

Successfully supplementing the Bauxite ISO 8685:1992 sampling standard with the more comprehensive iron ore ISO 3082:2017 standard: Inherent correlation of iron and aluminium elements for sampling West African Bauxite ores

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A contractual payment station sampling plant, built for a Guinean exporter of -100 mm bauxite ore, is designed to ISO 8685 offset-agreement requirements. However, where the dated ISO 8685 standard requires unknown input variables, is incomplete or silent, the sampling scheme designer uses the ISO 3082 sampling standard for iron ore to calculate the output parameters of number of sample increments and composite sample mass to supplement the bauxite sampling regime for chemical analysis of FeO_2 , Al_2O_3 and SiO_2 . The block flow diagram of the sampling scheme design is presented here.

Ongoing work on performance testing of the multi-stage sampling plant includes: 1) bias testing, 2) performance testing of throughput, sample size and crusher performance to design compliance and 3) chronostatistical analysis through variogram interpretation. The performance testing confirms that samples are bias-free (providing accurate samples and good data for further system performance evaluation), achieves the required standard deviation predicted by ISO 3082 and, importantly, that the correlation between FeO_2 and Al_2O_3 is consistent, to the extent that ISO 3082 can be successfully applied to supplement or replace ISO 8685 – for Guinean bauxite ore with similar correlation.

Keywords: ISO 8685, ISO 3082, variogram, quality variation, Guinean bauxite

INTRODUCTION

Aluminium is the most abundant metal in the world and international trade of its raw material, bauxite, is expected to increase due to the general use of the metal in not only general industry and alloys, but also the energy and strategic metal industries towards a greener environment. Guinea has around 80% of the global bauxite reserve.

An exporter of premium Guinean bauxite ore understood the importance of correct sampling as final quality assurance of bauxite grade between seller-and-buyer. The commitment to a world class QAQC programme can also serve as a strategic differentiator of quality product between competitors. Commercial payment terms are structured around ISO 8685 with maximum tolerable bias (MTB) specification for aluminium and silica grades. Compliance to ISO 8685 and sampling plant performance to sampling variance within the MTB levels are therefore important to control commercial trade risks. Compliance would require successful implementation of 1) sampling correctness, 2) adequate automated mechanical sampler design and 3) understanding the requirements for ISO compliance given system constants, variables and sampling parameters. However, coefficient of variation, increment variance and size range factor data are required as inputs to ISO 8685 methods to calculate the number of increments and composite sample mass – the vital parameters required for a sampling scheme design – but are not known for the greenfield

operation. Procedures explained by bauxite standards ISO 6138:1991 and 6139:1993 (experimental determination of the heterogeneity of constitution and distribution of a lot, respectively) require input data which are timely, costly and generally not available for greenfield projects. Therefore, the lacking ISO 8685 standard poses risks for defending quality parameters stipulated in commercial trade, particularly on greenfields projects where some of the required input constants and variables are not known and do not allow calculation of the sampling parameters.

Furthermore, the ISO 8685 standard is dated and compared to more recent standards, like ISO 3082 for iron ore sampling, may be considered incomplete because the latter standard has better guidelines around automated mechanical samplers' design and sampling plant ancillary considerations, sample preparation, maintenance, testing, etc. ISO 3082:2016 particularly requires less complicated input data and where greenfield operations are planned, states clear assumptions that can be made in the absence of quality variation and sampling variance data. Therefore, ISO 3082 is more comprehensive and easier to apply, particularly to greenfield projects. There may be a correlation between iron and bauxite grades for Guinean ore that can be exploited by applying the easier iron ore ISO sampling standard to greenfield projects.

LITERATURE

Holmes (2010) mentions that sampling must be given the necessary attention to produce representative samples for analyses. It should be noted that Holmes makes use of the word sampling not sampler, since a sampler is an equipment, mechanical or not, that can be used to collect a sample from a moving stream or stationary lot. The word sampling is the science of collecting representative samples which includes, but is not limited to the sampler, number of increments the sampler takes, mass of composite sample prepared, how the samples are collected, prepared, stored, transported to the lab and ultimately analysed. It is important to understand that deviation from an ideal sampling scheme is a cumulation of incremental error contributions towards the total sampling error as defined by the publicised Theory of Sampling (TOS). The degree of error accumulation and nature of the errors are ascribed to bias (accuracy deviation), precision (reproducibility) and ore variability. Therefore, the objective of any sampling system or protocol is to eliminate or minimise bias and standard deviation while measuring variance to the required precision level (Minnitt, 2007). Eliminating bias generating errors falls under the responsibility of the sampling equipment manufacturer (SEM) through unbiased equipment design and combatting segregation with the system design. Where material variation information is available (i.e. in the form of a nomogram), the SEM is also expected to design a sampling system that can sample the material variation to the required precision levels through sample increment frequency and sufficient sample mass. The quality variation information is not always available for the specific ore of concern; as a result, ISO standards are normally referred to for guidelines towards correct sampling of various bulk commodities (Steinhaus & Minnitt, 2014). According to them, SEMs need to familiarise themselves with the critical aspects of each ISO standard which relates to the minimum number of increments and the minimum sample mass to be collected per lot, the minimum number of sub-increments per preceding increment, quality variations, precision levels achieved relative to the material nominal top size, lot size, number of parts (or sub-lots) recommended per lot, etc.

