

Heterogeneity and Ingamells's Tests of Some Chilean Porphyry Ores

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ABSTRACT

Correct sampling practices are of vital importance to the mining industry; key decisions for the business are made on the basis of samples in all the value chain. Geology plays a relevant role in pre- and post-investment stages. Actually the understanding of natural variability is essential for geological modelling, geometallurgical modelling, mine planning, grade and process control. Improper sampling practices obstruct this objective introducing irrelevant variability components, which can cause monumental economic losses. A good approach to accomplish this understanding is the application of heterogeneity and Ingamells's tests, which are useful tools to quantify the variability of a certain *well-delimited geological unit* at different comminution scales. The application of heterogeneity test can help minimise Pierre Gy's fundamental sampling error and to study the impact of other sampling errors. Ingamell's test allows us to define the correct mass of sample for chemical analysis.

This paper presents the results of several heterogeneity and Ingamells's tests done in Chilean porphyry copper deposits that belong to CODELCO Chile (Chuquicamata, Mina Sur, Radomiro Tomic, El Salvador, Andina and Teniente), in different geological units (endogenous oxides, exotic oxides, supergene sulfides and hypogene sulfides) and for several elements (Cu, Co, Mo and As). These tests, considering the natural variability, have helped us develop accurate sampling protocols to minimise the fundamental error to acceptable levels. A major contribution of these tests (more than 30) is the generation of secure sampling protocols using the highest sampling constant obtained in the tests, for new porphyry copper deposits that don't have information given by heterogeneity and Ingamells's tests.

INTRODUCTION

Correct sampling practices are of vital importance to the mining industry; key decisions for the business are made on the basis of samples along the value chain from rock to cathodes. Sampling plays a relevant role in pre- and post-investment stages. Actually, the understanding of natural variability is essential for geological modelling, geometallurgical modelling, mine planning, grade, and process control. Improper sampling practices obstruct this objective introducing irrelevant variability components, which can cause monumental economic losses (Carrasco *et al.*, 2004). A good approach to accomplish this understanding is the application of heterogeneity and Ingamells's tests, which are useful tools to quantify the variance of the fundamental error in several *well-delimited geological units* at different comminution scales. The application of heterogeneity test can help minimise Pierre Gy's fundamental sampling error and to study the impact of other sampling errors. Ingamell's test allows us to define the correct mass of sample for chemical analysis and to minimise the impact of the segregation and grouping errors.

HETEROGENEITY TEST

The fundamental error of sampling is generated by constitution heterogeneity of the material being sampled (Gy, 1979; Pitard, 2003), which represents the variability in the content of the critical content, between individual fragments. The fundamental

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error depends on the weight of the sample, the weight of the lot to be sampled, the particle size distribution of the fragments, specially its top size of the fragments, the grade of the critical content in the lot, the liberation of the critical content as a function of the particle size, the shape and density of the fragments and the mineralogy of the critical content. *The main objective of heterogeneity test is to measure the fundamental error of a sampling protocol for the coarser top sizes generally the first and second splits.*

DIFFICULTY OF ESTIMATING THE LIBERATION FACTOR

Pierre Gy's formula is a triumph of thinking and a very useful tool for sampling, but it must be remembered that it is a simplification of a complex formula in five parameters. From this point of view the results obtained must be used with caution because the liberation is a complex process, therefore the liberation factor is very difficult to estimate, that is why so far universal liberation models do not exist.

For example, in porphyries liberation is strongly controlled by the style and type of the mineralisation. It has a different behaviour in hypogene sulfurs depending on the style of the mineralisation (veinlet type or disseminated), in supergene sulfurs (enriched zones) and also in endogenous oxides and exotic oxides (Carrasco, 2003).

Figure 1 shows the liberation behaviour studied by means of granulodensimetric analysis of two samples from El Salvador Mine. A detailed calculation of the liberation factor can be seen in Gy, (1967). The beta coefficient has been calculated as the slope of the straight lines of Figure 1.

Figure 1 suggests:

- Liberation behaviour depends on the critical content mineralogy, the ore texture and the number of *crystal families* (see Figures 2 and 3).
- The model $l = \left(\frac{d_l}{d}\right)^B$ (Gy, 1982; François-Bongarçon and Gy, 2001) does not work for the whole range of particle sizes because the beta coefficient varies as a function of the particle size. Nevertheless the *model works properly by intervals of particle sizes*.
- It is not possible to *a priori* define a beta coefficient for the calculation of the liberation factor by using the mentioned model. *It has to be measured performing experiments considering the geology*.
- For the coarser particle sizes (>0.3 cm) the beta coefficient is very small, therefore the increase of the liberation factor is small.
- It is very difficult to estimate the liberation factor by eyeballing or by using the geologist hand lens.
- The liberation curve is not just useful for sampling purposes, it is a good tool for geometallurgical modelling and optimisation of grinding and flotation circuits.

As the granulo-densimetric analysis is time consuming, expensive, and not useful for coarse material, based on the results of experiments done about liberation behaviour and because our

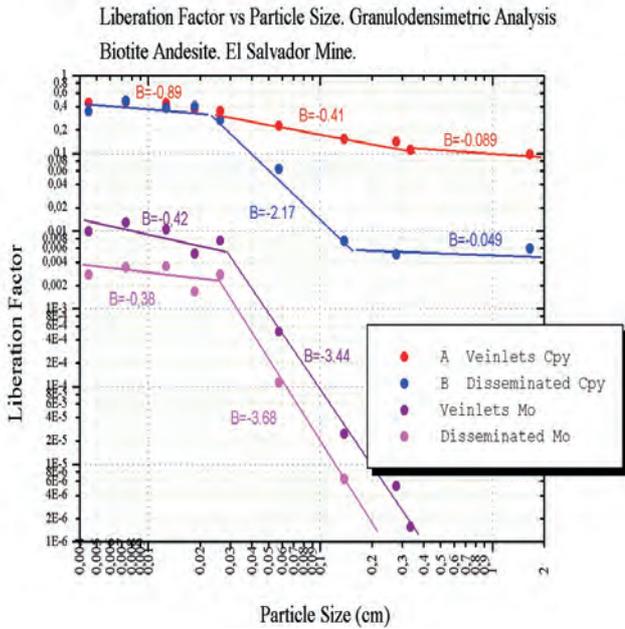


FIG 1 - Granulometry versus Liberation Factor. Sample A is a biotitised andesite with mineralisation of chalcopyrite veinlets (Figure 2) and sample B is a biotitised andesite with mineralisation of disseminated chalcopyrite (Figure 3). Notice that in sample B chalcopyrite is harder to liberate than sample A. This is because of the style of mineralisation. Chalcopyrite in veinlets liberates at coarser comminution stages than disseminated chalcopyrite.

At 0.02 cm the curves are very similar (modified from Carrasco, 2003).

