

# Quality control on assays: addressing some issues

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## ABSTRACT

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### *Article history:*

Received 1 July 2015

Received in revised form

Accepted 15 July 2015

### *Keywords:*

QA/QC

Quality control

laboratory

certified reference material

After several decades of research, papers, conferences and undoubtedly many millions of analyses of rock and soil samples globally, we know that the success of mining projects is largely dependent on the quality of the data on which they are modelled. The importance of proper sampling, data management, quality assurance and quality control have all been extensively addressed in the literature. However, as global standards change and mining companies have to be more and more innovative to extract value out of marginal projects, it is interesting to note that basic quality control concepts are still poorly understood, misapplied and lead to constant confusion in the exploration and mining sectors.

One such issue is how to interpret the quality of the data generated by the laboratories. Most geologists are familiar with control charts but not many realise that the most common approach for their use is disputed. It doesn't help that certified reference material providers provide conflicting advice on how to use their material and that most sampling and QA/QC courses, as well as information in the AusIMM latest Monograph on best practices are limited in their full explanation of the proper process.

Demonstrating and quantifying bias, accuracy and precision, and defining when standards really fail are critical to monitor quality and to communicate any issues between the various parties.

At the core of this lies a fundamental lack of understanding of the principles of quality control, sampling and basic statistics in the mining industry.

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## INTRODUCTION

Explorers spend many millions of dollars on gathering data, which forms the core value of an exploration project. Yet, the quality of this data is sometimes assumed by default to be suitable until proven otherwise.

This paper does not intend to sketch a new platform for the implementation and management of data and its quality, as this is well known. It has been documented in various publications over the last few decades, with the most recent reference work for the mining industry summarised in papers such as Roden and Smith (2014). Rather, this paper focusses on some key points where confusion still reigns and where the industry struggles to implement the correct guidelines. With some of these issues clarified and presented in some more detail, operators can be more confident in

their data assessments. The existence of such confusion also highlights some weaknesses in how the industry deals with training of its new professionals, which will have knock-on effects on issues such as productivity.

## THE ISSUES

There are a number of recurring issues that are quite common in the process of data quality review in the mineral industry. These issues are widespread and continue to limit the geologist's capability to make sound decisions based either on good or bad data, and are the main cause for frequent frustration in communications between laboratories and geologists. The default position sometimes even seems to be,

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surprisingly, that the data quality is adequate until an unmissable problem presents itself.

## QA/QC

Quality assurance and quality control, QA/QC in short, are two different things but are almost always used as a one-size-fits-all term for those situations where geologists talk about "something to do with standards, blanks and duplicates". The pervasiveness of this misuse of nomenclature in itself demonstrates the misunderstanding of the fundamental concepts of the management and safeguarding of quality.

Quality assurance means assuring the quality of the data by having a set of standard operating procedures (SOPs) in place, aiming to prevent errors being made in the sampling or measuring process. By making sure that the way each task is executed is documented, it can be demonstrated that no additional variance or bias should be introduced by the operator or the sampling mechanism. In other words: Billy samples in exactly the same way as Sally and we hope that they won't make any mistakes as they are following the same procedures.

In quality control, a certain number of checks and balances are implemented and are constantly reviewed and assessed, in order to identify whether the sampling system and the laboratory are indeed providing quality assays. In the mineral industry, the checks and balances that are commonly used to monitor sampling preparation and assaying processes include standards, blanks and duplicates. An important aspect of this is that the system can be controlled and adjusted during the process itself. As noted previously by Graindorge (2010), too many times QC results are analysed after a sampling programme has finished, and, besides being a complete waste of money, this is simply not acceptable practice.

In short, quality assurance is about prevention of errors, and quality control is about detection and correction/rejection of errors as they occur.

A drilling programme can, therefore, not be labelled as having "good QA/QC" when SOPs were hastily copied and pasted half-way through the programme, and standards, blanks and duplicates were submitted but only analysed after the programme finished, if at all. This practice is common throughout the industry, from junior explorers to larger producers.

Resource professionals, bound by codes of ethics and required to report in compliance with codes such as

JORC (2012), are required to demonstrate the level of quality of the data. However, because they often get involved late in the process, the resource professionals are left with the tough task of dealing with data sets which often contain a large number of questionable data, and because correction is no longer possible, have to make a tough judgement call as to what degree the issues have affected the overall confidence of the mineral resource estimation.

