Homogeneity and heterogeneity of crushed gold certified reference materials

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ABSTRACT

The use of control standards, or certified reference materials (CRM), are an important implement when assessing the quality of results from an on-site or commercial contract laboratory. While laboratories operate their own internal quality control, the submission of a blind CRM by the customer provides independent data and additional assurance.

CRMs on the market are pulverised to a fine particle size rendering them analysis-ready. These pulverised CRMs bypass the sample preparation step and are queued at the analytical stage of the process reducing the efficacy of a blind CRM. This is problematic. Sample preparation is a critical part of the laboratory process and a potential source of systematic error and bias.

In addition to pulverised CRMs, crushed iron ore and bauxite reference materials, typically less than 5 mm in particle size, have been included in quality control programs and submitted blind to laboratories for many years. For other commodities however, particularly gold, the manufacture of a crushed reference material has been elusive largely due to the disseminative mineralogical nature of the ores, the gold nugget effect and ineffective manufacturing techniques. For a crushed CRM to be an effective quality control tool, however, it needs to behave like a sample from the field which is, by nature, heterogeneous while maintaining effective between unit homogeneity.

In order to establish the potential for a crushed gold CRM to be used as part of routine quality control in a reverse circulation (RC) drilling program, a number of reference materials with specific gold concentrations were engineered then subjected to an inter-laboratory round robin exercise. The degree to which the materials are homogeneous or heterogenous is discussed in the paper, along with a comparison to a natural ore reference material crushed to 3 mm and subsampled. While providing some insights into the potential for systematic bias to occur in sample preparation for methods such as fire assay, the study also has implications for blind quality control of methods that analyse crushed samples, such as photon assay and cyanide leaches (PAL).

INTRODUCTION

The production of high-quality data models for grade control programs, block models, mine plans, blending and stockpile management relies on accurate and effective sampling and analytical protocols. The mine geologist largely takes responsibility for setting and monitoring protocols from the initial sampling at the drill rig or in-pit and face sampling, through to establishing the on-site or near-site laboratory methods. It is important these protocols maintain a high level of integrity so the mine geologist can confidently interpret the data. Sampling and analytical quality control is therefore fundamental to mining activities and are a critical activity for establishing data reliability and confidence. Incorrect sampling and sample handling procedures may have a significant impact on costs and decision made (Minnitt, 2007).

For the vast majority of cases, the laboratory contract includes two components agreed between the mine and the laboratory – sample preparation, and analysis. It is the responsibility of the mine geologist to include into the program, appropriate quality control tools to independently assess the laboratory results for trueness, precision, accuracy or bias. More so, it is generally not possible to achieve meaningful results without well controlled sampling and sample handling protocols. (Carswell, 2017; Dominy, 2016).

The primary purposes for the sample preparation process in the laboratory are, firstly, to ensure the samples are in a form ready for the analysis to proceed, and secondly the prepared sample is

representative of the original submitted sample. In recent years there have been developments in automation and robotisation that enable laboratories to prepare samples with less manual intervention (Knudsen, 2007). Either way, from a sample and quality control perspective, the two fundamental requirements of sample preparation to ready the sample for analysis are the same, particle size reduction (crushing and pulverisation), and mass reduction (splitting and sub-sampling). These two requirements typically involve a primary and/or secondary crusher to reduce the particle size of the sample from the as received size to a nominal top size of 2–3 mm, followed by a sub-sampling step to reduce the mass. Sub-sampling may involve a riffle splitter, manual or automated rotary splitters or linear divider with waste material discarded. The target mass depends on the size of the pulverising bowl, typically 1 kg for LM2 bowls and 2–3 kg for LM5 bowls. Lastly, pulverisation, or grinding, occurs in a sealed vessel containing a puck and/or ring, and rotated with a counterweight to cause the puck and rings grind at high speed against the bowl wall. Following sample size reduction to the powder form, a sub-sample is extracted from the bowl, typically 100 g or 200 g and stored in a labelled pulp packet ready for analysis. The remainder of the pulverised material is often discarded, or returned to the original bag and the pulveriser cleaned in readiness for the next sample.

