Optimization of sampling practices at Corrego do Sitio metallurgical plant

Marcus Félix Magalhães[1], Ana Carolina Chieregati[2], Vinícius Moreira Assis[3], Guilherme Costa Peixoto[4], Márcio Salgado Pereira[5]

[1] Process Engineer, AngloGold Ashanti, Córrego do Sítio Mineração, Santa Bárbara, Brazil, MFMagalhaes@anglogoldashanti.com.br

[2] Professor, Department of Mining and Petroleum Engineering, University of São Paulo, São Paulo, Brazil, ana.chieregati@usp.br

[3] Chief Metallurgist, AngloGold Ashanti, Córrego do Sítio Mineração, Santa Bárbara, Brazil, VMAssis@anglogoldashanti.com.br

[4] Metallurgy Manager, AngloGold Ashanti, Córrego do Sítio Mineração, Santa Bárbara, Brazil, GCPeixoto@anglogoldashanti.com.br

[5] Metallurgical Engineer, AngloGold Ashanti, Córrego do Sítio Mineração, Santa Bárbara, Brazil, MSPereira@anglogoldashanti.com.br

ABSTRACT

This paper describes the optimization of sampling practices and equipment at AngloGold Ashanti metallurgical plant and laboratory in Córrego do Sítio II (CDSII), which presented, mainly, the following problems: (i) inappropriate sampling equipment at the plant; (ii) absence of geostatistical study to define the optimal sampling interval; (iii) absence of heterogeneity study to calculate minimum sample masses and optimize sample preparation protocols; and (iv) inadequate chemical analysis technique for all ore types. Based on the Theory of Sampling (TOS), three studies were carried out at Córrego do Sítio: (1) variographic study to define the optimal sampling interval at the plant feed; (2) heterogeneity test to estimate minimum representative sample masses at each stage of the process and to optimize sample preparation protocols; and (3) comparative test between the standard fire assay and the screen fire assay techniques for gold grade analysis. The results of this work allowed the company to ensure the reliability of sampling and sample preparation equipment, to increase sample representativeness at all stages, to optimize sampling and sample preparation protocols and to enhance the operational control by minimizing grade estimation errors.

1. INTRODUCTION

The Córrego do Sítio (CDS) complex, located in the city of Santa Bárbara, state of Minas Gerais, Brazil, belonging to AngloGold Ashanti, for years has been extracting gold from sulphide and oxidizing ore from underground and open pit mines. The gold derived from sulphide ores, corresponding to more than 75% of the whole production, is processed in a metallurgical plant, passing through the stages of comminution, concentration, pre-treatment concentrate, leaching tanks and electrodeposition. The optimization of sampling procedures, equipment and protocols was required for each processing step, especially in the plant feed and tailings, according to the correct practices and standards required by the company.

According to Chieregati (2009), precious metals, especially gold, present several difficulties in sampling due to the very low gold concentrations, high segregation due to the gold density (19.3 g/cm^3) when gold is liberated, in addition to the contamination of sampling equipment by nuggets that are difficult to be comminuted. These problems can be solved and, at the very least, minimized with correct sampling equipment and procedures and with optimized preparation protocols that eliminate or mitigate sampling errors.

The process of gold grade estimation is divided basically in three main stages: (1) the primary sampling of a given lot, (2) the secondary sampling or preparation of the sample and (3) the chemical analysis. Each stage generates errors, characterized by their variance, which can be added, resulting in the overall estimation error

(OEE). Figure 1 illustrates the components of the overall estimation error, based on Pierre Gy's Theory of Sampling (TOS), showing systematic errors in light gray and random errors in dark gray.

