lots or open pit benches. Such lots may yield thousands of tonnes of mineralisation for processing (e.g., 10,000 t at Brucejack, Canada [81] and 3150 t at San Antonio [37,80]).

In cases where the nugget effect is high, such programmes may be the only way to assess gold grade. They are typically undertaken during feasibility studies and are seen as an insurance policy to avoid surprises once production commences by verifying grade estimates and metallurgical flow-sheets [77,81,84]. The tonnage of material extracted is dependent upon the heterogeneity and complexity of the mineralisation and project stage. For a small project a few 100 t may be taken, though this may rise to 10,000 t or greater for large projects (e.g., 40,000 t at Bendigo, Australia [100].

A well-planned bulk sampling or trial mining programme will account for mineralisation variability, resolution of which may require a number of sample collection areas in specific geometallurgical domains. In addition, the programme needs to account for grade variability and not just focus on high-grade or ROM mineralisation.

10.2. Sample Collection

During sample extraction/mining a number of matters should be considered (Figure 8) [77]:

- Planning of blasting in relation to stope width (underground) or bench height (surface pits) and minimising of dilution to match that which is likely during mining (e.g., DE reduction).
- Avoidance of over-blasting that results in the excessive liberation and loss of gold and/or gold-bearing sulphides (e.g., EE).
- Mucking of blasted material, in particular fines, with the potential use of vacuum devices to ensure maximum recovery (e.g., EE reduction).
- Management system, involving tagging and chain of custody actions, to ensure that bulk sample mucked material is kept separate from other broken rock and trucked to the required stockpile or bulk transport bag.

10.3. Sample Processing

Sample lot processing options range from the full lot to a sub-sample from the lot (Table 15). Option (1) provides the most defendable results given that, assuming rigorous design and operation, all material is processed and actual gold grade and recovery determined. Option (2) is likely to result in Option (1), providing that all individual lots are processed. The processing of selected lots of may lead to a high FSE from the lot. Option (3) may be problematic on a lot-by-lot basis, unless the sub-sampling protocol is rigorously defined (e.g., FSE and all other errors optimised). The decision of whether to use a plant or sample splitting tower is related to a number of issues which includes programme aims, availability, cost, minimisation of sampling errors, and nature of the mineralisation.

Pilot/demo programmes also require a QAQC system to be put in place. The scale of samples makes the use of CRMs impossible, but blanks may be passed through the circuit. In addition, all associated assays can be subjected to normal QAQC procedures. In the case of bulk samples, field duplicates can be taken and coarse duplicates after crushing.

10.4. Limitations

Whilst bulk sampling/trial mining programmes are important, they are not necessarily the panacea of metallurgical or grade evaluation. It is possible, for example, that a bulk lot may represent its local area well but not describe the wider orebody due to strong variability. During programme planning, the practitioner must consider the nature of the test area and what the results will mean. Sample collection and pilot/plant processing is likely to cost millions of dollars.

11. Case Study

11.1. Project Background

A vein-gold mine, located in South America, was evaluated to resume underground operations, including: 6800 m of HQ diamond core drilling, underground working re-access, and metallurgical sampling and testwork. HQ is a standard wireline diamond drill type, where the core diameter is 63.5 mm to yield approximately 8.5 kg/m for whole core. The resulting total Indicated Mineral Resource for the Main Reef was 350,000 t at 16.8 g/t Au ($\pm$22% at 80% reliability). Resource precision was estimated via Conditional Simulation, where the distribution of multiple-simulated grades allows the inherent estimation uncertainty to be reported at a given reliability, usually 80%. A geologically identical South Reef lies sub-parallel and 35 m away from Main Reef, with a drill-only Inferred Mineral Resource of 300,000 t at 15.2 g/t Au ($\pm$46% at 80% reliability). An underground mine plan was based on 50,000 t for year one, ramping up to 100,000 t early in year two and thereon. A pre-existing plant was relocated to site and updated to provide an annual capacity of 125,000 t.

Mineralisation is dominated by quartz veins with 1.5 m to 2 m widths. Economic grades are related to sub-vertical ore shoots, which extend around 75–150 m along strike and >350 m down plunge. The core high grade zone ("HZ") of the Main Reef contains high-grades of 20–60 g/t Au, compared to 4–8 g/t Au in the peripheral zones ("PZ"). Mineralisation contains coarse gold up to 2.5 mm in size, particularly in the HZ. Coarse gold up to 0.5 mm with a strong fine-gold component is common in the PZ. Gold is hosted in quartz and pyrite in both zones, though the pyrite host is substantial in the PZ. Stoping was across both the PZ and HZ mineralisation.

