

## Technical Note: Sample Preparation of 'Nuggety' Samples: Dispelling Some Myths about Sample Size and Sampling Errors

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### Introduction

During recent audits of numerous commercial laboratories, the first author has noticed that many laboratories prepare pulp samples from rock, drill core and drill cuttings of approximately 3 kg mass using large, fixed-bowl, shatter box-type, vibratory pulverizers. This preparation method is referred to by the laboratories as “total preparation”, because the complete 3 kg sample submitted by the geologists is pulverized before sub-sampling. During these laboratory audits, each laboratory manager was asked if this 3 kg pulverizing equipment produced a pulp equal to or better in quality than the smaller 1 kg shatter box pulverizers also in common use by commercial laboratories. Each of the laboratory managers indicated that the large pulverizer actually produced a pulp product that was *inferior* in grain size specifications to the 1 kg shatter box pulverizers. The laboratory managers furthermore admitted that the larger pulverizers were used solely because the clients requested the 3 kg pulp in the belief that it results in significantly better sub-sampling (preparation) precision than a 1 kg pulp. This purportedly improved precision was thought to be especially important for samples containing a significant “rare grain” or nugget effect.

Actual sampling, preparation and analytical quality control data gathered over the past 12 years by both authors from a wide variety of mineral deposit types, many of which contain a significant nugget effect, have shown this belief to be misguided. This brief communication presents the actual precision levels measured from two representative types of gold deposits using duplicate samples collected at each step of sample treatment (field sampling, preparation and analysis). In all cases, sample preparation involved the submission of samples larger than 3 kg, homogenization of the large sample before final pulverization, and the use of a 1 kg pulverizer. The overall precision levels observed in these case histories are then compared with the overall precision levels predicted (by Poisson statistics) to occur if a 3 kg pulverizer were used to prepare corresponding 3 kg samples of the same material. Results indicate that no substantial advantage is offered by preparation of a 3 kg pulp because the field sampling error represents the vast majority of the non-geological variations observed in the samples.

### Sample Preparation Scheme Observed

During auditing of the laboratories, several procedural problems were identified that could affect the quality of subsequent analytical results. The laboratories typically receive rock, drill core, or drill cutting samples in calico bags that are, in turn, shipped from the field in woven polypropylene bags. Unfortunately, calico bags containing dry drill cuttings sometimes exhibit contamination and sample loss problems, due to sample leakage via pores in the cloth, as well as splits in the bags (Figure 1). Consequently, it is recommended that sturdy plastic bags should be used for samples and sample shipment.



Figure 1- Dry samples received by the laboratory in calico bags may split and leak drill cuttings from one sample to the next during shipment. Spilled material is evident by the footprints on the floor.

In addition, the audited laboratories treat wet or damp samples in significantly different ways than dry samples, and this difference can produce additional errors during preparation. Some laboratories dry every sample in its calico bag before beginning sample preparation. Other laboratories remove the samples from their calico bags and place them in drying pans for drying. Other laboratories do not dry samples at all before beginning the preparation process. The amount of moisture in the samples will influence the efficiency of the sample preparation process, in particular pulverization, and this could cause significant errors and biases in the subsequent geochemical analyses.

The clients of the audited laboratories have been instructed to send no more than 3 kg of sample so that it fits directly into the pulverizer bowl. However, many samples received by the laboratories exceed 3 kg. This is particularly true for samples obtained from reverse circulation or blast hole drilling projects, as more than 3 kg of material is generally available. When samples exceed 3 kg, the laboratory should pass the entire sample through a jaw crusher to reduce its mean grain size, homogenize the sample using appropriate homogenization equipment, split off the excess reject, and then pulverize the 3 kg sample. However, several laboratories simply split off the excess sample without any pre-treatment (grain size reduction or homogenization) and discard it (Figure 2). This clearly imposes representativity problems that can severely affect overall measurement precision.