Recent analytical developments have interrupted historic sample preparation requirements. Photon Assay utilises a 400–500 g sample, and while largely incognisant of the particle size of the sample, bulk density and packing stability of the sample presented to the instrument are important variables that are monitored as part of the analysis. For photon assay, samples are crushed to a nominal top size of 2–3 mm, followed by splitting to the volume of the analytical jar. The pulverisation step is not required and not preferred (Tickner, Preston and Treasure, 2018).

Sample preparation is a critical part of the laboratory process. Samples damaged in sample preparation are difficult to recover, and errors carry through to analysis and compromise the results and therefore the trueness of the resource estimation. Sampling theory (TOS) studies (Gy, 1982; Pitard, 1993; Dominy *et al*, 2018; Minnitt, Rice and Spangenberg, 2007) have demonstrated that at each step in the field to data process a sampling error can occur and an understanding and minimisation of these errors should be a key objective in any quality control program. Dominy, Glass and Purevgerel (2022), in more detail summarise the application of the theory of sampling (TOS) to the sampling value chain including sample preparation and analysis. Within sample preparation, the potential for errors that contribute to analytical integrity to occur are significant. It is therefore critical that quality control strategies are implemented to monitor and assess all aspects of sample handling.

QUALITY CONTROL STRATEGIES

Quality control approaches currently used across the entire industry are fairly common and have been standard practice for many years (Thompson and Howarth, 1976; Abzalov, 2016; Dominy, Purevgerel and Esbensen, 2020) and in summary:

- Blank – the submission of a blank to assess contamination or carry-over, particularly for gold (trace element) and base metal (minor element) is common practice. Contamination can occur at any time within the laboratory, in all stages of sample preparation, and many stages within the analytical process, the severity dependent on the material types, sample preparation practices, laboratory cleanliness, analysis methods and elements of interest. Blanks measure neither precision nor accuracy in the laboratory.

- Field duplicate – a second submitted sample from the same drill interval can be collected submitted as a quality control sample in an attempt to measure laboratory variance and the quality of sample preparation. In practice, however, the splitting error from the field is often significantly greater than the variances within the laboratory, and the field duplicate should not be considered as an identical sample nor used for laboratory quality control. Field duplicates measure the variance in FSE and the sub-sampling technique in addition to compounding errors throughout sample preparation and analysis.

- Sample preparation split – in an attempt to assess quality in sample preparation, some mining geologists request the laboratory to extract a second split at the crushing step. Where a sample weight reduction is required and excess material is available, a second sample for pulverisation can be taken. These splits are taken at laboratory pre-defined sample intervals, and as such may not split a sample of grade or of interest to the exploration company. The sample

preparation split may be able to assess precision in the sample preparation process, but will not assess accuracy. If both the primary and split sample are compromised a systematic bias will not be detected.

- Analytical duplicate – a second split from the pulveriser, or a separately weighed aliquot from the sample pulp packet can be requested by the exploration company. The laboratory will label this sample as a duplicate or repeat. The duplicate data received can be used to assess the analytical precision of the laboratory and includes an extraction and weighing sampling error. The laboratory may also conduct their own internal repeats at designated intervals. However, neither a pulverising nor aliquot duplicate determine laboratory bias, but will only be able to assess precision. If the sample was compromised in sample preparation, both the primary and secondary aliquot will be biased.

- Submitted CRM – the primary quality tool utilised by exploration companies for the assessment of accuracy is a submitted CRM (Sterk, 2015). Purchased from a reputable supplier, CRMs are a stable pulverised material, sufficiently homogenised and characterised by a metrologically traceable procedure (ISO 17034:2016). The CRM is accompanied by a certificate containing consensus values, their associated uncertainties and a statement of metrological traceability. Uncertainties can be used to establish control limits that are monitored by the mine geologist. These CRMs are ideally 'blind' to the laboratory, and may have been sourced from the mining company's ore or manufactured from appropriate material. The CRMs are usually packaged in a foil or sachet, and submitted in the calico bags that would normally contain a field sample. The presence of pulverised CRMs in the submitted batch represents a dilemma for the laboratory as these samples need to bypass the sample preparation process, and be returned to the batch at a later step before analysis. The laboratory will exercise a number of sequencing options in order to optimise the re-insertion onto the batch.

- Laboratory QC – the laboratory will insert their own internal CRMs, blanks and repeat assays into the analysis batch at periodic intervals. A CRM will assess accuracy of the analysis, and only with sufficient statistical data collected over time, can they be used to determine method precision. Repeats assay will assess precision, and blanks will assess contamination.