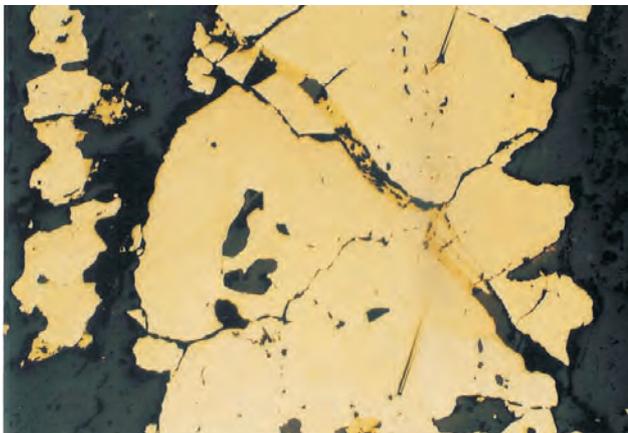


FIG 2 - Mineralisation dominated by chalcopyrite veinlets. The veinlet in the centre of the figure is 2 mm wide. (50 x) Crossed Nichols (Carrasco, 2003).

main interest is to optimise the critical stages of sampling protocols of blastholes, reverse circulation and diamond drill holes, we propose to calculate the variance of the fundamental error by means of the *heterogeneity test using two closed coarse relevant particle sizes classes* for different well delimited geological units, which can be found in porphyry copper deposits.

DESCRIPTION OF HETEROGENEITY TESTS (Based on Pitard, 1993 and Carrasco, 1998)

1. Inside a well defined geological unit prepare a sample of 250 kg. Use at least 50 increments of 5 kg. The sampled

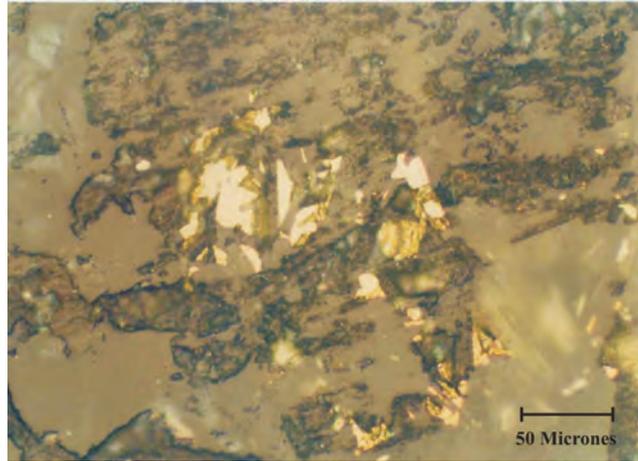


FIG 3 - Disseminated chalcopyrite mineralisation. Notice that some crystals are very fine (ten microns). This crystals are very hard to liberate even at 325 Tyler Mesh (45 microns). (400 x) Crossed Nichols (from Carrasco, 2003).

material can come from blastholes, diamond drill holes, reverse circulation drill holes, production faces of open pit mines, underground mines draw points, etc.

2. Prepare thin, polished sections for mineralogical analysis.
 3. Dry the sample for 24 hours.
 4. Crush the entire sample at 100 per cent -1" (-2.5 cm) using a jaw crusher. The crusher must be cleaned and sealed to prevent loss of fines and contamination.
 5. Screen all the sample to 1", 3/4", 1/2", 1/4" (-2.5 cm, 1.9 cm, 1.3 cm, 0.6 cm), 10#, 24# and 65# (# Tyler Mesh).
 6. Do the test in the fractions -1" +3/4" (-2.5 cm +1.9 cm) and -1/2" +1/4" (-1.3 cm +0.6 cm).
 7. Distribute the fraction(s) where the heterogeneity test is going to be done on a clean surface.
 8. In the chosen fraction(s), in order to minimise the other sampling errors, choose randomly:
 - 64 samples for base metals, and
 - 100 samples for gold, molybdenum, arsenic, antimony, silver, etc.
- Each sample must be formed of *n* fragments chosen *one by one* randomly. The weight of the samples must be similar between them, eg 200 g. Enumerate each sample from 1 - 64 or 1 - 100.
9. Pulverise each sample at -150# using a closed ring pulveriser. Don't use disc pulverisers which are dust generators and open. It is also known that gold sticks on them.
 10. Analyse each sample using atomic absorption, use volumetry (with preliminary separation) for ore grades higher than five per cent. For gold do screen fire assay with gravimetric finish. Be extremely cautious selecting the sample mass and the analytical method in order to minimise the sampling errors for analytical purposes and the analytical error, particularly for trace and ultra trace elements.
 11. Prepare the other fractions and analyse them.
 12. In order to study the impact of the segregation and grouping, delimitation, extraction and preparation error draw the curve, particle size versus grade.

13. Calculate the sampling constant for the particle sizes where the heterogeneity test was done:

$$C(d) = \frac{(\sum (A_i - A_L)^2 / (n-1) / A_L^2)}{d^3 (\frac{1}{M_s} - \frac{1}{M_L})}$$

where:

$d = \sqrt[3]{\frac{d_1^3 + d_2^3}{2}}$, d_1 and d_2 are the sizes of the screen chosen, where the heterogeneity was performed, eg (-3/4" +1/2") (-1.9 cm +1.3 cm)

C(d) sampling constant at size d (g/cc)

A_i is the grade of the i th sample (per cent)

A_L is the grade of the lot (per cent)

n = number of samples

M_s = weight of the sample (g)

M_L = weight of the lot

14. Design the sampling protocol using the largest sampling constant. In porphyry coppers arsenic minerals such as enargite-tennantite generally are the most heterogeneous (see Figure 4).

RESULTS OF HETEROGENEITY TEST CARRIED OUT IN CHILEAN PORPHYRY ORES

In Chilean porphyrys there is a vast experience carrying out heterogeneity test. These tests have been done in various mines and in the distinct geological units that have been recognised by geologists. Figure 4 resumes some of the results obtained.

We can conclude from Figure 4:

- Constitution heterogeneity is a function of the mineralogy of the critical content, grade of the element of interest, style of mineralisation, mineralisation associations, alteration processes, and ore textures. For example oxidation and supergene processes cause a diminution of heterogeneity.
- Arsenic minerals are the most heterogeneous followed by molybdenum minerals and then by copper minerals.
- The variability of the sampling constant for copper minerals is high (0.1 to 20). Sulfides are the most heterogeneous

followed by green oxides (atacamite, chrysocolla) and black oxides ('copper wad'), and then clay altered oxides.

- When no experimental tests are available, a safety line can be used to estimate the sampling constant for sulfides and oxides. A conservative beta coefficient of 0.1 can be assumed for coarser particle sizes then, the variance of the fundamental error can be estimated.