It is therefore critical to get data experts or the resource geologist introduced to the programme before the sampling commences. A brief review of some 20 recent mineral resource reports showed more than half to include ambiguous statements on data quality at best.

## PROCESS CONTROL CHARTS

When QC samples are analysed, key terms of accuracy, precision and bias are central to assessments of the results. However, these terms, too, are very frequently misunderstood and even in many code-compliant resource reports are used interchangeably and erroneously.

A full explanation of these concepts is outside the scope of this paper (a good starting point is Abzalov (2008)), but to address a few of the misunderstandings, the process control chart (sometimes called Shewhart plot) is investigated in more detail below.

Such a plot is used frequently in the mining industry to cast post-mortem judgments on accuracy and precision, however, the main purpose of a process control chart is to monitor the measurement process in order to detect changes in the process when they occur. Each of these changes presents an opportunity to improve the measurement system by discovering those extraneous factors that affect the measurement process without our knowledge. A process control chart can be used to monitor issues with drifting means, variance, bias and trends. Such plots are very easy to generate in Microsoft Excel™, various other spreadsheet software and commercially available process control software, and are easy to interpret. In terms of quality control, it is the first port of call for exploration geologists, plant metallurgists and resource geologists. However, even though its workings at first glance seem simple, it appears that many in the industry follow an incorrect approach, which leads to compromised datasets and missed opportunities for improvement.

The input data for process control charts often are the laboratory's assay results of certified reference

materials (CRM's), although other reference materials may also be used if they are fit-for-purpose. A CRM is sufficiently homogenised material and is stable with respect to one or more specified properties, which has been characterised by a metrologically valid procedure for those properties. It is accompanied by a certificate that provides the value of the specified property, its associated uncertainty, and a statement of metrological traceability (ISO, 2006). In the mining industry, both the laboratory and the client usually insert such CRM's in the sample sequence and both monitor the process separately using different tools, and this is a common cause of misunderstandings. The way these CRM's are produced and certified is important to understand when discussing the variability of the results. For more information, and a general summary on CRM usage the reader is referred to McWha (2014). Detail on the certification process can be found in ISO Guide 35 (ISO, 2006) and other related ISO documents.

It is obviously desired that laboratory results should be consistent from day to day. However, checking for such consistency is complicated by the inevitable occurrence of random errors (leading to "common cause variation"). Therefore, several statistical techniques have been developed to show whether or not time-dependent, or other non-random changes (e.g. "special cause variation") are occurring in the results, alongside the random errors. The laboratory needs to be able to stop and examine the analytical method quickly if it seems to be giving erroneous results. On the other hand, time and other resources will be wasted if the analyses are halted unnecessarily, so the quality control methods should allow their continued use as long as they are working satisfactorily (Miller and Miller, 2010). A measurement system is said to be in control when the process control chart shows no evidence of unpredictability. Because the term "in control" is often misused, some prefer to say that the process is predictable (Wheeler, 2015). Furthermore, contrary to statements in popular handbooks such as Miller and Miller (2010), for a system to be in control, it is not relevant to know whether the random errors in the measurements are too large. In simple terms: whether or not we like the total amount of variation present does not matter to determine whether a system is in control or not.

Once the first assay results for a CRM are available, they can be plotted against time (x-axis) and the results can be analysed. Control limits for use in the process control plots are defined and plotted together with a centre line, the latter generally being the process mean. These control limits define whether samples need to

be flagged or rejected and are often plotted using the following equation:

$$CONTROL\ LIMIT = \bar{x} \pm 3 \frac{\hat{\sigma}}{\sqrt{n}} \quad Eq.1$$

In explaining the principles behind this equation, it is sometimes stated that for a random variable with a normal (Gaussian) distribution that is not affected by special cause variation, there is theoretically only a 0.3% chance of a value falling outside three standard deviations. However, while this assumption is found in some reference materials (e.g. Kutz, 2015), it is at odds with the original work on statistical process control by Shewhart (1931). His approach is based on the fact that regardless of the shape of the histogram, three standard deviation limits will filter out virtually all of the routine variation, allowing us to detect those potential signals that are large enough to be of interest. We do not need to know the theoretical risk as long as it is reasonably small. Shewhart essentially provided global critical values that work with all types of data. His three standard deviation limits will filter out 99% to 100% of the routine background variation. There is neither a Gaussian distribution nor exact probability of a false alarm required.

The calculation of the standard deviation is where more confusion comes into play.

