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Sampling Errors and Control of Assay Data Quality in Exploration and Mining Geology

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1. Introduction

The fundamental cause of the errors of samples of rocks and minerals collected by geologists for evaluation of mining projects is heterogeneity of the sampled materials (Gy, 1982; Francois-Bongarcon, 1993; Pitard, 1993). Constitution heterogeneity and distribution heterogeneity (Pitard, 1993) both are important and cause geological sampling errors. The more heterogeneous the sampled material the more difficult it is to obtain a representative sample and infer characteristics of the geological object from samples. The current chapter overviews sampling theory explaining sampling error types and their likely causes, and also describes the practical approaches used in the mining industry for estimating sampling errors and monitoring them at an acceptably low level. It is based on numerous case studies by the author (Abzalov & Both, 1997; Abzalov, 1999, 2007, 2008; Abzalov & Humphreys, 2002; Abzalov & Mazzoni, 2004; Abzalov & Pickers, 2005; Abzalov et al., 2007; Abzalov & Bower, 2009) and also reviews of the recently published QAQC procedures used in the mining industry (Taylor, 1987; Vallee et al., 1992; Leaver et al., 1997; Long, 1998; Sketchley, 1998).

2. Types of sampling errors

Sampling errors are traditionally determined in terms of precision and accuracy of the data (Fig. 1). Precision, or repeatability, is a measure of how close sample values are to one another (Fig. 1) and accuracy is a measure of how close the sample value to the true grade (Fig. 1). Both of these parameters have to be estimated and strictly monitored during evaluation and the eventual exploitation of mineral deposits.

These errors can be generated at any stage of the samples extraction, preparation and the eventual analytical assaying. Depending on the factors causing sample errors they are grouped into three types (Eq. 1):

$$\text{TOTAL ERROR} = \text{Err. 1}^{\text{st}}\ \text{Group} + \text{Err. 2}^{\text{nd}}\ \text{Group} + \text{Err.3}^{\text{rd}}\ \text{Group} \tag{1}$$

Where:

Err.1st Group – are sampling errors related to a chosen sample extraction and preparation procedure, referred as sampling protocol. An example is poor repeatability of assays when sample sizes are disproportionately small in comparison with the degree of heterogeneity of

material. The main error of this type is known as Fundamental Sampling Error (Gy, 1982). It is always present and can not be fully eliminated as it is related to intrinsic characteristics of the sampled material, such as mineralogy and texture of mineralisation. The Fundamental Sampling Error (FSE) can be minimised through optimisation of the sampling protocols, which will be discussed in the next section. The first group also includes Grouping-Segregation error which is a consequence of the distribution heterogeneity of the sampled material (Pitard, 1993) and therefore this error also relates to the intrinsic characteristics of the sampled material.

Err.2nd Group – is the group of errors related to sampling practise, in other words the errors which depend on how rigorously the sampling protocol was developed, implemented and followed. The group includes delimitation, extraction, preparation and weighing errors. These errors are caused by incorrect extraction of the samples from a lot, their suboptimal preparation procedures, contamination and incorrect measurements. Human errors, such as mixed sample numbers, can also be included in this group. These types of errors can be minimised by upgrading practices of the samples extraction and preparation, which usually needs an improvement of the quality control procedures and often requires equipment upgrading.

Err.3rd Group – analytical and instrumental errors occurred during the analytical operations (Gy, 1982). The group includes assaying, moisture analysis, weighing of the aliquots, density analysis, precision errors and bias caused by suboptimal performance of analytical instruments. These errors are considered in the current study separately from the two first groups because of the different factors causing them.
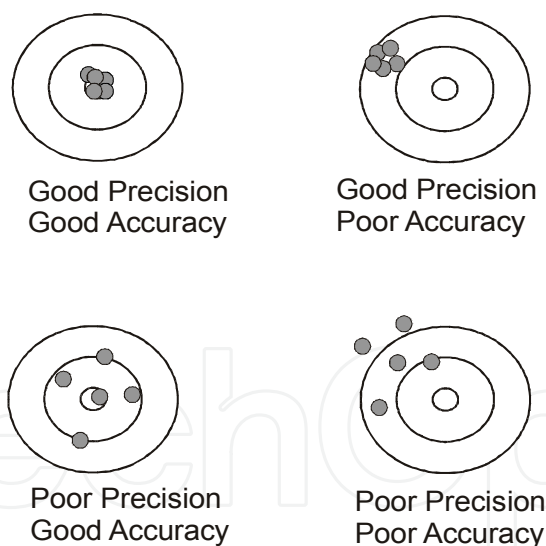

Fig. 1. Sketch explaining precision and accuracy of the data

2.1 Fundamental Sampling Error

Fundamental Sampling Error (FSE) is error related to constitution heterogeneity of the sampled material. It depends on the shape and size of the particles which constitute the sampled material, the size at which the critical components are liberated and also on mineralogy and density of gangue and valuable components. It is the only error which can be theoretically determined as it directly related to the constitutional characteristics of the sampled materials.

a.   Theoretical background.

The theoretical approach for estimating the FSE was proposed by P.Gy (1982) and further developed by F.Pitard (1993) and D.Francois-Bongarcon (1993, 1998, 2005). The theory states that FSE, representing precision of the samples expressed as their relative variance, can be estimated as follows (Eq. 2):

$$\sigma_{FSE}^2 = fgcld_N^3 \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \tag{2}$$

σ_{FSE}^2 - Fundamental Sampling Error, representing relative variance of the precision error;

f - shape factor. This parameter represents geometry of the particulate materials. It is a dimensionless factor varying from zero, when particles are ideal cubes, to one, when they represented by ideal spheres. Most types of mineralisation have shape factor varying in a narrow range from 0.2 (gold or mica flakes) to 0.5 (isometric grains).

g - granulometric factor, which is also called a particle size distribution coefficient or size range factor. This factor is dimensionless and taking into account the fact that fragments do not have the same size (d). If all fragments have had exactly the same size the factor (g) would be equal to 1. This theoretically is possible only in an ideal case when studied material is perfectly sorted. In practice it never happens, therefore the (g) factor is less than one and can be as small as 0.1 when particles show a wide range of distribution. Default values of (g) factor are summarised in Table 1. In the mining industry the value of 0.25 is usually used as default value as it suits for most types of mineralisation and corresponds to a case when 95% of particles pass the nominal mesh size.

Type	Explanation	Default (g) value
Non sorted material	Output of jaw crusher	0.25
Sorted material	Material between two consecutive screen openings	0.55
Naturally sorted material	Grains, e.g. rice	0.75

Table 1. Default values of the granulometric factor (Pitard, 1993)

d_N - a nominal particle size in centimetres. This is size (diameter) of a mesh retaining the upper 5% of particles.

M_S - mass of sample in grams.

M_L - mass of lot in grams.

c - mineralogical composition factor (g/cm³) calculated using (Eq. 3)

$$c = \left(\frac{1-t_L}{t_L}\right) \times \left(\rho_M \left(1-t_L\right) + \rho_G t_L\right) \tag{3}$$

where:

t_L - absolute grade of a lot expressed as decimal proportions of ore mineral, it changes from 0 to 1 (e.g. 1g/t = 0.000001), ρ_M - specific gravity of ore minerals, ρ_G - specific gravity of gangue.

The formula (Eq. 3) can be simplified (Francois-Bongarcon, 1998) and represented by it's concise version (Eq. 4).

$$c = (\frac{1\text{-}t_L}{t_L}) \times (\frac{\rho_M \times \rho_G}{\rho}) \tag{4}$$

In the equation (Eq. 4) (ρ) denotes the average specific gravity of mineralisation at a given grade (t_L), other variables are the same as in Eq. 3.
For low-grade ores, a mineralogical factor (c) can be further simplified and approximated as ratio of the density of the mineral of interest by the average grade of the studied material (Eq. 5):

$$c = \frac{\rho_M}{t_L} \tag{5}$$

The mineralogical factor (c) relates the sampling variance given by formula (Eq. 2) to the grade of mineralisation (lot) being sampled. D.Francois-Bongarson and P.Gy (Francois-Bongarson & Gy, 2001) have noted that 'any use of the formula, or any sampling nomogram derived from it, only makes sense when the grade level at which it is established is duly stated'.
l - liberation factor, estimated as ratio of liberation size to a nominal particle size (Eq. 6).

$$l = (\frac{d_L}{d_N})^A \tag{6}$$

where: d_N - a nominal particle size in centimetres, d_L - liberation size in centimetres, representing a liberation diameter of a mineral of interest, A - exponent.
Substituting liberation factor (Eq. 6) to equality defining FSE it becomes (Eq. 7):

$$\sigma^2_{FSE} = fgc \ (\frac{d_L}{d_N})^A d_N^3 \ (\frac{1}{M_S}\text{-}\frac{1}{M_L}) \tag{7}$$

If the exponent (A) is expressed as ($3-\alpha$) and after reduction of (d_N^3) the FSE formula becomes (Eq. 8)

$$\sigma^2_{FSE} = f \ g \ c \ d_L^{3-\alpha} d_N^\alpha \ (\frac{1}{M_S} \text{-} \frac{1}{M_L}) \tag{8}$$

Product of ($f \ g \ c \ d_L^{3-\alpha}$) is known as the sampling constant (Francois-Bongarson, 1993; De Castilho et al., 2005) and usually is denoted as (K).

$$K = f \ g \ c \ d_L^{3-\alpha} \tag{9}$$

Substituting sampling constant (K) to equality (Eq. 2) leads to formula of FSE (Eq. 10) which is most commonly used in practice.

$$\sigma^2_{FSE} = K d_N^\alpha \ (\frac{1}{M_S} \text{-} \frac{1}{M_L}) \tag{10}$$

The value of exponent (α) changes depending on (d_N). When (d_N) is smaller than liberation size (d_L) the exponent (α) is equal 3. Above the liberation size (d_L) the exponent (α) can be smaller, within the range of 1 to 3.

Equality (Eq. 10) can be further simplified by removing the ratio ($\frac{1}{M_L}$) which becomes negligibly small when the mass of a lot (M_L) is significantly larger than sample mass (M_S), which leads to concise version of the FSE formula (Eq. 11)

$$\sigma^2_{FSE} = \frac{K\,d_N^{\alpha}}{M_S} \tag{11}$$

Equality (Eq. 10) and its concise version (Eq. 11) are practically the most convenient tools for experimental definition of the FSE because parameters (K) and (α) can be calibrated experimentally (Francois-Bongarson, 1993, 2005). Methods of calibration are discussed in the next section of the book. When calibrated parameters are not available D.Francois-Bongarcon (1993) has suggested default (K) and (α) values for low-grade mineralisation, such as gold veins, which are K = 470 and α =1.5. However, great care should be taken as actual values of the sampling constant (K) can significantly differ from the default value (Sketchley, 1998).

b.   Experimental calibration of sampling constants.

Several techniques have been proposed (Gy, 1982; Pitard, 1993; Francois-Bongarson, 1993, 2005; Bartlett & Viljoen, 2002; Minkkinen & Paakkunainen, 2005; De Castilho et al., 2005; Minnitt et al., 2007) for experimental determination of sampling constants. The most common approach is the technique developed by Francois-Bongarson (2005), representing a modified version of the 'sampling tree experiment' (Francois-Bongarson, 1993), and 'heterogeneity test' of Pitard (1993). '30-Pieces Experiment' developed by D.Francois-Bongarson (1993) has many similarities to above mentioned 'heterogeneity test' (Pitard, 1993) representing a simplified version of it.