Under the ‘total preparation’ protocol, the 3 kg sample is pulverized so that nominally 95 % of the material is less than 105 microns (-150 mesh) or similar size. Then, a 250 g

continued on page 22

## Technical Note: Dispelling Some Myths...

continued from page 21



Figure 2 – Splitter used to reduce oversized samples to 3 kg before pulverizing. Samples are typically split without pre-treatment by crushing and homogenization. The excess sample is discarded.

scoop is taken from the top of the fixed pulverizer bowl and placed in a pulp bag for analysis. As the 250 g scoop cannot be collected in a geometrically representative manner because the large pulverizer bowl cannot be moved, further potential for sample bias is introduced. The excess pulp is placed either in the original calico bag or in a new plastic bag and stored for future reference.

Although all audited laboratories collect a pulp duplicate in each batch for analysis at some stage of the preparation process, only one laboratory actually routinely examines the long-term duplicate data to assess pulverized sample precision. Unfortunately, because of the presence of nuggets, the pulp from which these duplicates are derived may not be homogeneous, so these pulp replicates are affected by pulp and analytical error. As a result, an estimate of sample preparation precision is not typically possible using the 'total preparation' procedure. Nevertheless, geologists or geochemists who evaluate the pulp duplicate data can obtain an idea of the magnitude of pulp plus analytical error in their samples by using precision measurement techniques described below.

### Sample Preparation Scheme Employed

An alternative sample preparation scheme for the resource delineation projects used as examples in this paper permits the step-wise calculation of precision at each stage of sample size reduction (sampling, pulverizing, and analysis).

The initial field sample size is not critical to this protocol. A 10 kg split from a reverse circulation drilling program can be handled in the same manner as a 4 kg sample of 1 m of half BQ core that is split or cut in the field. Nevertheless, a

duplicate sample must be obtained at each stage of the sample size reduction process, from the field sample to the final, weighed analytical pulp, in order to allow estimation of the actual sampling/sub-sampling error introduced at each sample size reduction stage. This preparation scheme is as follows:

1. Field samples are collected from splits of drill core, reverse-circulation (RC) or blast hole (BH) cuttings or outcrop. Ideally, RC and BH samples should be obtained directly from the drill discharge as drilling progresses. Drill core samples should be obtained by cutting, as this prevents biases associated with 'hand cobbing' from taking place.
2. A 'field' or 'sampling' duplicate is collected from the same sampling interval as the original sample at a frequency commensurate with the size of the exploration program. In RC or BH drilling programs, two representative splits of mass equal to normal samples represent adequate duplicate samples. These splits are best obtained by passing the material from the entire sampling interval through a riffle splitter of Jones or Gilson design. Opposite halves of the same core comprise ideal sampling duplicates from drill core; however, quarters or thirds of core sampling duplicates have been collected by some companies and suffice as long as their weaknesses are appreciated. Because of the different sample masses of these smaller duplicates, calculated precision levels overestimate sampling errors in larger half core samples. Fortunately, these biases imposed by different sample supports can be corrected using ideal theoretical (Poisson statistical) models. Duplicate samples are selected according to a random-stratified regimen to ensure that one duplicate sample occurs in each analytical batch. Each sampling duplicate is given a separate number so that it is blind to the laboratory.
3. Once received by the laboratory, the entire sample is removed from the bag and placed in a drying pan of sufficient size and robustness to handle the larger sample. The entire sample is then dried.
4. After drying, the sample is removed from the drying pan and jaw crushed to reduce its fragment size so that 95 % of the sample is less than 2 mm in size (monitored by subsequent screen tests).
5. This crushed 2 mm material is then passed through an appropriately sized riffle splitter several times to homogenize it before a final split of 1 kg is collected. The residual material is stored as a coarse reject, lest sample miss-ordering or other mishaps to the pulp occur (in one case, a fire) during analysis.
6. As above, a 1 kg duplicate of the coarse reject is again collected randomly for each analytical batch. This 'preparation' duplicate is subsequently given a distinguishing sample number and treated like a normal sample.
7. The one kilogram sample is then pulverized using a one kilogram shatter box bowl such that at least 95 % of the material is less than 105 microns (-150 mesh) in size (again, monitored by screen tests).