Rather than estimating the standard deviation from the data itself, the standard deviation is often taken from the CRM datasheet. This value is reported as part of the results from the round robin tests that CRM producers collect to communicate the mean (declared value) and variability of a CRM in its grade determination process. Even though this is the approach favoured by most resource professionals, it is dangerous to use such values for process control limits as they do not relate to a particular laboratory's own measurement process and data. One should consider that if the assay process is really in control and being operated consistently at some other standard deviation (e.g. from the CRM), then the analyst may spend considerable effort looking for assignable causes that do not exist (Montgomery, 2012). For the purpose of within-laboratory process control, and identification of trends and outliers, the standard deviation is therefore recommended to be estimated from the laboratory's own measurements of the CRM.

Another incorrect approach to establishing control limits from the laboratory's own measurements of the CRM is if the control limits are estimated by computing the overall standard deviation of a long range of single assay values. The occurrence of any special cause variation during the period in which the

assay values were obtained could lead to overestimation of the process standard deviation. In other words: by calculating a simple standard deviation over the entire dataset, that non-random, special cause variance that we're trying to detect by establishing appropriate control limits is now actually included into the control limits themselves. This leads to control limits that are too far apart, entirely defeating the purpose of the control chart. This mistake is very common in the industry because the papers and courses on the subject state the formula but not the calculation of the standard deviation, leaving room for free and often incorrect interpretation of this critical factor by industry professionals.

Importantly, it is not even possible to estimate the inherent variability in the process from a single measurement. In mineral exploration, only a few companies, if any at all, ever request the CRM to be analysed in replicate (i.e.  $n$  in Equation 1 is always 1). A standard deviation or a range can only be calculated from two or more measurements.

Therefore, a better, practical and statistically viable approach when control charts for individual results (e.g. no replicates and  $n=1$ ) are required is to assume that the variation between successive measurements may be used to estimate the process standard deviation. This is called a moving range approach and the formula is simply calculated as follows (Equation 2):

$$CL = \bar{x} \pm 3 \frac{\overline{MR}}{1.128} \quad \text{with } MR = |x_i - x_{i-1}| \quad \text{Eq. 2}$$

Even though this creates statistically sound control limits, the quality of such a plot depends on the spacing of the standards. When the spacing is large (i.e. several hours or days), undesired non-random variance introduced by the laboratory assaying process is included into the values. The first principle for an effective moving range chart is that successive values need to be logically comparable. The second is that the moving ranges need to isolate and capture the local, short-term, routine variation that is inherent in the data. When these principles are ignored, the moving range chart can miss signals that it would otherwise detect (Wheeler and Chambers, 2010).

It should be noted that the moving range approach is one of several approaches, however, it is the one in most common use for this purpose. Additionally, it is relevant to note that special cause variation is not limited to one value falling outside properly constructed control limits; other criteria exist that also

indicate the occurrence of special cause variation that lead to the system not being in control (e.g. the measurement system has changed), regardless of the control limits being breached. These include: two out of the last three points above/below two sigma, four out of the last five points above/below one sigma, eight consecutive points on one side of the mean or target value, etc.

If a properly constructed control chart shows the run of laboratory assay results to be predictable, then we can evaluate how far the laboratory mean result differs from the assigned value of the CRM, and test if this is of both statistical and practical significance. We can also assess the standard deviation of the laboratory results, and check that against desired performance, often specified by the client as based on the CRM-certified standard deviation.

If the laboratory results show the presence of special causes, we would expect the laboratory to have reacted to the control chart signals, and attempted to identify and remove the causes, to attempt to return the process to statistical control. If the laboratory has failed to do this, the data will have one or more "special causes" present. If a particular "special cause" is of the "single mistake" variety, then such values can be removed from the data and the new series tested for statistical control. However, if special causes like drifting or step changes are present in the data, the overall mean has little practical meaning, and a more complicated breakdown of the data may be required to work out which of the results are showing differences from the standard value.

Figure 1 shows such a situation, where, during a laboratory and QC audit, a step change was evident at around 30 samples for a gold CRM. The operator had not picked up on this *during* the process and instead had assessed the performance of all measurements together in a monthly "QC meeting report" (Figure 2). If appropriate criteria to indicate the occurrence of special cause variation would have been applied (e.g. such as four out of the last five points above one sigma), using a moving range approach, then it would have been clear at sample 35 that the system was not in control.

In this post-mortem case, where the operator had used a standard deviation calculated from the entire set of single measurements, the monthly QC reports showed no issues with the standard (i.e. Figure 2 shows all values fall within the thick dashed lines).