PARTICLE SIZE DISTRIBUTIONS VERSUS COPPER GRADE

Another useful product of the heterogeneity test is the knowledge of the particle size distribution as a function of grade.

Figure 5 shows several curves for various cases. Figure 5 suggests:

- For ores the grade content increases when the particle size decreases.
- The slope of the curve is a function of the *liberation behaviour* of the copper mineralogical species; therefore it depends on the ore texture, crystal size, mineralogy of the critical content and number of crystal families.
- In addition, the slope of the curve gives an idea of the impact of the segregation, grouping, delimitation, extraction and preparation errors in the sampling process. As long as the slope increases those sampling errors could increase.
- On the contrary when the curve is flat with respect to particular analyte, the under or over representation of one size fraction in a sample will cause only relatively small levels of bias in the assay result for that analyte (ie Mina Sur altered oxides).
- The tail has a different behaviour. As the particle size decreases the copper grade decreases. As the copper minerals are linked to the coarse fractions, not uncommon incorrect sampling systems rejecting the coarser fragments, conducts to underestimation of the tail grades, hence to overestimation of the metallurgical recovery.
- For ores, the slopes are always *very flat for the coarser size fractions*.
- For finer particle sizes the slopes increase as a consequence of the liberation process of the critical content.

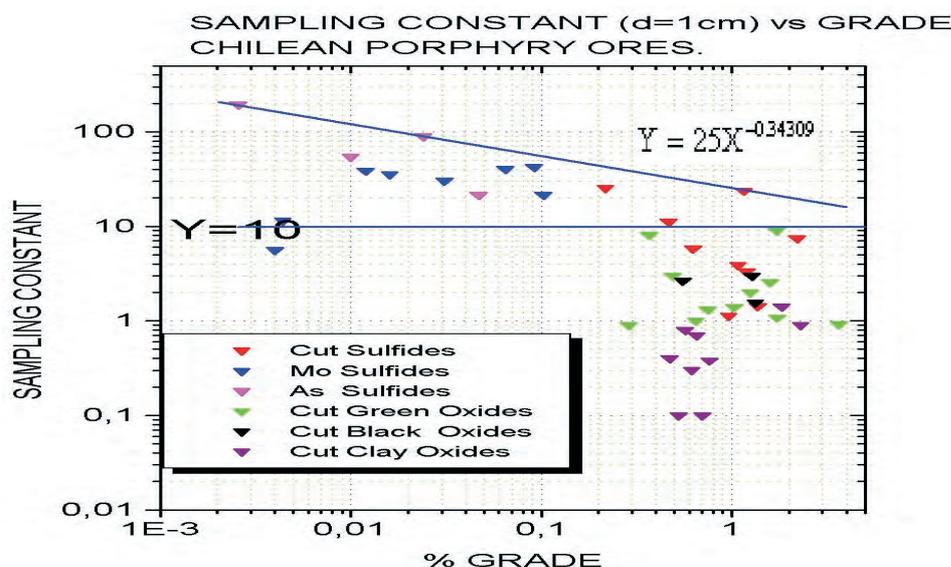


FIG 4 - Sampling constant versus grade, d = 1 cm (from Carrasco, 2003).

COMPARISON OF THE ESTIMATED FUNDAMENTAL ERROR VERSUS THE TOTAL SAMPLING ERROR FOR BLASTHOLES AT CHUQUICAMATA MINE

To compare the magnitude of the relative fundamental error of the sampling process of blastholes with the total sample error the systematic duplicates of blastholes sampling where studied. Figure 6 shows the results.

The study suggests:

- As expected the fundamental error for the sampling process of blastholes is always lower than the total error.
- The total relative sampling, plus analytical errors increase as the content decreases.
- For copper, molybdenum and arsenic the total sampling and analytical errors are acceptable on the grade ranges of business relevance.
- For very low grades the total sampling and analytical errors increase above 16 per cent, probably due to analytical imprecision. A study must be performed in order to find out the causes of such an increase.

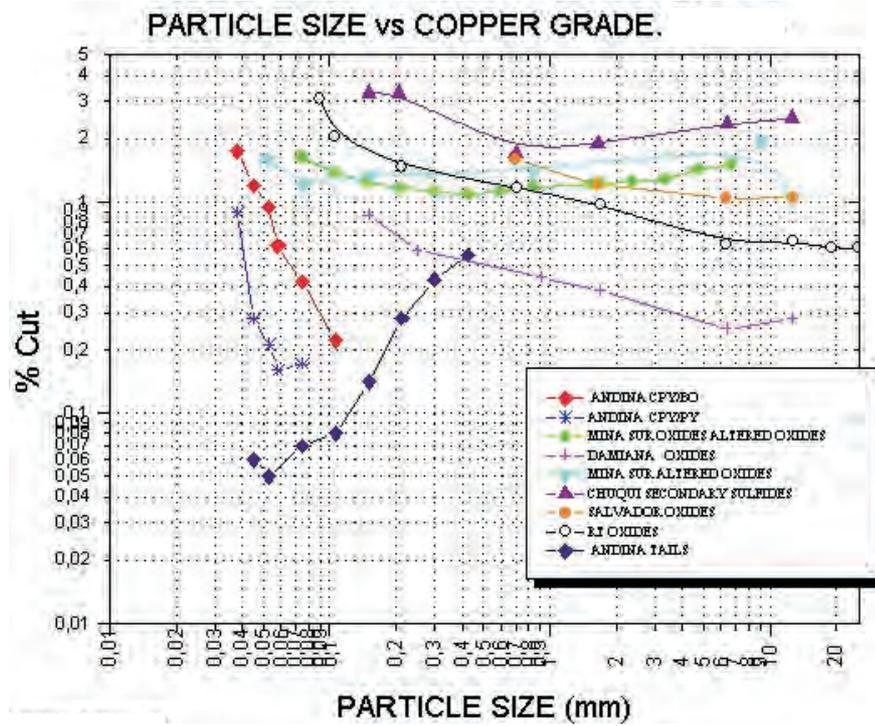


FIG 5 - Copper content versus particle size for several ores and a tail.