The Risk of Using Unknown System Variables to Calculate Sampling Scheme Parameters into an Inflexible Plant Design

Where data is not available, assumptions towards unknown input variables are the designers' only option to allow parameter calculation as the basis for the sampling scheme design. The scheme design will dictate the equipment selection and throughput capacity for the sampling plant. Once operational, the sampling plant must produce accurate results without bias. The accurate data from the sampling campaign will allow calculation of sampling parameters and the

sampling scheme design must be verified for ISO compliance using the new quality variation and performance data (increment sampling-, preparation- and analysis variance). The results may render the original sampling scheme non-compliant and larger sample weight and/or more sample increments may be required. Where more increments are required over a constant lot size, the sampling throughput requirement will increase, affecting equipment and system selection. It may also happen that production or in-sampling-plant crushers do not perform to targeted size reduction and increased particle sizes may have to be sampled and/or prepared at larger incremental and composite sample mass. It is therefore important that the sampling equipment, crushers and sampling plant throughput design capability be conservative on the first iteration and adaptable to accommodate the new throughput rates, sample weight and actual particle top size. Adaptability should come without major modifications and capital cost. Designing green field sampling plants to meet (not exceed) minimum compliance requirements is a great risk if reliable input variables are not available. Where major plant upgrades must be done to a new sampling plant, following commissioning and sampling scheme parameters verification, capital is not likely to be approved, resulting in undersized and underperforming (obsolete) sampling plants which the industry should not tolerate.

With input variables unknown, the sampling parameter calculation must be done from experience, reasonable assumption or using ISO standard recommended values - if an ISO standard is available for the mineral in question.

Calculating Sampling Design Parameters from Known and Unknown Constants & Variables

All sampling system designs require known and unknown inputs (constants, variables, parameters) to allow a compliant sampling scheme design. The two most important sampling scheme design outcomes are the number of sample increments required and the mass of sample needed to obtain the targeted precision level for the lot size, particle nominal top size and material variation. The reverse-progression diagram for calculating the sampling parameters is shown in Figure 1.

Some of the input constants and variables are readily known because they are essential as (non-sampling scheme design) production plant and equipment design inputs. The likely unknown variables are the sampling scheme design requirement of quality variation and sampling variance (also preparation and measurement variances are unknown for systems and laboratories that are not built yet). Even though various methods exist to obtain the variables, they are essentially unique to each sampling scheme design and require timeous and costly practical experimentation which is likely not considered on greenfield projects to the point where the already lagging-priority sampling plant is then on the project critical path (Slabbert, 2022).

