

# Good sampling is good business: reconciling economic drivers of productivity and quality using fundamentals of sampling theory

O. Dominguez<sup>a</sup> and K. Smith<sup>b</sup>

<sup>a</sup>[Oscar.R.Dominguez@bhpbilliton.com](mailto:Oscar.R.Dominguez@bhpbilliton.com)

<sup>b</sup>[Kathleen.Smith@bhpbilliton.com](mailto:Kathleen.Smith@bhpbilliton.com)

Iron Ore supply is outpacing global demand, reinforcing the importance of product quality and reliability as critical factors that distinguish Iron Ore producers in a competitive market. This expectation calls for a shift in industry attitudes toward sampling in bulk commodities, beginning with a greater emphasis on optimisation of sampling processes from Exploration to Port. Business initiatives aimed at optimising processes often call upon technological innovation, such as mobile sampling and analysis modules at the drill rig. Such technologies indeed represent an exciting frontier in the business of minerals exploration; however their merits must be critically compared to existing sampling protocols before implementation if sample quality is to be maintained. Quantifying the Fundamental Sampling Error (FSE) of the sampling protocol is a minimum requirement to achieve this and should be preceded by experimental calibration of the sampling constant  $K$  and the exponent  $\alpha$ . Here, we present a case study in which the Segregation Free Analysis (SFA) calibration methodology proposed by Minnitt et al.<sup>3</sup> was used to determine  $K$  and  $\alpha$  for a Channel Iron Deposit (CID) and a Brockman Iron Formation-hosted Bedded Iron Deposit (BID) from the Pilbara region of Western Australia. Following three experimental calibrations of  $K$  and  $\alpha$ , liberation size was calculated for iron oxides and deleterious minerals using Gy's formula. Validation of liberation size is critical if the resulting FSE calculation is to inform business decisions. Electron beam instruments such as QEMSCAN have been proposed as a relatively quick and low cost way to estimate liberation size<sup>2</sup>. An "off-the-shelf" QEMSCAN analysis was trialled as a validation method against the SFA calibration results. Good agreement was achieved between liberation sizes determined by the SFA calibration method and the QEMSCAN analysis. Furthermore, the QEMSCAN results proved to be a beneficial source of supplementary information, in the form of particle size analysis, which indicates the degree of aggregation that persisted in the calibration material despite best efforts to eliminate it, as well as mineral abundance analyses, which either confirmed or highlighted uncertainty around critical mineralogical assumptions made in the calibrations. These observations emphasise the importance of validation when assessing FSE. The case study presents an industry perspective on the applications of sampling theory in response to an increasingly competitive Iron Ore market.

## Introduction: economic drivers of technology, productivity and product quality

Commodity prices have fallen in recent times, leaving mining companies with an imperative to cut costs, improve productivity, innovate technological solutions, all whilst improving the quality of their product. Iron Ore in particular is an increasingly competitive market as global supply outpaces demand; thus product quality and reliability are becoming increasingly relevant factors that distinguish iron ore producers in the marketplace. Such economic conditions indeed necessitate challenging the status quo and looking to optimisation of processes aimed at lowering cost and raising productivity; however this must be done with great care where technical considerations such as sampling protocols are involved. Furthermore, adopting new technologies designed to streamline processes – such as mobile sample preparation and analysis equipment at the drill rig – must be carefully measured against traditional sampling and analytical methods; otherwise mining companies may find initiatives geared towards productivity in direct conflict with initiatives geared towards improving product quality.

To critically compare a conventional sampling protocol against a novel, technologically innovative protocol, one must first determine some measure of error which can be critically compared. Gy's Theory of Sampling (1979)<sup>1</sup> suggests that the single most influential

error is the Fundamental Sampling Error (FSE) of the sampling design, and application of Gy's formula to calculate FSE must be preceded by experimental calibration of the sampling constants  $K$  and  $\alpha$  for the particular ore type (Minnitt et al., 2011)<sup>3</sup>. Here we present a case study in which the BHP Billiton Iron Ore Exploration group, in collaboration with Dominique Francois-Bongarcon of Agoratek International, conducted three experimental calibrations of  $K$  and  $\alpha$  in order to quantify the FSE of our sampling protocol. The determinations of  $K$  and  $\alpha$  were ultimately used to calculate the liberation size of deleterious materials present in iron ores, chiefly alumina, silica, and phosphorous. For proprietary reasons, iron results will not be published.