'Sampling Tree Experiment' was first proposed by D. Francois-Bongarcon in 1993 and then modified in 2005 (Francois-Bongarson, 2005). The modified version represents analysis of the series of the duplicate samples (Fig. 2) cut from a lot at various comminution degrees (Table 3) allowing to experimentally obtain the (K) and (α) parameters of the Fundamental Sampling Error (Eq. 11).
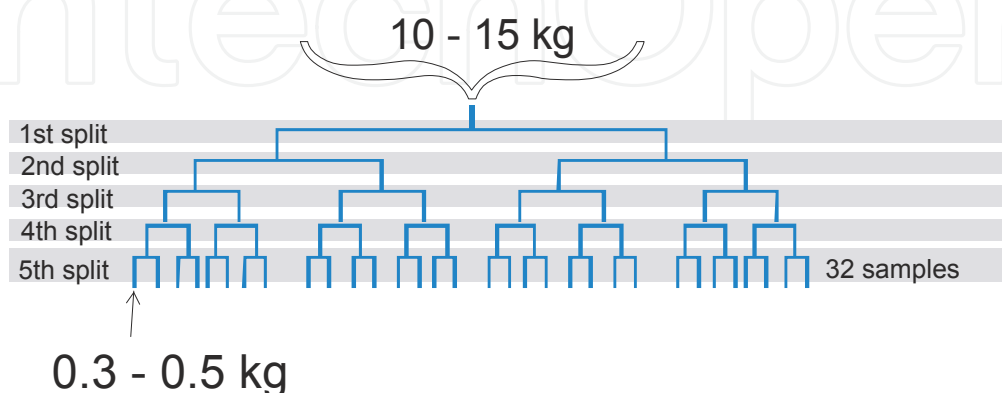

Fig. 2. Flow sheet of the 'Modified Sampling Tree Experiment' (MSTE). The shown binary sampling tree is applied to each of the four nominal size fractions (Table 2)

Theoretical background of this method is as follows. Firstly the formula (Eq. 11) can be logarithmically transformed to the equality Eq. 12:

$$\text{Ln}\,(M_S\sigma_{FSE}^2) = \alpha\,\text{Ln}(d_N) + \text{Ln}(K) \tag{12}$$

According to this expression the values of $[\,\text{Ln}\,(M_S\sigma_{FSE}^2)\,]$ are plotted against the particle sizes $[\,\text{Ln}\,(d_N)\,]$ as a straight line because the equality (Eq. 12) represents equation of the line ($Y = AX + B$). Tangent of the angle between this line and the abscissa axis (A) is equal to exponent (α) in the equality Eq. 12 and constant (B) is equal to $[\,\text{Ln}(K)\,]$. The objective of the 'MSTE' is to deduce parameters (A) and (B) of a linear function describing relationships between ($M_S\sigma_{FSE}^2$) values and the particle sizes (d_N). In practise, to infer parameters of a linear function $[\,\text{Ln}\,(M_S\sigma_{FSE}^2) = \alpha\,\text{Ln}(d_N) + \text{Ln}(K)\,]$ it is sufficient to experimentally obtain several points which are plotted onto the diagram $\text{Ln}\,(M_S\sigma_{FSE}^2)$ vs. $\text{Ln}(d_N)$ and then a linear function is inferred by a suitable best fit algorithm.

'MSTE' method is based on collecting a representative sample of 40-60kg which is then dried, successively crushed and split following the flow sheet shown on the Fig. 2. The nominal particle sizes for the four groups of subsamples depend on mineralogy and texture of the mineralisation. Examples of the particle sizes that have been used at the 'MSTE' are shown in Table 2 which can be used as a reference when 'MSTE' is planned; however, best practise is to determine experimentally the sample weight and the nominal particle size of each sampling series.

		Sampling Series				Elements of Interest	Reference
		First	Second	Third	Forth		
Deposit type	Orogenic Gold	2.5	0.3	0.1	0.05	Au, As	a, b
	Ni-S: Komatiitic-type	3	1	0.5	0.1	Ni, Cu, As	b
	Cu-Au-U: IOCG-type	2.5	0.5	0.1	0.05	Cu, U, Au, S	b
	U: unconformity-type	2.5	1	0.1	0.01	U	b
	Bauxite	3	1	0.5	0.1	Al_2O_3, SiO_2, Fe, LOI	b
	Iron ore: BIF-derived	3	1	0.5	0.1	Al_2O_3, SiO_2, Fe, LOI, P	b
	Cu-Au: porphyry-type	2.5	1	0.1	0.05	Cu, Mo, Au, As	b
LOI – Loss on Ignition							
Data used: a – (Minnitt et al., 2007); b – Abzalov, M. (unpublished data).							

Table 2. Examples of the nominal particle sizes (cm) of the samples at the "Modified Sampling Tree Experiment"

Procedure of the 'Modified Sampling Tree Experiment' is as follows (Fig. 2):
Representative sample of 40-60kg is collected and dried;
- The whole sample is crushed at jaw crusher to a nominal size of 95% passing the mesh size chosen for Series 1 (Table 2);
- One-quarter of the sample (lot) is split out and forms the first subsampling series;
- Remaining material is crushed to a nominal size of 95% passing the mesh size chosen for Series 2 (Table 2);
- One-third of these secondary crushed material is split out and forms the second subsampling series;

- Remaining two fractions are recombined and crushed to a nominal size of 95% passing the mesh size chosen for Series 3 (Table 2);
- The crushed material is split by riffle splitter onto two equal subsamples, one of them split out and forms the third subsampling series;
- The remaining material is crushed to a nominal size of 95% passing the mesh size chosen for Series 4;
- Using a riffle splitter each of these portions is now split into 32 samples (Figure 2). Each of the produced samples is weighed, pulverised and assayed. Minnitt et al. (2007) recommends to use 2 samples for granulometric analysis. These samples are randomly chosen from the group of 32 and remaining 30 samples are assayed and used for statistical inference of the (K) and (α) parameters (Fig. 3).

This approach produces 4 groups of 32 samples. Each group includes samples of the same nominal size of the particles and approximately of equal weight.
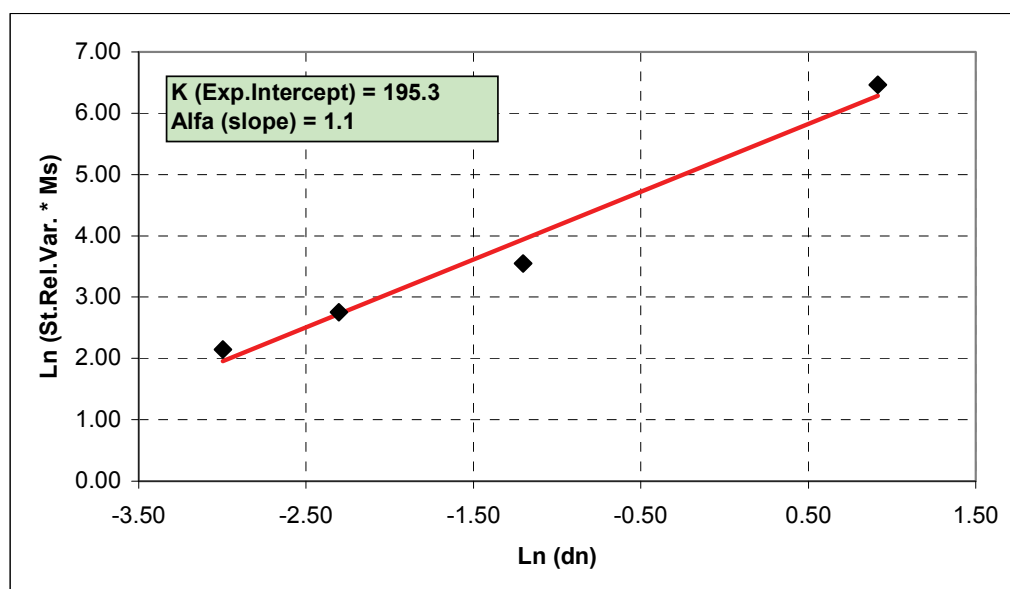

Fig. 3. Experimental calibration of the (K) and (α) parameters of the Eq. 12

c. Sampling nomogram.

Variance of the fundamental sampling error (FSE) can be graphically expressed as a function of sample weight (M_S) and the nominal particle size (d_N). Diagram representing relationships between these parameters is called nomogram (Fig. 4). This is achieved by plotting the FSE vs. the given sample mass (Pitard, 1993; Francois-Bongarcon, 1993). For practical reasons all values are plotted on the nomogram in the logarithmic coordinates (Fig. 4).

On the sampling nomogram (Fig. 4) the crushing and grinding stages, which do not contribute to sampling variance, are represented by vertical lines. The sample reduction stages, when a smaller sample is extracted from a larger sample, in other words a sample mass reduction at constant rock particle size, are represented on the diagram as a path along the straight lines of a slope -1. The actual position of the line depends on particle size (d_N) and also sampling constants (α) and (K) therefore only one line can be constructed for

each sub sampling stage at the given sample particle size (d_N). Every stage when particle size is reduced (i.e. comminution) is lowering the FSE and therefore it is represented on the nomogram by vertical line which extended until it intersects a new straight line of slope -1 corresponding to a new particle size. Combination of the vertical and diagonal segments corresponding to all stages of the sampling protocol graphically visualises the entire sample preparation protocol. Each stage contributing to the overall precision error is clearly represented on the nomogram and this allows to use this diagram as an effective tool for assessment, control and improvement of the sampling procedures.
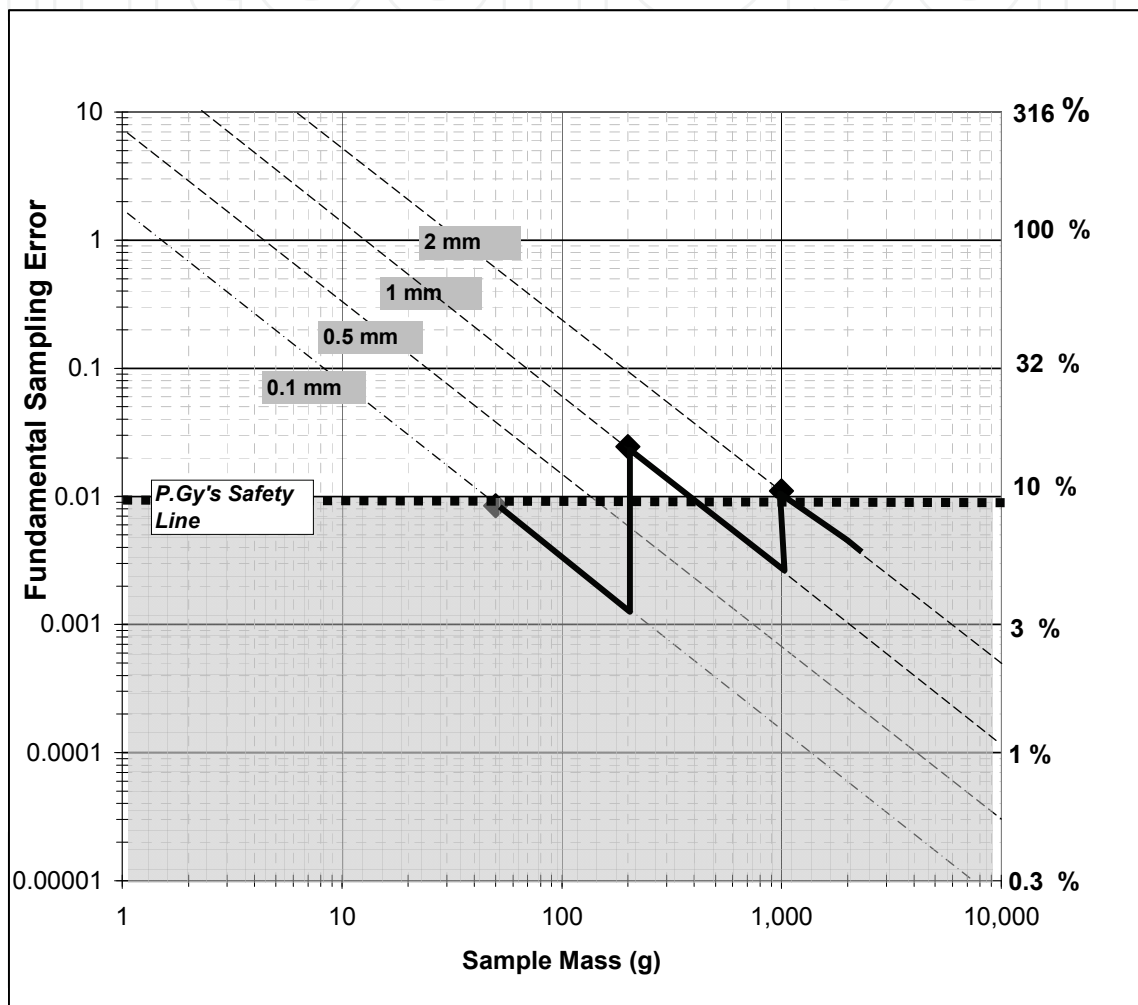

Fig. 4. Sampling nomogram, gold deposit, Canada

To facilitate interpretation of this diagram certain values of acceptable precision errors can be shown. A common practice is to draw a line corresponding to 10% relative error, or in other words the relative variance equal to 0.01 (Fig. 4). This threshold is known as P.Gy's safety line (Gy, 1982). All suboptimal sample preparation stages which FSE exceeds the chosen threshold are easily diagnosed on the nomograms. The example presented in Fig. 3 shows that the largest FSE is introduced at the second subsampling stage when 200g of sample is collected from a pulp ground to -0.5mm. This stage introduces approximately 20% precision error whereas first sub-sampling, when 1 kg was split from the material crushed to

-1mm causes a smaller error, less than 10%. Final stage when 50g aliquot was collected from a 200g pulp pulverised to -0.1mm is also characterised by less than 10% of FSE. Based on this nomogram it is obvious that improvement of the sampling protocol should be focused on optimisation of the stage 2, which can be easily achieved by collecting larger sub-sample, approximately 500g.