11.2. Metallurgical Sampling and Testwork

During evaluation, two 50 kg diamond half-core and two 50 kg underground panel sample composites were collected from the HZ mineralisation. Two 50 kg diamond half-core sample composites were collected from the PZ mineralisation. Core quality was excellent, with total core recovery above 95%. Samples were placed into large polyweave sacks and tied with wire. They were transported by road to the laboratory. No chain-of-custody was in place and there were no written protocols for the process.

All samples were crushed to P_{90} -3 mm and 20 kg split off for three-stage GRG testing. A 10 kg crushed sub-sample was split from all samples and subjected to flotation (rougher and cleaner) testwork. The results show high levels of GRG associated with high grades from the HZ (Table 16). Similarly, reasonably high grades were obtained from the PZ. A small amount of gold was recovered in pyrite from the PZ.

Table 16. Summary of original evaluation testwork results.

Sample Nos.	Number of Samples	Domain	Head Grade Range (g/t Au)	GRG Range (%)	Flotation Range (%)
C.01–04	4	HZ	43–95	91–98	Trace
C.05B and 06	2	PZ	15–26	40–79	3–11

11.3. Plant Design and Performance

Based on the testwork results, a gravity plant was re-located and commissioned based on two Knelson concentrators processing 15 t per hour. The tailings from the primary Knelson were passed through the secondary to facilitate additional gold recovery. The gravity feed product was P_{90} -250 µm. The design expectation of the plant was to achieve an overall gold recovery in the range 80–85%, with the increased gold recovery compared to the testwork results based on using two Knelson units. Within a few months of commencement, a head grade of 6 g/t Au was achieved with gold recoveries in the 45–55% range. The original mine plan was for a 15 g/t Au head grade with 85% gold recovery.

11.4. What Went Wrong?

A number of issues relating to metallurgical sampling and geological interpretation were identified:

- HZ samples and sub-samples were unrepresentative, as they were biased to very high grade mineralisation which gave high GRG values.
- PZ samples and sub-samples were unrepresentative, as they were biased to relatively high-grade material that was occasionally present in the PZ, and thus gave high GRG values.
- Primary PZ samples and sub-samples did not represent the sulphide mineralisation, thus the flotation response was low.
- Too few samples were collected.
- Testwork laboratory used was not certificated and had a poor reputation.
- No QAQC was undertaken anywhere in the sampling chain, including during assaying.
- Geological interpretation of the HZ focused on a continuous zone down the centre of the shoot (considered to be 50% of the shoot). This was not the case, in reality there is a series of high-grade sub-shoots, forming a discontinuous zones within the main-shoot forming 25% of the shoot zone. The mine plan did not honour the geology or grade distribution.
- No mineralogical studies were undertaken.

During sample preparation, the crushing and splitting of the primary 50 kg samples to 20 kg and 10 kg sub-samples yielded a high FSE (Table 17). In all cases, the FSE values were greater than ±25%.

Table 17. FSE values for sub-sampling. All FSEs at 90% reliability.

Domain/FSE Value	Grade Range (g/t Au)	d_{95Au} Range (mm)	GRG Sub-Sampling FSE (%)	Flotation Sub-Sampling FSE (%)
HZ	43–95	1–2.5	±25–35	±40–55
PZ	15–26	0.2–0.5	±25–45	±40–70

Based on knowledge of both ore types, the samples should not have been split, but tested in their entirety. Taking a representative primary split for flotation testwork was problematic. However, it was clear that flotation should only be used after gravity concentration and thus a tailings sub-sample of 5 kg could be optimally split with an FSE of less than ±5%. The splitting process after crushing was based on shovelling 20 kg directly into buckets from a pile. The splitting of sub-samples from a primary composite is a key issue, where the split process was not representative. This is further compounded when the primary sample is not representative either. In this case, the sub-sampling process was prone to enhanced FSE, GSE, DE and PE.

11.5. Corrective Metallurgical Sampling and Testwork Campaign

11.5.1. Historical Data Review

A review of historical data and inspection of mine workings revealed that previous stoping in the early 1900s was highly selective in the HZ (30–90 g/t Au head grade). Historical plant recoveries were in the range 50–60% using a simple gravity and amalgamation circuit. It was clear from the historical stoping patterns that the HZ comprised small, potentially 100–400 t high-grade discontinuous steeply-plunging pods.