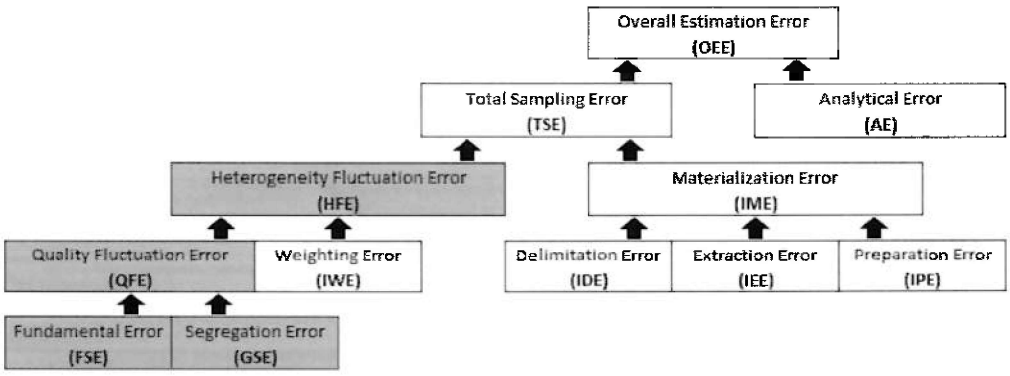


Figure 1: Components of the overall estimation error
(modified from CHIEREGATI & PITARD, 2018).

The present study aims to the optimization of sampling equipment and practices in CDS plant and laboratory, based on the Theory of Sampling (TOS), including a variographic study to define the optimal sampling interval of the tailings and plant feed, heterogeneity test to estimate the representative minimum masses at each stage of the process, optimization of the sample preparation protocols and comparative tests between fire assay and screen fire assay to determine the most suitable analytical technique for gold grade analysis.

2. METHODOLOGY

The first step of the optimization process was the variographic analysis of the automatic samplers installed in the metallurgical plant, aiming at the calculation of the heterogeneity fluctuation error (HFE). Samples were collected from the main flows of the plant, which compose important data for the mass balance process: feed grade, flotation tailings and leach tailings. The campaign generated 48 samples per flow, separated by 2.5 minutes, to elaborate the variograms.

The second step was the heterogeneity test, which aims to calculate the fundamental sampling error, estimate the minimum mass of the samples and optimize the sampling and sample preparation protocols, defining the optimal protocol for the laboratory.

To carry out the heterogeneity test, the following steps were adopted:

1. Selection of approximately 300 kg of representative sample;
2. Screening of the material in the following size fractions:
   - –19.0 +12.7 mm
   - –12.7 +6.35 mm
   - –6.35 +4.75 mm
   - –4.75 +3.35 mm
3. Selection, for each size fraction, of 50 groups with at least 100 fragments each, randomly collected one by one;
4. Calculation of the $\mathbf{IH_L}$ (constant factor of constitutional heterogeneity).

The third step was the comparison between the fire assay and the screen fire assay techniques. The objective of this comparison was to evaluate the possible existence of bias due to the analysis method employed for gold grade determination. The main difference between the standard fire assay technique and the screen fire assay technique is that the first one analyzes a single aliquot of 30 g (or the average of two or three aliquots) collected

from an initial mass; while the second one analyzes the entire mass retained in 200 mesh from an initial 600g mass, in addition to an aliquot of 30g collected from the passing mass in 200 mesh, resulting in a balanced final grade.

2.1. Variographic Analysis

The variogram is a function of time between two points located on an axis and allows the characterization of the one-dimensional heterogeneity of chronologically ordered data. The function of the experimental variogram is defined by:

$$v(j) = \frac{1}{2N}\sum_{q}(t_{q+j} - t_q)^2 \quad (1)$$

Where **j** is the lag or time interval between two increments, **v(j)** is the variogram function for the time interval **j**, t_q is the grade of the increment **q** and t_{q+j} is the grade of the increment separated by **j** from the increment **q**. Note that there are several pairs of values separated by **j**, therefore **N** represents the number of pairs.

Then, the variance, the absolute standard deviation, and the relative standard deviation of the heterogeneity fluctuation error should be calculated at a 95% confidence interval, assuming **Q** as the total number of increments collected to a given range of time **j**. The variance of the heterogeneity fluctuation error is described by the following equation:

$$s^2(HFE) = \frac{W(j)}{Q} \quad (2)$$

W(j) is called "error generator" and can be defined as a function of the variographic interval **j**. When the masses of the collected increments do not show variations greater than 20%, we can assume that the variogram of the grade t_q is practically the same variogram of the heterogeneities of the sampled material (Gy, 1998).