2.2 Grouping and segregation error

Grouping-Segregation error is generated by small scale distribution heterogeneity and reflects the differences in the content of a metal of interest between groups of fragments (increments) collected at very small intervals (Gy, 1982; Pitard, 1993). Examples of Grouping – Segregation error includes separation of fine particles from larger fragments when they are discharged from the sampling devices or conveyor belts (Fig. 5A). Another example of a possible error caused by segregation factor is accumulation of the heavier and smaller particles at the bottom of blast hole cones (Fig. 5B). This can be gold grains or sulphide minerals liberated from a gangue. Segregation can also be caused by different physical or chemical properties of the sampled materials, including magnetic or electrostatic properties, moisture content, adhesiveness (Pitard, 1993).
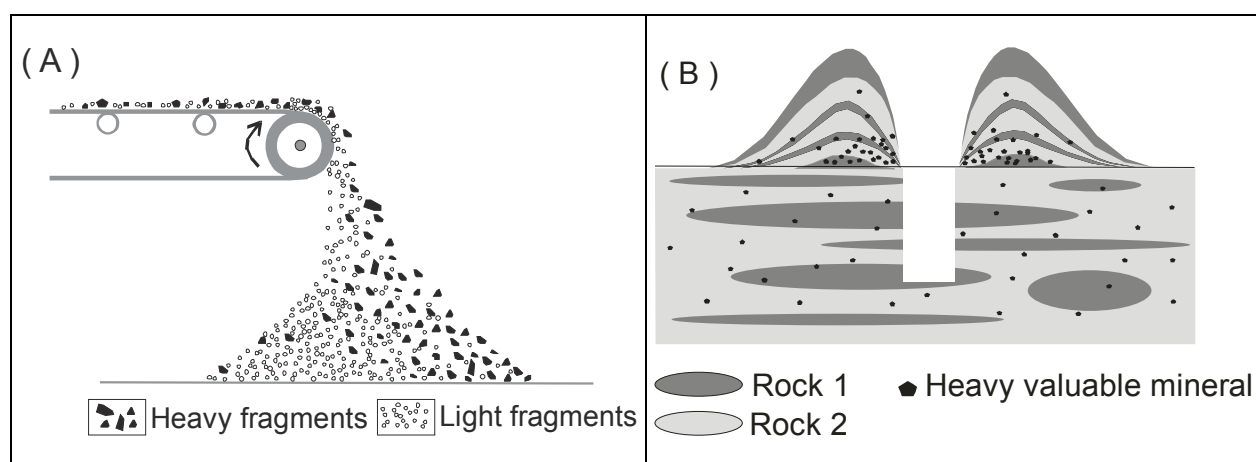

Fig. 5. Examples of segregation of the rock fragments: (A) segregation of the heavy and lighter fragments at the discharge from conveyor belt, modified after Pitard (1993); (B) uneven distribution of fragments in a blast hole cone caused by segregation of the smaller and heavier fragments (e.g. liberated gold particles, or sulphides grains) and their accumulation at the bottom of the cone

The Grouping-Segregation error represents a complementary part of the Fundamental Sampling Error (Eq. 13) and therefore has been assigned here to the first group of errors (Eq. 1) because of its direct relationships with fundamental error. However, in practice this error as much depends on sampling protocol, being controlled by the value of fundamental error (σ_{FSE}^2), as it is a consequence of the practical implementation of the sampling protocol. For example Grouping-Segregation error can be generated by insufficient homogenisation of the material prior to sampling.

The Grouping-Segregation error by definition is a function of two factors, grouping factor (f_G) and segregation factor (f_S), both being a consequence of the small scale distribution heterogeneity of the material (Eq. 13).

$$\sigma_{GS}^2 = f_G f_S \sigma_{FSE}^2 \tag{13}$$

Grouping factor in practise characterises the size of the increments (i.e. fragments) making up the sample. A segregation factor characterises the number of increments. Both of these factors are dimensionless and can not be dissociated from each other (Pitard, 1993).

Grouping-Segregation error can not be theoretically estimated, however, in practice, it can be minimised if factors causing this error are well understood. F.Pitard (1993) has pointed out that grouping factor can be minimised by taking as many and as small increments as practically possible, assuming that all aspects of extraction and preparation of these increments is carried out correctly. Practical implication of this finding is that when selecting a riffle splitter preference should be given to that with a more riffles. When collecting samples by randomly chosen fragments, as a rule of thumb, the sample should be made of at least 30 increments (Pitard, 1993). The same rational applies to the choice of rotary splitter. To minimise the grouping-segregation error it is necessary to assure that sample was collected by at least 30 cuts from the lot.

The minimisation of errors caused by segregation factor is more difficult than for errors related to the grouping factor (Pitard, 1993). The only practical approach is to homogenise the material prior to sampling, which is not always technically possible. It is important to understand the segregation mechanisms in the proposed sampling operation and adjust the procedures so that a samples extraction will be less affected by inhomogeneous (segregated) distribution of the sampled particles. For example, to minimise the errors caused by segregation factor the blast hole in Figure 5B should be sampled by using a sampling device which is radial in plan view and taking several increments positioning them at random around the blast hole pile (Pitard, 2005).

2.3 Errors related to sampling practices

This group includes delimitation, extraction, preparation and weighing errors, which occur as a result of incorrect extraction of samples from a lot and their suboptimal preparation procedures. In other words the errors included in this group are not directly related to the sampling protocol but in fact are associated with the practical implementation of the protocol. Simply this means, that having the best possible protocol does not guarantee that your samples will be accurate and repeatable as this requires that protocol should be implemented in a practical way that eliminates, or at least minimises, the errors related to sampling practices which are described here as Group 2 errors. The most common example of this type of error is contamination of the laboratory samples during their preparation. Contamination of the samples caused by incorrect preparation procedures can completely destroy the integrity of the samples and all efforts to obtain the samples with the lowest possible fundamental error will be wasted.

Delimitation error occurs when not all material in the sampled lot has equal probability to be selected to the sample (Gy, 1982; Pitard, 1993). The most common situation leading to delimitation errors is sampling of broken ore by collecting material from the top of the pile (Fig. 6A). The bottom of the pile is not represented in the sample causing biased results due to delimitation error. Another common example of delimitation error is shown in Figure 6B. When the sample is taken by a rounded scoop the material at the bottom of the pile has less likelihood to be part of sample then fragments at the top of the pile.
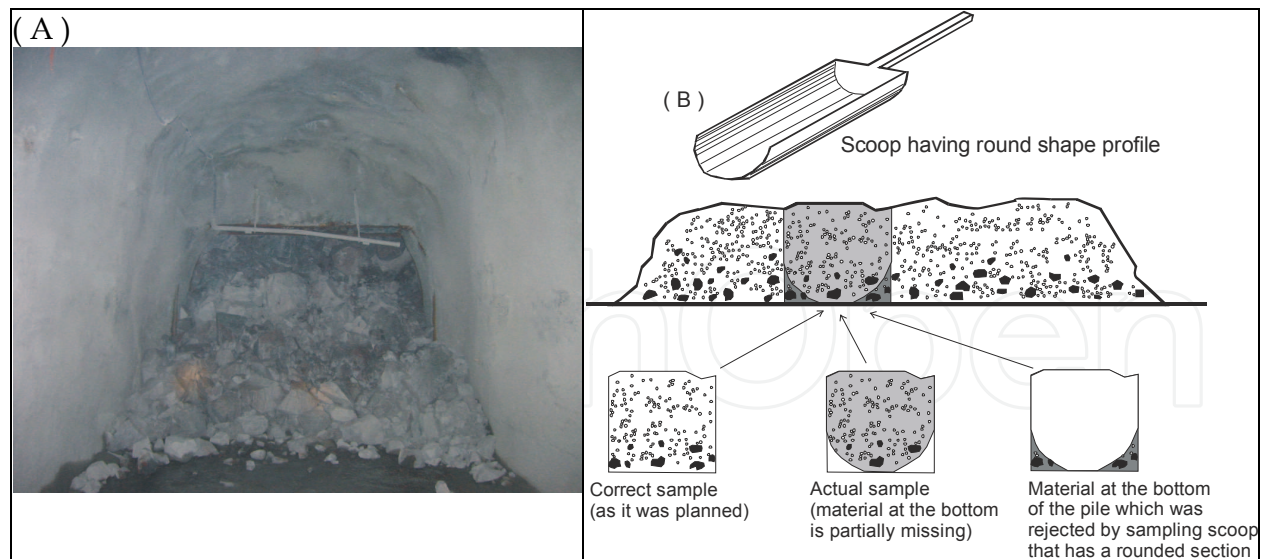
Fig. 6. Examples of delimitation errors: (A) sampling broken ore at the draw point of underground stope represents an unresolvable issue of the delimitation error.; (B) sampling of crushed material (e.g. blast hole cone) by a scoop having round shape profile

Extraction error is the result of a sampling tool which is selectively taking fragments, therefore these errors also known as sample recovery errors (Pitard, 1993) because they are caused by selective sampling systems. This type of error can be frequently observed in geological exploration and mining geology applications (Fig. 7). One of the most common examples of extraction error is a preferential caving of soft material, such as clay pods, when drilling rocks of variable strength. Sampling of blast hole cones using incorrectly designed auger drill, which rejects large fragments is another type of extraction error often occurring in geological applications. In all these cases the extraction error can cause significant biases of the analytical results. It is important to note that having the correct equipment does not guarantee high quality results because inappropriate use of equipment can also lead to significant extraction error. The example shown in Figure 7B demonstrates an extraction error caused by the incorrect use of a riffle splitter which is quickly fed on one side. Such an approach leads to disproportional distribution of the fragments and segregation of heavier particles on one side.
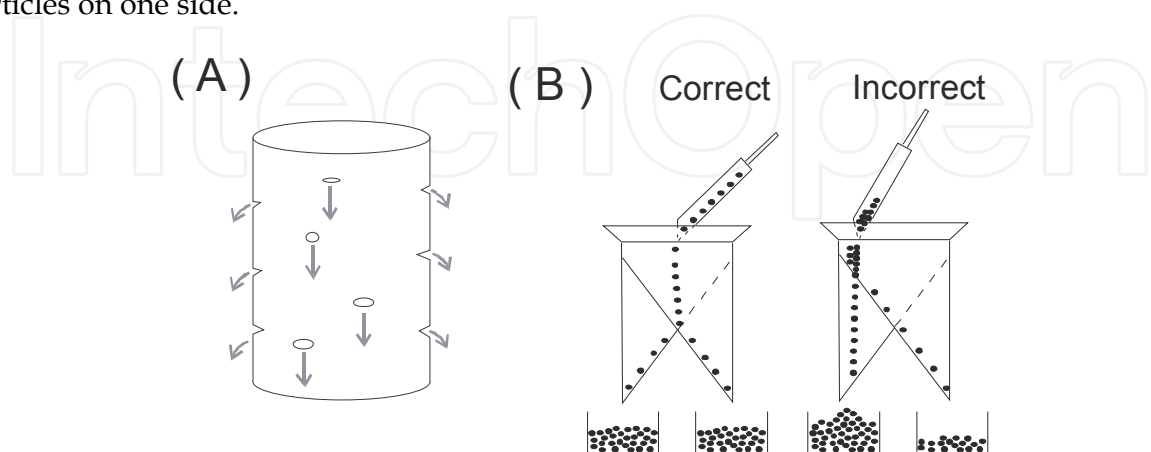

Fig. 7. Examples of Extraction errors: (A) extraction error caused by selective plucking of mineral grains (e.g. gold grains) from the drill core surface; (B) extraction error caused by incorrectly used riffle splitter, modified after Pitard (1993)

Preparation errors are the changes of chemical or physical characteristics of the material caused by its crushing, grinding, pulverising, homogenising, screening, filtering, drying, packaging and transportation. These errors take place during processing of the samples, and include contamination of the samples, preferential losses and alteration of sampled material. For example, in the blast holes some portion of the drill cuttings fall back into the hole which can be a source of sample biases due to preparation error.
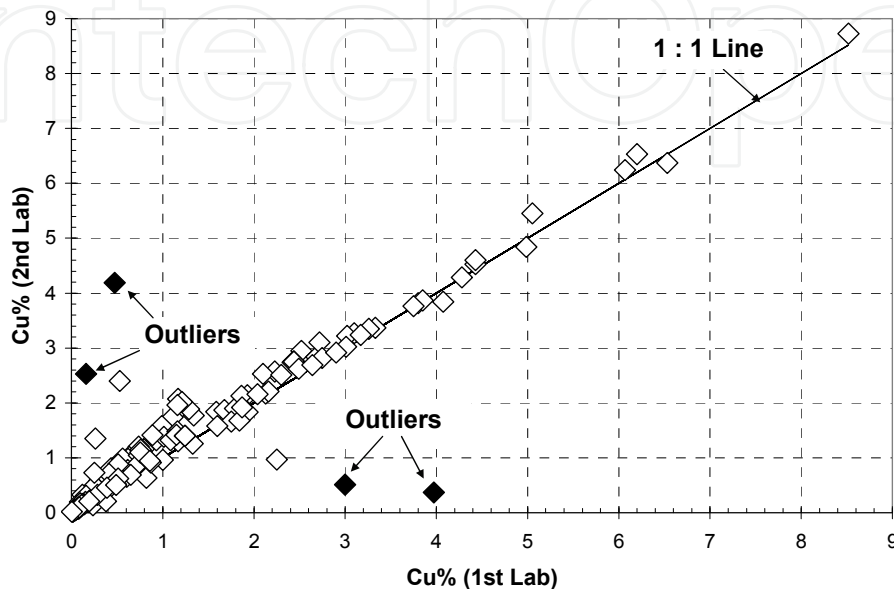

Fig. 8. Scatter-diagram of the Cu grades determined in the drill core samples and their duplicates. Cu-Au bearing skarn, Russia. All data, except several outliers, are compactly distributed along the 1:1 line indicating a good repeatability of the sample grades. Several erratic results (outliers) have been caused by mixing of sample numbers in the lab

Weighing errors are the errors which are introduced by weightometers and scales. The author has observed a case where a laboratory had been equipped by the state of art analytical equipment including robotic XRF instruments whereas the sample preparation stage was still very old, out of date and poorly calibrated scales were used, entirely eliminating all benefits of having high precision analytical instrument.