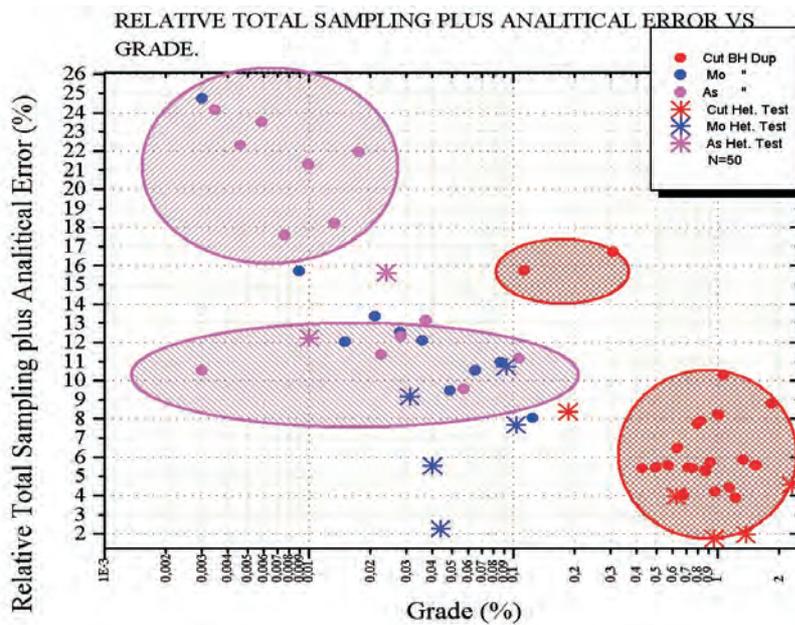


FIG 6 - Fundamental error versus total sampling plus analytical errors from BH duplicates at Chuquicamata Mine.

INGAMELLS' TEST

Chemists, when sampling for analytical purposes generally work with the paradigm of homogeneity neglecting the influence of the fundamental, grouping and segregation error. Therefore, some times the sample selection process and the sample weight is far away from the principles of the sampling theory (Gy, 2000).

The analytical mass sample for chemical element determination is established previously by several reasons: instrumentation, system of measurement, dissolution times, reagent consumption and others conventions without any consideration of the constitution and distribution heterogeneity of the sample.

As discussed above, liberation is a complex process conducting to several heterogeneity distributions as a function of several factors, but never to homogeneity. Therefore, it is necessary to establish experimentally the mass of sample for analysis. Ingamells' test is a good practical tool for this purpose (Ingamells and Pitard, 1986).

Figure 7 shows in a very explicit way the influence of the sample weight for analytical purposes on the total sampling errors of anodes of El Salvador refinery.

The graph indicates the huge contribution of irrelevant or artificial variability or noise to the process when using an improper small sample weight for chemical analysis. For example, selecting 0.25 grams for oxygen assay introduces a big sampling error, which impels the observation of the process. When the sample weight increases the relative nugget effect decrease in such a way the process can be studied properly. Because the smelting process does not work at random the good negative correlation between relative nugget effect and sample weight for chemical analysis explains by itself the need for experimental determination of the sample weight.

Prior to explaining the basis of Ingamells' test, it is important to point out the relevance of analytical accuracy and precision. In order to assure the quality of the chemical analysis the atomic absorption spectrometry (AAS) method for copper was tested by electrogravimetry, a primary absolute method on 47 standard reference materials belonging to several geological units of several CODELCO porphyry coppers. Figure 8 shows the results.

This figure indicates the excellence of the AAS method for copper when the principles of analytical chemistry are applied properly.

DESCRIPTION OF INGAMELLS' TEST

1. The pulverise sample – 100# Ty is spread on a very clean surface.
2. Prepare a very thin cake (around of 0.5 cm high) as shown in Figure 9.
3. Divide the cake in N column and N files, for copper use N = 10.
4. Take from each square a sample of 0.25 g by using a stratified random mode.
5. Register the mass and the positions of each sample.
6. Dissolve with nitric, perchloric and sulfuric acid, avoid to dryness. Dissolve the salt with water and diluted hydrochloric acid. Transfer the solution to 100 ml volumetric flask and complete the volume with water. Homogenise.
7. Determine the concentration of the copper by AAS using the principal wavelength (324.8 nm).
8. Plot and observe the results.
9. Study the data of the histogram.
10. Composite at random samples of 2n, 3n, 4n, etc. Calculate means, variance, and mode. Study the evolution of the shape of the histogram.
11. Calculate the variogram function of the grade along several directions.
12. If variogram is pure nugget effect or the nugget component is higher than 70 per cent of the total variance calculate the variance of the sampling error by:

$$\sigma_e^2 = \frac{\sigma^2}{n} \quad (\text{Central Limit Theorem})$$

where:

s = standard deviation of the grade using the original sample weight (0.25 g) = w

σ_e = standard deviation of the sampling error using mass W, n = W/w

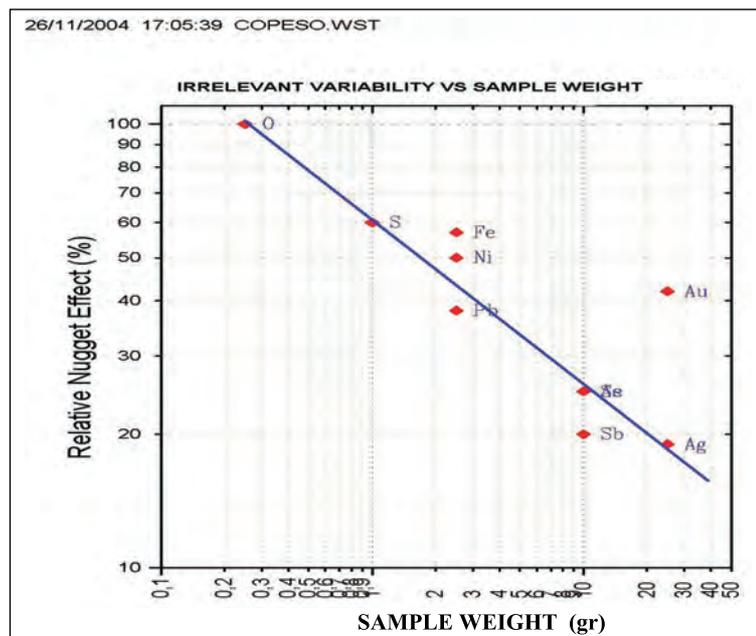


FIG 7 - Relative nugget effect of the temporal variograms of several elements versus the sample weight for chemical analysis. El Salvador refinery.

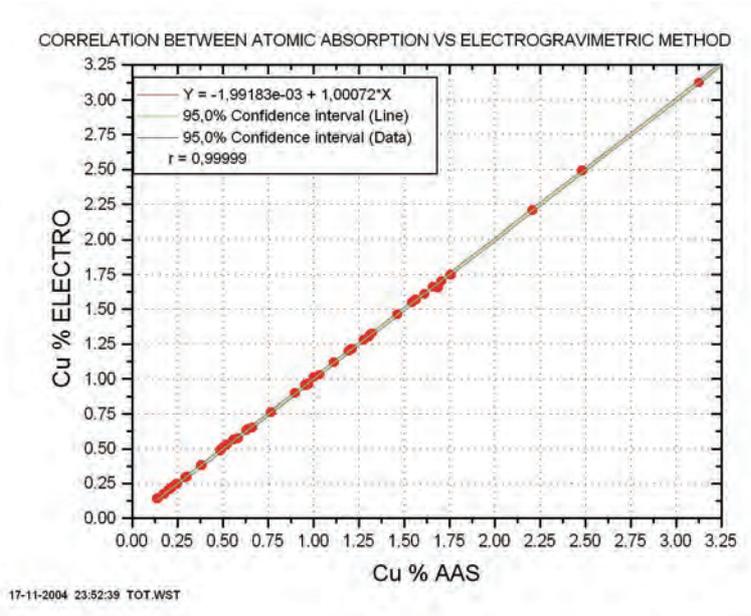


FIG 8 - AAS method versus electrogravimetry method.