2.2. Heterogeneity test

According to Chieregati and Pitard (2018), constitutional heterogeneity (CH_L) is a type of of heterogeneity that exists when we consider the fundamental properties of the fragments in a lot, observing them one by one. By definition, the zero of CH_L would be a lot consisting of fragments identical in shape, size, density, etc. Therefore, the CH_L of the fragments of a lot at a given comminution degree is an intrinsic property of the lot and does not vary unless another comminution stage is performed (Pitard, 1993). Mixes or homogenizations have no influence on CH_L.

When a sample mass, M_S, is randomly collected, fragment by fragment with the same probability, from a lot of fragmented material, M_L, a sampling error arises between the real (and unknown) grade of the lot and the grade of the selected sample. This error is the lowest error for a sample collected under ideal conditions, and therefore is called the fundamental sampling error (FSE). The FSE is specifically related to the CH_L of the same lot and is the only error that is never zero. Its importance may be secondary to most constituents, but usually it becomes larger to constituents occurring in smaller proportions, and much larger to trace elements contained in high-purity materials or to low-grade precious metals.

The FSE is generated at each sampling stage and generally presents an insignificant average, being characterized by its variance, s^2_{FSE}, calculated according to Gy's formula:

$$s^2_{FSE} = \left(\frac{1}{M_S} - \frac{1}{M_L}\right) cfgld^3 \quad (3)$$

Where **c**, **f**, **g**, and **l** are the Gy's factors, M_S and M_L are, respectively, the mass sample and the initial mass of the lot, and **d** is the maximum size of the fragments, top size or d_{95}.

In the case of precious metals or very low-grade ores, the greatest difficulty is to estimate the liberation factor **1**, that varies greatly and therefore it is difficult to assign it an average value. The heterogeneity tests were developed in order to calculate the **FSE** variance without needing to estimate the liberation factor, and its result is the calibration of the sampling constants K and α in Equation 4.

To calculate the minimum masses and nomographs, the following equation can be used, where IH_L is calibrated by the heterogeneity test.

$$s^2_{FSE} = kd^{\alpha} \left(\frac{1}{M_S} - \frac{1}{M_L}\right) = IH_L \left(\frac{1}{M_S} - \frac{1}{M_L}\right) \quad (4)$$

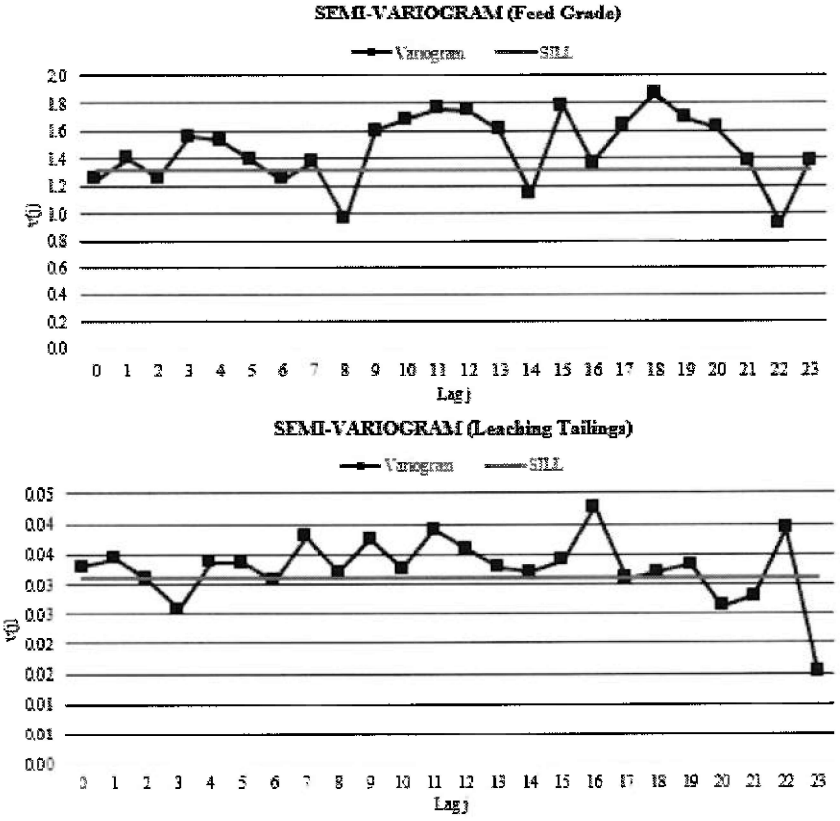
A value of 16% is accepted as the maximum relative deviation of the fundamental error recommended for gold ores.