It is the form and use of submitted CRMs that are of particular focus in this study. Despite the warnings in previous sampling studies (Dominy, Glass and Purevgerel, 2022), a quality control tool to adequately assess systematic and random biases in sample preparation is lacking in the industry. CRMs on the market are pulverised to a fine particle size rendering them analysis-ready. Pulverised CRMs bypass the sample preparations step and are queued at the analytical stage of the process reducing the efficacy of a blind CRM. In addition, for the case of analytical methods that test a crushed sample portion (Photon Assay, PAL), the use of a pulverised CRM unlike routine samples is problematic and it is not just from a representative and matrix matching point of view. These methods rely on the crushed nature or rock/particle properties of the sample to maintain method integrity. For best practice QA/QC, the properties and nature of the CRM used should be similar to the routine samples – as prepared and analysed.

Crushed CRMs containing a certified concentration of gold have been elusive, largely due to the disseminative mineralogical nature of the ores, the gold nugget effect, or ineffective manufacturing techniques. To state the conundrum from another perspective, for a crushed CRM to be an effective quality control tool, it needs to behave like a sample which by its nature is heterogeneous, while at the same time maintain fit for purpose between unit homogeneity. The manufacture of such a CRM is not trivial.

For bulk ores (iron ore, bauxite), crushed CRMs with consensus values and their uncertainties have been used for many years and are available. Crushed CRMs have been designed to eliminate the use of a blank that presents a high risk to submitted sample contamination. Furthermore, crushed CRMs of bulk ores allow the quality of the reported results to be assessed throughout the whole laboratory process not just the analytical component. The practice of using a crushed iron ore CRM for sample preparation quality control has been demonstrated through a case study by Carter and Armstrong (2023). The case study demonstrated the effectiveness of a crushed CRM to detect a systematic bias brought about by faulty dust extraction on a primary crusher in an automated

laboratory. The equipment fault had a significant bearing on the accuracy of the analytical results for samples that passed through the crusher. The error would not have been detected without the use of a crushed CRM passing through the complete sample preparation process.

CRUSHED REFERENCE MATERIAL FOR GOLD

While the case for crushed reference materials in bulk commodities has been well established, for gold bearing ores the manufacture of a crushed CRM requires some thought. To be similar to a field RC sample, a crushed CRM product would need to have a maximum size of 5 mm and packaged in 500 g up to 2 kg units. For routine submission of a crushed CRM, the product needs to be homogeneous between units, at a variance level at or below the typical level of variance of the laboratory method. Between unit variance is difficult to achieve for gold ore based certified reference materials, due to the nugget effect (Brand, 2015).

To circumvent the problem of manufacturing a crushed certified reference material from gold ore stocks, Independent Mineral Standards engineered and manufactured three crushed gold CRMs with a nominal top size of 3 mm, where the distribution of gold was at the microscopic level. The patented product was manufactured from an engineered rock, with dispersed gold embedded within the mineralogical structure. The material was further prepared by multi-stage homogenisation and sub-sampling. The final product was packed at nominal 500 g and 2 kg units, in labelled heat-sealed bags for individual use in their entirety. The units were then subjected to a variety of within batch (between unit), and within unit tests to understand the level of inherit homogeneity and heterogeneity and therefore their suitability as a routine quality control tool.

Homogeneity and certification study

During the packaging stage, units were selected for homogeneity and characterisation studies. The homogeneity of each crushed gold CRM was performed by sending 15 samples selected throughout the batch to a single laboratory and conducting analysis of each sample in triplicate. The results are shown in Table 1. The variances determined are applicable only when the entire contents of each unit are analysed or prepared. In the case of pulverisation, subsequent sub-sampling of the prepared units were taken, then analysed.

TABLE 1

Crushed gold CRM homogeneity study results, grade and between sample mean variances.