FIG 9 - Sample for Ingamells' test.

EXAMPLES

Example1: Determination of the sample mass for copper chemical analysis – Head of the flotation plant at Andina Mine

Figure 10 shows the 'spatial' distribution of the copper grade.

One can observe in this figure a high degree of segregation. As a matter of fact the high grades are located in the upper part of the sample and in some specific spots. The picture also indicates how dangerous is to take a sample for analytical purposes based only in a single increment. The copper grade of 0.25 grams samples varies in this particular case from 0.9 per cent to 1.025 per cent. It is clear that the assumption of homogeneity in pulps for chemical analysis does not stand in this case. Our experience after many Ingamells's tests is that homogeneity is an illusion. The experiment indicates that proper homogenisation and the selection of the sample by taking as much increments as possible in a random stratified mode is essential to minimise the grouping and segregation errors.

Figure 11 shows the variogram functions in the vertical and horizontal directions.

The strong zonal anisotropy indicating a higher variability in the vertical direction, the radial drift in the same direction, and the spherical behaviour in the horizontal direction confirms the existence of segregation. In other words, in this particular case a high-grade sample is surrounded by a high-grade zone and a low-grade sample is surrounded by a low-grade zone. The magnitude of the nugget effect is less than 50 per cent of the total variance; hence the random component is smaller than the structured components.

Figure 12 shows the evolution of the copper grade distribution as a function of the sample mass.

One can observe the evolution of the probability distribution function as a function of the increase of the sample weight. At 0.25 grams, the distribution is very asymmetric with a mode higher than the mean. Therefore, when small samples are taken the probability of overestimation of the sample grade is around 60 per cent. When the sample weight increases above 0.75 grams, the distribution has a symmetrical normal behaviour therefore the probability of underestimation is equal to the probability of overestimation. *The sample weight where the mode converges to the mean is then the minimum sample weight to use.*

- 13. If variogram function presents special structures for the grade calculate the variance of the sampling error by:

$$\sigma_e^2 = \frac{1}{N} \sigma^2(0/s) + \frac{Co}{N} \quad (\text{Matheron, 1970})$$

where:

$\sigma^2(0/s)$ = dispersion variance of point sample grade inside the cell S (Random Stratified Sampling Mode)

Co = nugget effect

N = number of cells = number of samples

- 14. Plot the cumulative histogram for different sample sizes on probabilistic normal paper. Note when the distribution becomes normal.
- 15. Plot the mean, mode and the 95 per cent confidence interval ($\bar{x} \pm 2\sigma_e$) as a function of the sample weight.
- 16. Define the sample mass for chemical analysis.

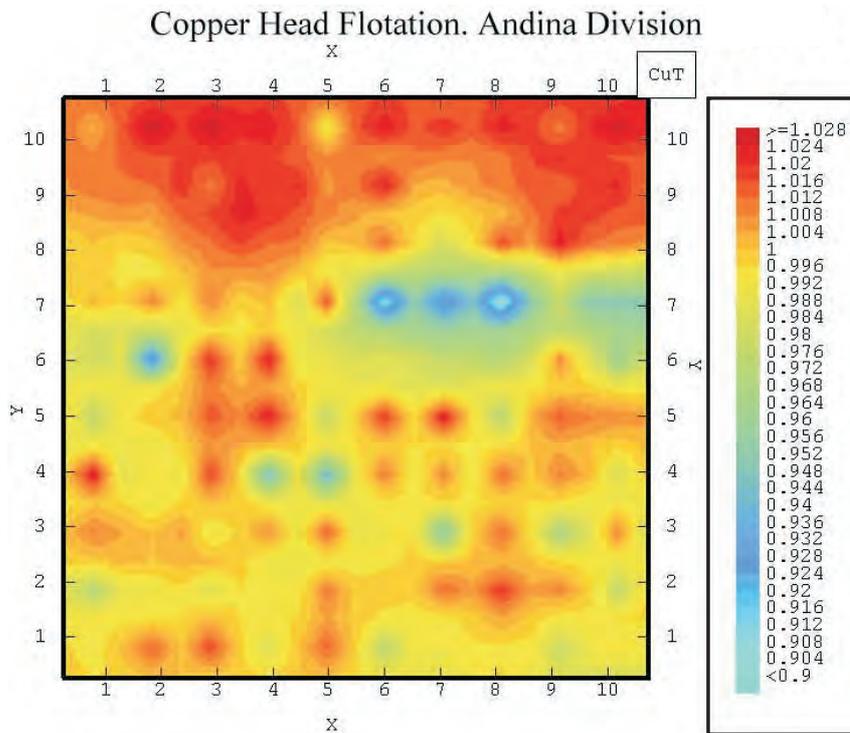


FIG 10 - 'Spatial' distribution of the copper grade.

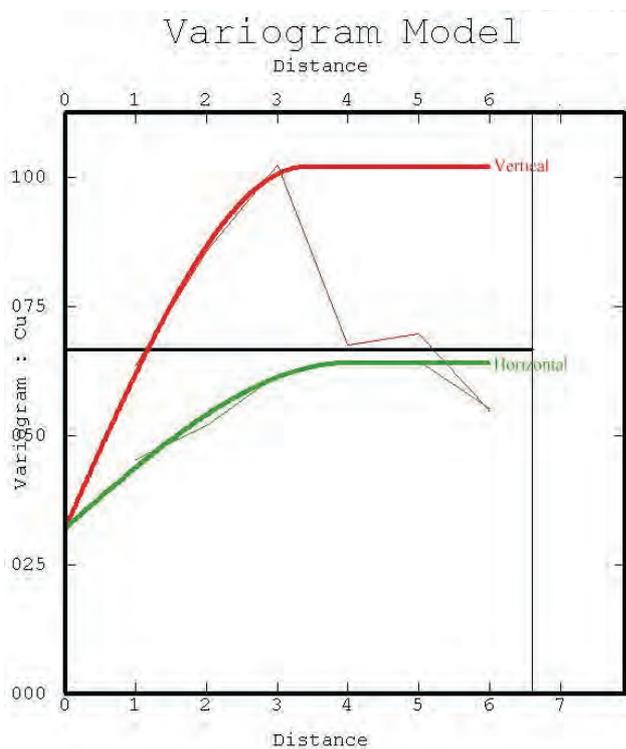


FIG 11 - Variogram of copper grade.