As an influential variable in the FSE calculation, it was critical to validate the liberation sizes generated by the calibration experiments. QEMSCAN technology has been suggested as a novel approach to heterogeneity in the past (Lyman, 2011)<sup>2</sup> and was trialled as a validation technique using an "off-the-shelf" suite of analyses provided by Bureau Veritas Australia. The use of QEMSCAN proved to be a beneficial source of supplementary information, including particle size distribution analyses, which appear to confirm that aggregation persisted in the material used in the calibration experiments despite best efforts to eliminate it. Furthermore, the QEMSCAN work confirmed some mineralogical assumptions, which influences the FSE calculation as the density input associated with the sampling

constant K. In light of achieving good agreement between experimental and QEMSCAN liberation sizes, the QEMSCAN analysis is considered to be worthwhile.

### Experimental calibration of k and alpha

Three calibration experiments were conducted according to the Segregation Free Analysis methodology proposed by Minnitt et al.<sup>3</sup>. These experiments will be referred to as Experiment 1, Experiment 2 and Experiment 3. Each experiment utilised high-grade iron ore sourced from BHP Billiton exploration projects or active mines. Two calibrations were conducted for Brockman hosted bedded iron formation; the first sourced bulk sampled material from Reverse Circulation (RC) drilling and the second sourced material from coarse-crushed diamond core, previously used for metallurgical test work. The third experiment assessed blasted Channel Iron Deposit (CID) material sourced directly from an active pit at the BHP Billiton Yandi mine. Details of sample collection and preparation for each experiment are presented below. All samples were assayed by XRF at the SGS Newburn Laboratory in Perth.

#### Experiment 1: RC sourced Brockman ore

The first calibration experiment utilised approximately 100 kg of high-grade Brockman ore generated by Reverse Circulation (RC) drilling from a BHP Billiton exploration project in the Pilbara. RC sourced bulk material was chosen for the initial trial for its easy availability and low cost. The bulk material was de-aggregated using a steel roller and split into four lots using a rotary splitter for easier handling. Approximately one half of the original 200 kg lot was run through a nest of sieves in geometric progression ( $r = 2$ ); however, due to the destructive nature of the RC drilling method, the maximum grain size was smaller than anticipated with a  $d_{max}$  of 0.95 cm. This constrained the first calibration experiment to four size fractions and a pulp series, which is used to approximate analytical variance (Minnitt et al., 2011)<sup>3</sup>. A total of 30 samples were collected from each fraction by spooning approximately 100 g of sieved material into a sample bag. Each sample weighed approximately 100 g.

#### Experiment 2: Diamond sourced Brockman Ore

The second calibration experiment utilised approximately 100 kg of high-grade Brockman ore sourced from coarse-crushed diamond core from a BHP Billiton exploration project in the Pilbara. Partially mineralised shale material contained within the lot was highly

subject to aggregation. Following de-aggregation using a steel roller, the diamond sourced material yielded a  $d_{max}$  of 1.9 cm. The material was then rotary split for easier handling, and run through the nest of sieves as per experiment 1, resulting in five size fractions and a pulp series. Between 16 and 32 samples were collected from each size fraction as per experiment 1, depending on the volume of material generated in that fraction during sieving. Each sample was approximately 120 g.

#### Experiment 3: Mine sourced CID

The third calibration experiment utilised approximately 100 kg of high-grade CID ore sourced from an active pit of the BHP Billiton Yandi mine. Aggregation was not apparent upon visual inspection; however the material was subject to de-aggregation with the steel roller as a precaution, followed by rotary splitting and sieving as per experiments 1 and 2. The mine material yielded a  $d_{max}$  of 3.8 cm, resulting in six size fractions and a pulp series. A total of 32 samples were collected from each size fraction as per experiments 1 and 2 at approximately 130 g per sample.