Au	Sample preparation and Pb fire assay (2 kg unit)			Photon assay (500 g unit)		
CRM	Mean (g/t)	SD (g/t)	Relative SD (%)	Mean (g/t)	SD (g/t)	Relative SD (%)
IMS-235	0.24	0.004	1.72	0.23	0.019	8.12
IMS-236	0.74	0.012	1.57	0.74	0.022	3.05
IMS-237	2.14	0.035	1.65	2.10	0.050	2.37

Following these studies, the batches were found to be sufficiently homogeneous to proceed to the characterisation study. Relative standard deviations for sample preparation and fire assay were all below 2 per cent which is exceptionally low, and similar to the performance of pulverised CRMs that only measure the variance in the analytical component. The homogeneity results did not identify any nugget effects in the material. The higher standard deviations for photon assay are typical for the method for gold grades at these levels.

Certified values were obtained for 2 kg bagged material using traditional sample preparation and fire assay. A total of ten laboratories received five 2 kg samples and were requested to conduct routine sample preparation, followed by fire assay for the determination of gold concentration. Australian based laboratories tended to crush and split the sample to a mass for pulverisation in an LM5, nominally 2.5 kg mass. Canadian based laboratories tended to crush then split the sample to a mass

for pulverisation in an LM2, nominally 1 kg mass. In both regional cases, fire assay was conducted between 25 g and 50 g masses with either an AAS or ICP finish.

Values for Photon Assay were certified using 500 g bagged materials with no further sample preparation performed. Samples were transferred to the photon assay jars in their entirety. A total of seven photon assay machines were used to analyse five samples. The property values and associated uncertainties from the characterisation studies are shown in Table 2.

TABLE 2

Crushed gold CRM characterisation study results.

Au	Sample preparation and Pb fire assay (2 kg unit)			Photon assay (500 g unit)		
CRM	Certified (g/t)	Within lab SD (g/t)	Relative SD (%)	Certified (g/t)	Within lab SD (g/t)	Relative SD (%)
IMS-235	0.23	0.006	2.6	0.22	0.025	11.4
IMS-236	0.72	0.024	3.3	0.72	0.028	3.9
IMS-237	2.08	0.063	3.0	2.07	0.072	3.5

The characterisation results in Table 2 show good agreement between fire assay and photon assay for the multi-laboratory exercise. The results demonstrate the materials are fit for purpose as quality control tools, with relative standard deviations below 4 per cent, with the exception of the low-level CRM from photon assay where the variance of the measurement method dominates uncertainty as the grade approaches the detection limit. The relative standard deviation of 3–4 per cent is higher than those obtained during the homogeneity study and higher than typical pulverised CRMs (1–2 per cent). This was attributed to the contribution to variance from the ten laboratories who processed the samples, possibly due to differences in sample preparation and fire assay method.

Comparison to natural ore – homogeneity

To illustrate the homogeneity performance of the engineered material, a crushed CRM was also manufactured from a natural ore. Precured from mill feed material in a greenstone-hosted gold mine in Western Australia, the ore was subjected to the same crushing to a top size of 3 mm followed by the same multi-stage homogenisation and sub-sampling steps as the engineered material. The natural ore reference material was packaged in 2 kg with further sub-sampling to 500 g units. The 2 kg units were submitted to two commercial laboratories for sample preparation and fire assay. The 500 g units were analysed by the PAL method without any further sample preparation or sub-sampling. In this way the homogeneity can be compared to the engineered reference material of a similar grade, with difference attributable to the between unit homogeneity. The results are shown in Figure 1.
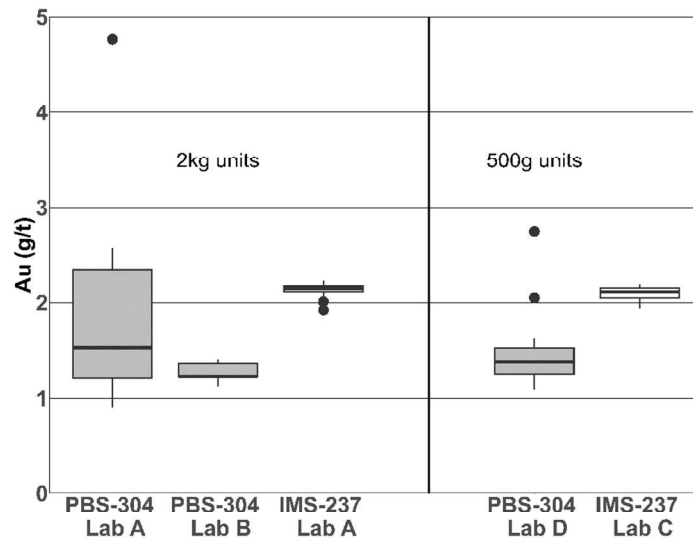
FIG 1 – Comparison of homogeneity study results for a natural ore reference material (PBS-304) and an engineered reference material (IMS-237). Outliers are shown as dots.