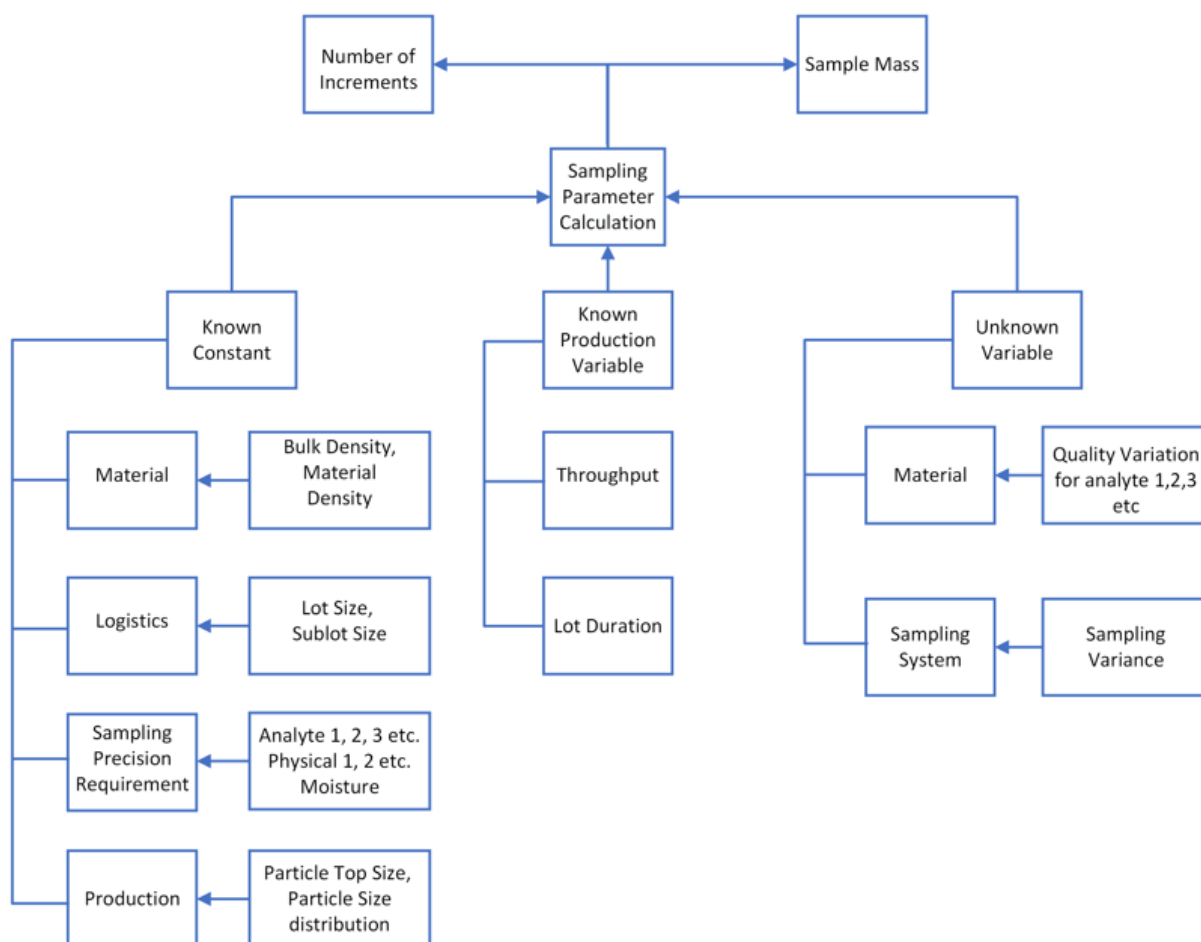


Figure 1. Reverse progression of calculating the two sampling scheme parameters from categorised, known and unknown constants and variables.

SAMPLING SCHEME DESIGN: ASSUMPTIONS, CALCULATIONS AND OUTCOMES

Calculating the Sampling Scheme Parameters - Comparing ISO 8685 to ISO 3082 Requirements and Outcomes

The number of sample increments and minimum sample mass requirements ~~is~~ are calculated from the respective ISO standards, using the equations compared in Table I and II, respectively.

Assumptions, risks and conclusions from the respective number of increments calculations

The constant sampling variance (or precision) requirement should be known from commercial trade, industry standards and user (client) provided. For the system under discussion, the more stringent requirement of 0.2% (absolute) standard deviation on SiO_2 is used and not the 0.5% on Al_2O_3 .

The unknown increment variance (or quality variation) requirement is more difficult to determine for an unbuilt sampling system with unknown procedures, but ISO 3082 provides some solid guidelines for values and can be used as conservative assumptions with low risk. ISO 8685 lists some example numbers for sampling variance but does not provide firm guidelines on what value to use. An alternative variogram method is proposed, but again requires pre-existing sampling data before the sampling scheme is designed – let alone the sampling system built and operational to produce results. For this project, an existing neighbouring operation's increment variance is known and under reasonable assumption is used. The assumption is a project risk and

would later have to be confirmed (which is part of this paper).

The number of primary sample increments calculated from the respective standards are the same.

Table I. Comparison of ISO standards number of primary increment equations, symbols, values and results

ISO 8685		ISO 3082	
$n = \frac{V_I}{\sigma_S^2}$	Number of primary increments	$n_1 = \left(\frac{2\sigma_W}{\beta_S}\right)^2$	Number of primary increments
where			
$V_I = 1.75$	Increment Variance	$\sigma_W = \sqrt{1.75} = 1.323$	Quality variation
$\sigma_S^2 = 0.04 \%$	Desired Sampling Variance	$B_S = 0.4 \%$ (Absolute)	Desired sampling precision
Calculating			
$n = 43.75$ primary sample increments		$n_1 = 43.75$ primary sample increments	

Assumptions, risks and conclusions from the respective composite sample mass calculations

The tabulated comparison evidently concludes that the ISO 3082 calculation is simpler (mathematically) but more importantly – provides the known constant (standard deviation of division) clearly and leaves no project risk.

The ISO 8685 standard requires various known constants (relative sampling error, particle density), but also unknown variables. The known variables are often known for production related purposes and particle nominal top size of the production ore is a known variable. However, the particle size distribution variables are not known and had to be determined experimentally for this project (costly and time consuming on a project that is already on the critical path). Most importantly, the unknown variable (coefficient of variation) is too costly to determine and left the sampling scheme designer exposed with an assumption from a neighbouring operation.