11.5.2. Sampling Strategy

A corrective metallurgical sampling and testwork programme was undertaken. Estimates of theoretical sample mass for HZ and PZ ROM ore yielded 100 kg to 1 t, and up to 50 kg respectively to achieve ±20% at 90% reliability. At the extreme, a sample mass of 4 t could be required for cut-off grade mineralisation with gold clustering present, though this was considered rare. A mass of 150 kg was accepted for the HZ and PZ mineralisation, given that it theoretically represented the critical cut-off (5 g/t Au) and ROM (11 g/t Au) grades, and was a practical mass to collect and handle. In the underground sampling area, the vein width varied between 1.7 and 2.2 m, which equates to a mass per unit width of 75–85 kg/m. Up to 0.3 m of wallrock material was included in the samples to dilute to the mining width of 2 m.

Given access to four mine levels which represented the first year of production, the samples were collected from development drives. In addition to the underground samples, four half HQ core composites were also collected and tested. These were taken from intersections outside of the underground development area, representing years 2 and 3 of production.

11.5.3. Underground Metallurgical Samples

The PZ samples were collected on an approximate 15 m (dip) by 15 m (strike) grid, with 7–9 samples per level. The HZ were collected on an approximate 15 m (dip) by 5–25 m (strike) grid, with 6–8 samples per level. The uneven grid of the HZ samples reflects its poddy nature. The sampled area represented 60,000 t of Indicated Mineral Resource.

All samples were panel cut by hand with the assistance of a compressed air pick from walls and backs [37]. Sample material was captured on tarpaulins laid on top of rubber matting (Figure 3). All material was then placed into 25 kg capacity double-plastic bags. Each set of six 25-kg bags comprised one composite sample, which were packed into steel drums, sealed and transported to the laboratory. Samples were collected from the same location, hence they were variability samples. Thirty samples were collected from the HZ and twenty-eight from the PZ. Sample collection was fully documented and independently supervised.

11.5.4. Core Composite Metallurgical Samples

Five composites were taken from half HQ core retained from the previous drilling campaign. The composites allowed for the relative proportions of HZ and PZ mineralisation across low-grade/cut-off grade, ROM grade and ROM high-grade. Intersections were selected based on spatial location, domain and grade. Given the difficulty of preparing 150 kg composites from half-core, a composite mass of 70–75 kg was accepted. This approximated to 9–12 intersections per composite, based on half-core yielding 4 kg/m. All core pieces were placed into 25 kg capacity double-plastic bags. Each set of three 25-kg bags comprised one composite sample, which were packed into steel drums, sealed and transported to the laboratory. Sample collection was fully documented and independently supervised.

11.5.5. Testwork Programme

Entire samples were subjected to three-stage GRG testwork. Tailings sub-samples of 20 kg were cut by rotary sample splitter for both leach and flotation testing. Testwork data variability is quantified via the RSV (Table 18). Head grades determined from the GRG test display the highest variability for both HZ and PZ. However, compared to the original half-core drilling results, which yielded RSVs of 179% and 165% respectively, the variability is reduced and within expectation for coarse-gold mineralisation. The flotation RSV is relatively low and reflects the more disseminated nature of the gold-bearing sulphides, particularly in the PZ. The GRG shows higher variability in the HZ, with small variability in the PZ where there is less coarse gold.

Table 18. RSV values for underground sampling and testwork programme.

Domain	Number of Samples	Ave. Primary Sample Mass (kg)	RSV Head Grade (%)	RSV GRG (%)	RSV Flotation (%)
HZ	30	156	46	35	15
PZ	28	154	32	17	27

The core composites displayed similar results, albeit lower overall recoveries likely due to the smaller sample mass (Table 19).

Table 19. Summary of core composite testwork results.

Sample	Domain	Domain Fraction	Grade Type	Composite Mass (kg)	Est. Head Grade (g/t Au)	Head Grade (g/t Au)	GRG (Au %)	Flotation Recovery (Au %)	Total Recovery (Au %)
DCM.1	PZ	1.0	Low/COG	71	3.9	4.2	15	66	81
DCM.2	HZ/PZ	0.2/0.8	ROM	71	10.5	11.5	61	26	87
DCM.3	HZ/PZ	0.3/0.7	ROM	75	13.3	13.6	60	24	84
DCM.4	HZ/PZ	0.4/0.6	ROM high	69	17.4	18.1	65	20	85
DCM.5	HZ/PZ	0.5/0.5	ROM high	73	19.9	17.7	71	15	86
DCM.6	HZ/PZ	0.6/0.4	ROM high	71	23.7	21.0	76	13	89