Figure 13 shows the evolution of the confidence for the mean grade of the sample as a function of the sample mass.

It can be seen from the figure that the sample weight has a very important role in the magnitude of the sampling error. In this particular case the optimum sample weight is around 1.25 grams. Note the convergence of the mode to the mean.

Example2: Determination of the sample mass for copper chemical analysis – Porphyry ore

Figure 14 shows the 'spatial' distribution of the copper grade.

There is no apparent segregation in the sample, never the less the variability of the copper grade is extremely high. As a matter of fact a 0.25 grams sample can give back chemical analysis results between 0.6 per cent and 1.28 per cent copper.

The variograms (Figure 15) are stationary and pure nugget effect, then the spatial correlation between grades is nil. In other words, it means a high-grade sample does not represent a high-grade zone neither a low-grade sample a low-grade zone indicating no segregation.

Figure 16 shows the evolution of the copper grade distribution as a function of the sample mass.

It can be seen the evolution of the probability distribution function as a function of the increase of the sample weight. At 0.25 grams the distribution is very asymmetric with a mode lower than the mean. Therefore when small samples are taken the probability of underestimation of the sample grade is around 60 per cent. When the sample weight is two grams, the distribution has a symmetrical normal behaviour, therefore the probability of underestimation is equal to the probability of overestimation. Never the less, two grams still conduct to a big fundamental error, ± 0.1 per cent copper at a confidence level of 95 per cent. In this particular case a proper sample weight is eight grams (see Figure 17).

Perhaps the main factor contributing to the high heterogeneity of this sample is over grinding. As a matter of fact, the sample has 60 per cent of the fragments under 11 μm (see Figure 18). As segregation is minimal, the over grinding probably causes a high degree of liberation of the critical content increasing then the fundamental error variance. Such over grinding is unnecessary for analytical purposes. Therefore is advisable to control grinding when preparing pulps.

Not over crushing, avoid mineral liberation; in order to minimise the segregation and grouping error.

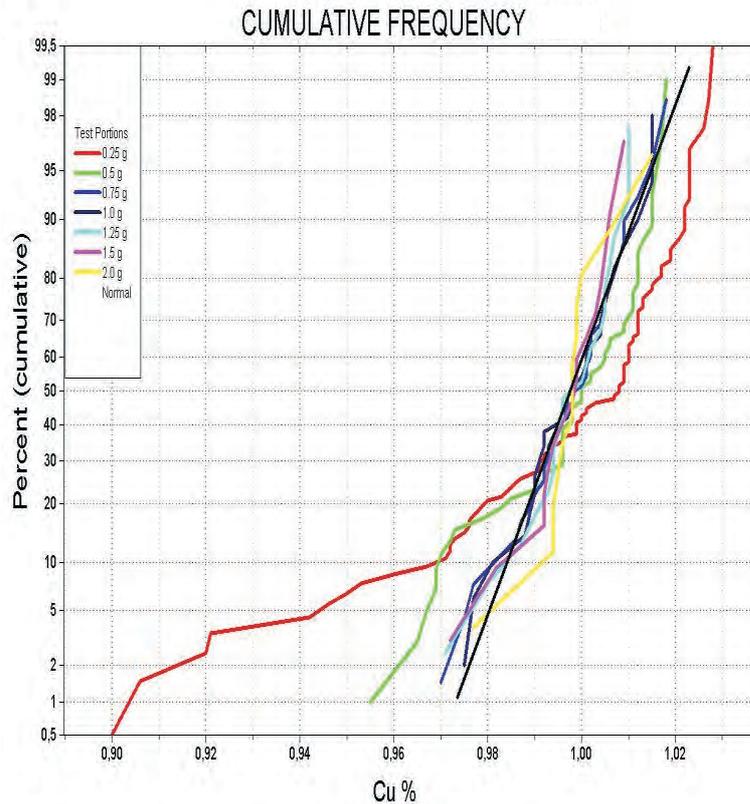


FIG 12 - Distribution of the copper grade versus sample mass.

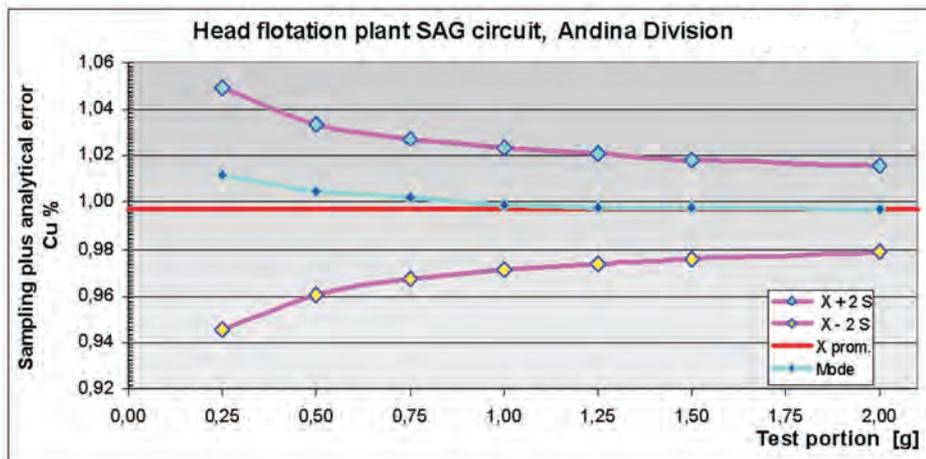


FIG 13 - Confidence of the mean grade versus sample weight.

COMPARISON OF ESTIMATED SAMPLING ERROR FOR CHEMICAL ANALYSIS PURPOSES – INGAMELL TEST VERSUS BLASTHOLES DUPLICATES AND STANDARD REFERENCE MATERIALS, CHUQUICAMATA MINE

The relative sampling plus analytical error was calculated for copper, molybdenum and arsenic from Ingamells’ test, blastholes duplicates and standard reference materials. The results are shown in Figure 19.

The principal conclusions from Figure 19 are:

- the results of Ingamells’ test are coherent with duplicates and SRM;

- relative error decreases while grade of the element increases;
- heterogeneity has a great increase for low grade for all elements;
- Ingamells and RSM’s results have less error than duplicates because they are analysed with special care, not at routine;
- in general, for CODELCO Chilean porphyry ores, the sample mass for analytical purposes, which give good results, is one gram for copper, one to two grams for molybdenum and two to five grams for arsenic; and
- for geochemical purposes more than four grams are always needed.

