Improved Reliability of Resource Estimates Based on Assay Quality Control

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ABSTRACT

All resource models depend on assays and an assumption that representative subsamples were assayed accurately. Resource estimation, for gold projects in particular, requires an understanding of routine sample preparation and assay methods.

Assay quality control programs have become near ubiquitous since NI43-101 reports were introduced nearly 20 years ago. There are no specific rules for the rate of insertion, actions required for quality control failures and other questions that frequently arise. Statistics from studies of publicly available NI43-101 reports will be presented that have helped Analytical Solutions Ltd. develop guidelines for clients to achieve "industry standard" compliance.

Achieving a representative sample for assay is a significant challenge for some gold projects. Part of the challenge is balancing the cost of sample preparation and maximizing the probability of achieving a representative sample. Justifying higher costs for sample preparation should be based on duplicate data and the correct estimation of precision. The industry uses a wide range of statistics to estimate precision; this has caused confusion about optimum results.

The preferred methods of calculating precision using the coefficient of variation and visualization of the data are presented.

Key Words: Assays, sampling, precision, duplicates

INTRODUCTION

In 1999 Canadian regulators introduced National Instrument 431-101 to define the preparation of technical reports for mining projects. Other jurisdictions have similar codes such as Australia (JORC), South Africa (SAMREC) and an international code, Committee for Reserves International Reporting Standards ("CRIRSCO"), is expected to be completed shortly. Beginning in 2021, the SEC will require disclosure for U.S. mining properties consistent with global standards.

All of these systems include a requirement to report on assay quality control programs. They all mention the use of reference materials (standards), blanks and duplicates but none provide specific instructions. Regulations only state that quality control and assay methods are "appropriate" and "justified." Published best practices provide general guidelines but none specify rates of insertion, required precision, use of accredited laboratories or other specifics.

Geologists and project managers are required to design,

implement and document sampling, assaying and quality control methods that will manage the project risk as well as satisfy regulatory disclosure.

Over the last 20 years there has been wide adoption of assay quality control programs. To monitor industry norms, Analytical Solutions Ltd. reviewed over 100 NI43-101 reports with a focus on technical reports that included drilling activity. Insertion rates of blanks and reference materials were compiled in both 2014 and 2018 (Figure 1).

There was little change from 2014–2018. In both surveys, companies have mostly included reference materials and blanks at a rate of one in 20 or one in 25. About 5% of the technical reports did not include any information about quality control programs. Where the technical report was a feasibility study, reporting on the quality control programs may have been included in previous technical reports where there was more emphasis on disclosure about mineral resource estimation. In 3% of cases, the only quality control information discussed was the laboratory's internal quality control.

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Figure 1. Insertion Rates for Reference Materials From NI-43-101 Reports.

In 2015, the Ontario Securities Commission conducted a survey of NI43-101 technical reports and identified items that were common disclosure issues. Assay quality control procedures not disclosed as required was the tenth item listed. In addition, the Ontario Securities Commission provided new guidance that "If data integrity and geological and grade continuity are good but QA/QC is poor the QP [Qualified Person] can downgrade a measured or indicated resource to an inferred resource but the QP must still take steps necessary to verify that the data meets the requirements of an inferred resource."

ASSAY QUALITY CONTROL IN PRACTICE

The basics of inserting blanks and reference materials are well established for most mineral exploration projects. The subtleties associated with matrix-matched materials, how to define quality control failures and required actions for failures are not as well defined and often vary between operators. Differences occur based on the deposit type, deportment of the ore minerals and a company's risk tolerance.

There has been less attention paid to monitoring of precision. Guidelines state that geologists must ensure that samples are "representative". The term is not defined and no quality expectations are provided. It is just as well since every commodity and deposit type will have different expectations.

A quality control program can monitor the precision of duplicates to understand how sample preparation and assaying procedures impact whether a sample is representative.

Samples are processed in different ways but the example for drill core is informative. First, drill core is generally sawn in half. A 3–6 kilogram drill core sample is then crushed and a portion is split to be pulverized. The pulverized sample, or pulp, is then sub-sampled for assaying. There are a number of criteria that will impact whether the final assay represents the metal concentration in the original drill core sample including sample size, grain size specification for crushing and pulverizing, splitting method, weight of the split, weight of the analytical aliquot and analytical method.