3. RESULTS AND DISCUSSION

3.1. Variographic Analysis

The variograms were calculated using the results of the gold grades of the samples collected sequentially by the automatic samplers in the plant.

The values **v(0)**, also called the "nugget effect", were estimated by the regression of the first three points of the variogram to x = 0. The variograms are represented in Figure 2.



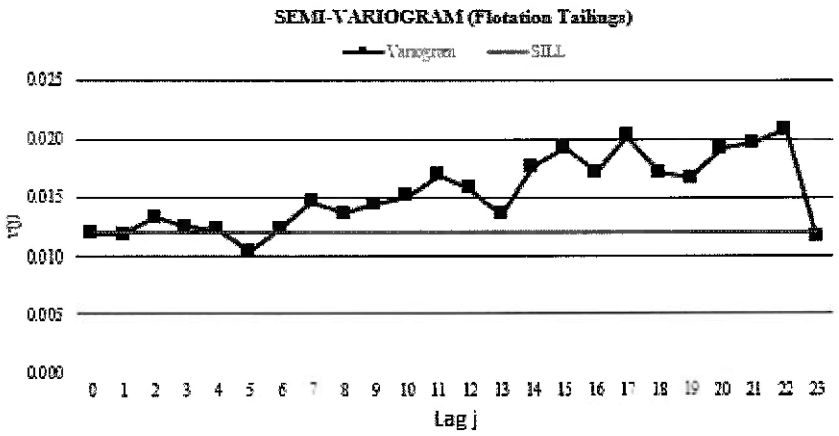


Figure 2: Experimental variograms representing the plant streams.

The horizontal red line of the variograms is called the "sill", characterizing the global heterogeneity of the data. This line represents the statistical variance of the data and can be seen as the model of a variogram in which its values are not correlated. The variogram **v(j)**, in turn, characterizes the sequential heterogeneity of the data.

Table 1 presents the results of the heterogeneity fluctuation error variances for each time interval **j** considered. Multiplying the value of **j** by the minimum interval between sample collections (in this case, 2.5 minutes) results in the value of the time interval considered to the variance calculation. The last column of each stream represents the 95% confidence interval, assuming that the errors present normal distribution.

Table 1: Variance of the heterogeneity fluctuation error for each plant stream.

Lag j	Time	Feed grade			Flotation tailings			Leaching tailings		
	(min)	s^2(HFE)	s(HFE)	s(HFE)95%	s^2(HFE)	s(HFE)	s(HFE)95%	s^2(HFE)	s(HFE)	s(HFE)95%
0	00:00:00	-	-	-	-	-	-	-	-	-
1	00:02:30	0.0259	0.1610	9.15%	0.00023	0.0151	8.38%	0.0007	0.0262	4.63%
2	00:05:00	0.0281	0.1676	9.52%	0.00024	0.0154	8.52%	0.0007	0.0268	4.74%
3	00:07:30	0.0291	0.1705	9.69%	0.00025	0.0157	8.71%	0.0007	0.0273	4.83%
4	00:10:00	0.0288	0.1696	9.64%	0.00026	0.0162	8.98%	0.0008	0.0275	4.86%
5	00:12:30	0.0290	0.1704	9.68%	0.00028	0.0167	9.24%	0.0007	0.0274	4.83%
6	00:15:00	0.0307	0.1752	9.95%	0.00029	0.0170	9.41%	0.0007	0.0270	4.76%
7	00:17:30	0.0327	0.1809	10.28%	0.00030	0.0172	9.54%	0.0007	0.0268	4.73%
8	00:20:00	0.0346	0.1859	10.56%	0.00030	0.0174	9.63%	0.0007	0.0272	4.79%
9	00:22:30	0.0361	0.1900	10.80%	0.00031	0.0175	9.67%	0.0008	0.0277	4.89%
10	00:25:00	0.0371	0.1927	10.95%	0.00030	0.0174	9.64%	0.0008	0.0282	4.97%
11	00:27:30	0.0377	0.1942	11.04%	0.00030	0.0174	9.63%	0.0008	0.0286	5.04%
12	00:30:00	0.0380	0.1949	11.07%	0.00031	0.0175	9.69%	0.0008	0.0288	5.09%
13	00:32:30	0.0383	0.1956	11.12%	0.00032	0.0178	9.83%	0.0009	0.0292	5.16%
14	00:35:00	0.0389	0.1973	11.21%	0.00033	0.0181	10.04%	0.0009	0.0299	5.27%
15	00:37:30	0.0394	0.1986	11.29%	0.00034	0.0185	10.27%	0.0009	0.0306	5.40%
16	00:40:00	0.0393	0.1982	11.26%	0.00036	0.0189	10.44%	0.0010	0.0310	5.48%