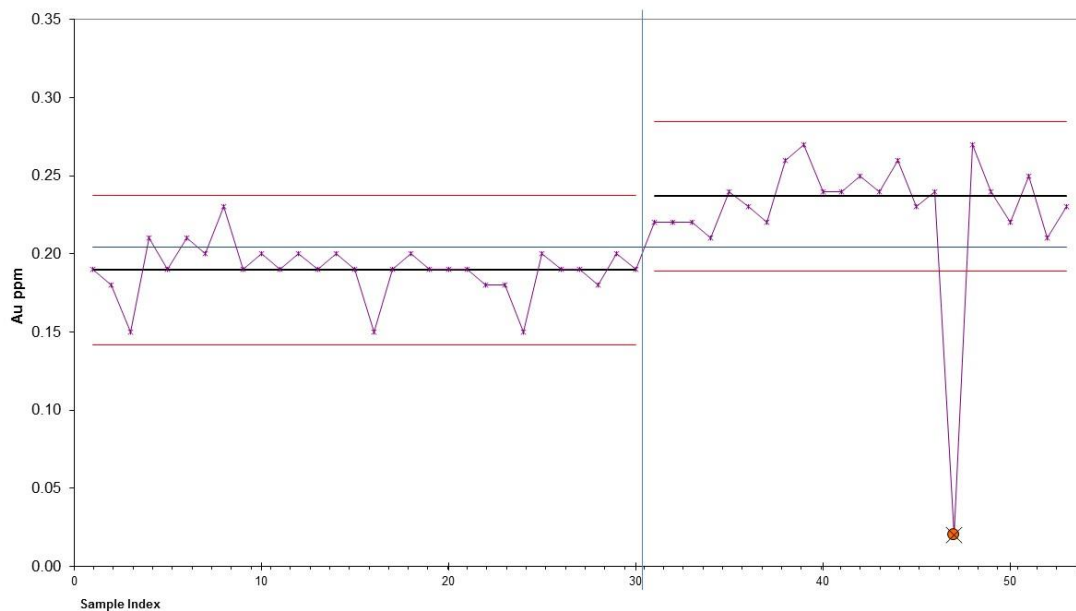


Figure 1 Laboratory results for a gold standard at a typical resource definition operation. The thin red lines show limits defined by the moving range approach.

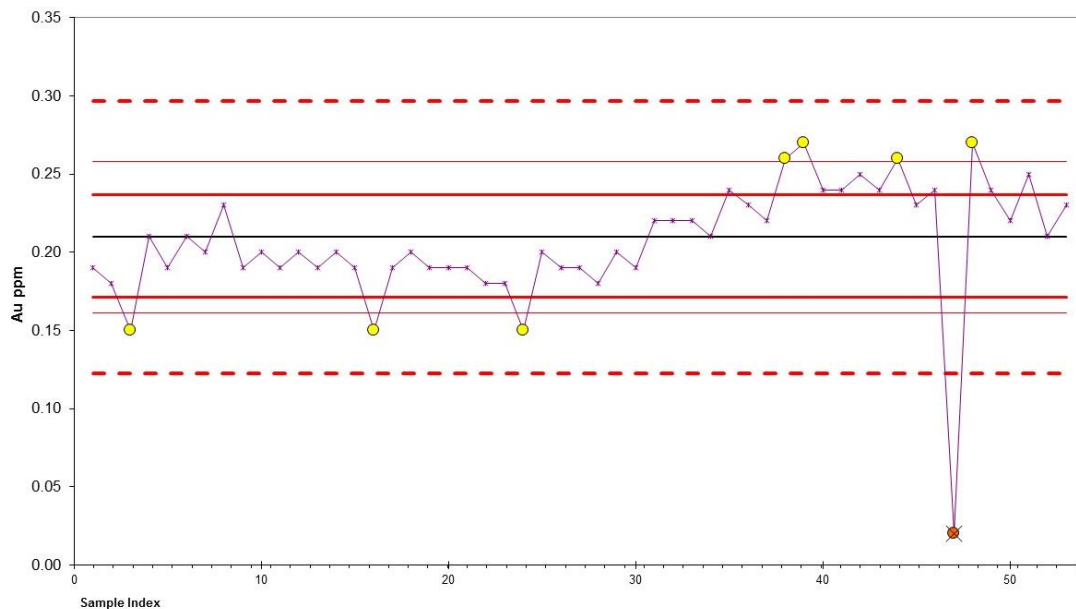


Figure 2 Same results as in Figure 1, assessed altogether (i.e. without noting the step change). Solid red lines show upper and lower control limits established by using the standard deviation from the CRM datasheet. Dashed thick red lines show upper and lower control limits if established by using incorrect calculation of the standard deviation. The thin red lines show limits defined by the moving range approach.