continued on page 23

## Technical Note: Dispelling Some Myths...

continued from page 22

8. When pulverized, the removable shatter box bowl is turned upside down on a clean mat so that the entire pulverized pulp forms a conical pile. A representative split of the pulp, usually 250 g, is then taken from the pile on the mat for analysis. The remaining pulp reject (approximately 750 g) is stored for future reference or for screen metallics analysis if a severe nugget effect is suspected. Note that a second 250 gram pulp duplicate sample does not routinely need to be collected, because laboratories routinely collect and analyze 'pulp' duplicates as part of their analytical quality control measures.
9. Finally, for gold, a 50 gram sub-sample from each 250 g sample is weighed out and analyzed by fire assay techniques. Note that because no grain size reduction actually takes place after collection of the 250 grams of pulp, this sub-sampling stage is actually 1 kg to 50 g, instead of 1 kg to 250 g, or 250 g to 50 g.

### Calculation of Step-Wise Measurement Precision

Actual precision estimates for the case histories presented in this paper have been made using duplicate samples from all stages of the sample size reduction process (initial sampling in the field, sub-sampling to a 1 kg sub-sample after jaw crushing, and further sub-sampling to 50 g analyte after pulverization; Figure 3, Path A). In this instance, three stages of duplicates are required for the sample preparation scheme described above.

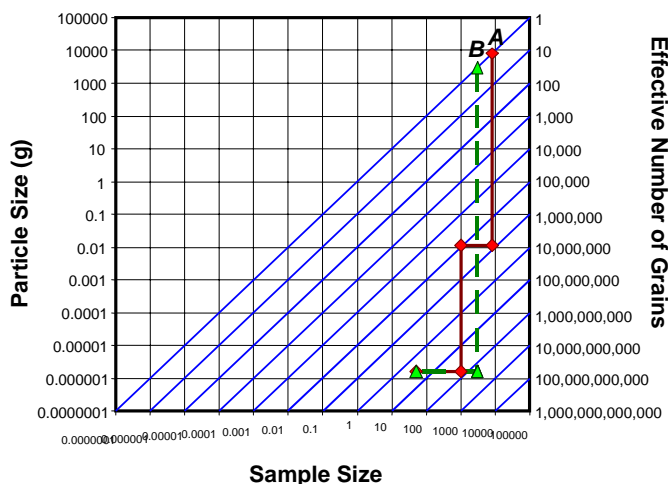


Figure 3 – Sample preparation diagram illustrating the particle and sample size reduction path followed by the initial 8 kg samples from the intrusion-hosted Au deposit example presented (A), and a hypothetical path followed by 3 kg samples that undergo 'total preparation' (B), as defined by the audited laboratories. In both cases, a 50 gram sub-sample is ultimately used in analysis. Diagonal lines and the axis on right describe the number of equant grains that would occur in the samples/sub-samples.

'Sampling duplicates' are collected by the geologist, and record the cumulative uncertainties associated with the entire sampling and analytical process. That is: (i) the error introduced through the collection of only part of the core, RC or BH cutting, or rock mass for assay; (ii) the error in sub-sampling a jaw crushed sample for pulverizing; and (iii) the error in sub-sampling the pulverized sub-sample for assay

analysis, plus analytical error. 'Preparation duplicates' are collected by the laboratory, and report the errors due to sample size reduction after crushing, and the errors associated with weighing and analysis of the pulp. 'Pulp duplicates' are also collected by the laboratory, most of the time routinely, and report the magnitude of sub-sampling error associated with taking 50 grams from a 1 kg pulp, and any subsequent weighing and analytical error.