The second group also includes different types of human errors, such as mixing sample numbers, transcript errors, and incorrect instrument readings. These errors, except in the cases of deliberate fraud, are accidental by their nature and can create extremely erratic values, abnormally high or low in comparison with the true sample grades. When such extreme values are present, these accidental-type of human errors can be easily recognised by presence of outliers on the scatter-diagrams where sample duplicates are plotted against the original samples (Fig. 8).

2.4 Instrumental errors

This group includes errors related to various analytical and instrumental measurements during weighing of the final aliquots and their assaying. A typical example of these types of error is instrumental drift causing biased assays. It also can be incorrect calibration of the instruments. The instrumental errors also include those which are caused by use of the out-of-date equipment, in particular if it is characterised by poor detection limits.

A special case of instrumental errors is represented by incorrectly chosen analytical techniques, which is suboptimal for the given type of mineralisation and/or grade ranges. For example, fire assay with atomic absorption finish is used for low-grade gold mineralisation, whereas high grade gold is better assayed by fire assay with gravimetric finish. Application of the atomic absorption finish to high grade gold samples can lead to their incorrectly determined grades because of instrumental error.

3. Control of analytical data quality (QAQC)

Control of analytical data quality is usually referred to in the mining industry as Quality Assurance and Quality Control (QAQC) and this involves quantification and systematic monitoring of the samples accuracy and precision (i.e. repeatability), and timely diagnostics of sample errors and identification of error sources.

3.1 Accuracy of the analytical data

Accuracy of samples is usually monitored by including samples with known grade to the assayed samples batch (Kane, 1992; ISO Guide, 1989; Leaver et al., 1997; CANMET, 1998; Sketchley, 1998). The samples with *a priori* known grade are called standard samples or simply standards. They can be obtained from commercial laboratories where standards have been prepared and assayed rigorously following the appropriate procedures (Kane, 1992) and results statistically certified. The best practice is to prepare standards from material mineralogically similar to that of the studied mineralisation; these are called the matrix matched standards.

Possible contamination is controlled by inserting blank samples. Blanks are samples where the grade of a metal of interest is negligibly low, usually below detection at the given laboratory. It is a common practise to make the blank samples from barren quartz.

Standards and blanks are called reference materials and used for detection a possible sample biases and quantification the accuracy errors. Leaver et al. (1997) have suggested a definition of a reference material as 'a substance for which one ore more properties are established sufficiently well to calibrate a chemical analyser or to validate a measurement process.' A reference material is classified as a certified reference if it was issued and certified by agencies, such as Canadian Centre for Mineral and Energy Technology (CANMET), the National Institute of Standards and Technology and other governmental agencies, whose technical competency is internationally recognised (Leaver et al., 1997).

Accuracy of laboratory analyses is assessed by statistical tests which are largely based on a comparison of the arithmetic mean of the replicate analyses of a certified standard against its certified mean. The statistical tests of the standard samples repeatability can be based on replicate assays of a certified standard in one laboratory or, conversely, inter laboratory (Round Robin) analyses. Different tests are available (Kane, 1992; ISO Guide, 1989; CANMET, 1998; Sketchley, 1998) and most common are summarised in the current section of this book. It is noted that a good practice is to support testing of the assay accuracy by estimation of standard deviation of the replicate analyses of the standard samples, as poor precision of the standard sample assays can prohibit reliable detecting of analytical biases.

a. Statistical tests for assessing performance of standard samples: single laboratory case

The most common situation is when one or several certified standard samples have been included in a batch of samples and analysed together in the same laboratory. In such case when several repeat assays of a given standard sample are available the analytical accuracy

is assessed using a statistical test (Eq. 14). If this condition (Eq. 14) is satisfied the analytical results are considered acceptable with regard to accuracy.

$$|m - \mu| \leq 2\sqrt{\sigma_L^2 + \frac{S_W^2}{n}} \tag{14}$$

where:

μ - certified mean of a given standard sample;

σ_L - certified between-laboratory standard deviation of a given standard sample;

m - arithmetic mean of the replicate analyses of this certified standard sample in the assay batch;

S_W - estimated within-laboratory standard deviation of the replicate analysis of the standard sample included in assay batch;

n - number of replicate assays of a given certified standard in analytical batch.

The base formula (Eq. 14) can be simplified as the usual empirical relationships is $\sigma_L \approx 2 S_W$ (CANMET, 1998). Consequently, for large (n > 10) number of replications the above condition (Eq. 20) can be simplified as follows (Eq. 15):

$$|m - \mu| \leq 2 \sigma_L \tag{15}$$

Certified value of the between-laboratory standard deviation (σ_L) of the reference materials is not always available. If this is the case, the condition of the accuracy acceptance (Eq. 15) can be further simplified (Eq. 16):

$$|m - \mu| \leq 4 S_W \tag{16}$$

When multiple analyses of a certified standard have been made in the same laboratory the results can be used for estimation of analytical precision of laboratory. Analytical precision is acceptable if results of the replicate analysis of the certified standards assayed in this laboratory satisfy the statistical test (Eq. 17) (ISO Guide 33):

$$(S_W / \sigma_C)^2 \leq \frac{\chi^2_{(n-1);\ 0.95}}{n-1} \tag{17}$$

where:

S_W - standard deviation of the replicate analysis of the standard

σ_C - the certified value of the within-laboratory standard deviation of the standard

$\chi^2_{(n-1);\ 0.95}$ - critical value of 0.95 quartile ($\alpha = 0.05$) of the (χ^2) distribution   at (n-1) degrees of freedom, where (n) is the number of replicate analysis of the standard.   In practise, at least 3 repeat analysis of the certified standards should be available for this test (CANMET, 1998).

b.   Statistical tests for assessing performance of the standard samples: different laboratories case

When exploration samples and standards inserted to the samples batch have been analysed in (*p*) several different laboratories, the overall accuracy of the analytical results can be tested (Kane, 1992; ISO Guide, 1989; CANMET, 1998) using the following statistical condition (Eq. 18):

$$|m - \mu| \leq 2\sqrt{\frac{S_{LM}^2 + \dfrac{S_W^2}{k}}{p}} \tag{18}$$

where:

μ - certified mean of a given standard sample;

m - arithmetic mean of the replicate analyses of this certified standard sample in the assay batch;

S_W - estimated within-laboratory standard deviation of the replicate analyses of the standard samples;

S_{LM} - estimated between-laboratory standard deviation of the replicate analyses of the standard samples;

$k = \dfrac{n}{p}$, is a ratio of total numbers of replicate analysis (n) of certified standards to a

number of laboratories (p) participating in Round Robin test.

The assays of the certified standards obtained from different laboratories can be used for estimation analytical precision (Eq. 19).

$$(S_W / \sigma_C)^2 \leq \frac{\chi_{p(k-1);\ 0.95}^2}{p(k\text{-}1)} \tag{19}$$

where:

S_W - estimated standard deviation of the replicate analysis of the standard;

σ_C - the certified value of the within-laboratory standard deviation of the standard;

$k = \dfrac{n}{p}$, is a ratio of total numbers of replicate analysis (n) of certified standards to a

number of laboratories participating in Round Robin test (p);

$\chi_{p(k-1);\ 0.95}^2$ - critical value of 0.95 quartile ($\alpha = 0.05$) of the (χ^2) distribution at $p(k\text{-}1)$ degrees of freedom.

Inter-laboratory precision can be assessed indirectly using the equality Eq. 20. Precision is considered satisfactory if this equality is true.

$$\frac{S_W^2 + kS_{LM}^2}{\sigma_C^2 + \sigma_L^2} \leq \frac{\chi_{(p-1);\ 0.95}^2}{(p\text{-}1)} \tag{20}$$

where:

σ_C - certified value of within-laboratory standard deviation of reference standard;

σ_L - certified value of between-laboratory standard deviation of reference standard;

S_W - estimated standard deviation of the replicate analyses of the standard samples;

S_{LM} - estimated between-laboratory standard deviation of the replicate analyses of the standard samples;

$k = \dfrac{n}{p}$, is a ratio of total numbers of replicate analysis (n) of certified standards to a

number of laboratories participating in Round Robin test (p);

$\chi^2_{(p-1);\ 0.95}$ - critical value of 0.95 quartile ($\alpha = 0.05$) of the (χ^2) distribution at (p-1) degrees of freedom.

c.   Statistical tests for assessing performance of the standard samples: single assay case

Repeat assays of certified standards may not be available in a single analytical batch, which often can contain only one standard sample. In such case, when repeat assays of certified standards are not available a decision on the analytical accuracy should be made using a single value (*X*) of the certified standard included into the analysed batch the above mentioned statistical tests are inapplicable. Decisions on the accuracy of the method need to be based on the different statistical test (Eq. 21) which uses a single assayed value of the certified standard (Kane, 1992; CANMET, 1998):

$$| X - \mu | \ \leq \ 2\ \sigma_C \tag{21}$$

where:

X - assayed value of the certified standard;

σ_C - certified within-laboratory standard deviation of the standard;

μ - certified mean of a given standard sample.

d.   Assay biases diagnostic diagrams: pattern recognition method

The pattern recognition technique is applied to certified standards (Leaver et al., 1997; Sketchley, 1998; Abzalov, 2008).This method is based on the fact that specific types of analytical problems have recognisable patterns on the special types of the diagrams. The different distribution patterns of analytical results are indicative of error sources and types.

The assayed values of certified standards are plotted onto the diagram on a batch/time basis (Fig. 9). Good quality analysis will be characterised by random distributions of the data points around the certified mean value on this diagram (Fig. 9A) and 95% of the data points will lie within two standard deviations of the mean and only 5% of assays should lie outside the interval of two standard deviations from the mean. It is essential that the same number of samples should occur above and below mean. When distribution of the standard sample assays differ from the pattern shown on the Figure 9A this likely is indicate a possible analytical errors. For example, the presence of 'outliers' (Fig. 9B) is most likely an indication of data transcription errors. This feature does not imply data bias but nevertheless indicates a poor data management system suggesting possible random errors in the database. Consistent shift of a standard sample assay (Fig. 9C) is indicative of analytical bias which could be caused by equipment calibration issues or changed analytical procedures in the lab.

Less common distribution patterns occur when dispersion of the standard sample grades rapidly decrease (Fig. 9D). Such a decrease in the standards variability is commonly interpreted (Sketchley, 1998) as indicative of data tampering, or suggests that standard samples have been recognised by laboratory personnel and treated more carefully than other samples (Abzalov, 2008). Such assay values of standard samples cannot be used as confirmation that the assayed samples lack bias.

Accurate analyses are also characterised by lack of systematic data trends on grade versus order of analysis diagrams. Trends can be recognised by a systematic increase or decrease of the assayed values of the standards (Fig. 9E). Another commonly used criteria for identification of possible trends are distributions where two successive points lie outside of the two standard deviation range or four successive points lie outside the one standard deviation range(Leaver et al., 1997).