The well-entrenched and global mineral exploration industry phenomenon of applying incorrect pass/fail criteria leads to either the laboratory being requested to spend a large amount of money on re-assaying of

samples that are perfectly predictable (e.g. when using CRM-certified standard deviation data), or it leads to exploration companies, processing plants, etc., allowing values that are statistically out of control to

go by unnoticed (i.e. by incorrectly estimating the standard deviation and creating control limits that are too far apart).

Inserting QC samples into the sample stream by the client (e.g. exploration company) is widely considered to be good practice and can serve an important purpose in terms of independent verifications of laboratory results. However, in terms of real-time process control, their use should be treated with caution. The laboratory is performing its own statistical process control (using CRM's sourced from the same or different manufacturers), and, unlike the client, is in a good position to react immediately when the process is out of control. Rather than blindly resubmitting entire batches or ranges of samples where a client CRM performs outside three standard deviations (especially when using CRM-certified standard deviation for control lines), the client would benefit more from reviewing the laboratory's internal QC results frequently, openly and in collaboration with laboratory management.

Resource reports often show statements on the performance of CRM's (e.g. as a post-mortem), and if the standard deviations of the entire population have been used to establish control lines, unsurprisingly, these statements are positive in nature. If CRM-certified values are used to define control lines, only when the process has been shown to be in control, can a judgment be made on the performance of the laboratory in terms of precision. The CRM-certified standard deviation then effectively acts as a customer specification and can be used to determine whether the precision is fit-for-purpose.

Sampling and QA/QC courses the world over include insufficient background on this critical quality control step. CRM producers give conflicting advice as to how to use their CRMs (with some suggesting to use the process standard deviation and others to use the CRM standard deviation).

The distinction between the right and wrong ways of computing limits in statistical process control was first made by Shewhart (1931). It should not be an issue some 85 years later.

## BIAS

Much has been written on the subject of bias, as recently as a short article in the AIG magazine by Long (2015) on quality control for pXRF; yet it is astounding that out of a review of some 50 of the latest globally published resource reports, many showed

basic errors in approach or at least a lack of scrutiny in the assessment of bias.

Of note is also that almost all these reports have a Table 1 (in the case of reports based on compliance with the JORC Code), in which the mandatory Competent Person's comment on the important aspect of bias mostly boils down to the quantitatively unsubstantiated opinion of "there is no bias".

The concept of bias is indeed discussed rather freely in most mineral industry contexts. For instance, mineral resource reports simply state the author's visual impression of the data, quite often by referring to a simple scatterplot of dataset A vs. dataset B, supported perhaps by comparisons of the means of both datasets (i.e. exact bias). Other reports may show a quantile-quantile comparison, for instance, when populations are not of the same size. Loose guidelines exist as to how to interpret the results from such approaches (e.g. results showing exact bias larger than 5% are considered not acceptable), however, it is important that the statistical significance of any differences are addressed.

A simple and sound visual approach to determining bias is possible using the process control plot discussed above, however it is not used consistently in the mining industry. By using the CRM-certified target value as the centre line on an  $\bar{x}$ -chart (rather than the population mean), any evidence of lack of control (e.g. one sample outside 3 standard deviations, two out of the last three points above/below two sigma, etc) is an indication of bias. It is important to make sure that the process is in control before such an assessment is made. More on this approach can be found in Wheeler and Lyday (1989).

Several methods exist to quantitatively assess bias and they are not applied enough in the mineral exploration industry. Take for instance, the comparison of two datasets containing assay data: one from laboratory A and the other from laboratory B. It is easy to determine the relative bias (which is simply  $(A-B)/B \times 100\%$ ). However, to determine whether this bias is statistically significant, several other tests can and should be performed. The variance of each of the data sets is key to such calculations as this determines whether the difference in the means is *statistically significant* at a certain confidence level. Most practitioners should be familiar with Z-scores and the Student's t-test, however, they are rarely applied to data comparisons in the mineral industry and most assessments seem to be limited to subjective visual assessments of the "rough size" of the exact bias. Depending on the statistical characteristics of the datasets being

compared (i.e. normality, outliers, variance), several other tests may be required (e.g. Grubbs, Fisher, Cochran, etc.).