17	00:42:30	0.0395	0.1987	11.29%	0.00037	0.0192	10.62%	0.0010	0.0315	5.57%
18	00:45:00	0.0409	0.2024	11.50%	0.00038	0.0195	10.80%	0.0010	0.0322	5.68%
19	00:47.30	0.0431	0.2075	11.79%	0.00040	0.0199	11.02%	0.0011	0.0329	5.81%
20	00:50:00	0.0454	0.2130	12.10%	0.00041	0.0203	11.25%	0.0011	0.0335	5.91%
21	00:52:30	0.0479	0.2188	12.44%	0.00043	0.0208	11.51%	0.0012	0.0341	6.03%
22	00:55:00	0.0507	0.2251	12.79%	0.00045	0.0213	11.81%	0.0012	0.0350	6.17%
23	00:57:30	0.0537	0.2317	13.17%	0.00048	0.0219	12.12%	0.0013	0.0359	6.34%
24	01:00:00	0.0569	0.2384	13.55%	0.00050	0.0224	12.42%	0.0014	0.0368	6.49%

The variographic analysis of the three analyzed streams (feed grade, flotation tailings and leach tailing) indicates the error associated to the current sampling interval of the automatic samplers, programmed for 5 minutes:

- Feed grade: 9.52%
- Tailings (flotation): 8.52%
- Tailings (Leaching): 4.74%

It is important to note that, with the exception of the variogram of the flotation tailings, the others presented a "pure nugget" effect. In these cases it is difficult to evaluate the optimal sampling interval. For the flotation tailings, the curve reaches the sill at $j = 1$, therefore, the optimum sampling interval can be considered as 2.5 minutes. Given the relatively low error of the CIL tailings, the sampling interval can be maintained as 5 minutes.

3.2. Heterogeneity Test

Figure 3 shows the correlation between the constant factor of constitutional heterogeneity, IH_L, and the fragment nominal size, constructed as a result of the heterogeneity test and the calibration of the sampling constants, where $K = 311.82$ and $\alpha = 2.4841$.

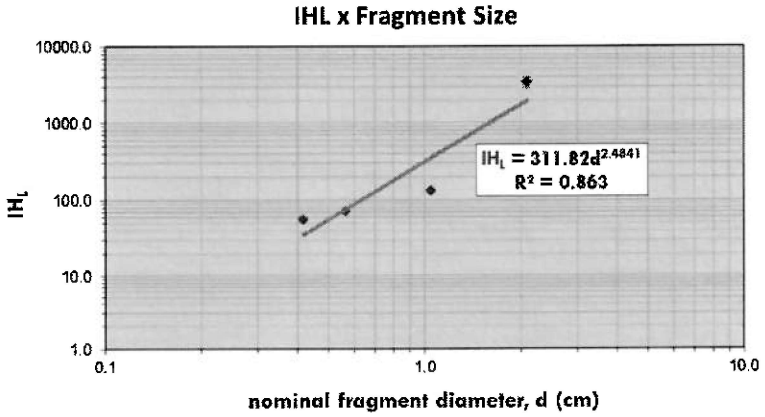


Figure 3: Correlation between IH_L and the fragment size.

According to Equation 5 for a large lot mass M_L, the minimum feed grade sample mass is calculated as follows:

$$M_S = \frac{311{,}82 \times d_{95}^{2{,}4841}}{0{,}16^2} = 33{,}3\ \text{kg} \tag{5}$$

Table 2 shows the relative standard deviations and the relative variances of the fundamental sampling error for the current sample preparation protocol, highlighting in yellow the critical step of the protocol (the secondary division of the primary sample). Table 3 presents the optimized protocol ensuring a maximum relative standard

deviation of the fundamental sampling error of 16% considering the summed variances of the protocol steps. The protocol was optimized by selecting an aliquot of 600 g instead of 300 g in the secondary division stage.