The testwork in the HZ confirmed the high-GRG nature of the mineralisation (61–76%), but with lower values than previously. The PZ testwork indicated low GRG (<15%), with most of the gold being sub-100 μm and dominantly sulphide-hosted. Flotation testwork on the gravity tails indicated that an additional 13–26% of gold could be recovered in a pyrite concentrate depending on feed grade and relative proportion of PZ ore. After initial grinding to P_{100} −850 μm for the GRG test, 5 kg of sub-sample was split off by rotary sample splitter from each sample. The sub-sample from the 150 kg primary sample yielded, at the 90% reliability, FSE values of ±6% and ±10% for the PZ and HZ respectively. Each sample was then fed to a micro-panner and the concentrate isolated. The tails were re-ground to P_{90} −100 μm and fed through the micro-panner and the concentrate isolated. Both concentrates were set in resin and inspected by both optical and automated mineralogy. In the PZ, studies confirmed the deportment of gold (20–150 μm and rarely up to 500 μm in size) within pyrite.

11.5.6. Pilot Programme

Based on the new testwork results, seven one-tonne master variability-composite samples were collected underground for pilot testwork. These allowed for the relative proportions of HZ and PZ mineralisation across very-low grade, low-grade/cut-off grade, ROM grade, ROM high-grade and very-high grade ore (Table 20).

Samples were collected and composited from various locations to provide the required sample grade. All samples were collected from pre-mapped and channel sampled drive backs using hand and compressed air pick(s). In a few cases, very small explosive charges were used to pre-split the rock prior to sampling. Broken material was collected on the floor by tarpaulins underlain by rubber matting. This material was subsequently placed into cut down drums designed to contain 150 kg of sample, which were transported out of the mine on rail wagons. At surface the drums were emptied into purpose-built plastic-lined wooden crates, each designed to contain 750 kg. The crates were placed into a secure shipping container for transport to the laboratory.

Table 20. Summary of pilot plant testwork results.

Sample	Domain	Domain Fraction	Grade Type	Head Grade (g/t Au)	GRG (Au %)	Flotation Recovery (Au %)	Total Recovery (Au %)
PVC.1	PZ	1.0	Very low	1.9	5	49	53
PVC.2	PZ	1.0	Low/COG	5.3	18	70	88
PVC.3A	HZ/PZ	0.3/0.7	ROM	11.0	64	29	93
PVC.3B	HZ/PZ	0.4/0.6	ROM	13.2	66	24	90
PVC.5	HZ/PZ	0.5/0.5	ROM high	23.4	73	21	94
PVC.5	HZ/PZ	0.8/0.2	ROM high	21.6	77	18	95
PVC.6B	HZ	1.0	Very high	32.9	82	14	96

The pilot plant was designed to mirror the production plant across crushing, grinding and screening, primary and secondary Knelson units, followed by tailings grinding and flotation. The flotation sulphide concentrates were intensive leached to recover the gold.

11.5.7. Quality Assurance/Quality Control

All bench- and pilot-scale samples were sent to an independent accredited laboratory. The laboratory was audited by both company staff and its consultants. It instigated a QAQC programme throughout testwork and associated assaying (Table 21). Sample preparation and testwork equipment was cleaned between every sample.

Table 21. Summary of corrective programme QAQC.

Action/Activity	Rate/Responsibility	Performance Expectation	Actual Performance
Sample security: chain of custody	Project and laboratory	Full compliance	No security breaches No seals broken or samples lost
Duplicates	See next Section 11.5.8		
Certified reference material (CRM): Tails and concentrate assays from GRG and flotation testwork	1 in 5 Project and laboratory	Limits: ±2δ–3δ (“warning”) >±3δ (“action”) Relative bias: within ± 10%	Within limits: 95% of CRM results < ± 2δ 5% of CRM results ± 2δ–3δ 0% of CRM results > ± 3δ Relative bias: Low CRM (2.9 g/t Au) + 5.5% Moderate CRM (8.7 g/t Au) + 3.1% High CRM (24.4g/t Au) + 7.9%
Blanks	1 in 5 Project and laboratory	Blank assay <0.1 g/t Au	98% <0.1 g/t Au
Barren “sand” flush of GRG circuit for all samples	1 in 10 Laboratory	<1% gold loss in blank material compared to sample head grade	100% <1% gold loss
Laboratory audit	Project	Full compliance across all procedures	A number of minor non-material issues were noted. All matters resolved

11.5.8. Duplicate Sample Analysis of Underground Panel Programme

Duplicate sample analysis across the sampling and laboratory process allows errors to be apportioned to each component of activity [21,48]. The results demonstrate the dominance of sampling error (component error 38%), which provides 78% of the total variability (Table 22). The preparation and analytical component errors are within expectation (Table 14). The theoretical sample mass of 150 kg (refer Section 11.5.2.) is essentially validated, where it was optimised to achieve ±20% precision. The sampling error calculated includes the collection errors (e.g., DE and EE), whereas the theoretical sample mass value only reflects in-situ variability (e.g., the GNE).