All duplicates were obtained at random, without regard for mineralization type, grade, or lithology. Any bias in the duplicate sampling program will negate the validity of subsequent precision calculations, so all duplicate pairs were analyzed with the original samples in the same sample analytical batch. Duplicates that were analyzed by a different laboratory, or at a later time, (such as in check assays) can introduce variance artefacts that are not related to sampling precision, and so were not included in these data evaluations.

The statistical technique used to estimate the actual precision levels at each stage of sample treatment was the modified Thompson-Howarth method (Stanley 2003). It, or Thompson and Howarth's original method (Thompson and Howarth 1973, 1976a, 1978; Thompson 1973, 1982; Fletcher 1981; Stanley and Sinclair 1986), represents the preferred precision estimation procedure. In all cases, the regression employed initially fits a line through the data using both slope and intercept. However, in all cases, the magnitude of the intercept was so small that the error was virtually always proportional in character. As a result, a second 'proportional' model was subsequently regressed (involving fitting only a slope, or relative error term), as a simple proportional error model significantly simplifies the subsequent error calculations in this paper.

By assembling and evaluating the field sampling, preparation, and pulp duplicate results, the errors associated with each step of sample treatment can be estimated by subtracting 'downstream error' from each composite error estimate. The source of the greatest uncertainty in the sampling and analytical program can thus be quantified and targeted for reduction through procedural modifications, if necessary.

### Examples of Sampling and Analytical Precision

Two representative case history examples of sampling precision estimation programs are presented from different types of gold mineralization. Both mineralization types exhibit significant 'nugget effects' in the gold analyses. The deposit names and locations remain confidential.

#### Intrusion-Related Gold Deposit

The hosts to gold and copper mineralization at this deposit include altered quartz diorite and granodiorite, and breccias that cross both the intrusive and surrounding andesitic volcanic rocks. Gold mineralization occurs as inclusions in disseminated sulphides and as free gold or electrum on fracture surfaces or in breccia matrices. The gold inclusions appear to be well distributed and do not present a significant sampling problem. Gold within the volcanic rocks and enclosed breccias typically has a larger grain size that contributes to a significant amount of sampling

continued on page 24

### Technical Note: Dispelling Some Myths...

*continued from page 23*

and sub-sampling variability at all stages of sample treatment. The average weight of the two meter long, half core samples was 8 kg.

The three types of duplicate pairs: ‘sampling’ ( $n = 3488$ ), ‘preparation’ ( $n = 1573$ ) and ‘pulp’ ( $n = 1410$ ), were collected during a major drilling project. Relationships between sampling errors and concentrations were determined using the modified Thompson-Howarth technique (Stanley 2003). In all cases, a proportional model was fitted to the duplicate data. One standard deviation relative errors for sample, preparation and pulp duplicates are 17.1 %, 9.0 % and 4.6 %, respectively. By converting these to relative variances via squaring the relative one standard deviation errors, subtracting the relative pulp variance from the relative preparation variance, subtracting the relative preparation variance from the relative sampling variance, and re-expressing the results as relative errors by taking their square roots, the actual relative sampling, preparation and pulp errors can be determined. Total relative error, measured by the sample duplicates, is thus 17.1 %, and all component errors (14.5 %, 7.7 % and 4.6 %, respectively) are presented in Figure 4 to illustrate the relative magnitudes of each error component.