Table 2: Variance of the fundamental error and deviations for the current sampling protocol.

Stage	Initial mass	Final mass	d	IHL	Rel. Var.	Std. Dev.
	(g)	(g)	(cm)	(g)	(s^2 FSE)	(s FSE)
1. Primary sampling	640000000	36000	0.30	15.67	0.000435	2.09%
2. 1st split	36000	3000	0.30	15.67	0.004788	6.92%
3. 2nd split	3000	300	0.30	15.67	0.047005	21.68%
4. Pulverization	300	300	0.0074	0.0016	0.00000	0.00%
5. Selection of analytical sample	300	30	0.0074	0.0016	0.00005	0.69%
TOTAL (s^2 FSE)					**0.052275**	**22.86%**

Table 3: Fundamental error variance for the suggested protocol.

Stage	Initial mass	Final mass	d	IHL	Rel. Var.	Std. Dev.
	(g)	(g)	(cm)	(g)	(s^2 FSE)	(s FSE)
1. Primary sampling	640000000	36000	0.30	15.67	0.00044	2.09%
2. 1st split	36000	3000	0.30	15.67	0.00479	6.92%
3. 2nd split	3000	600	0.30	15.67	0.02089	14.45%
4. Pulverization	600	600	0.0074	0.0016	0.00000	0.00%
5. Selection of analytical sample	600	30	0.0074	0.0016	0.00005	0.71%
TOTAL (s^2 FSE)					**0.026164**	**16.18%**

The following nomographs refer to the current sampling and sample preparation protocol (Figure 4) and to the optimized protocol (Figure 5).

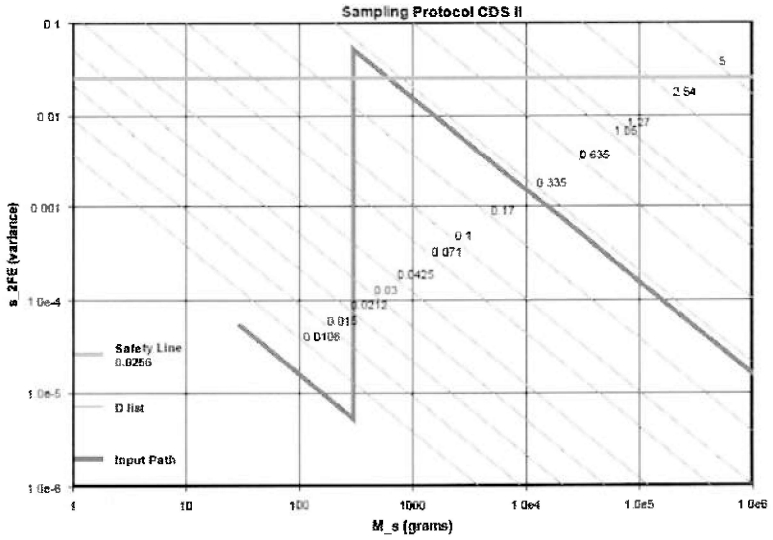


Figure 4: Nomograph representing the current sampling protocol (OSP. FPSC ®).