At each stage of the process, when samples are subdivided, there is an introduction of uncertainty. It is easier to achieve a representative sample when particle size is reduced and many sub-samples are recombined. Thus if a sample is crushed to 90% passing 1 mm, the sample split is more likely to be representative of the entire sample than if the sample is crushed to a specification of 75% passing 2 mm.

Similarly if a sample is passed through a riffle splitter with 30 chutes, the sample split is more likely to be representative of the entire sample than if the splitter has 16 chutes. Although only reported in one incident, there was a case where the number of chutes in the riffle splitter was an odd number. Obviously, to achieve a 50:50 split the number of chutes needs to be an even number.

The proportion of the material split is also a consideration. A one-meter length of split HQ core can weigh 3 kilograms. If 250 grams is sub-sampled, then less than 10% of the sample is pulverized. The other extreme would be if a 0.3 m length of HQ core is crushed and sub-sampled; then a 1,000 gram split that is pulverized would be almost the entire sample.

To measure the impact of different preparation and analytical procedures, samples are analyzed in duplicate to assess the magnitude of relative error. Another term used to describe the measurement of differences between sample pairs is precision. Precision is used to describe the closeness of the measurements to each other.

Precision includes:

- *Repeatability*—the variation arising when all efforts are made to keep conditions constant by using the same instrument and operator, and repeating during a short time period; and
- *Reproducibility*—the variation arising using the same measurement process among different instruments and operators, and over longer time periods.

Precision is usually described in terms of the probability that a result can be reproduced. The statistics are reported as a function of two standard deviations. Based on a normal distribution, two standard deviations from the mean should include 96% of the results.

There is a wide variety of calculations performed to communicate precision in the mineral exploration industry. Stanley and Lawie (2007) summarized many of the statistics that are used in the literature and reports (Table 1). Table 1 shows the relationship of these statistics with respect to the Coefficient of Variation.

Stanley and Lawie recommended the use of the coefficient of variation (CV) but warned it provides poor estimates of relative error and large data sets are recommended if the average relative error is large. The use of CV to estimate precision is more robust than the other calculations in Table 1 and less prone

Measurement	Conceptual	Single Duplicate	Average Formula for	R elationship
	Formula	Pair Formula	Several Duplicate Pairs	with CV
Coefficient of Variation	$CV = \frac{\sigma}{\mu}$	$CV = \frac{2}{\sqrt{2}} \frac{ x_1 - x_2 }{(x_1 + x_2)}$	$\overline{CV} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} \left(\frac{2}{\sqrt{2}} \frac{ x_{1i} - x_{2i} }{(x_{1i} + x_{2i})}\right)^2}$	CV
Relative Precision	$RP = \frac{2\sigma}{\mu}$	$RP = \frac{4}{\sqrt{2}} \frac{ x_1 - x_2 }{(x_1 + x_2)}$	$\overline{RP} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} \left(\frac{4}{\sqrt{2}} \frac{ x_{1i} - x_{2i} }{(x_{1i} + x_{2i})}\right)^2}$	$2 \times CV$
Relative Variance	$RV = \frac{\sigma^2}{\mu^2}$	$RV = 2\frac{(x_1 - x_2)^2}{(x_1 + x_2)^2}$	$\overline{RV} = \frac{1}{n} \sum_{i=1}^{n} \left(2 \frac{(x_{1i} - x_{2i})^2}{(x_{1i} + x_{2i})^2} \right)$	CV ²
Absolute Relative Difference	$ARD = \frac{ x_1 - x_2 }{\mu}$	$ARD = 2\frac{ x_1 - x_2 }{(x_1 + x_2)}$	$\overline{ARD} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} \left(2 \frac{ x_{1i} - x_{2i} }{(x_{1i} + x_{2i})} \right)^2}$	$\sqrt{2} \times CV$
Half Absolute Relative Difference	$HARD = \frac{1}{2} \frac{ x_1 - x_2 }{\mu}$	$HARD = \frac{ x_1 - x_2 }{(x_1 + x_2)}$	$\overline{HARD} = \sqrt{\frac{1}{n} \sum_{i=1}^{n} \left(\frac{ x_{1i} - x_{2i} }{(x_{1i} + x_{2i})} \right)^2}$	$\frac{\sqrt{2}}{2} \times CV$

Table 1. RELATIONSHIP OF THESE STATISTICS WITH RESPECT TO THE COEFFICIENT OF VARIATION.