It is noted that the field and coarse duplicate data are relatively small. Both data sets represent the grade frequency distribution, reflecting 80% of the population above 1 g/t Au. Therefore their application to error analysis is deemed appropriate.

Table 22. Evaluation of errors across the underground-panel metallurgical-sample testwork programme based on duplicate samples. All FSE values reported at 90% reliability. Component relative error calculated via method presented in [14].

Duplicate Type	Explanation	Mass	Number (Frequency)	Estimated Stage FSE	Component Error	Component Relative Error	Proportion of Total
Field	Panel samples collected at the same location	150 kg	15 (1 in 4)	-	Sampling	38%	78%
Coarse	Panel samples crushed to P_{90} −1.5 mm and rotary sample divider split	75 kg	15 (1 in 4)	<±15%	Preparation	18%	18%
Laboratory pulp 1: concentrate	GRG concentrate split prior to intensive leach	30 g	58 (1 in 1)	<±5%	Analytical	4%	1%
Laboratory pulp 2: tailings	Core and panel sample tails after GRG stage-3 at P_{90} −100 μm rotary sample divider split	10 kg	58 (1 in 1)	<±5%	Analytical	8%	3%
-	-	-	-	<±15%	Total	43%	100%

In reality the total error is closer to 39%, since during testwork the entire sample was processed through the GRG protocol, therefore the preparation error component is close to zero. Some minor error may be imparted through gold contamination or loss, though the QAQC programme records this at a minimal level (Table 21). The component errors are sampling at 38% and total analytical at 9%. In this case the sampling error represents 95% of the total variability.

11.5.9. Sampling Validation and Ore Characteristics

The pilot study results were used to validate the sampling strategy, providing characterisation data across the domains (Table 23). The estimated d_{95Au} values were established from size-by-assay of the gravity concentrates. The results validate the practical choice of 150 kg samples to support the new sampling and testwork campaign. For higher grades, the precision values for ROM high- and very-high grades are ±28–31%, which is not unreasonable.

Table 23. Summary of sampling characteristics based on pilot plant results.

Sample	Head Grade (g/t Au)	Est. d_{95Au} (μm)	Theoretical Mass (kg)	Theoretical Precision Based on 150 kg Sample
PVC.1	1.9	120	15	±1%
PVC.2	5.3	250	25	±3%
PVC.3A	11.0	1000	140	±14%
PVC.3B	13.2	1100	155	±16%
PVC.4	23.4	2000	530	±28%
PVC.5	21.6	2100	660	±31%
PVC.6B	32.9	2300	570	±29%

The grade RSV values for the HZ and PZ domains are 46% and 32% respectively (Table 18), where the RSV includes all sampling errors (Table 3). The overall results are deemed to be acceptable. In addition, the duplicate sample data also provide validation of the programme (Table 22).

11.6. Communicating Sample Representativity, Testwork Quality and Fit-for-Purpose Results

It is critical that sample representativity, testwork quality and overall fit-for-purpose application of results are communicated to stakeholders. In many cases, results will be publically released requiring transparent and material reporting. Tables 24 and 25 provide an overview of representativity and fit-for-purpose nature of testwork results for the case study, for both the original and corrective programmes.

Table 24. Risk review of metallurgical sampling and testwork for case study original programme.

	Key Parameter	Comment	Material Errors	Risk Rating
1	Spatial distribution and number of samples	Samples biased to spatially restricted (clustered) areas and high grades Too few samples. Only 6 in total	GNE	High
2	Sample mass	Samples too small compared to theoretical mass	GNE	High
3	Degree of domaining	HZ and PZ defined, but unrepresentative HZ domain interpretation incorrect	GNE	Moderate
4	Collection and handling	Original core samples moderate-poor quality based on core recoveries in the 60–80% range No details on how underground samples were collected No written protocols to comply with TOS All samples placed in plastic sample bags with wire ties	DE, EE, PE	Moderate
5	Transport and security	Bags placed in unsecured wooden boxes on the back of an open truck and transported to the laboratory No chain of custody recorded	PE	Moderate
6	Preparation	Composite blending via shovelling Sub-sample splitting sub-optimal	FSE, GSE, DE, EE, PE	High
7	Testwork (incl. QAQC)	Potential for some contamination between samples due to poor laboratory practice No QAQC Non accredited laboratory	PE	Moderate
8	Assay (incl. QAQC)	No issues with assay procedure used No QAQC Non accredited laboratory	AE	Moderate
9	Validation	Via plant, poor reconciliation Grade reconciliation in the −60% to −75% range on a monthly basis Recovery 50% of that predicted on a monthly basis	-	High
			Sample representativity (1)–(5)	Low/poor
			Testwork-measurement quality (6)–(8)	Moderate
			Fit-for-purpose rating (1)–(9)	Low/poor

Table 25. Risk review of metallurgical sampling and testwork for case study corrective work programme. Refer to Tables 21 and 22 for QAQC performance.