Systematic drifts (Fig. 9E) of assayed standard values usually indicate a possible instrumental drift. Alternatively this can be also caused by degradation of the standard samples. The author of the current paper is familiar with cases when characteristics of the standard samples have degraded in comparison with their certified values because of inappropriate storing conditions of the standards when they were kept in the large jars and have not been protected against vibrating due to operating equipment.
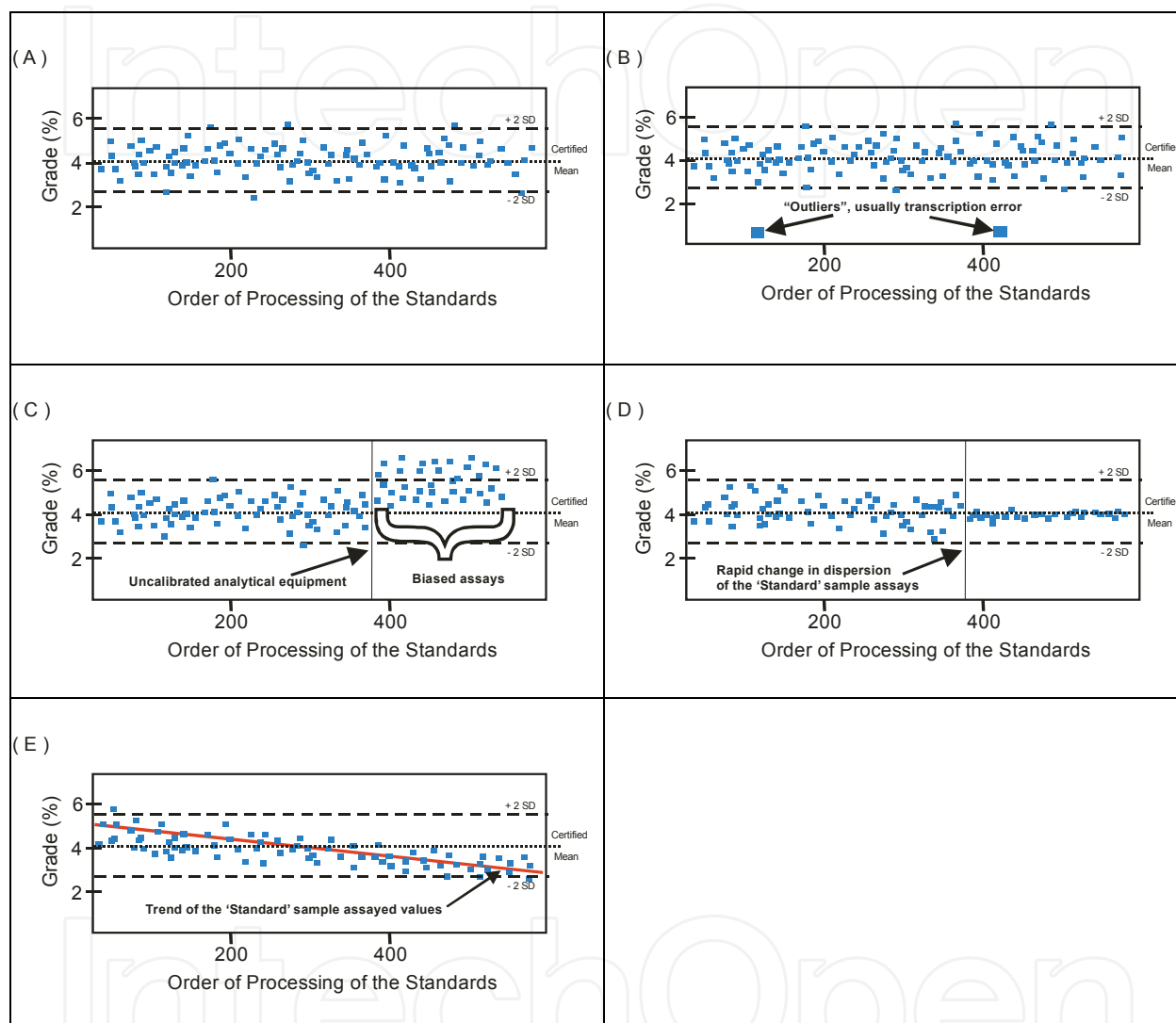

Fig. 9. Schematic diagrams showing quality control pattern recognition method (Abzalov, 2008): (A) Accurate data, statistically valid distribution of the standard values; (B) Presence of 'outliers' suggesting transcription errors; (C) Biased assays; (D) Rapid decrease in data variability indicating for a possible data tampering; (E) Drift of the assayed standard values

Blank samples (i.e. material that have very low grade of a metal of interest) are usually inserted after high grade mineralisation samples. The main purpose of using blanks is to monitor laboratory for a possible contamination of samples which is mainly caused by poor house keeping and insufficiently thorough cleaning of equipment. Blank assays are also presented on the grade versus order of analysis diagram (Fig. 10) and if equipment have not been properly cleaned the blank samples will be contaminated which is reflected on the diagram as increased value of a metal of the interest. The case presented on the Figure 10

shows that laboratory procedures have degraded during the course of the project because the blank samples after approximately blank with sequential number 150 have started to systematically indicate contamination by base metals derived from processed Ni-Cu sulphide samples.
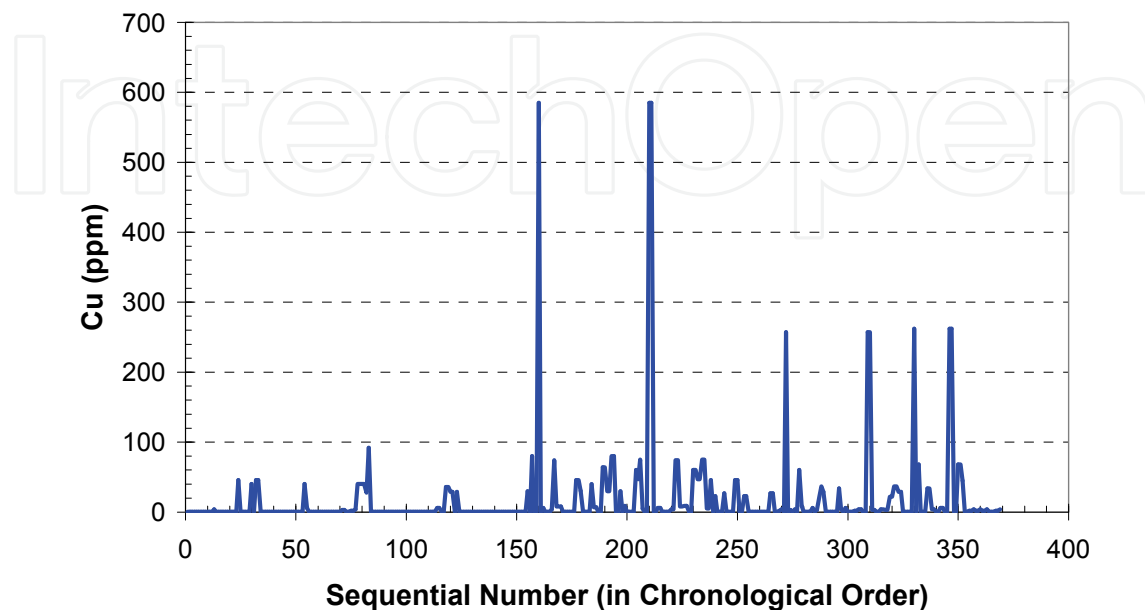

Fig. 10. Cu grade of blank samples plotted versus order of their analysis, Ni-Cu project, Australia

3.2 Precision of the analytical data

Precision error of assayed samples is estimated and monitored by using matching pairs of samples (Abzalov, 2008). Sample pairs (duplicates) represent the most common type of matching pairs of data. The first sample is usually referred to as original sample, and the second is called duplicate. The duplicate sample is merely another sample collected from the same place and following the same rules as used for collecting the initial (original) sample. In practise, the duplicate sample can be a second blast hole sample collected from the same blast hole cone or another half of the drill core. It also can be a duplicate sample collected at a particular stage of the sampling protocol, such as coarse rejects after crushed material has been split using an appropriate sample reducing devise or second aliquot collected from the same pulverised pulp. When sampling protocol includes several stages of comminution and subsampling it is a good practise to take duplicate samples at each subsampling stage. Another type of matching data pairs are twin drill holes (Abzalov, 2009a).

a. Statistical analysis of sample pairs

The most common approach in assessing paired data is to plot the duplicate sample results vs. original assays and the precision error can be quantified from the paired data through assessing the scatter of the data points with corrections for their deviation from y=x line. The differences between an assayed sample and it's duplicate is caused by errors associated with sample preparations and assaying. Precision error is mathematically deduced from differences between matching pairs of data, and is usually represented as

variance of the assayed values normalised to the means of the corresponding pairs of the data.

The different statistical methods are available for estimation precision error from paired data (Garrett, 1969; Thompson & Howarth, 1978; Bumstead, 1984; Shaw, 1997; Francois-Bongarson, 1998; Sinclair & Bentzen, 1998; Sinclair & Blackwell, 2002). All these methods have been reviewed by the author (Abzalov 2008) and compared by applying the same sets of duplicated pairs of samples collected in operating mines and mining projects. The study (Abzalov, 2008) concurs with the suggestion of Stanley and Lawie (2007) that the use of average coefficient of variation (Eq. 22 and 23) as the universal measure of relative precision error in geological applications is appropriate.

$$CV\% = 100\% \times \frac{\text{Standard Deviation}}{\text{Mean}} = 100\% \times \frac{|a_i - b_i| / \sqrt{2}}{\dfrac{a_i + b_i}{2}} \qquad (22)[1]$$

Average CV% can be calculated from the (N) pairs of the duplicated samples using equality (Eq. 23).

$$CV\% = 100\% \times \sqrt{\frac{1}{N}\sum_{i=1}^{N} \frac{\sigma_i^2}{m_i^2}} = 100\% \times \sqrt{\frac{2}{N}\sum_{i=1}^{N}\left(\frac{(a_i - b_i)^2}{(a_i + b_i)^2}\right)} \qquad (23)$$

Stanley (2006) has shown that absolute difference between paired data is directly proportional to standard deviation $(|a_i - b_i| = \sqrt{2} \times \text{Standard Deviation})[2]$ and consequently the various measurements representing a ratio of the absolute differences between paired data to the means of the corresponding pairs (Table 3) are directly proportional to coefficient of variation.

Hence, such statistics as AMPD and HARD (Table 3), commonly used by geoscientists (Bumstead, 1984; Shaw, 1997; Roden & Smith, 2001), represent nothing else but products of CV% by constants ($\sqrt{2}$) and ($\frac{\sqrt{2}}{2}$) respectively (Table 3) and therefore offer no more information than the CV% itself.

---

[1] For consistency with other measurements coefficient of variation (CV) (one standard deviation divided by mean) is expressed as percentage (CV%)

[2] Variance (σ^2) is determined by the following equality: $\sigma^2 = \dfrac{\sum_i (x_i - \dfrac{\sum x_i}{N})^2}{n-1}$. In case when only a single pair of data is available the formula can be transformed as follows:

$$\sigma^2 = \frac{(a_1 - \dfrac{a_1 + b_1}{2})^2 + (b_1 - \dfrac{a_1 + b_1}{2})^2}{2-1} = \frac{(\dfrac{2a_1 - a_1 - b_1}{2})^2 + (\dfrac{2b_1 - a_1 - b_1}{2})^2}{1} = \frac{(a_1 - b_1)^2 + (b_1 - a_1)^2}{4}$$

$$= \frac{(a_1 - b_1)^2}{2};$$

Taking square root of the variance we arriving to a final formula of standard deviation of the paired data: $\text{Standard Deviation} = \sqrt{\sigma^2} = \dfrac{|a_1 - b_1|}{\sqrt{2}}$

Conventional Name of the Error Estimator	Single Duplicate Pair Formula and relationship to CV%	Reference	Comments
AMPD	$AMPD = 100\% \times \dfrac{\mid a_i - b_i \mid}{(a_i + b_i)}$ $AMPD = \sqrt{2} \times CV\%$	Bumstead, 1984	This estimator is also known as MPD (Roden and Smith, 2001) or ARD (Stanley & Lawie, 2007)
HARD	$HARD = 100\% \times \dfrac{\mid a_i - b_i \mid}{(a_i + b_i)}$ $HARD = \dfrac{\sqrt{2}}{2} \times CV\%$	Shaw, 1997	HARD is simply the half of the corresponding AMPD
where (a_i) is original sample and (b_i) is its duplicate			

Table 3. Measures of Relative Error Based on Absolute Difference Between Duplicated Data Pairs

b.　Reduced Major Axis (RMA)

This is a linear regression technique which takes into account errors in two variables, original samples and their duplicates (Sinclair & Bentzen, 1998; Sinclair & Blackwell, 2002). This technique minimises the product of the deviations in both the X- and Y- directions. This, in effect, minimises the sum of the areas of the triangles formed by the observations and fitted linear function (Fig. 11). Calculation of the RMA parameters is explained in detail in (Abzalov, 2008) and only briefly summarised below.