Overall conclusion on sampling parameter calculation

The number of sample increments from both standards are the same. The composite sample masses are comparable. The iron ore standard is user-friendly, easier to implement and removes project risk by clearly specifying reasonable assumptions for unknown constants or variables. The iron ore standard is also more conservative in composite sample mass at lower particle nominal top size and will allow for better precision levels of -75 mm ore. It may therefore be possible to use the ISO 3082 to supplement or replace the ISO 8685 as a more user-friendly standard.

The Resulting Sampling Scheme Design

The minimum gross sample mass and number of primary increments calculates that the minimum sample increment mass must be: $m_G/n = 3769/44 = 85.7$ kg. The sample increment frequency is calculated as the lot duration (four hours) divided by the number of primary increments required. For this system design, 44 increments must be taken in four hours' time which requires an increment every five minutes and 27 seconds.

For chemical sample compliance, the sampling plant must take a primary cut every five minutes and 27 seconds to total 44 increments over a four-hour lot period. Each increment must have a

minimum mass of 85.7 kg. The total composite sample mass over the lot must be 3769 kg at 100 mm particle size, 59 kg at 25 mm and 0.81 kg at 6 mm nominal top size.

Table II. Comparison of ISO standards composite sample mass equations, symbols, values and results. Please note that units are as set out in the respective ISO standards unless otherwise listed

ISO 8685		ISO 3082	
$m_G = \left(\frac{C_V}{\sigma_S}\right)^2 \rho g D^3 \times 10^{-6}$	Minimum gross sample mass	$m_s = \frac{0.00032d^{2.5}}{\sigma_D^2}$	Minimum mass of divided sample
Where			
$C_V = 15$	Coeff. of variation between particles of the quality characteristic under investigation.		
$\delta_S = 0.2$	Required relative sampling error	$\sigma_D = 0.1$	Desired standard deviation of sample division in %Fe - the major component of the STD of sample prep
$\rho = 2.68$	Particle density (not bulk density) (ton/m ³)		
D = 100, 25, 6	Nominal top size of the ore in the lot (mm)	d = 100, 25, 6	Nominal top size of the sample (mm)
g	Size range factor.		
Where the size range factor g is read off Table 2 of ISO 8685, after calculating:			
If $D/D' > 4$	g=0.25		
If $4 \geq D/D' \geq 2$	g=0.50		
If $D/D' < 2$	g=0.75		
If $D/D'=1$	g=1.00		
Where			
D = 100, 25 & 6 mm	Nominal top size of the ore		
D' = 1-3 mm*	Sieve size retaining 95% of the ore. *D'=3 mm for D=100, 25; D' = 1 mm for D=6.		
Calculating composite sample mass required at 100, 25 and 6 mm nominal top size respectively			
m_{G_100}	3768.75	m_{S_100}	3200
m_{G_25}	59.89	m_{S_25}	100
m_{G_6}	0.81	m_{S_6}	2.82

However, there is a requirement for a process control physical sample to monitor the particle top size from the mining operation. To extract this physical indicative sample, a secondary hammer (cross-belt) sampler is designed onto the sampling plant primary feeder conveyor. This sample is not required to be ISO 8685 complaint but the removal of material from the chemical composite sample stream affects the final sample weight rendering the plant non-compliant. The physical sampler would extract a single physical sample increment, per alternating primary sample increment. The mass of each physical sample increment is calculated to be 8 kg and 21 kg at minimum and maximum production loading, respectively. Over the lot period consisting of 44 primary sample increments, it would mean that 462 kg of the total primary sample increment would be removed from the system and not reported to the ISO 8685 complaint chemical leg of the sampling plant. To account for the removal of material three additional primary sampler increments (now totaling 47 increments) would be extracted to substitute (257 kg per increment

times 3) 771 kg of primary increment through the system at 10 kton/h throughput rates. The same proportionality also applies at the minimum throughput rates of 3.75 kton/h. To allow the three additional sample increments over the lot period of four hours, the increment time had to improve from five minutes and 27 seconds to the final design five minutes and 11 seconds. Previous increment minimum- and composite sample masses remain unchanged. The resultant sampling scheme design is depicted in Figure 2.