	Key Parameter	Comment	Material Error	Risk Rating
1	Spatial distribution and number of samples	Even spatial resolution along development drives More samples across domains. 58 panel variability samples, six core composite and seven pilot variability composites	-	Low
2	Sample mass	Optimised to theoretical mass. Dominance of 150 kg samples, supported by 1 t pilot samples	-	Low
3	Degree of domaining	Refined HZ and PZ domains	-	Low
4	Collection and handling	All samples collected according to protocols written to comply with TOS All samples placed in steel drums and sealed prior to transportation	-	Low
5	Transport and security	Chain of custody procedures in place All sample drums secured into locked container for transportation	-	Low
6	Preparation	Composites blended via rotary sample splitter All splitting optimised to FSE All equipment cleaned between samples	-	Low
7	Testwork (incl. QAQC)	Good QAQC, with documentation across sample collection, preparation and testwork Rigorous cleaning of laboratory equipment No contamination Accredited laboratory	-	Low
8	Assay (incl. QAQC)	Good QAQC, with full documentation across sample collection, preparation and assaying Rigorous cleaning of laboratory equipment Accredited laboratory No contamination Acceptable analytical error and CRM relative bias	-	Low
9	Validation	Pilot programme verified testwork Ultimately optimised plant acceptable reconciliation, where grade reconciliation in the ±15% range and recovery as predicted on a monthly basis	-	Low
			Sample representativity (1)–(5)	High/good
			Testwork-measurement quality (6)–(8)	High/good
			Fit-for-purpose rating (1)–(9)	High/good

Parameters (1) to (5) provide a measure of sample representativity and parameters (6) to (8) the quality of the testwork. A rating of overall programme fit-for-purpose is given. Any activity that is deemed as low risk is clearly acceptable and contributes to the overall ratings. In the context of the case study, Table 24 indicates the high risk and overall low quality of the entire original programme. The corrective programme indicates a low risk throughout (Table 25).

11.7. Conclusions

Key issues related to:

- Poor liaison between geologists and metallurgists for sampling programme design.
- Poor geological interpretation and over-reliance on an old model.
- Too few samples collected.
- Unrepresentative (too small) sample composites and sub-samples.
- Poor laboratory practice during preparation and testwork.
- Lack of documentation and QAQC

A geological reinterpretation of the Main Reef was undertaken to account for the nature of the HZ sub-shoots. A revised Indicated Mineral Resource estimate totalled 200,000 t at 11 g/t Au (±20% at 80% reliability) representing the first 2 years of production. An Inferred Mineral Resource of 400,000 t at 15 g/t Au (±42% at 80% reliability) provided a future base for the project. A new flotation circuit was added, preceded by a grinding circuit. After further controls on mining, head grades increased to around 10 g/t Au and recoveries up to 90% (gravity and flotation combined; up to 75% and 68% respectively dependent upon feed grade). Subsequent optimisation of mining, stockpile blending and processing has led to an increased head grade of 12.5 g/t Au and recovery to 94%. As of the current date (April 2018), the operation has stabilised at 120,000 t per annum production to yield around 1400 kg gold (45,300 ounces gold). On-going diamond drilling and underground development incrementally increases the resource base each year.

These issues led to disruption over 18 months and are estimated to have cost the company around US$ 36M in lost gold sales (US$ 31M) and corrective expenditure (US$ 5M) during 2013–2014. Intangible losses related to company and staff reputation, particularly with the workforce, local community, government regulators and investors, and reduced project value (estimated to be around 60% in 2013).

12. Public Reporting of Metallurgical Sampling and Testwork Programmes

The public reporting of metallurgical sample and testwork results are as important as grade results, but frequently receive less rigor. The 2012 JORC Code [26] has global application for the reporting of exploration results, resources and reserves. It requires the Competent Person(s) to provide commentary on any sampling and testwork/assaying process. From a metallurgical perspective, Table 1 Section 4 of The Code requires disclosure as part of reporting Ore Reserves (Table 26).

Table 26. Extract from The 2012 JORC Code Table 1 (Section 4) pertaining to metallurgical testwork [26].