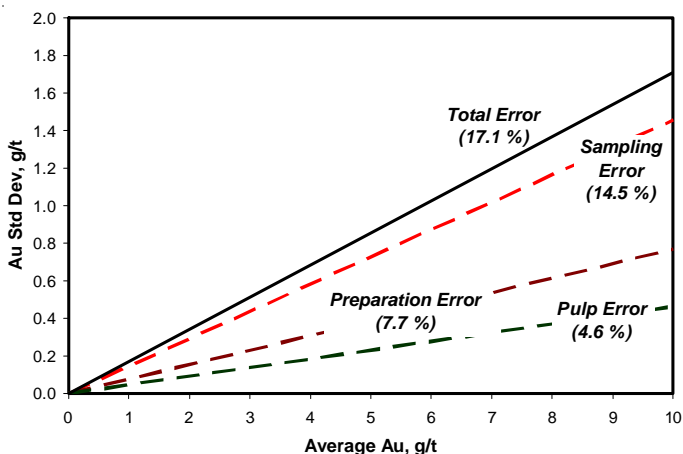


Figure 4 – Modified Thompson-Howarth-type plot illustrating the magnitude of total, sampling (8 kg), preparation (1 kg) and pulp (50 g) error, as determined by duplicate quality control monitoring for preparation protocol (A) on Figure 3, from an anonymous intrusion-related gold deposit.

Note that, based on the associated relative variances, more than 72 % of the total variation created by the entire sampling and analytical process is introduced during cutting (sampling) of the drill core. Only 20 % of the total variation is attributed to splitting the crushed core to 1 kg, and 7 % of the variation is attributed to the splitting and weighing of 50 g of pulp and analysis.

Given the above sampling, preparation and analytical errors, hypothetical errors can be deduced for alternative sampling, preparation and analysis protocols using Poisson statistics. The variance of the number of rare events (a Poisson variable, such as nuggets) is known to be:

$$\sigma_x^2 = x$$

(Speigel 1975), where  $x$  is the number of ‘equant’ nuggets in the sample (Clifton *et al.* 1969; Stanley 1998). Rearranging this equation by inverting it and distributing the variance into standard deviations, we obtain:

$$\frac{\sigma_x}{x} = \frac{1}{\sigma_x} = \frac{1}{\sqrt{x}}$$

Because the relative error on the effective number of nuggets in a sample ( $\delta_x/x$ ) must equal the relative error on the element concentration ( $\delta_c/c$ ) in the sample, the relative sampling error on the concentration is related to the number of equant nuggets in a sample. Equation 2 thus allows one to deduce the change in relative error that will occur as a result of a change in sample size.

For example, if 3 kg samples from this intrusion-related gold deposit were collected (instead of 8 kg) and prepared using the ‘total preparation’ method described above (Figure 3, Path B), one can estimate the new sampling error that would result using Equation 2. The original sampling error of 14.5 % allows one to determine that, on average, 47 equant grain nuggets would be present in an 8 kg sample (Clifton *et al.* 1969; Stanley 1998). As a result, if 47 equant grain nuggets were contained in 8 kg,  $3/8 \times 47 (= 18)$  nuggets should, on average, be contained in a 3 kg sample. Using Equation 2, again, the relative sampling error associated with collection of 3 kg samples containing 18 nuggets should be 23.8 %. This is obviously larger than the 14.5 % sampling error for the 8 kg samples because the 3 kg samples are smaller.

Given the above, the total sampling error associated with the ‘total preparation’ protocol, had it been applied to samples from the intrusion-related gold deposit, can be deduced. Sampling error, pulp error, and total error are presented in Figure 5. Note that no preparation error is included, because no sample size reduction occurs during preparation, as the entire 3 kg sample is pulverized. Furthermore, because the pulp in both cases is 50 g in mass, the pulp error observed in the original sample preparation protocol can be used for the ‘total preparation’ protocol.