The general form of the reduced major axis (RMA) is as follows (Eq. 24):

$$b_i = W_0 + W_1 a_i \pm e \tag{24}$$

where (a_i and b_i) are matching pairs of the data, (a_i) denotes a primary samples plotted along X axis and (b_i) is duplicate data, plotted along the Y axis. (W_0) is Y-axis intercept by the RMA linear model, (W_1) is the slope of the model to X-axis, (e) standard deviation of the data points around the RMA line.

The parameters (W_0, W_1 and e) estimated from the set of the matching pairs of data (a_i and b_i), plotted along X-axis and Y-axis, respectively. The slope of RMA line (W_1) is estimated as ratio of standard deviations of the values (a_i and b_i) (Eq. 25).

$$W_1 = \frac{\text{St.Dev}(b_i)}{\text{St.Dev}(a_i)} \tag{25}$$

Intercept of the RMA model with Y axis is estimated from (Eq. 32)

$$W_0 = Mean(b_i) - W_1 Mean(a_i) \tag{26}$$

RMA model allows quantifying the errors between matching data pairs (Eq. 27 – Eq.30). Dispersion of the data points about the RMA line (S_{RMA}) is estimated using Eq. 27.

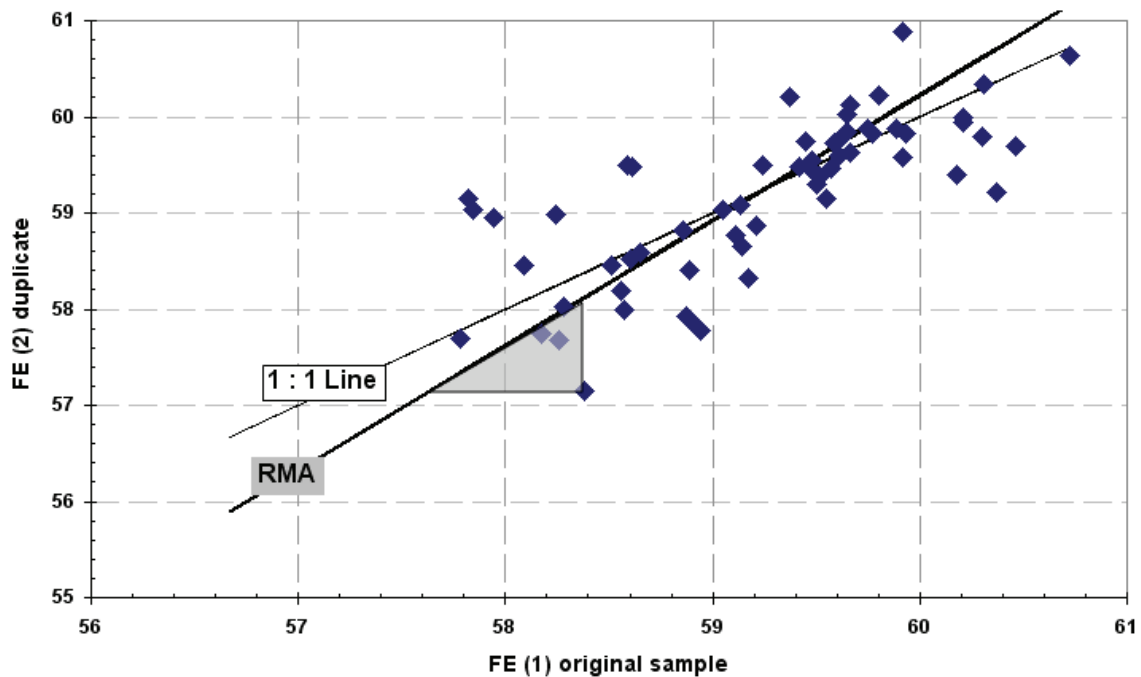$$S_{RMA} = \sqrt{[2(1-r)(Var(a_i) + Var(b_i)]} \tag{27}$$

Fig. 11. Scatter- diagram and the RMA model (thick line) fitted to paired Fe grades of the blast hole samples (Abzalov, 2008). 1:1 line (fine) is shown for reference. Grey triangle shows an area formed by projection of the data points and RMA line. RMA technique minimises the sum of the areas of the all triangles

Error on Y-axis intercept (S_0) is estimated using Eq. 28. and the error on the slope (S_{SLOPE}) is estimated using Eq. 29.

$$S_0 = St.Dev(b_i) \sqrt{\left[\frac{(1-r)}{N}\right] \left[2 + \left(\frac{Mean(a_i)}{St.Dev(a_i)}\right)^2 (1+r)\right]} \qquad (28)$$

$$S_{SLOPE} = \frac{St.Dev(b_i)}{St.Dev(a_i)} \sqrt{\frac{(1-r^2)}{N}} \qquad (29)$$

where (r) is correlation coefficient between (a_i and b_i) values, (N) is the number of the data pairs and (*Var*) are variances of the (a_i and b_i) values.
The relative precision error ($P_{RMA}(\%)$)[3] can be estimated from the RMA model:

$$P_{RMA}(\%) = 100 \times \frac{\sqrt{\frac{S_{RMA}^2}{2}}}{\frac{(\sum_i^N a_i) + (\sum_i^N b_i)}{2N}} \qquad (30)$$

---

[3] For consistency with CV% and other estimators discussed in this section the ($P_{RMA}(\%)$) value is estimated at 1 standard deviation and reported as per cents (i.e., multiplied by 100 in Eq. 30).

c.   Relative Difference Plot (RDP)

Relative difference plot (RDP) has been suggested by F.Pitard (1998) as graphic tool for diagnostics of the factors controlling precision error (Fig. 12). Relative difference is estimated as the differences between matching pairs of data which are normalised to the means of the corresponding pairs of the data (Eq. 31). This relative difference is expressed as per cents.

$$\text{RD (Relative Difference) } \% = \ \frac{1}{N}\sum_{i}^{N}[100 \text{ x } \frac{(a_i - b_i)}{(a_i + b_i)/2}] \qquad (31)$$

where (a_i) and (b_i) are the matching pairs of the data and N is the number of pairs.

Calculated RD(%) values are arranged in increasing order of the average grades of the data pairs and then RD(%) values are plotted against sequential numbers of the pairs whereas the calculated average grades of the data pairs are shown on the secondary y-axis (Fig. 12). Link between average grades of the data pairs and their sequential numbers are established by adding a 'Calibration curve' to the RDP diagram (Fig. 12). RD(%) values usually exhibit a large range of variations; therefore, interpretation of this diagram can be facilitated by applying the moving window technique to smooth the initially calculated RD(%) of the data pairs (Fig. 12).
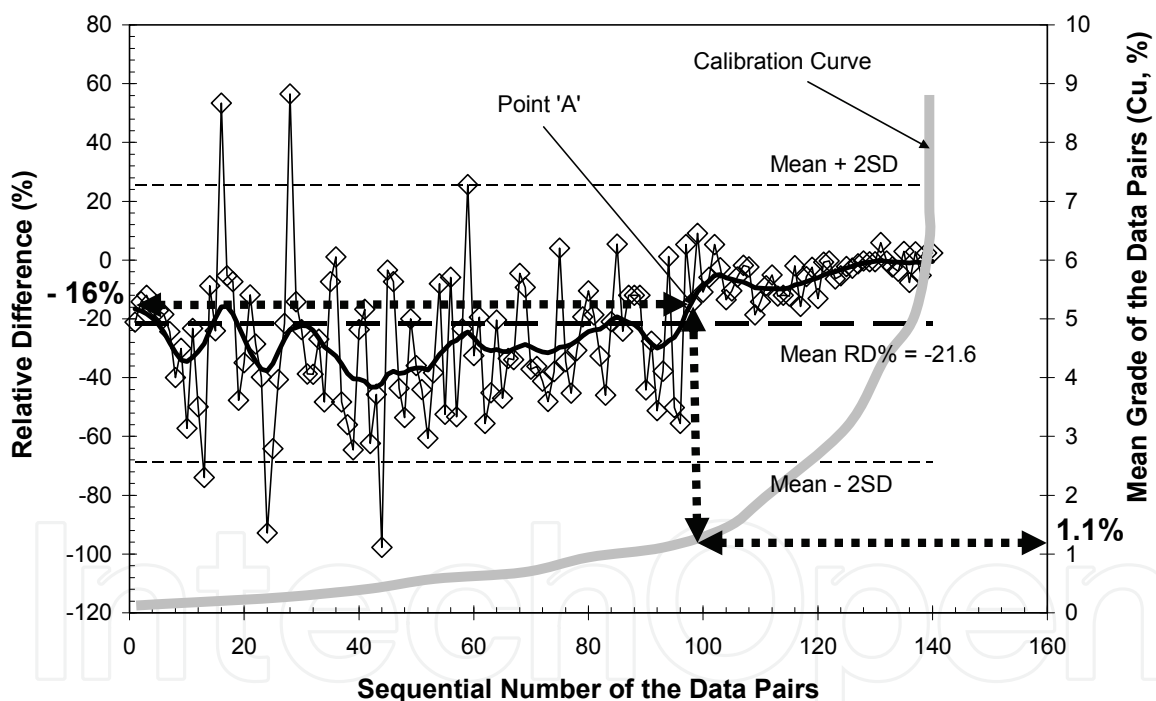

Fig. 12. Relative Difference Plot (RDP) showing Cu(%) grades of duplicated drill core samples assayed in different labs (Abzalov, 2008). Open symbols (diamond) connected by fine tie-lines are RD(%) values calculated from matching pairs of data (i.e., original sample and duplicate). Average RD(%) value (thick dashed line) and ± 2SD values (fine dashed lines) are shown for reference. The solid line is a smoothed line of the RD(%) values calculated using a moving windows approach. The 'Calibration Curve' sets the relationship between RD% values on the primary y-axis, the sequential number of the data pairs plotted along the x-axis and the average grades of the data pairs plotted on the secondary y-axis. For example point 'A' (sequential number of the data pairs is 100) is characterised by RD% value equal to -16% and average grade of that pair of data is 1.1% Cu

The example in Figure 12 is based on data collected from a massive Cu-sulphide project in Russia. Approximately 140 core duplicate samples have been analysed in an external reputable laboratory as part of project due diligence. The results, when plotted on an RDP diagram (Fig. 12), show that copper assays of low grade samples (Cu < 1.1%) are biased. Assayed values have significantly underestimated the true copper grade of those samples. Another feature revealed by this diagram is that low grade samples (Cu < 1.1%) exhibit excessive precision error which is not shown by higher grade samples (Cu >1.1%). These findings have triggered a special investigation of the laboratory procedures, with a particular emphasis on differences between high-grade and low-grade samples.

The RDP diagram can be used for testing the impact onto data precision the different factors, for example sample size, core recovery, depth of the samples collection. In that case the RD% values are arranged according to a factor under investigation. In Figure 12 the RD% values are plotted against the sequential number of the samples arranged by their sizes (i.e. length of the sampling interval). The diagram (Fig. 13) shows that small samples, less than 0.5m in length, are characterised by approximately twice as larger precision error than samples of 1 m in length.
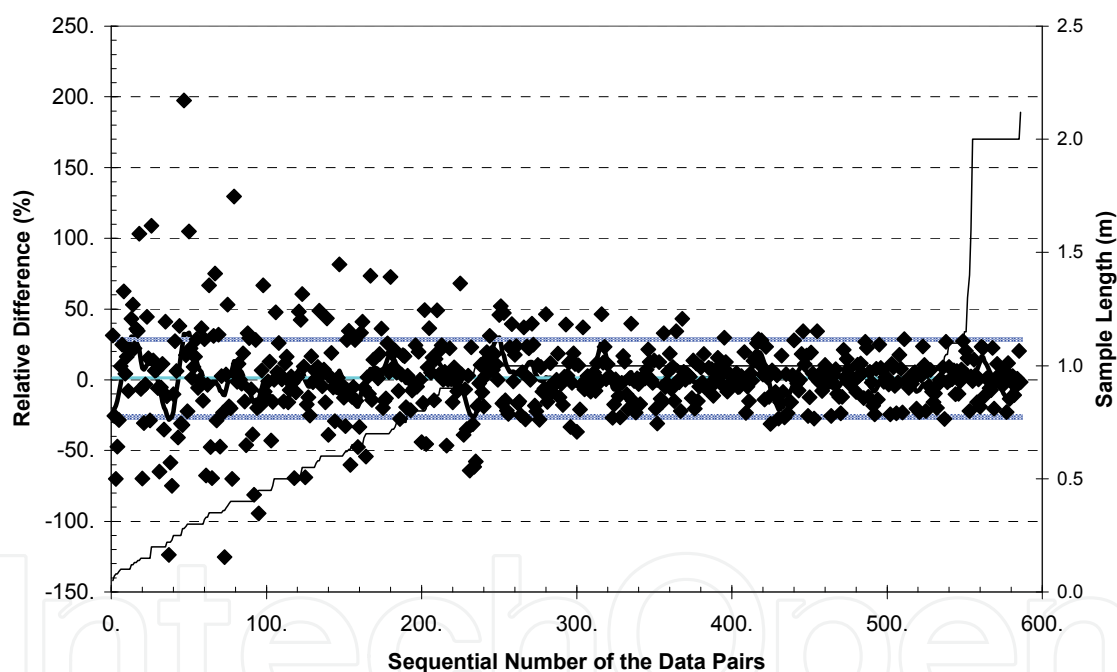

Fig. 13. Cu% grades of the duplicated drill hole samples plotted on the RDP diagram. RD% values are arranged by length of the samples

4. Twin holes

Drilling of twinned holes (i.e., drilling of a new hole, or "twin", next to an earlier drill hole) is a traditional technique used in exploration and mining geology for verification of mineralization grades. The importance of this technique is emphasized in the JORC Code, which specifically queries if twinned holes have been used for 'verification of sampling and assaying' (JORC, 2004, p.15). A well known example of a (negatively) successful twinned holes program is the confirmatory drilling at the Busang project of Bre-X Minerals Ltd.