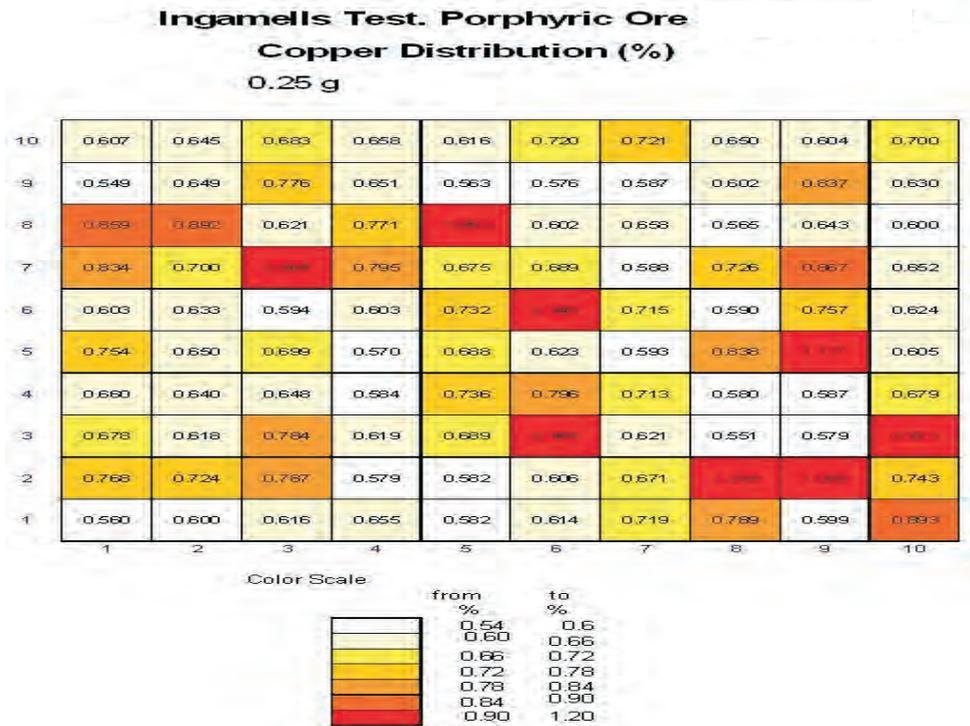


FIG 14 – 'Spatial' distribution of copper grade.

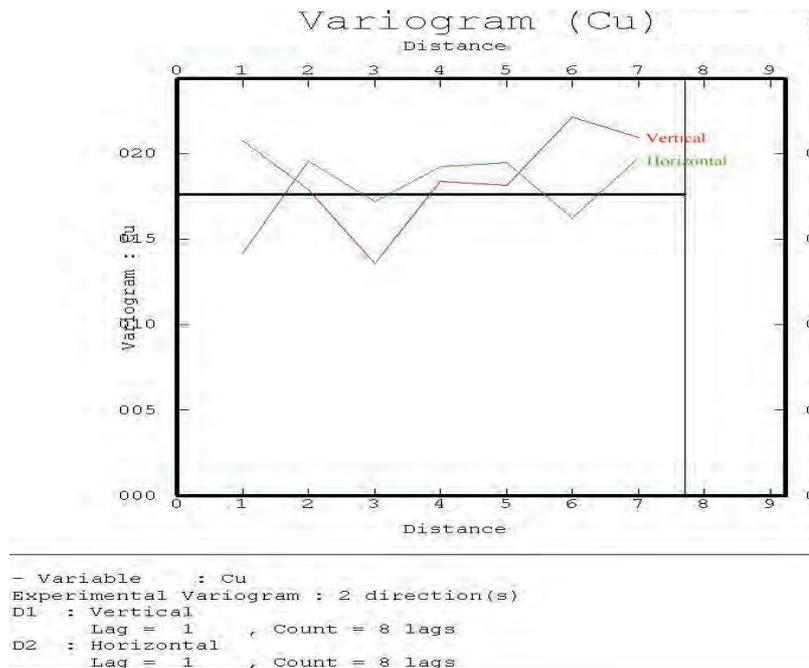


FIG 15 - Variogram of copper grade.

CONCLUSIONS

- Ore forming processes determine the constitution and distribution heterogeneity at several scales. Therefore, geological knowledge is essential for the determination of the uncertainty of the sampling processes.
- It is very difficult to estimate the liberation factor by eyeballing or by using the geologist hand lens. Therefore the variance of the fundamental error must be measured performing experiments considering the geology. The

heterogeneity test is a reliable tool for this purpose.

- Liberation behaviour depends on the critical content mineralogy, the ore texture and the number of *crystal families*.
- The model $l = \left(\frac{d_l}{d}\right)^\beta$ does not work for the whole range of particle sizes because the beta coefficient varies as a function of the particle size. Never the less the *model works properly by intervals of particle sizes*.

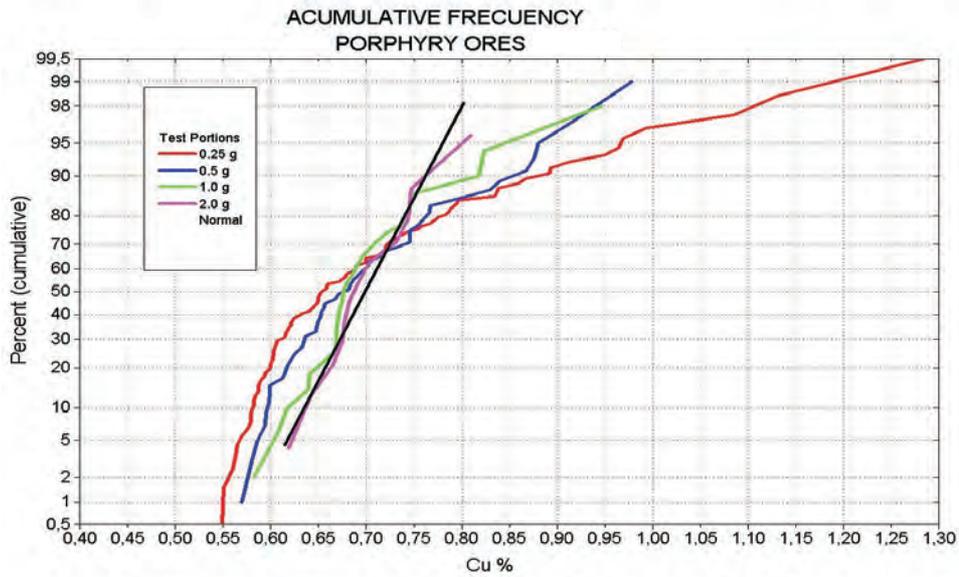


FIG 16 - Distribution of copper grade versus sample mass.