The study demonstrates the between unit homogeneity of the engineered product is superior to the natural ore. The variances observed in the data for the natural ore CRM render the product unsuitable as a candidate reference material. Seemingly random spikes in grade are indicative of the material containing poorly disseminated gold throughout the material. The natural ore material was not subjected to a characterisation study.

Heterogeneity study

In order to understand the potential for contribution to uncertainty from sample preparation, the engineered reference material was subjected to a within bag variance test. This test was designed to understand the level of homogeneity-heterogeneity within each unit. A random selection of 2 kg units for IMS-236 were selected from the batch and split into 4 × 500 g samples followed by analyses in their entirety by photon assay. The first study involved careful splitting by rotary splitter divider into eight segments, with two segments from opposite sides of the carousel combined into each 500 g sample. The second study involved taking a 'grab' sample from the 2 kg unit by pouring the sample into each of the four 500 g photon assay jars for analysis. This latter technique would be considered poor sampling practices, failing Gy's test for each particle to have equal opportunity to be included in each sub-sample (Gy, 1982). For each of the 500 g jars, multiple analysis was conducted in order to understand the contribution to uncertainty from the analysis and the sample splitting technique. In this way, the within, and between jar variances could be visualised and assessed. The results are shown in Figure 2. Each figure is shown on the same y-scale.
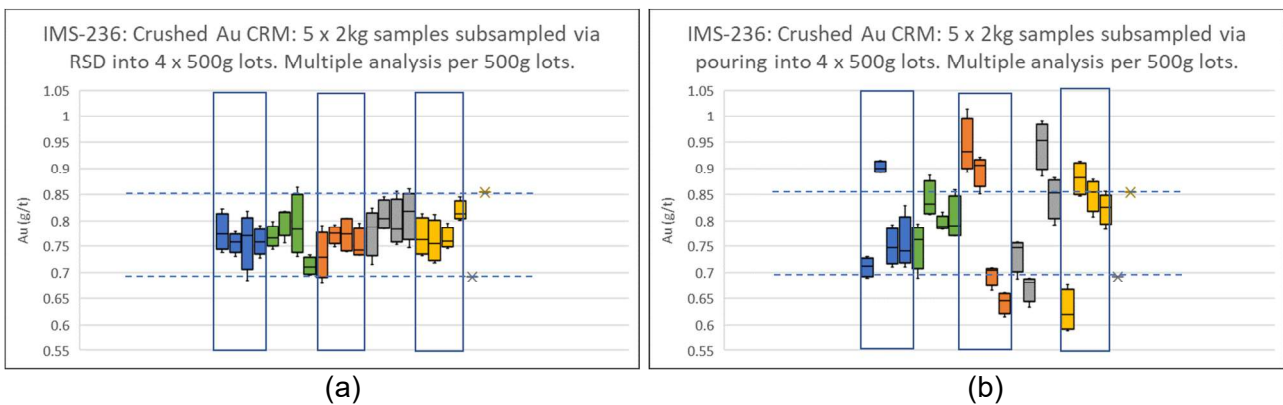

FIG 2 – (a) Photon assay results – 2 kg IMS-236 CRM split to 500 g lots using rotary splitter divider. (b) Photon assay results – 2 kg IMS-236 split to 500 g lots using a grab sample approach.

The test results in Figure 2, is objective evidence of the impact of splitting method, and quality of the sampling practices on the variance between replicate aliquots. The results demonstrate that when the CRM is split correctly, the within jar variance is similar to the between jar variance. The dotted lines in Figure 2a are three times the standard deviation from the mean and would represent a set of control limits that could be applied to these CRMs. It is clear from Figure 2b, when the 2 kg CRM is poorly sub-sampled into the 500 g jars, a significantly higher between aliquot variance occurs, with some of the results falling outside the control limits and therefore failing the quality control test. The results are confirmation the design of the crushed CRM enables sample preparation biases to be detected.