An associated and commonly underappreciated issue is that of sample selection that lay the foundation for check sampling programmes and such bias assessments. This is critical, for instance in determining whether Laboratory A is providing results similar to Laboratory B, or if results for an old core drilling programme can be reproduced by check sampling. By using a grade cut-off or bin-wise approach to sample selection, an artificial bias can actually be introduced into the process; hardly something that helps when trying to determine a bias! Instead, geologically defined mineralised intercepts should be selected randomly, or randomly select samples within mineralised zones, without regard for the assay grade of the individual samples (Long, 2015). None of the reviewed mineral resource reports which discuss check sampling elaborated on the method of sample selection.

It should be noted that none of the approaches are silver bullets in assessing bias, however, the current default approach seems to be a very basic one and should be improved on.

## LABORATORY ERRORS AND ISSUES

When discussing quality control on assays, clearly the laboratory plays an important role. Even though the laboratory may well have some issues and room for improvement in important areas, when samples are sent to international ISO-accredited commercial laboratories, it is rare to see their practices influencing the quality of the overall sample results to such a negative degree where they should not be used in resource evaluations. It can also be easily demonstrated that sampling errors introduced in the laboratory are typically small when compared to those that get introduced at earlier sampling stages, such as the primary drilling and sampling.

For this reason, laboratory audits are not employed very often as companies seem to put their faith in the accreditation system and internal quality control process. Out of 850 recent mineral resource reports reviewed by the author, only five contained statements on a laboratory audit. When they *do* get carried out, they often do not go further than some comments on the cleanliness of the sample preparation area, as mostly such audits are carried out by geologists who usually have limited understanding of a fire assay process, XRF machine or analytical geochemistry.

Geochemists on the other hand, may not always have a full appreciation of the sampling theory, which is important for any assessments of errors that get introduced in the sample preparation phase.

Carrying out a full laboratory audit can add significant value to any quality assurance system, however, it is particularly important for small, privately run laboratories. When mineral resources located outside the Committee for Mineral Reserves International Reporting Standards (CRIRSCO) regulated jurisdictions need to be brought into a transparent format, such audits and proper reviews of the laboratory are especially important.

When dealing with commercial laboratories, the most important issue is most commonly not a technical one. A lack of communication between the laboratory and its client, in combination with a limited understanding of the quality control process is often where much of the misunderstanding on *perceived* quality issues comes from. For instance, the geologists have determined that their standards are failing and the finger is quickly pointed at the laboratory. In turn, the laboratory can't see any problems as its own control samples show that there are no problems. In light of the earlier comments on quality control mistakes, the problem lies in a misunderstanding *on both sides* of how to actually assess the data. They include incorrect handling of samples, swapping of standards, database errors and other poor data handling issues.

Why is this issue so common? It is simply not possible to have a discussion on quality control if neither party speaks the language to a required level of competence. Neither party has a full understanding of how the process works and a lot of time is wasted by discussing non-issues. This is where properly constructed control charts change the game by helping everyone to ask the right questions.

## DISCUSSION & CONCLUSIONS

These issues are only a few of many that have an impact on the overall confidence we have in the resource models we build. They serve to illustrate the issues we face as resource professionals and are only the tip of the iceberg when it comes to the quality of our data and management of errors. This approach leads to what statisticians call Type I and Type II errors (failure to make the correct decisions based on the data) and will affect the downstream commercial evaluations of projects. There is no excuse for mismanagement of such errors. For instance, the internet is flooded with sources of information on

process quality control (although caution is advised) and most university degrees include a sound component of statistical data analyses.

Clearly, the actual impact of each of the examples in isolation on overall confidence in a mineral resource estimate is unlikely to be large enough for results not to be reported or included in evaluations (see discussion in McWha, 2014). But equally, without a sound understanding of the foundations of QA/QC, a Competent/Qualified Person's discretion on quality assessments and uncertainties isn't always an effective safeguard to address important data quality matters. It demonstrates a much broader problem: acceptance of the marginal approach to validation of data, which is the most critical ingredient in resource evaluation (i.e. rubbish in = rubbish out).

## ACKNOWLEDGEMENTS

The author would like to thank mineral resource consultants John Graindorge, Mike Stewart, Mike McWha, geochemist Richard Holdsworth, and statistical process control consultants Robin Henderson, Tim Ball, Dr. John Henstridge and Dr. Don Wheeler for improvements, discussions and review of this paper.

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