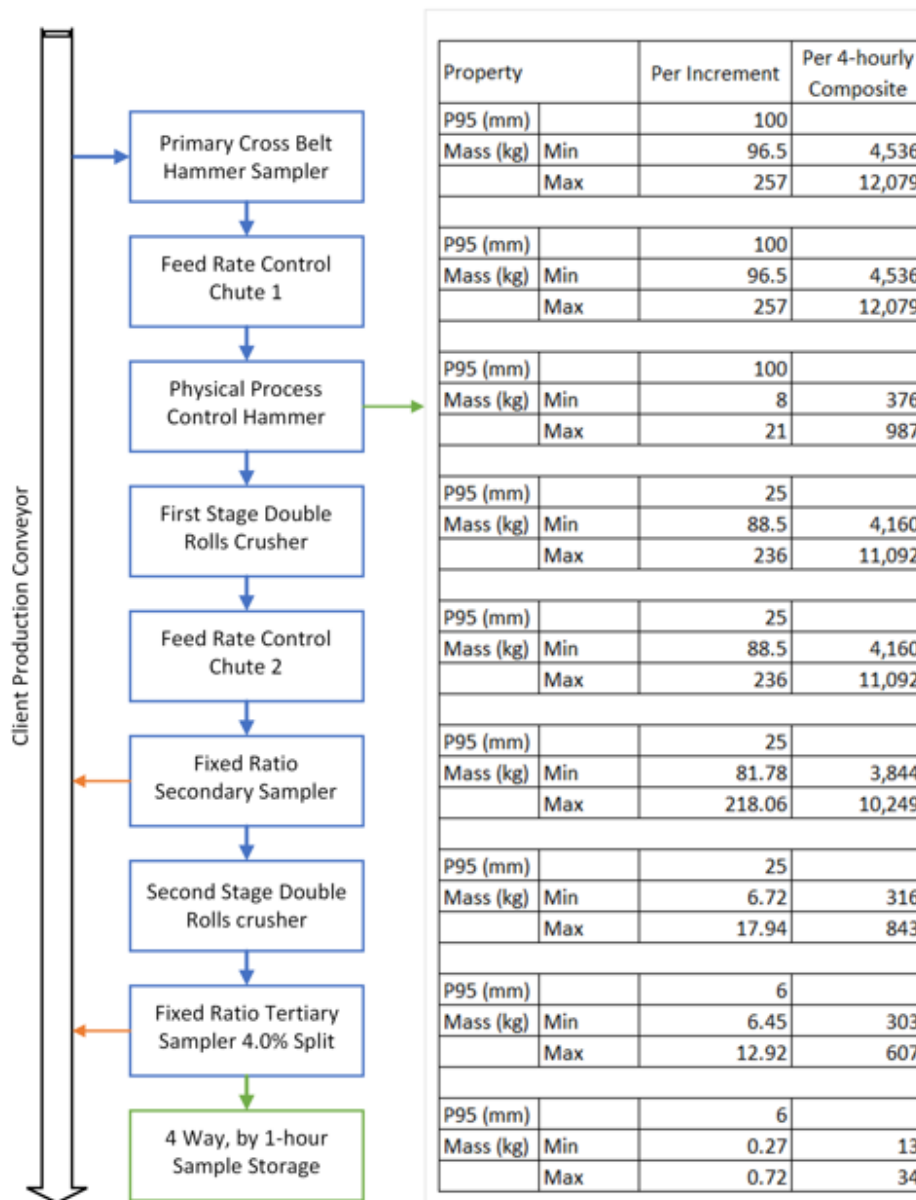


Figure 2. Block flow diagram of physical and chemical ISO 8685 compliant sampling scheme design.

SAMPLING SCHEME PERFORMANCE RESULTS AND VERIFICATION OF ASSUMPTIONS

Bias-free Results

The sampling plant, sample preparation and analytical procedures were bias tested six months after commissioning, by an independent, experienced third party. Two reference samples were

extracted from the stopped production conveyor as per standard industry practice and in compliance of ISO 10226:1991 aluminium ores – experimental methods for checking the bias of sampling. The composite chemical sample results ('Method B') are compared to the average of the two profile plate reference belt cuts ('Method A') over 60 sample sets (one set comprising the composite system sample and reference samples 1 and 2). The entire sampling plant (as depicted in Figure 2), including all process steps and equipment performance was tested. The bias test results from the 2021 campaign are reported by Slabbert (2022): "...A bias from a statistical perspective is observed where the average and standard deviation (position and span of the yellow line) does not intercept zero. However, from a practical point of view the data renders the bias irrelevant since the confidence interval falls within the MTB limits – listed as absolute percentages."

The bias test was performed again in 2022 with outcomes in Figure 3. The bias test concludes that no statistical or practical bias exists, and the samples generated by the sampling plant, preparation steps and analysis is accurate. Therefore, the data used to calculate and report results below are an indication of true increment variance and quality variation without accuracy concerns.