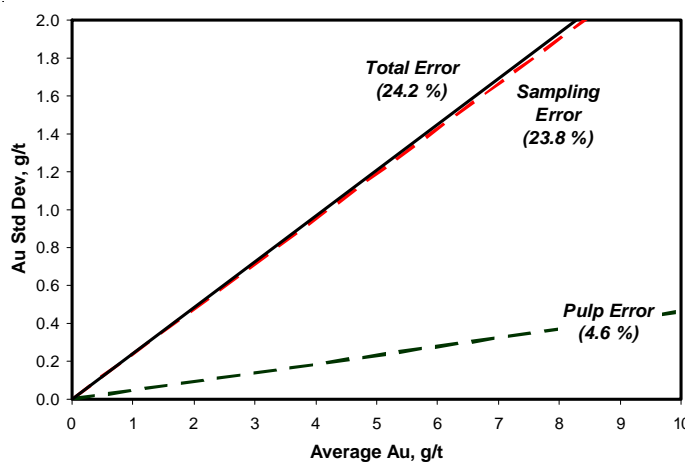


Figure 5 – Modified Thompson-Howarth-type plot illustrating the Poisson-predicted magnitudes of total, sampling (3 kg), and pulp (50 g) error if the ‘total preparation’ protocol (B) on Figure 3 were used to process the samples from the intrusion-related gold deposit.

## Technical Note: Dispelling Some Myths...

*continued from page 24*

Comparing the results in Figures 4 and 5, we see that the ‘total preparation’ protocol (Figure 3, Path B) actually introduces 24.2 % relative error, and thus creates 62 % more error than the original 8 kg => 1 kg => 50 gram sample preparation protocol (Figure 3, Path A). Although the ‘total preparation’ protocol does not introduce the 7.7% preparation error that the original protocol did, the larger sampling error caused by a smaller initial sample overwhelms this preparation error reduction, primarily because sampling error is the largest component of error. Although the ‘total preparation’ protocol appears superior at first, the limited sample size required by this approach actually makes it significantly inferior!

### Epithermal Low Sulphidation Gold Deposit

This gold deposit is hosted by structurally controlled quartz silica breccia and banded quartz veins. Some gold mineralization exhibits high to very high grades (3 to 1000 g/t) and significant visible gold occurs in the core. Consequently, a very high nugget effect can occur in these samples. During core cutting, the core was orientated so that the veins, if present, were cut as evenly as possible.

A similar sample preparation protocol was followed at this deposit. Because core sample masses range from 3 to 6 kg depending on the core length, an average core mass of 4.5 kg has been used. Sample preparation also involved jaw crushing to 95 % less than 2 mm, homogenization and splitting of 1 kg of material for pulverization to 95 % less than 105 μm, and then splitting of 50 grams for analysis (Figure 3, Path A). A similar duplicate sampling procedure was also followed to determine the magnitude of sampling, preparation and analytical errors associated with assaying.

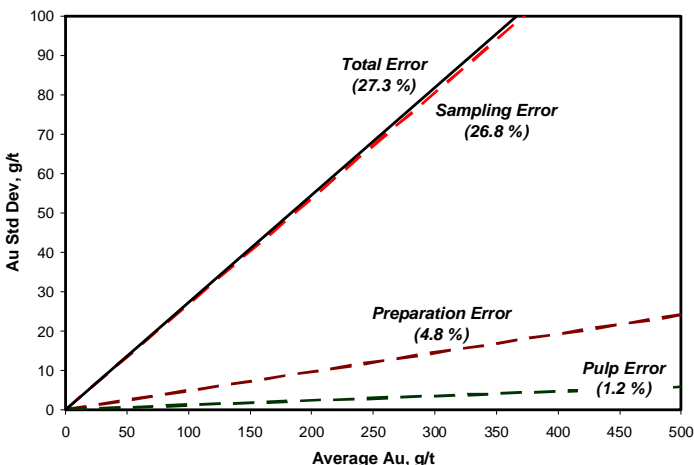


Figure 6 – Modified Thompson-Howarth-type plot illustrating the magnitude of total, sampling (4.5 kg), preparation (1 kg) and pulp (50 g) error, as determined by duplicate quality control monitoring for a preparation protocol similar to that of Path A on Figure 3, from an anonymous low sulphidation epithermal gold deposit.

Modified Thompson-Howarth analysis of 637 field sampling duplicates, 501 preparation duplicates, and 501 pulp duplicates was undertaken to determine the various component errors. Results from analysis of sample, preparation and pulp duplicates indicate that these relative

errors are 27.3 %, 4.9 % and 1.2 %, respectively. Converting these to variances (as above), subtracting pulp error from preparation error, and subtracting preparation error from sampling error, to isolate the individual errors, results in 26.8 %, 4.8 % and 1.2 % errors for sampling, preparation and pulp procedures. Because of the larger nugget effect in this deposit, this sampling error constitutes 96.7 % of the variance, whereas preparation and pulp errors constitute only 3.1 and 0.2 % of the variance, respectively.