Notes x1, x2 = duplicate pair results; μ = duplicate mean; σ = duplicate standard deviation; CV = coefficient of variation (σ/μ ; mean/standard deviation); n = number of pairs of duplicate samples (referenced with index i). Stanley and Lawie, 2007.

to overestimation using alternative approaches like Thompson and Howarth (1973; 1978).

A small number is indicative of a high probability that results can be reproduced, whereas a high value for precision shows that the result is not as reliable. As an example, consider a case where the percent coefficient of variation (CV%) is stated as 5% and the assay is reported as 10 g/t gold. You could then state "I have confidence that 96% of the time, when the same sample is assayed, the gold results will vary between 9 to 11 g/t gold". The CV% is doubled to reflect that the two standard deviation precision is presented. And the result is expected to be confirmed within \pm 10% only 96% of the time since \pm two standard deviations represents 96% of a normal distribution.

Another important factor in assessing precision for analytical results is that it changes depending on concentration. As the detection limit of the method is approached, precision will increase asymptotically. This applies to all analytical methods and measurement systems.

The major commercial laboratories provide guidance on allowed tolerances for different analytical methods. Figure 2 shows how the precision increases as gold concentrations decrease. Ideally, samples would have gold concentrations where the precision is optimized, generally about 50 times the detection limit.

As the precision is expected to change with concentration, any declaration of sample precision should be respectful of the limitations of the analytical method. Figure 3 is a CV% plot vs. gold concentration for laboratory duplicates analyzed by fire assay for a large project with 4,500 pairs of data. The calculated precision using all of the duplicates pairs is 28% which implies that assays are only likely to repeat on pulps within \pm 28% for 96% of samples. However, when the average CV% is calculated for different grade ranges, it becomes clear that one can have far more confidence in the assays that are likely to impact resource estimation. As expected, precision is poor at 31% for gold assays less than 0.5 g/t gold. Confidence in the assays improves to 9% precision for samples with more than 2 g/t gold. Most commercial laborato-



Figure 2. Change of Precision with Gold Grade and Method Detection Limit.

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Figure 3. Precision Estimates Determined by CV% for Pulp Duplicates.

ries acknowledge that the fire assay method is not expected to report pulp duplicates to within better than 7 to 10% for concentrations at least 50 times detection limits.

Calculation of precision using the coefficient of variation can be skewed by a few cases where results were poor. It can be useful to visualize the repeatability of the assays using a Relative Percent Difference (RPD) graph and check sample distributions against the calculated precision.

The Relative Percent Difference can be calculated as:

[Original Assay – Duplicate Assay]/[Average] × 100

Figure 4 shows the RPD plotted against the average of the assays for 1,565 laboratory duplicates on the same pulp. As expected, the RPD increases as the detection limit is approached. The calculated precision, using CV% for gold between 0.1–1 g/t, was 22%. The graph shows samples within \pm 22% in this grade range; 94% of the samples fall within \pm 22%. Similarly, 90% of the samples with gold grades between 1 to 5 g/t gold, fall within \pm 14%, which is the calculated precision.

The sample distributions are consistent with the estimated precision and provide confirmation that the CV% method was appropriate for the data set.



Figure 4. Relative Percent Difference Graph for Pulp Duplicates.

CONCLUSIONS

The mineral exploration industry has adapted well to regulations introduced 20 years ago that pertain to assay quality control. Whereas monitoring of accuracy has been rigorously adopted, there are still opportunities to improve the use of duplicate assay data.

Duplicate data for pulps and splits of the coarse crush material are readily available from commercial laboratories which use the information for internal quality control. Mine laboratories can often provide pulp duplicate assays but are less likely to have preparation duplicates assayed routinely. Other types of duplicate data, such as field duplicates, blast hole duplicates and check assays, can be evaluated using the techniques described.

Several aspects of interpreting, calculating and presenting

precision data have been discussed. These data can be used effectively to understand the risks associated with using assays for resource estimation, grade control and other decisions. Precision information can justify changes to sample preparation and analytical procedures to achieve the desired precision at grade ranges where important decisions are made.

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