The splitting of a 2 kg unit received by the laboratory, into a 500 g sub-sample for pulverisation is not an uncommon practice. What these results demonstrate is that a bias may occur and be undetected when a single subsample is pulverised and analysed. If the exploration company requests a crushing split duplicate to be analysed and split biases are occurring, it may be possible to detect the poor practices are the cause. Random biases can be somewhat accommodated over a large drilling campaign with sufficient data over time and only if the magnitude of the bias is acceptable. However, more importantly, if the bias includes a systematic component, that is, all samples are biased high or low in sample preparation, the nature of the problem is more serious for mining geology. We were unable to confirm with these results, however, if there was any systematic bias because the analysis order was not tracked against the loading order and a trend from analysis to the split sample was not established. A subsequent investigation was conducted to establish if the sample preparation biases observed are potentially systematic.

It is worth noting a similar trend in Figure 2b could result from a field sample that contains nuggety gold which could mask the detection of splitting quality. In fact, methods of analysis for extremely nuggety gold ores sometimes includes preparation and analysis to extinction, or collection of bulk samples, so that a better measurement of gold grade can be obtained (Roberts, Dominy and Nugus, 2003). With the use of a crushed CRM demonstrating correct splitting practices, a comparison of the CRM duplicate data to the variance between a sample split could be used as a measure of the 'nuggetiness' of the samples and determine if an alternate sample preparation and analytical regime is warranted.

INVESTIGATION INTO SAMPLE PREPARATION BIAS
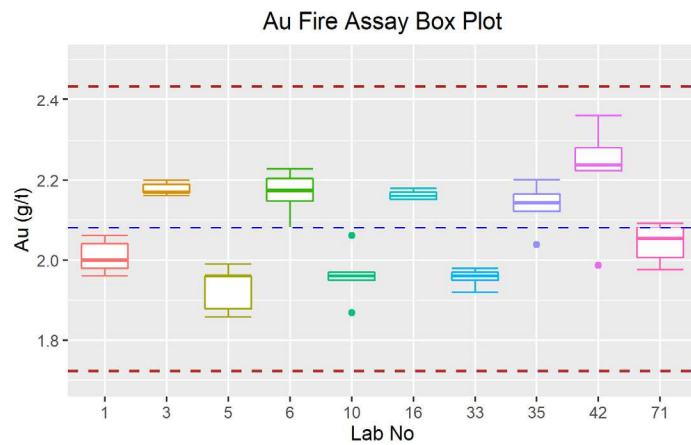
Characterisation study results

In order to determine if systematic biases can occur in sample preparation, and if crushed CRMs can detect them, an additional investigation was conducted using the samples analysed during the characterisation study. The characterisation study for sample preparation and fire assay involved sending five units to ten laboratories. The certified values and uncertainties for all of the CRMs are shown in Table 2, however, when a comparison between the results from all of the laboratories are visualised in box plots (Figure 3), an interesting trend emerges.

Au Fire Assay Box Plot

(a)

Au Fire Assay Box Plot

(b)

Au Fire Assay Box Plot
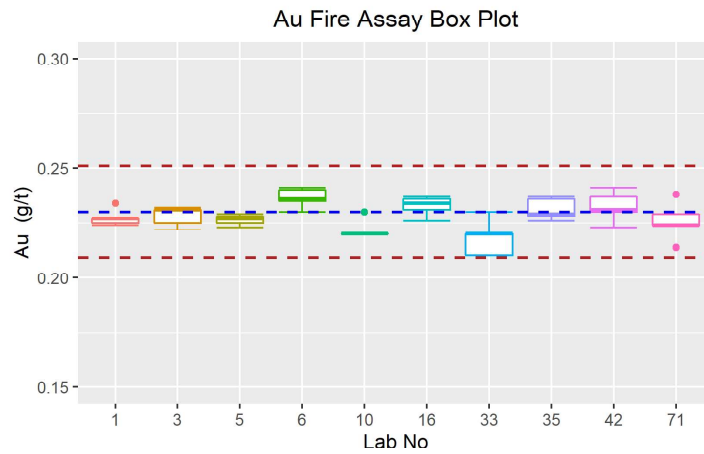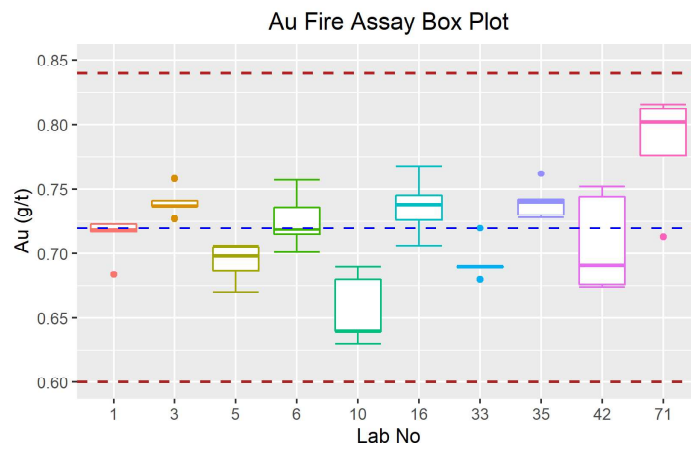
(c)