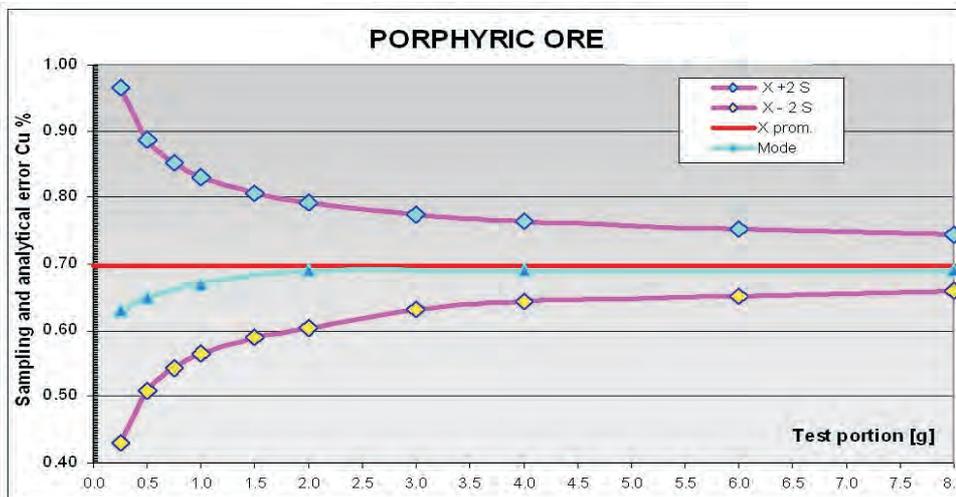


FIG 17 - Confidence of the mean grade versus sample weight.

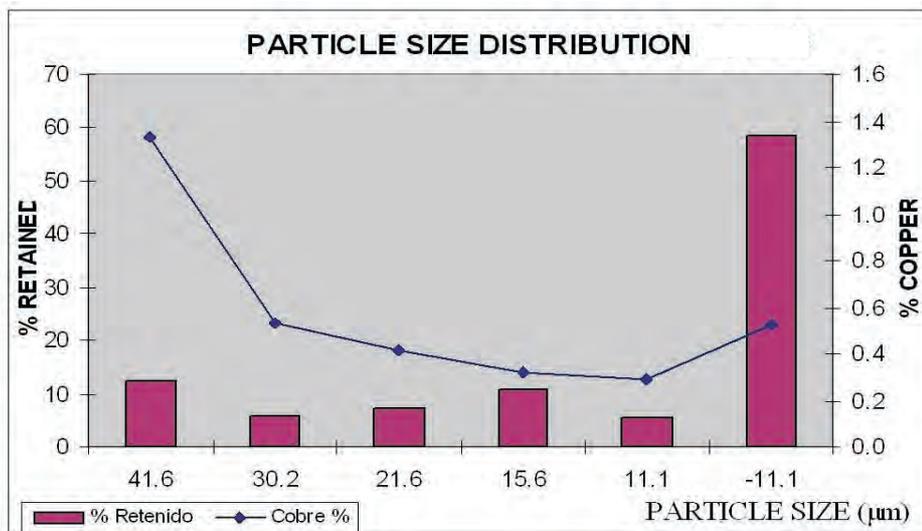


FIG 18 - Particle size distribution.

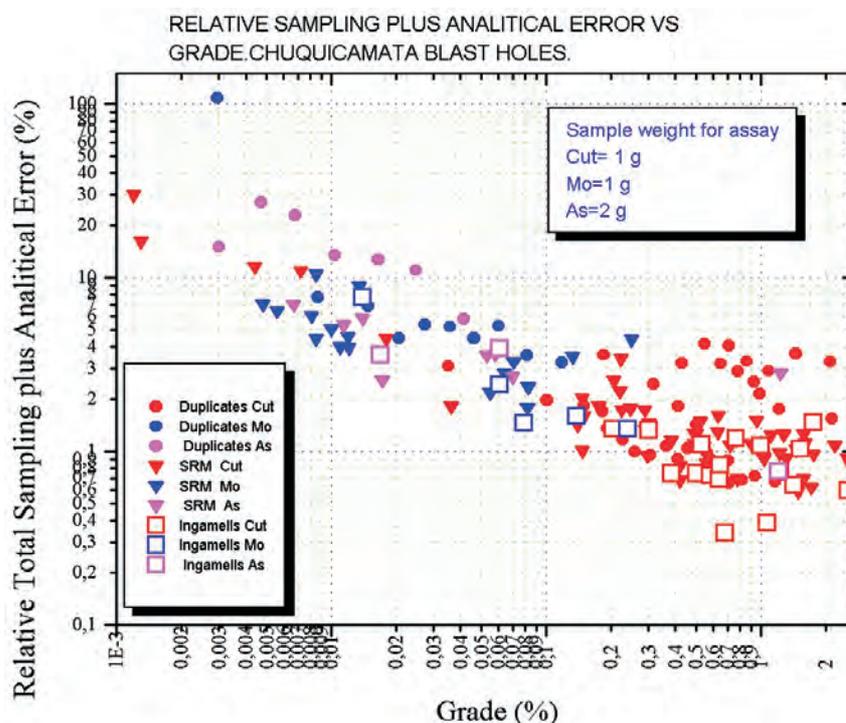


FIG 19 - Relative sampling error for analytical purposes. Duplicates, SRM and Ingamells' test.

- For the coarser particle sizes (>0.3 cm) the beta coefficient is very small, therefore the increase of the liberation factor is small.
- The liberation curve is not just useful for sampling purposes. It is a good tool for geometallurgical modelling and optimisation of grinding and flotation circuits.
- The sampling constant at $d = 1$ cm for CODELCO porphyry coppers varies between 20 and 100 for arsenic minerals, eight to 50 for molybdenite and 0.1 to 20 for copper minerals. The variability of the sampling constant for copper minerals is high (0.1 to 20). Sulfides are the most heterogeneous followed by green oxides (atacamite, chrysocolla), black oxides (wad), and then clay altered oxides.
- When no test is available a safety relation between the sampling constant and the grade at $d = 1$ cm exists:

$$C(d) = 25X^{-0.34309}$$

where:

X is the grade of As, Mo or Cu in per cent.

For coarser particle sizes ($d > 0.3$ cm) a beta coefficient of 0.1 could be used to calculate the sampling constant for different particle sizes.

- Other useful product of the heterogeneity test is the knowledge of the particle size distribution as a function of grade. The slope of the curve is a function of the *liberation behaviour* of the mineral species of interest; therefore it depends on the ore texture, crystal size, mineralogy of the critical content and number of crystal families. In addition, the slope of the curve gives an idea of the impact of the segregation, grouping, delimitation, extraction and preparation errors in the sampling process. As long as the slope increases those sampling errors increase.
- It is necessary to establish experimentally the sample mass for analysis. Ingamells' test is a good practical tool for this purpose.

- Generally, for CODELCO Chilean porphyry ores, the sample mass for analytical purposes, which give good results, is one gram for copper, one to two grams for molybdenum and two to five grams for arsenic.
- For geochemical purposes more than four grams are always needed.

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