Metallurgical factors or assumptions	**1**	The metallurgical process proposed and the appropriateness of that process to the style of mineralisation
	2	Whether the metallurgical process is well-tested technology or novel in nature
	3	The nature, amount and representativeness of metallurgical test work undertaken, the nature of the metallurgical domaining applied and the corresponding metallurgical recovery factors applied
	4	Any assumptions or allowances made for deleterious elements
	5	The existence of any bulk sample or pilot scale test work and the degree to which such samples are considered representative of the orebody as a whole
	6	For minerals that are defined by a specification, has the ore reserve estimation been based on the appropriate mineralogy to meet the specifications?

Items (3) and (5) in Table 26 are the most relevant to metallurgical sampling and testwork programmes. Item (3) looks for clarification around the "what, where and how" of sampling, whereas (5) focusses on validation via bulk sampling and/or pilot work. Both look for some discussion on representativity. The general format presented in Tables 24 and 25 provide a framework in which to communicate a number of The 2012 JORC Code Items (e.g., 3 and 5).

Within Canada, the CIM Definition Standard [101] is referenced in context of National Instrument 43-101 (NI 43-101) [27], which in turn references best practice guidelines [102]. Like The 2012 JORC Code, reporting within the NI43-101 framework requires disclosure of sample selection and testwork (Table 27).

Table 27. Extract from NI43-101 Item 13 pertaining to metallurgical testwork (from [27]).

Mineral processing and metallurgical testing	(a)	the nature and extent of the testing and analytical procedures, and provide a summary of the relevant results
	(b)	the basis for any assumptions or predictions regarding recovery estimates
	(c)	to the extent known, the degree to which the test samples are representative of the various types and styles of mineralisation and the mineral deposit as a whole
	(d)	to the extent known, any processing factors or deleterious elements that could have a significant effect on potential economic extraction

In NI 43-101, Items 13(a) and 13(c) are the most relevant to this discourse, again requiring disclosure with respect of sample representativity and testwork quality.

Both The 2012 JORC Code [26] Section 4 Item 3 and NI43-101 [27] Item 13(c) require specific comment on the representativity of metallurgical samples. Project study reports often fail to detail the spatial extent of metallurgical sampling, or the number of composites collected and their relative masses. It is not unusual for a feasibility study report to state; "the sampling and testwork programmes have been extensive and due care was taken in selecting and compositing representative samples" or "the testwork samples were deemed to be representative" with little or no justification as to why samples are considered representative.

The 2012 JORC Code [26] Table 1 Section 1 (Sampling techniques and data) is aimed at the geologist and ultimate resource model. However, it covers disclosure on key matters such as: sampling techniques; sub-sampling techniques and sample preparation; quality of assay and laboratory tests; verification sampling and assaying; location of data points; data spacing and distribution; orientation of data in relation to geological structure; sample security; and audits or reviews. These criteria are relevant to metallurgical sampling programmes. The authors contend that the Table 1 Section 1 criteria should be used to support Table 1 Section 4 criteria, particularly Items 3 and 5.

13. Driving Representativity: Design of a Sampling and Testwork Programme

A truly representative sample generally does not exist, given that we are trying to sample extensive and often complex geological entities. The GNE impacts in-ground sample representativity, where representativity in low-GNE mineralisation is likely to be high, whereas in high-GNE mineralisation representativity is likely low and may require pilot or bulk sampling. Practitioners can make substantive progress into making samples more representative and reduce project risk by good planning, realistic implementation of TOS guidelines and evaluation of the data produced. The design and execution of a programme can be based on five steps to achieve representative samples and fit-for-purpose testwork results (Table 28).

Table 28. Summary of steps to achieve optimal metallurgical sampling programmes (modified from [37]).

	Steps/Key Activity
1: Scope	• Present business case to support introduction or improvement • Define data quality objectives and what is required to ensure that results will be fit-for-purpose • Understand deposit geology, grade and mineralogical distribution (as appropriate for project stage) • Undertake a characterisation programme to determine preliminary metallurgy, gold particle size distribution metrics and domains
2: Develop	• Select a sample strategy based on deposit characteristics and heterogeneity • Consider sampling method(s) • Ensure that sample collection, testwork and assaying protocols are designed with reference to the expected ore type(s) • Ensure TOS and QAQC are applied • Document within the framework of DS3077
3: Execute	• Ensure that staff are properly equipped and trained, with a focus on reducing sampling errors and increasing sample quality • Ensure regular staff supervision during sample collection/compositing • Ensure measures to reduce risk of sample tampering and enforce chain-of-custody protocol
4: Validate	• Ensure regular review and auditing of sampling strategy • QC analysis and action • Reconciliation with pilot or demo plant testing, or production (if appropriate) • Review sample representativity and confirm that data are fit-for-purpose
5: Refine	• Update as required and/or return to stages 2 and 3 for further work