FIG 3 – (a) Low-grade IMS-235 characterisation study box plot. (b) Medium grade IMS-236 characterisation study box plot. (c) High-grade IMS-237 characterisation study box plot.

While not as evident in the lower grade, as the grade of the CRM increases, there appears to be two populations of data. The spread of the data explains why the standard deviations of the characterisation studies are significantly higher (2.6–3.3 per cent) than the homogeneity studies (<2 per cent). Some laboratories have reported results above the mean and others below. The trend appears to be similar for all three grades, but more pronounced at the highest grade. This is an interesting development. Table 2 shows the mean or certified values between fire assay, and photon assay are in very close agreement. Both of these methods are expected to determine the total gold

concentration of the sample. The hypothesis is the bias occurs in sample preparation with the loss, or concentration of gold-bearing material. Alternatively, there could be an analytical bias between each of the laboratories.

In the case of IMS-237 (Figure 3c), the z-scores for sample preparation fire assay method of the individual laboratory means were calculated using the global mean and standard deviation from the photon assay results. The results suggest for two of the laboratories, the bias is significant (z-score > 2).

Umpire analysis

To test if the bias is occurring in sample preparation, or during subsequent analysis, pulverised samples were returned from four of the laboratories and subsequently labelled as A, B, C and D respectively. The selection was made from four laboratories belonging to different global groups and where biases were both positive and negative.

The returned pulverised samples prepared at laboratories A, B, C and D, were then sent to an alternate single laboratory for analysis. The results are shown in Figure 4. The samples were re-numbered and submitted blindly and routinely to the alternate re-analysis laboratory without reference to the nature of the investigation.
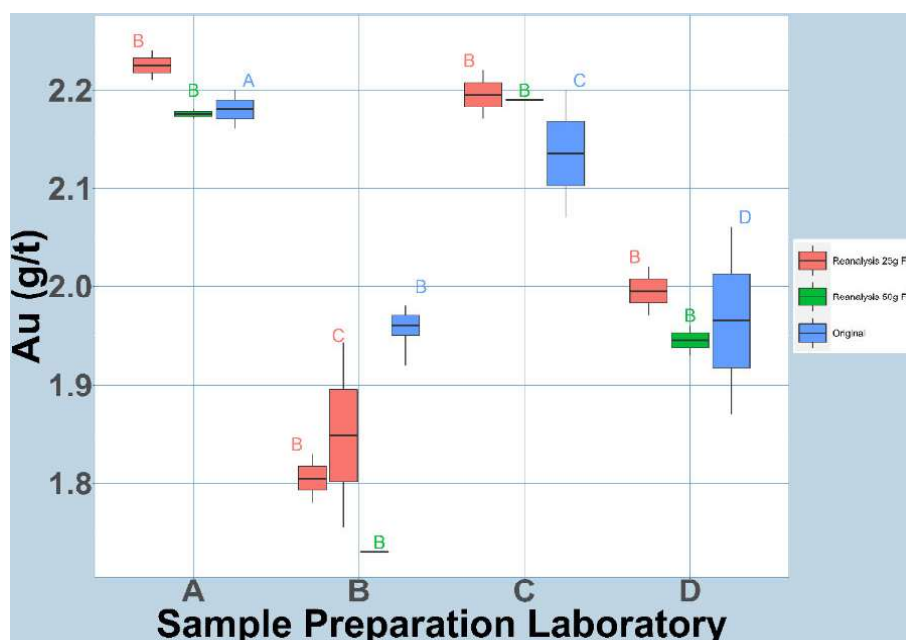

FIG 4 – Fire assay results for pulverised samples prepared at laboratories A, B, C and D (x-axis), then resubmitted and analysed at laboratory B and C as indicated adjacent to each box. Re-analysis was performed at both 25 g and 50 g masses.