Fraud was revealed by seven twinned holes (Lawrence, 1997). Each of the seven new holes was drilled only 1.5 m away from supposedly extensively gold mineralized holes, and all failed to substantiate the originally stated gold values (Lawrence, 1997). Application of the twinned holes technique is not limited to verification of the high-grade intersections and also includes verification historic data (Abzalov, 2009a). Such a need usually occurs where results from earlier drill holes are causing concerns either by being noticeably different to other drilling campaigns in the same area, or where poor quality of drilling has been revealed from previously documented parameters, such as core recovery. In some cases, historic data might be suspect (e.g., biased) because of suboptimal drilling technology or inappropriate drilling parameters. For example, air-core drilling of a mineral sands deposit might be biased, and the issue could be examined by twinning the historic air-core holes with sonic drill holes.

Verification of earlier drilled holes by twinning them with new holes is a common technique used by geological due diligence teams when reviewing third party projects (Gilfillan, 1998; Abzalov, 2009a). Drilling twinned holes becomes particularly important where the reviewed project is based on historical data that were collected without rigorously applied sampling quality control procedures, or where the relevant quality assurance/quality control (QAQC) documentation is lacking. Where bias of historic data has been proven, the data from twinned holes can be used for quantification of this bias, and integrated into the resource model using multivariate and/or non-stationary geostatistical methods for correcting the resource estimates (Abzalov & Pickers, 2005; Abzalov, 2006).

The number and locations of twinned holes, drilling methods, and the tested variables are selected to accomplish the objectives of the proposed study. For example, if the purpose of drilling twinned holes is to verify high grade intersections, new holes should be drilled next to previous holes that reportedly intersected high grade mineralization. Conversely, if the objective is to test and statistically quantify a possible bias in historic data, the twinned holes should be drilled in such a manner that assures that a wide range of grades is tested, including both low- and high- grade mineralization. In this latter case, twinned holes should be distributed throughout the entire deposit, and the number of twinned holes should be sufficient for statistical analysis of the data.

It should be remembered that twinned holes should be located as close as possible to the original holes to minimize the effects of short range variability in the studied variables. A study undertaken by Abzalov et al. (2007) at the Yandi iron-ore open pit has shown that CV% of Al_2O_3 grades estimated from matching pairs of blast holes and reverse circulation (RC) holes increase from 23.8% to 35.7% when the distance between twinned holes increases from 1 m to 10 m. These results suggest that many unsuccessful twinned holes studies could have failed because twinned holes have been drilled too far apart. Personal experience of the author indicates that good practice is not to drill matching pairs of holes more than 5 m apart.

When planning twinned drill holes it is necessary to remember that important decisions may have to be made based on a limited number of verification holes. Therefore, it is necessary to ensure that their quantity and quality are sufficient to make a conclusive decision. It is necessary to ensure that verification holes are of the best practically achievable quality. This often requires the use of a different drilling technique, commonly a more expensive one to that used for the previous drilling. For example, air core holes in mineral sands projects are preferably twinned by sonic drilling, auger holes in bauxite deposits by diamond core holes, and RC holes in coal deposits by PQ diamond core holes. It is

important to realize that verification holes, even if they are drilled using the most up to date technology, can still produce biased and/or excessively variable results. For example, the quality of confirmatory diamond drilling can be marred by poor core recovery, or sonic drilling can be adversely affected by self-injection of sands into core barrels caused by lithostatic pressure.

The number of twinned holes depends on the aim of the twinning program and the range of variation in the studied variables. Where studied variables are characterized by large short range variability, the number of twinned holes should be sufficient for conclusive statistical and geostatistical analysis. In the author's experience this may often be achieved by drilling a relatively small number of holes. However, special studies that require 20–30 twinned holes are not uncommon (Abzalov, 2009a).

Prior to comparing assayed grades in the twinned holes it is necessary to compare geological logs, because the geological features are usually more continuous than grades (Sinclair and Blackwell, 2002). Twinned holes are initially compared by mineralized intersections. Comparisons should not be limited to average grades of intersections, but should also include thicknesses and positioning of the contacts. The latter is particularly important when down hole contamination of the samples is suspected. More detailed studies include comparison of twinned holes by matching intervals. Practically achievable outcomes are often obtained through grouping samples by geological units (Abzalov, 2009a). This approach is particularly useful when twinned holes are drilled through stratified mineralization or regularly intercalated low- and high-grade zones.

In particular cases, for example when twinned holes are drilled through mineralization exhibiting gradational zoning, comparison can be made directly between samples where they are of an equal length; alternatively comparisons may be made between equal length composites (Abzalov, 2009a). Compositing of samples of small size into composites of a larger size smoothes away the impact of outliers and converts samples into common length data, which are necessary for geostatistical resource estimation. In twinned holes analysis, when comparing variables with high statistical variation, grouping samples to larger composites is commonly necessary to minimize the noise exhibited by individual samples (Abzalov, 2009a). It is important to ensure that samples are composited to geological boundaries.

5. Database

Modern mining operations use a vast amount of different sampling data, coming from drill hole, trench, surface, grade control and mine workings samples. The total number of collected samples can be in the hundreds of thousands which are supported by hundreds of QAQC samples and they all need to be stored together in a relational database which is used for storing assayed sample results and for data grouping and analysis using different selection criteria (Lewis, 2001; Abzalov, 2009b,c). Therefore, the quality control of the analytical data in the mining industry should include procedures of relational databases management.

Good relational databases should not be limited to storing data which are required for geological modelling and resource estimation. They should also include metadata or "data about the data". A vast amount of auxiliary information is usually received from the drilling contractors, field geologists and analytical laboratories, all which need to be systematically stored in the relational database. S.Long (1998) has suggested a practical way to store all

additional information without making the database tables overly cumbersome. Most of the important auxiliary information are recorded in the headers of the data sheets, received from the personnel and laboratories undertaking drilling, documentation, sample collection and assaying. For example almost all auxiliary information related to analytical techniques is usually reported at the header of assay certificate received from the laboratory. Entering this information into a separate table within the same relational database enables storage of all key facts related to analytical laboratory, technique, personnel and dates of analyses.

6. Discussion and conclusions

6.1 Planning and implementation of sampling protocols

The sampling protocol should be optimised and rigorous quality control systems implemented at the early stage of project evaluation just after it has been realised that a discovered mineral occurrence warrants a detailed delineation and technical and economic evaluation. At this stage, usually referenced as Order of Magnitude or Scoping study, the given deposit has to be systematically drilled and sampled, based on which the geological characteristics of the deposit will be quantified and the resources estimated. The biased or otherwise suboptimal assay results can lead to incorrectly assessed mining projects with costly consequences. Use of suboptimal sampling protocols or a lack of rigorous QAQC procedures can cause delays in the mining project development because of additional work requested by auditors to verify the earlier drilling results.

The first step is to design sample preparation procedures assuring that they are optimally suited for the studied mineralisation. The chosen protocol should allow the cost effective collection of representative samples of a quality sufficient for the correct evaluation of the deposit. A good starting point is to estimate Fundamental Sampling Error and plot the proposed protocol on a sampling nomogram (Fig. 4). This approach allows the optimisation of such parameters as weight of the initial sample, particle sizes after each comminution stage and sizes of reduced samples. Based on this study all stages of the sample preparation can be clearly determined and parameters of the process are quantified. These parameters need to be considered when the project team chooses equipment for sample preparation. In particular it is necessary to assure that capacity of equipment matches the proposed protocol.

Particular attention should be given to the minimum size of the samples which can be taken without dangerously increasing the samples precision error. The latter is unfortunately often forgotten when geologists are sampling drill core. It is very important to record the length of the sampled intervals and mass of the collected samples. Good practice is when both weights of the samples wet and dry, are measured and documented.

Established sampling protocols need to be documented and represented graphically, as the sample preparation flow chart (Fig. 14) and be made easily accessible to the geological team. The next step after the sampling protocol has been optimised is to add quality control procedures. At this stage it is necessary to decide the stages within the sampling protocol where duplicates are collected and the procedures and frequency of collecting of duplicates. It is also necessary to decide how many reference materials shell be inserted with each sample batch and develop procedures allowing the disguise of standards and blanks. The project management at this stage are making the decision to develop matrix matched standard, specially prepared for the studied mineralisation, or use commercially available certified standards.
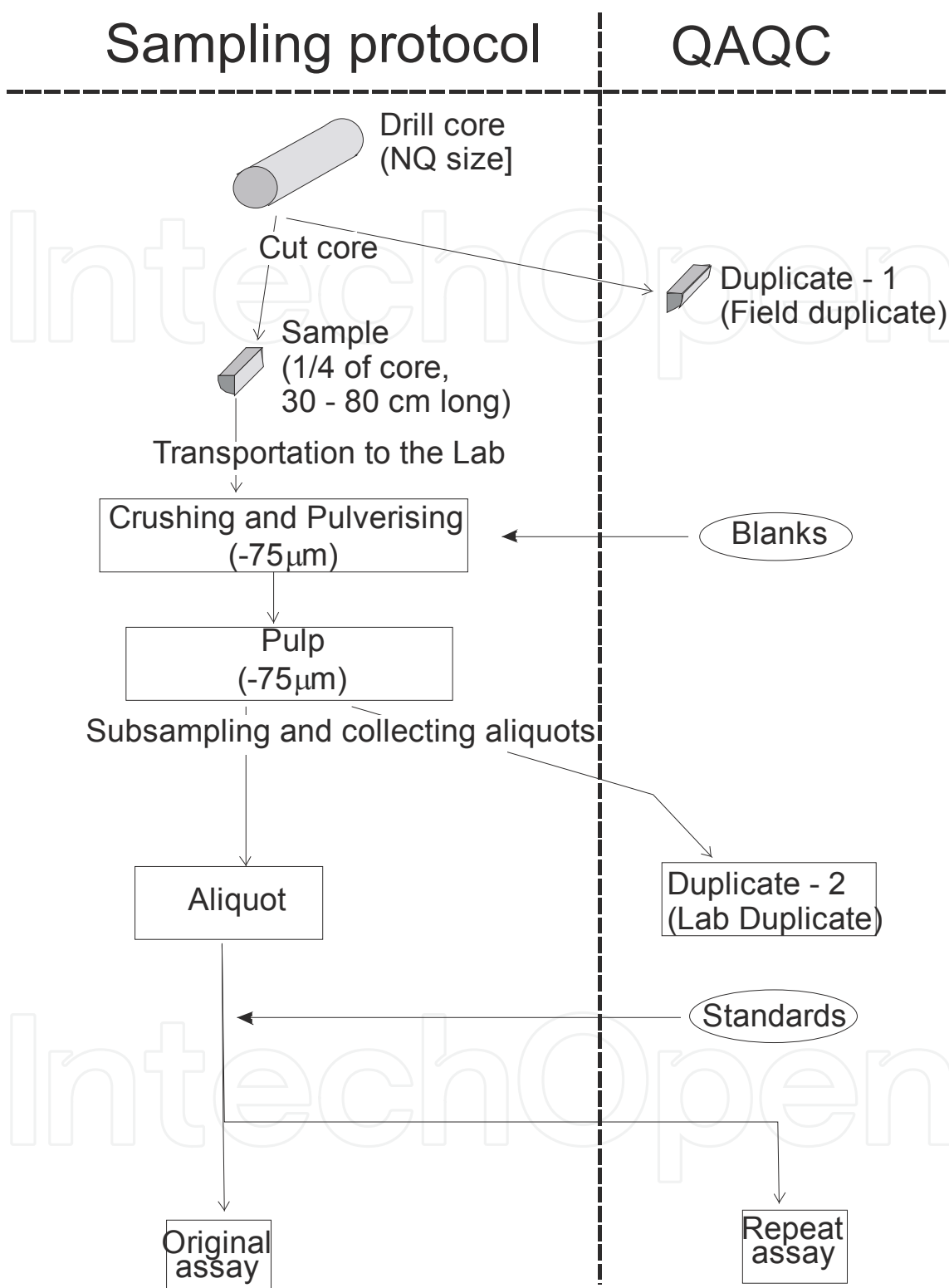
Fig. 14. Sample preparation flow chart combined with the samples quality control map, West Musgrave Ni-sulphide project, Australia

Quality control procedures should be shown on the sample preparation flow-chart (Fig. 14). Such combined diagrams, where sampling protocol is joined with the samples quality control map, are useful practical tools helping to implement and administrate the QAQC

procedures assuring that all sampling and preparation stages are properly controlled. It is also important to note establishing the references between the quality control map and sample preparation flow chart help to better understand stages monitored by different quality control actions facilitating interpretation of QAQC results. When the sampling protocol is changed it should be documented and the associated QAQC procedures updated.