14. Conclusions

(1) Correctly collected and prepared metallurgical samples to support testwork are critical to effective gold processing plant design. There is often a paucity of material for sampling and often only half-core from resource drilling. Not only is sample material required for recovery testwork, but also for comminution and geoenvironmental testwork. Metallurgical sampling programmes should be integrated into strategic geometallurgical studies to ensure maximisation of data usage and better decision making. Early stage consideration of metallurgical sampling and testwork will lead to better decision-making with respect of resource delineation and development, more focused metallurgical studies as a project develops and ultimately optimised shareholder value.

(2) During programme scoping, a sampling for sampling step should be undertaken to permit preliminary characterisation. Any in-situ coarse to very-coarse fraction (or similarly gold-bearing sulphides) will be the most material to testwork result quality. Reviewing geological and grade information as early as possible helps determine the likely heterogeneity (e.g., degree of nugget effect) and potential domains. Multi-disciplinary core logging will go a long way to resolve domains and their variability in consideration of (2) above.

(3) Representativity is based on appropriate sample mass to improve precision and sampling protocols to reduce bias. An estimate of theoretical sample mass can be defined by the application of Poisson statistics, with a target precision of ±20% at 90% reliability. Subsequent sub-sampling for testwork can be controlled via the FSE equation with a target precision of ±15% at 90% reliability. The selection of precision and reliability levels depend on mineralogical assessment and practitioner judgement.

(4) All samples must be collected within the framework of TOS. Where broken rock samples (e.g., crushed material) are used, the FSE equation can be applied. Large diameter diamond drill core provides the best quality samples on which to undertake testwork. The traditional paradigm is to form metallurgical composites from quarter resource evaluation core. Improved core logging, digital photography and data collection methods now provide an opportunity to use half or whole core samples for metallurgical testwork. For high-GNE mineralisation, multiple large diamond drill core and/or bulk samples may be the most appropriate.

(5) During testwork, laboratory crushing, grinding, splitting and blending should be optimised in the light of TOS. Reduction of sample loss and contamination is paramount, and should be controlled via appropriate hygiene procedures. Testwork errors must be minimised. Ensure that testwork sub-samples are representative of the original sample. Particular attention is required across: equipment maintenance and operating parameters, cleaning of equipment and associated pipework, and reducing careless work practices.
(6) Testwork must be accompanied by mineralogical studies which play a critical role in optimising gold recovery processes. Mineralogy is widely applied in the characterisation of gold ores and mill products, and to determine potential issue(s) that may cause gold losses. The information acquired can be used as a basis for metallurgical testwork programme design or optimisation. Ideally, a mineralogical study should be conducted prior to the start of, or at the early stage of a testwork programme.
(7) A QAQC programme must be introduced to ensure on-going quality control of sampling and testwork. The association of QAQC with TOS is unequivocal. A key part of QAQC is documentation and training. Written protocols and procedures, staff training, periodic auditing of protocols and people, and re-training are required. The new DS3077 provides a framework on which to produce transparent protocols [53]. Control is through the examination of laboratory and audit procedures results, and adherence to standard or industry-accepted operating procedures. Samples must be handled to ensure their integrity and security. A chain-of-custody must be enacted.
(8) Measures are recommended to reduce risk of the tampering of samples. These include: maintaining increased security between the sample site (e.g., exposure and/or drill rig) and sample shipment; recording who has access to samples between collection and shipping, and maintaining a secure copy of that record through the project life; and employing an outside agency with no vested interest in the project, to maintain custody and security over samples.
(9) Technical teams should consider bulk sampling to support pilot or trial testing programmes to evaluate grade and metallurgy as part of pre-feasibility or feasibility studies. This may be particularly important in high GNE mineralisation or where high variability relates to mineralogical and/or textural complexities that impact on recovery.
(10) Early collaboration across technical disciplines is required to design and implement metallurgical sampling and testwork programmes. As a multi-disciplinary approach, geometallurgy emphasises technical collaboration and is gradually producing more dual discipline professionals. Discipline sharing should be considered a key development for the future of the mining industry.
(11) There is now a need to move towards proper quantification of sampling and analytical errors. A first step is the application of the RSV defined in DS3077 [53]. Beyond this, resolution of component relative errors across sampling, preparation and analysis can be gained from duplicate sample pairs [21,47,48]. Measurement uncertainty analysis also provides value in this quest [21,50,55,103].

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