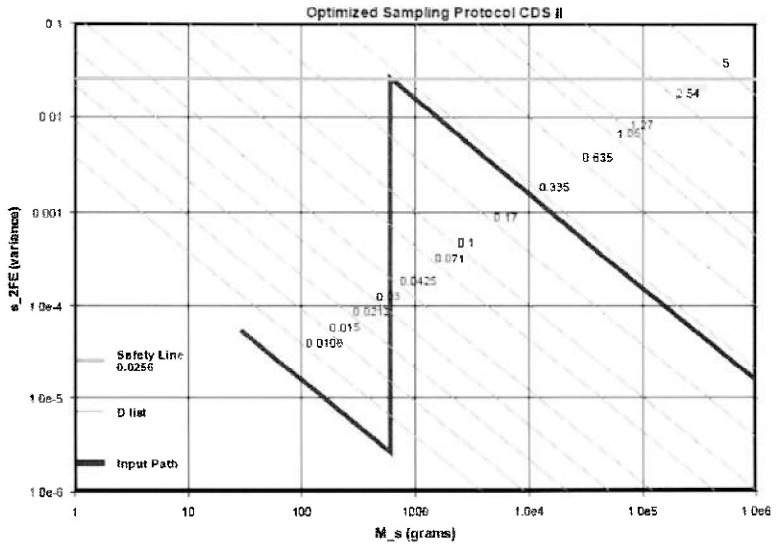


Figure 5: Nomograph representing the optimized sampling protocol (OSP, FPSC ®).

3.3. Chemical Analysis Technique

The procedure employed to study the chemical analysis techniques was the comparison of 198 samples pairs that were analyzed by fire assay and by screen fire assay. The relative difference plot, ordered by gold grade, is shown in Figure 6.

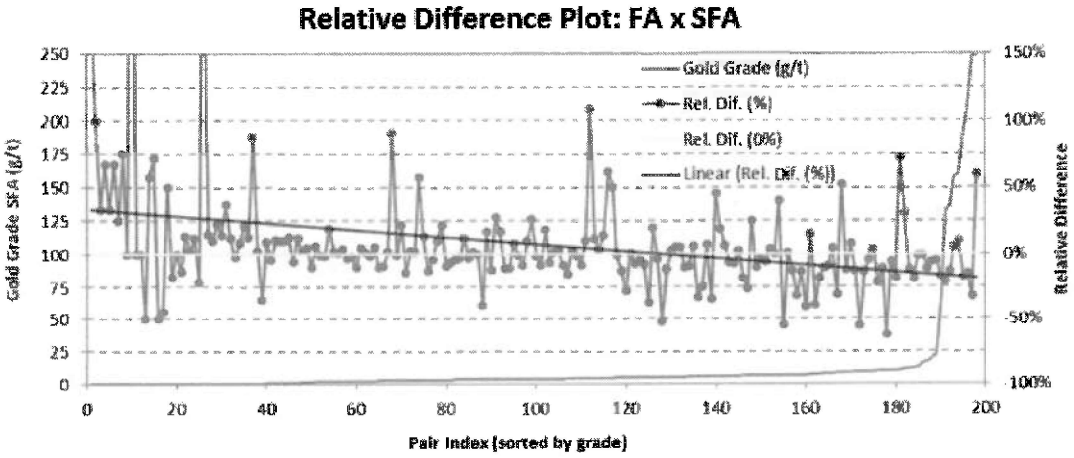


Figure 6: Relative difference plot comparing the fire assay and the screen fire assay.

The linear regression line shows that the higher the gold grade, the greater the underestimation trend of the fire assay technique; and the lower the gold grade, the greater the overestimation trend of the fire assay technique. The analysis indicates that the fire assay technique tends to overestimate grades below 5 g/t and to underestimate grades above 5 g/t. The average bias of the 198 duplicate samples was +7.68%, which means that the fire assay technique tends to overestimate gold grade by 7.68% relative, on average.

4. CONCLUSIONS

Based on the variographic analysis, the optimum sampling interval in the flotation tailings can be considered as 2.5 minutes. For the leach tailings, the sampling interval may remain as 5 minutes.

Through the sample constants calibration for the CDS sulphide ore, based on the results of the heterogeneity test, it was possible to determine a minimum feed sample mass of 33.3 kg in order to guarantee the representativeness of the plant feed samples, considering there is no systematic error at the sample collection stage. These results also showed that the division stage during sample preparation generated errors that exceeded the maximum s_{FSE} recommended by Francis Pitard and Pierre Gy for gold ores (s_{FSE} max = 16%). Thus, the selection of an aliquot of 600 g in the secondary split stage was determined to optimize the sample preparation protocol.

The results of the comparative study between gold analysis techniques showed that the fire assay tends to overestimate the gold grades below 5 g/t and to underestimate the grades above 5 g/t. The average bias of the 198 duplicate samples was +7.68%. These results highlighted the errors that can be generated by using the standard fire assay technique to determine the gold grade, allowing the recommendation of the screen fire assay technique to avoid biases and to guarantee sample representativeness.

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