



A COMPARISON OF PULVERISED GOLD CERTIFIED REFERENCE MATERIALS IN 2023

The insights laboratories need to achieve results they can trust

Certified Reference Materials (CRMs) are a critical part of the quality control process in any mining laboratory.

As you might expect, each CRM on the market promises consistent and repeatable results. But, how do you know your CRM will actually deliver results you can trust time and time again?

When it comes to Pulverised Gold CRMs, the comparisons have already been made and the results are in.

Not all gold CRMs are equal.



LET'S START WITH HOMOGENEITY AND HOW ITS MEASURED

When it comes to the reliability of one gold CRM over another, homogeneity is one of the most important properties to consider.

Why?

Because a CRM with the lowest possible variance in homogeneity will enable the quality control manager to better evaluate the variance, or level of precision, in their analytical process.

Homogeneity can be assessed throughout a batch of CRM by replicate analysis from multiple samples collected, and then evaluating the results.

The challenge, however, when designing experiments to measure the inherent variance of certified reference materials, is to distinguish the variance in the reference material from the uncertainty of the analytical method itself.

THE ROLE OF RELATIVE STANDARD DEVIATION WHEN COMPARING CRMS

One of the most common ways of assessing, and hence comparing, the homogeneity of CRMs is to determine the relative standard deviation (RSD) from replicate analysis. The more homogeneous the CRM, the lower the RSD. The RSD between repeat analysis will also be dependent on the mass used for the analysis.

When analysing for gold in minerals and ores, the concentration levels are often at unit parts per million, or even at parts per billion. For this reason, analysis using large mass samples provides for sufficient detection of gold during the analysis. The traditional technique for determining gold content in ores and minerals is fire assay. Fire assay is a concentration method via a collection and cupellation process enabling precise measurements of the gold concentration to be made at trace levels

But there's an even more important reason why high mass samples are used when analysing for low level gold.

Gold is often present in samples at a high particle size and large mass samples are required to ensure the analysis is representative. Fire assay is typically 25-50g, and photon assay and PAL is up to 500g. Cyanide leaching and bottle roll techniques can be a few kilograms.

This 'nugget effect' can be problematic as it will cause sampling errors during sample splitting, particularly when the final aliquot for analysis is taken at the end of the sample preparation process.

It follows, therefore, for a CRM to be considered fit for purpose -and homogeneous – that it should not contain any nugget of gold, but rather, the gold should be finely dispersed throughout the batch, ideally at the microscopic level.

USING HOMOGENEITY TO COMPARE CRMS

A study comparing the homogeneity of CRMs from various manufacturers was conducted in 2015- Brand (2015).

The study involved the analysis of a number of gold CRMs using neutron activation. Neutron activation is a highly precise assay method that can utilise small sample masses.

In the study a 1g mass aliquot was analysed with the RSD calculated from repeat analyses. In addition, using sampling theory equations, the minimum sample mass required to measure gold in each CRM and achieve an RSD of 1% was also calculated. This is referred to as the sampling constant from Ingamells and Switzer (1973). It allows for a direct comparison to be made between CRMs regardless of grade.

The more homogeneous the CRM is, the lower the sampling constant. Ideally the sampling constant should be significantly lower than the mass used in the method. If the sampling constant is greater than the method mass, then the gold in the CRM is considered to be 'nuggety'.



INTRODUCING A NEW COMPARISON FOR 2023

Back in 2015, CRMs from IMS weren't available and therefore not included in the study.

So in 2023, we decided to run a similar investigation using our Pulverised Gold CRMs to see how it would have stacked up against the competition.

We conducted a 1g Instrumental Neutron Activation Analysis (INAA) study from three production batches of Pulverised Gold Certified Reference Materials manufactured by IMS with 28 x 10g samples randomly extracted from throughout each batch. All of the batches selected are available to the market.

A single analysis was conducted for each 10g sample by INAA at a 1g mass. The analysis was performed at Bureau Veritas Canada.

Calculations were made in this study for the following:

- Homogeneity expressed as percent relative standard deviation (%RSD) at 1g.
- Mass at which the RSD is 1% (sampling constant in g).
- Homogeneity RSD extrapolated to 30g and 50g.

As a standardised method of dispersion of the gold throughout the batch, independently of grade, the RSD indicates the level of precision or repeatability of the analysis. The lower the RSD, the more homogeneous the CRM, and vice-versa.

The sampling constant (in g), is the mass of sample to use for the analysis, where the RSD would be 1%. The higher the sampling constant, the more unreliable the CRM is for analysis, and the greater the contribution from inhomogeneity to uncertainty in the results

SO HOW DID IMS COMPARE?

We've added our results to those published by Brand (2015). As you can see, this demonstrates the homogeneity provided by IMS Pulverised Gold CRMs is more than a match for other industry suppliers.

	Mean RSD (%)	Mean Sampling Constant (g)	Based on 1g INAA data and the sampling constant	
Manufacturer			30g 2RSD (%)	50g 2RSD (%)
Manufacturer A	1.61%	3	0.60	0.47
IMS	2.17%	5	0.79	0.61
Manufacturer C	2.35%	9	0.88	0.68
Manufacturer D	3.09%	14	1.16	0.89
Manufacturer E	9.70%	129	3.64	2.82
Manufacturer F	12.08%	251	4.53	3.51

THE KEY TAKEAWAYS

- Homogeneity is crucial and a key property of any CRM
- IMS is committed to the assessment of the homogeneity of its manufactured batches.
- IMS makes its assessment data readily available, so users can be confident in the product and achieve results they can trust.

HOW DOES IMS PREPARE ITS PULVERISED GOLD CRMS?

- The raw materials undergo a multi-stage homogenisation process and are discharged into storage drums.
- During the discharge the material is sub-sampled at regular intervals from which homogeneity and characterisation samples are drawn.
- The samples taken are randomised before being submitted to independent ISO/IEC 17025 accredited laboratories for homogeneity and inter-laboratory round-robin testing.

FIND OUT MORE

For more information on IMS CRMs or to discuss the results of the survey, please contact Dr John Carter at john@imstandards.com.au

References

Ingamells, C.O. & Switzer, P. 1973. A proposed sampling constant for use in geochemical analysis. Talanta, 20, 547- 568.

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