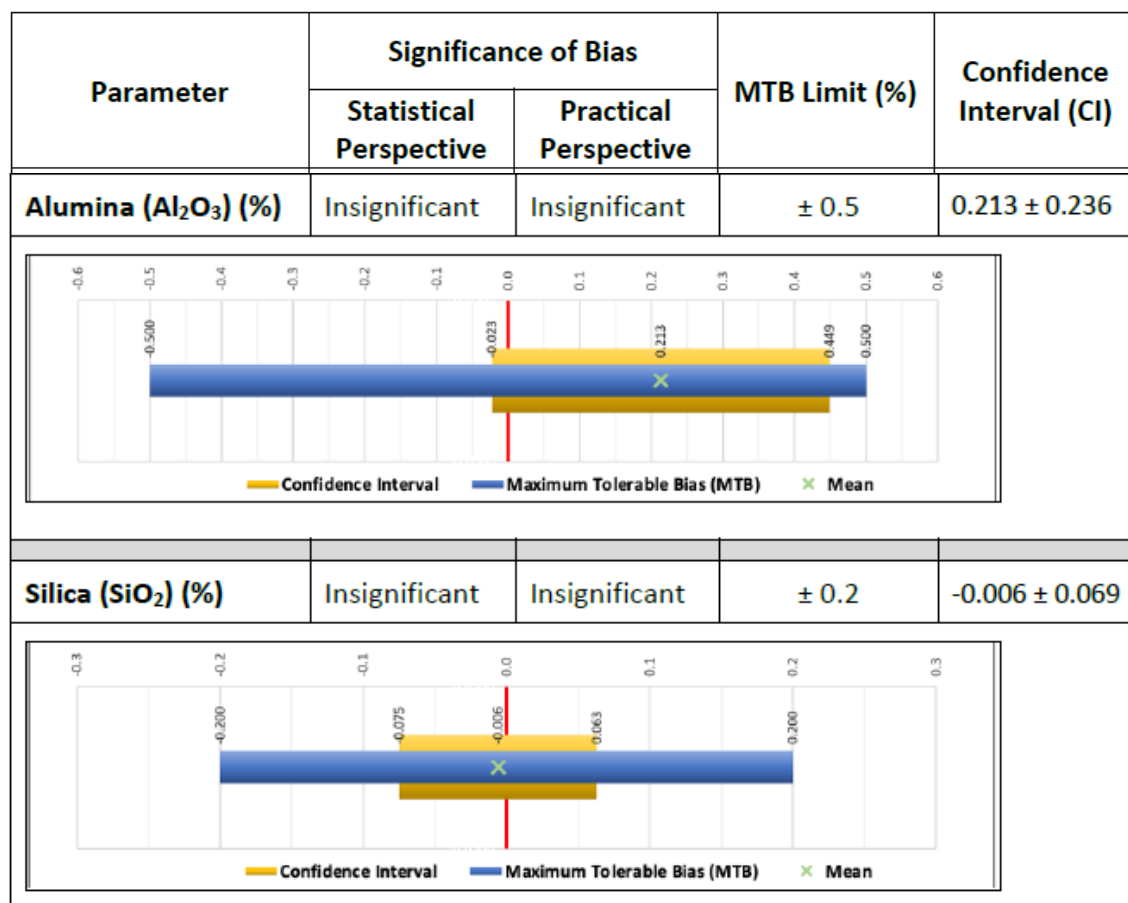


Figure 3. Bias test (2022) results of the sampling scheme showing client specified MTB with the blue line; bias test results with the yellow line.

ISO 6138: Calculation of Coefficient of Variation versus Conventional Statistics

ISO 6138 requires grade by size analysis for each data point which, even after performance testing of the sampling plant and despite being in operation for some years, is not readily available and the workload falls outside of normal analysis routines/contracts and is therefore resource-taxing and expensive. The impracticality of ISO 6138 must be circumvented with coefficient of variation calculation with conventional statistics by calculating the standard deviation of the system-

produced sample data set, and then dividing by the mean of the data set (values remain anonymous for confidentiality reasons).

The result is a C_V of 0.030 for the 2022 data set (the better of the two acceptable sets of results). The value is much lower than the assumed C_V of 15 used in the design phase. The difference between the generated and assumed value is likely due to the additional blending operation at the project compared to the neighbouring operation which has a straight loading facility without the blending quality assurance step. Also the variation over the bias test period (limited to three days) is low compared to expected variation over a continuous production period. The coefficient of variation is well below the design value and sampling exceeds the minimum ISO 8685 requirement during the bias test period.

Given that ISO 8685 then uses C_V to calculate composite sample mass, and that ISO 3082 does not need C_V to calculate composite sample mass, it is concluded that ISO 8685 and ISO 6138 are cumbersome, and ISO 3082 is more user-friendly and recommended.

ISO 6139: Calculation of Increment Variance

ISO 6139 allows the experimental determination of the heterogeneity of distribution of a lot of aluminium ore. From a set of analytical data, the variance of individual analysis can be calculated using:

$$V_e = \frac{\sum_{i=1}^n (x_i - \bar{x})^2}{n-1} \quad [1]$$

Where:

V_e is the variance of individual analysis

n is the number of individually analysed increments

x_i is the analytical value of the increment

$\bar{x}$ is the geometric mean of the data set of x_i

The data set of 58 individually analysed, system-produced increments from the 2022 bias test is used to calculate the variance of individual analysis. The analytical value of percentage alumina is used to calculate the variance that equals 1.835. It is also known that:

$$V_I = V_e - V_{PM} \quad [2]$$

Where:

V_I is the increment variance

V_e is the variance of individual analysis

V_{PM} is the preparation and measurement variance.