In Figure 4 the original results are shown in blue (the furthest box plot to the right of each laboratory indicated on the x-axis), and represents the original analysis for the submitted samples. In these cases, sample preparation and fire assay analyses were conducted at the same laboratory in the same submitted batch. The original results from laboratories A and C biased high compared to the mean, and laboratories B and D where biasing low. All of the pulverised samples returned were then submitted to laboratory B for repeat analysis. To complete the re-analysis exercise, pulverised samples prepared at laboratory B were also sent to laboratory C for analysis. Repeat analysis results are shown in orange for 25 g fire assay (first box from the left for each preparing laboratory), and green for 50 g fire assay (second box from the left for each preparing laboratory).

The first observation to make is the bias does not trend significantly with the mass used to conduct the analysis. Both 25 g and 50 g fire assay results are aligned in Figure 4. The instruction to conduct 25 g assay, or half weight, included a request to maintain the same flux quantities so that if there were any difficulties with chemistry of the material and extraction during fire assay, the results should

bias low for 50 g fire assay. While there is a slight low bias for the larger mass, the bias is not noteworthy.

The more significant trend in the box plots from Figure 4 is that the repeat analysis at laboratory B and C matches the bias that occurred in the analysis from where the original samples were prepared. In addition, as a quality control check, the repeat analyses at laboratory B, for the resubmitted samples are consistent in bias with the original results prepared at laboratory B. This informs us that the analysis performed at laboratory B is providing consistent results, and there are no biases occurring from analytical batch to batch.

To determine if the overall high/low bias was occurring in sample preparation, or in the analysis, the alignment between the repeat analysis (orange and green boxes), and the identification of the preparing laboratory on the x-axis should be noted. If the bias was occurring in the analysis, one would not expect the re-submitted sample results to closely align with the original results from where the samples were first prepared. However, the repeat analyses in the case of laboratory B and C closely align with the origin of the prepared samples in both cases, therefore indicating a systematic bias has occurred in sample preparation.

Umpire analysis implications

One of the common quality control strategies exploration companies employ is to conduct duplicate analysis of prepared samples from the main contract laboratory at a repeat or umpire analysis. The comparison of the results is given as confirmation of trueness of the main contract laboratory assay. This study suggests there could be a confirmation bias at the umpire laboratory. If a systematic bias occurs in sample preparation from the original submitted sample, the umpire analysis will not be able to detect this sample preparation bias and will confirm the original analysis. The submission of a field duplicate for sample preparation at an umpire laboratory may not be ideal as the variances from the field are included in the umpire result.

While evidence for a potential systematic bias to occur in sample preparation has been obtained, further investigations are required to understand the root cause. Closer inspection of the laboratory techniques used during crushing, splitting, pulverisation and sub-sampling are required, in addition to a comprehensive inspection of equipment conditions including dust collection systems.

CONCLUSIONS

This study describes the homogeneity and heterogeneity properties of a crushed gold CRM that has the potential to be utilised as a quality control tool during the preparation and analysis of mine geology samples. The study describes typical protocols utilised for quality control, and identified a potential for current practices to be deficient in the detection of random and systematic biases in sample preparation. The analysis of the crushed gold CRM was performed by fire and photon assay methods, with good agreement in the total gold for each of the materials produced.

Sub-sampling, splitting and sample preparation trials have indicated that there is a potential for both random and systematic biases to occur in sample preparation, that may be difficult to detect with current quality control practices. The systematic bias from a crushed gold CRM with certified value of 2.1 g/t could be as high as ± 0.2 g/t, representing a total relative bias of 10 per cent which is significant for mine planning and delineation of ore and waste.

While this study has identified the potential for a systematic bias to occur, the root cause of the bias has not been identified. Evidence suggests crushing and splitting processes may be a contributor, however pulverisation and sub-sampling processes cannot be ruled out. Further investigations are required to determine the parameters in the laboratory that are contributing to the bias and find evidence to confirm or refute the observations.

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