Finally, all procedures should be documented and personnel responsible for their implementation and control determined and instructed. It is necessary to assure that geological team, working at the mine or developing project, regularly reviews the QAQC results. Alternatively, all good efforts can be wasted if sampling errors have not been timely diagnosed. The author after reviewing many different mines found the most effective and practically convenient periodicity for the data quality review is when the QAQC results were checked by mine geologist with every analytical batch and summary QAQC reports have been prepared for approval by the chief geologist on a monthly basis. The monthly reports should contain several diagnostic diagrams showing performance of the reference materials and duplicates. The reports also should present the calculated precision variances which shell be compared with their acceptable levels. The levels of acceptable precision errors and deviation of the standards from their certified values should be clearly determined and documented as part of the QAQC procedures.

6.2 QAQC practice

a. Frequency of inserting QAQC materials into the sample batches

The reliable control of sample precision requires approximately 5% to 10% of field duplicates, and 3% to 5% of pulp duplicates. Duplicate samples should be prepared and analysed in the primary laboratory. The bias in analytical results is detected by including 3% to 5% of standard reference materials with every sample batch. Anonymity of the standards is a serious issue, because standards can be easily recognised in the sample batches and be treated more thoroughly by laboratory personnel. To overcome this problem some duplicate samples should be analysed in an external, reputable laboratory as part of accuracy control. It is suggested that at least 5% of the total analysed duplicates, including pulp duplicates and coarse rejects, should be analysed in a reputable external laboratory.

b. Distribution of reference materials

Standards should be inserted with such frequency that allows the constant monitoring of possible instrumental drifts and biases. Distribution of reference material within the batch should allow the detection of possible biases of the results and at the same time these reference samples should remain anonymous. In general, the best practice is to insert standards and blanks to every sample batch. A good practice is to use more than one standard, so that their values span the practical range of grades in the actual samples. However, standard samples alone cannot identify biases introduced at different stages of sample preparation.

c. Distribution of duplicate samples

Duplicate samples should be chosen in a manner whereby all stages of data preparation are properly addressed and the precision errors associated with all sub-sampling stages are adequately estimated by the sample duplicates. This means that sample duplicates should include field duplicates, coarse reject duplicates and the same-pulp duplicates (Long, 1998). Special attention should be given to the field duplicates as they are mostly informative for estimation of the overall precision of samples. When rotary drilling is used, the field

duplicates, which in this case are called "rig" duplicates, are collected from the sample splitting devices built-in to the drill rigs. These can be rotary, cone or riffle slitters. In case of diamond core drilling the field duplicates are represented by another portion of core. Field duplicates of the blast hole samples should be another sample, taken from the same blast hole cone as the original sample and following exactly the same procedures.

Coarse reject duplicates consists of material representing output from crushers. There often can be more than one type of coarse reject when a samples preparation requires several stages of crushing and splitting. In this case, coarse reject duplicates should be collected for each stage of crushing and/or grinding followed by sample reduction. Operations, using large pulverisers, usually don't have coarse rejects as all collected sample is pulverised to a fine pulp. In this case, it is extremely important to collect and analyse field duplicates to understand the overall repeatability of the assayed results.

Same-pulp duplicates should be analysed in the same laboratory with the original samples and externally, sending a certain portion of them to an independent and reputable laboratory.

When collecting sample duplicates it is necessary to remember that they should be identical to the sample which they are duplicating. Unfortunately this is not always possible as the chemical or physical characteristics of the remaining material can be altered after sample has been collected or its amount is simply insufficient to make a representative duplicate. For example, if drill core is sampled by cutting a half core it is unwise to collect quarter core as a duplicate as this will produce a duplicate samples of a twice smaller weight than the original samples. Such duplicates are likely to produce a larger precision error than that of the original samples. Using all remaining second half of core as duplicate is also suboptimal practice as it breaches auditability of the data. The problem can be partially resolved if to take duplicate using the approach shown on the Figure 15.

In case when all drill samples are assayed, the latter is a common case at the bauxite mines, the only possibility to assess precision of the drill samples is to use twin holes (Abzalov, 2009a).
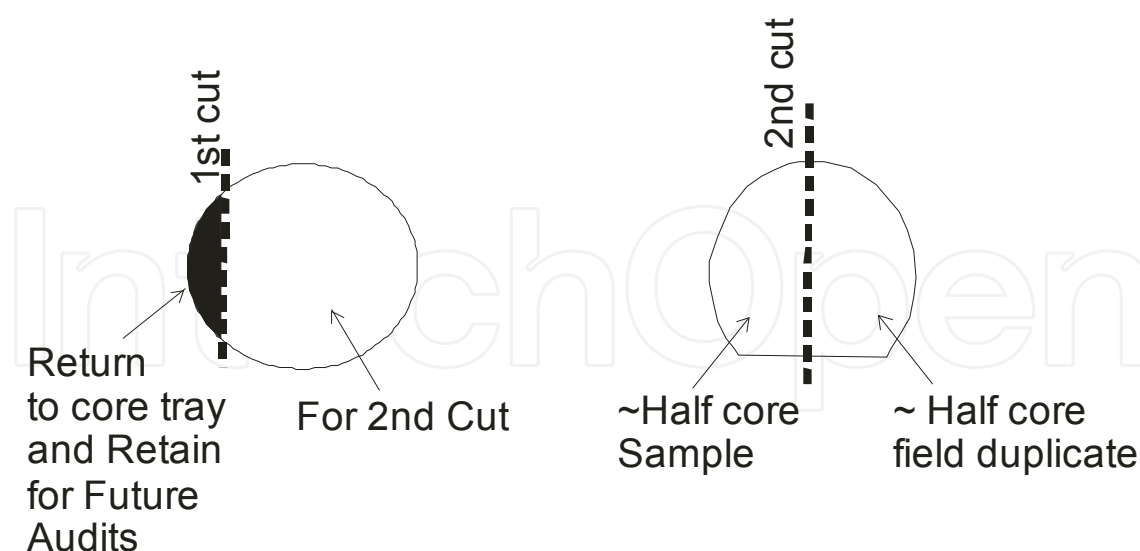

Fig. 15. Sketch explaining collection the duplicate drill core samples in case if routine samples represent the half of the core cut by diamond saw

It is important to assure that duplicate samples should be representative for the given deposit covering the entire range of the grade values, mineralisation types, and different geological units and have a good spatial coverage of the deposit. It is difficult to achieve the

representativeness of the duplicates when they are collected at random from the sample batches. The disadvantage of randomly selected duplicates is that most of them will represent the most abundant type of rocks, usually barren or low-grade mineralisation. The ore grade and, in particular, high-grade intervals are often poorly represented in randomly chosen duplicates. They can also be non-representative regarding mineralisation types, their spatial distribution or grade classes. To meet these two conditions, a dual approach is recommended: approximately half of the duplicates should be collected by a project geologist with instructions to ensure that they properly represent all mineralisation styles and grade ranges, and that they spatially cover the entire deposit. The other half of the duplicates should be selected and placed at random in each assay batch.

The position of sample duplicates in a given analytical batch should not be immediately after their original samples and also should not be systematically related to it. It is important that the original samples and their duplicates are included in the same analytical batch which will allow using them for within-batch precision studies.

Numbering of the duplicate samples should disguise them in the analytical batch. The duplicate samples should be taken through the same analytical procedure as the original sample.

6.3 Precision and accuracy estimates

Repeatability of the samples assay is best represented by coefficient of variations of the sample assays estimated from differences between original samples and duplicates (Eqs.22 - 23). Based on numerous case studies, the appropriate levels of sample precisions are proposed (Abzalov, 2008) for different types of deposits which are summarised in Table 4. It is suggested that these levels be used as approximate guidelines for assessing analytical quality. When the estimated CV% exceeds the allowed tolerance level (Table 4), an RDP diagram (Figs. 12 and 13) can be used for a more detailed analysis and diagnosis of errors.

It is important to remember that the values in Table 4, although based on case studies of the mining projects, may not be always appropriate because mineral deposits can significantly differ by the grade ranges, statistical distribution of the studied values, mineralogy, textures and grain sizes.

The Reduced Major Axis (RMA) model (Fig. 11) is another QAQC technique that is becoming increasingly popular among geoscientists (Sinclair and Bentzen, 1998; Sinclair and Blackwell, 2002). This method consists of the calculation of the RMA line and related parameters (Eqs.24 - 30), and is particularly useful for identifying bias between paired data. This method can be used to calculate the precision error estimate from dispersion of the data points about the RMA function. However, it was shown (Abzalov, 2008) that the errors estimated from the RMA model can significantly differ from CV% values. These differences are likely due to sensitivities of the RMA model to the presence of outliers, which makes this technique inappropriate for strongly skewed distributions.

Accuracy of the data should be controlled using standard reference materials and also by assaying a certain portion of the pulp duplicates in a reputable external lab. Standard reference materials alone don't provide full coverage of all possible causes of the sample biases and therefore don't guarantee their timely detections. For example, even matrix matched standards can create a false impression of security when they not fully reflect the level of problems and complexity presented in the actual samples.

Analysis of the standards should be routinely carried out by the mine geologists after results are received from a laboratory. Analysis of standard performance should commence with

graphic presentation of the standard assay results (Fig. 9) and a review their distribution patterns. A pattern recognition method is applied for qualitative diagnostics for possible accuracy issues. When distribution of assayed values of reference standards don't exhibits any systematic drifts or other types of specific patterns indicating possible accuracy errors the statistical techniques are applied for quantitative estimation of accuracy (Eqs. 20 – 27). Obtained statistical estimates should be compared with the certified means and the standard deviations. Only when the assayed values of the standards match their recommended values the analytical batch can be considered sufficiently accurate.

All obtained data should be stored together in a relational database which is regularly backed up assuring transparency of the data handling procedures and their auditability. It should be remembered that database is a final storage for large volume of data and therefore the quality control of the analytical data in the mining industry impossible without accurate management of the data flow and database administration procedures.

Mineralisation Type / Deposit	Metal	Best Practise	Acceptable Practise	Sample type
Gold, very coarse grained and nuggety	Au (g/t)	20 (?)	40	Coarse rejects
Gold, coarse to medium grained	Au (g/t)	20	30	Coarse rejects
	Au (g/t)	10	20	Pulp duplicate
Cu-Mo-Au porphyry	Cu (%)	5	10	Coarse rejects
	Mo (%)	10	15	
	Au (g/t)	10	15	
	Cu (%)	3	10	Pulp duplicate
	Mo (%)	5	10	
	Au (g/t)	5	10	
Iron Ore, CID type	Fe (%)	1	3	Field duplicate
	Al2O3 (%)	10	15	
	SIO2 (%)	5	10	
	LOI (%)	3	5	
Cu-Au-Fe scarn and Iron oxide associate Cu-Au	Cu (%)	7.5	15	Coarse rejects
	Au (g/t)	15	25	
	Cu (%)	5	10	Pulp duplicate
	Au (g/t)	7.5	15	
Ni-Cu-PGE - sulphides	Ni (%)	10	15	Coarse rejects
	Cu (%)	10	15	
	PGE	15	30	
	Ni (%)	5	10	Pulp duplicate
	Cu (%)	5	10	
	PGE (g/t)	10	20	
Detrital Ilmenite sands	Total Heavy Minerals (%)	5	10	Field duplicate

Table 4. Best and acceptable levels of the precision errors (CV%) at the mining projects (Abzalov, 2008)

7. Acknowledgements

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