However, the ISO 8685 and ISO 6139 standards do not list values for preparation and measurement variance. The standards are again supplemented with ISO 3082, Table 5, where, for a <15 kton lot size of -6.3 mm ore and a sample mass of 145 kg, $B_{PM} = 0.66$. Then,

$$\sigma_{PM} = \frac{\beta_{PM}}{2} \quad [3]$$

Where:

σ_{PM} is the preparation and measurement variance.

$\beta_{PM} = V_{PM}$ is the precision of preparation and measurement.

Which allows calculation of V_I equal to 1.727. The design assumption of V_I equal to 1.75 is therefore verified and slightly improved upon in practice. The increment variance is calculated only over the bias test regime and does not yet accurately represent the variance over the continuous operation.

Chrono-Statistical Verification

The above calculations are done only on the data sets for the duration of the bias test which may mean that coefficient of variation and increment variance are both underreported compared to prolonged operation across various grades and performance of the sampling system.

Two sets of chrono-statistical results are presented to 1) compare the chrono-statistical calculation of the parameters to the methods described above for the bias test duration data and 2) calculate the parameters using only chrono-statistical methods over a longer production period using extended production data.

Calculation of increment variance and coefficient of variation for bias test data set

An absolute variogram (Figure 4) of aluminium content from the bias test results are drawn for system-produced samples. The lag period is therefore consecutive bias test results and in chronological order.

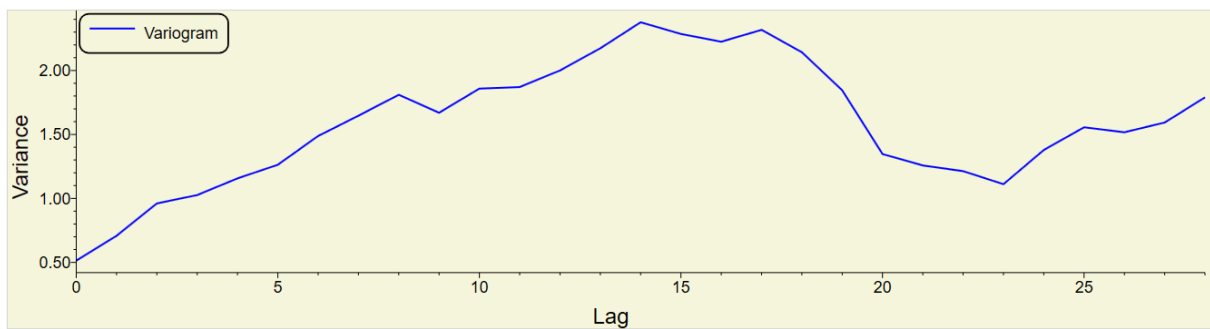


Figure 4. Absolute variogram of alumina content (%) for the reference samples over the bias test. (Pitard, 2023)

The coefficient of variation is calculated as the standard deviation divided by the mean (values to remain anonymous for confidentiality reasons) and calculates a C_V of 0.030 (the same as the number calculated using classical statistics method).

The increment variance is determined from the variogram as 1.71 and compares well to the ISO 6139 method that calculated 1.727.

It is concluded that both C_V and V_I calculated with variogram methods compare well to conventional statistics and ISO 6139 methods, respectively. The variogram calculation steps can therefore be used to calculate C_V and V_I over longer production periods.

Calculation of increment variance and coefficient of variation for a three-month production period

An absolute variogram of aluminium content from the bias test results are drawn (Figure 5, top graph) for system-produced samples. The lag period is 1-hourly analysis of the system produced samples in chronological order.

The coefficient of variation is calculated as the standard deviation divided by the mean and is 0.019! Over the larger data set the C_V is lower than over the bias test period. Re-evaluation of Figure 4 does however indicate a drifting variance and the actual (confidential) data does show a change in analytical data values around halfway through the bias test that can account for the higher variance of the shorter bias test period.

The increment variance (V_I) is determined from the variogram as 0.80 and is again lower than the bias test value and below the design assumption of 1.75.

It is concluded that the sampling plant exceeds minimum ISO 8685 and ISO 3082 requirements on sample mass and number of increments, from fair assumptions made for C_V and V_I , at the design stage, respectively.

Correlation of Alumina and Iron for the Ore

Given that actual analytical values are confidential (and actual data, regression analysis or other means of reporting cannot be shared), variogram comparison (Figure 5) of alumina and iron indicates the very precise correlation between the two elements for this ore (and general Guinean bauxite).