As above, Equation 2 can be used to determine the total error that would occur if the ‘total preparation’ sampling, preparation and analysis protocol were employed for samples from this low sulphidation epithermal gold deposit (Figure 3, Path B). Results are presented in Figure 7, and indicate that the smaller 3 kg initial sample creates virtually all of the error observed. This error (32.9 %) is again substantially larger than the error produced using the original sampling protocol involving a 4.5 kg sample (26.8 %). Thus using a 3 kg pulverizer does not guarantee the geologist or geochemist that they are using the ideal sample preparation protocol!

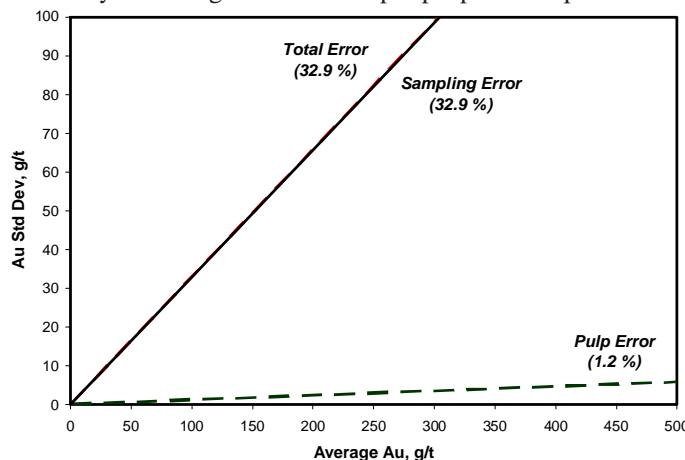


Figure 7 – Modified Thompson-Howarth-type plot illustrating the Poisson-predicted magnitudes of total, sampling (3 kg), and pulp (50 g) error if the ‘total preparation’ protocol (B) on Figure 3 were used to process the samples from the low sulphidation epithermal gold deposit.

### Conclusions

Experience by the authors has shown that the two gold deposit case histories presented involve sampling, preparation and pulp error magnitudes that are representative of many mineral deposits exhibiting nugget effects. At these deposits, geologists should expect the largest component of measurement error to be introduced during the initial field sampling stage. As a result, significant efforts to reduce overall measurement error should focus on this initial stage of the sample treatment procedure, as this is the place where the most benefit can be achieved with the least amount of effort (and cost). Field samples that are as large as is economically possible to handle should be submitted to the laboratory for crushing, homogenization, splitting and pulverization.

Efforts to reduce measurement error by pulverizing large sample masses are shown to be mis-guided, as the initial limitation on sample size (3 kg) typically imposes far more error than is eliminated through the subsequent pulverization

*continued on page 26*

## Technical Note: Dispelling Some Myths...

continued from page 25

of large sample masses. Furthermore, problems associated with pulverization of large sample masses (cross-contamination due to incomplete cleaning of unwieldy equipment, lack of representativity due to arbitrary reduction of sample masses to fit the pulverizing equipment by the laboratory, etc.) can be easily avoided using smaller volume pulverizing equipment and a two-stage sample reduction protocol, without imposing un-acceptable measurement errors. Use of the 'total preparation' sample reduction protocol can actually increase measurement error, resulting eventually in higher capital costs to the future mining venture because the larger errors result in more uncertainty and higher risk.

In summary, geologists and geochemists undertaking drilling programs and assaying samples on exploration projects should familiarize themselves with the sampling theory necessary to understand the limitations of the techniques they employ, so that they can effectively produce quality assay information with acceptable errors for use in subsequent mineral resource evaluations.

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