The variograms (and not reported: analytical data, trends and correlation) of the two elements are so close that the author had to double check if the same data set was not accidentally used to calculate both variograms. But indeed, at close inspection, subtle differences between the variograms can be seen and it can be concluded that the correlation of the element grades in the ore is uncanny.

Guinean bauxite has a very close correlation between the elements and justifies that ISO 3082 for iron ore be used to substitute or even replace ISO 8685.

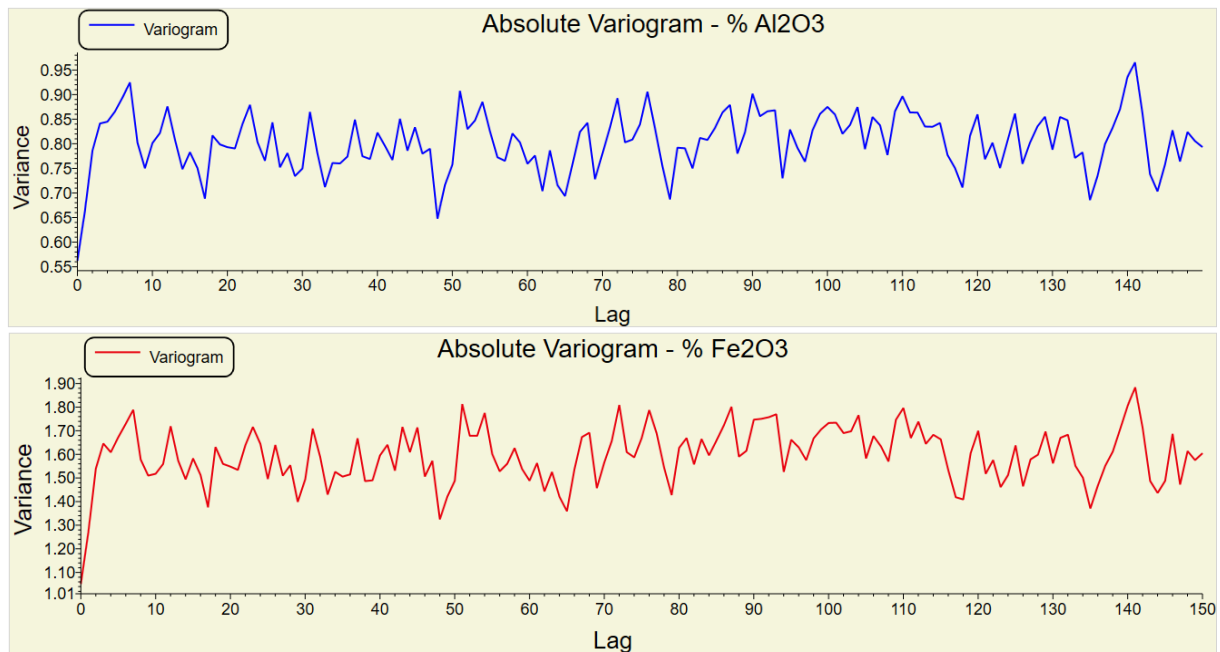


Figure 5. Comparison of absolute variograms for elemental content of alumina % (top) and iron % (bottom) over a three-month production period. (Pitard, 2023)

CONCLUSIONS AND RECOMMENDATIONS

Calculating sampling scheme parameters: coefficient of quality variation and increment variance, from unknown constants and variables pose a risk for the sampling designer which can result in risk for the user to the extent of obsolete sampling systems that cannot process the required number of increments to yield the required composite sample masses.

ISO 3082 is more user-friendly, complete and requires less, and less complicated inputs to significantly reduce the risk of sampling parameters calculation over ISO 8685. Guinean bauxite ore has a strong correlation between iron and alumina content. Comparative sampling parameter calculations - using the respective ISO standards - result in the same number of increments and sample masses to be collected and conclude the same sampling scheme design.

The sampling plant is bias tested for accuracy and performance tested for operational compliance

to the ISO design schemes and confirmed to pass both performance parameters. The results produced by the sampling plant are then used to verify that the design was correct in its assumptions for unknown ISO 8685 input requirements. The outcomes confirm that the coefficient of quality variation and increment variance calculated with three methods - 1) ISO 8685 or derivatives, 2) ISO 3082 and 3) results obtained during variogram preparation - conclude the same sampling parameter results.

It is proposed that ISO 3082 may be used to supplement/replace ISO 8685 as a sufficient and more user-friendly sampling standard, where iron- and alumina content are correlated, especially for greenfield projects where sampling related input variables are unknown.

It is recommended to repeat this study for other global ore bodies with or without correlation between iron and alumina so that the application constraints of the proposal can be better understood before recommendation to industry.

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