### Data processing

Data processing was conducted according to the procedure outlined by Minnitt et al.<sup>3</sup>; thus the data reduction process by which a single-stage variance is calculated for each series will not be discussed in detail here. Perhaps of greater interest to the calibration is the removal of outliers. Outliers were removed according to the "Outlier Modes" method discussed in Minnitt et al.<sup>3</sup>, under the supervision of Agoratek International. Using this approach, a total of 46 outliers were removed from a data-set of 584 assays; over half of these were observed in Experiment 2, perhaps due to the substantial aggregation observed in this material. Following outlier removal, calibration curves with slope alpha and intercept K were compiled according to the procedure outlined by Minnitt et al.<sup>3</sup> (Figure 1).

Liberation size was then calculated according to the formula (from Minnitt et al.<sup>3</sup>, p. 144):

$$d_i = \left( \frac{K}{c.f.g^i} \right)^{\frac{1}{3-\alpha}}$$

Where K is given by the intercept of the calibration curve, composition factor c is given by the density (as grade has been normalised

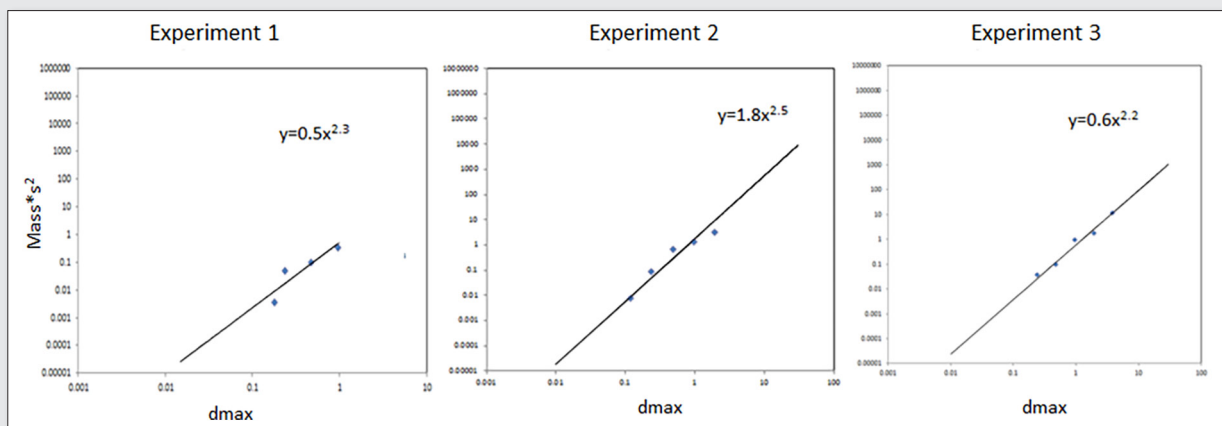


Figure 1.  $Al_2O_3$  calibration curves for each SFA experiment

**Table 1.** Experimental alpha and liberation size (dL) for key analytes as determined by the SFA calibration experiments

SFA Experiment	Analyte	alpha	dL SFA ( $\mu\text{m}$ )
Experiment 1	$\text{Al}_2\text{O}_3$	2.3	8
	$\text{SiO}_2$	2.3	17
	P	2	2
Experiment 2	$\text{Al}_2\text{O}_3$	2.5	14
	$\text{SiO}_2$	2.4	21
	P	1.8	16
Experiment 3	$\text{Al}_2\text{O}_3$	1.8	25
	$\text{SiO}_2$	2	2
	P	1.8	3

to 1 according to the data processing procedure), shape factor  $f$  is assumed to be 0.5 and granulometric factor  $g'$  is equal to 0.44 according to the relationship of  $g'$  and the ratio of sieve sizes  $d_{\text{max}}/d_{\text{min}}$  (Minnitt et al.<sup>3</sup>, p. 139). Experimental alphas and calculated liberation sizes are given in Table 1.

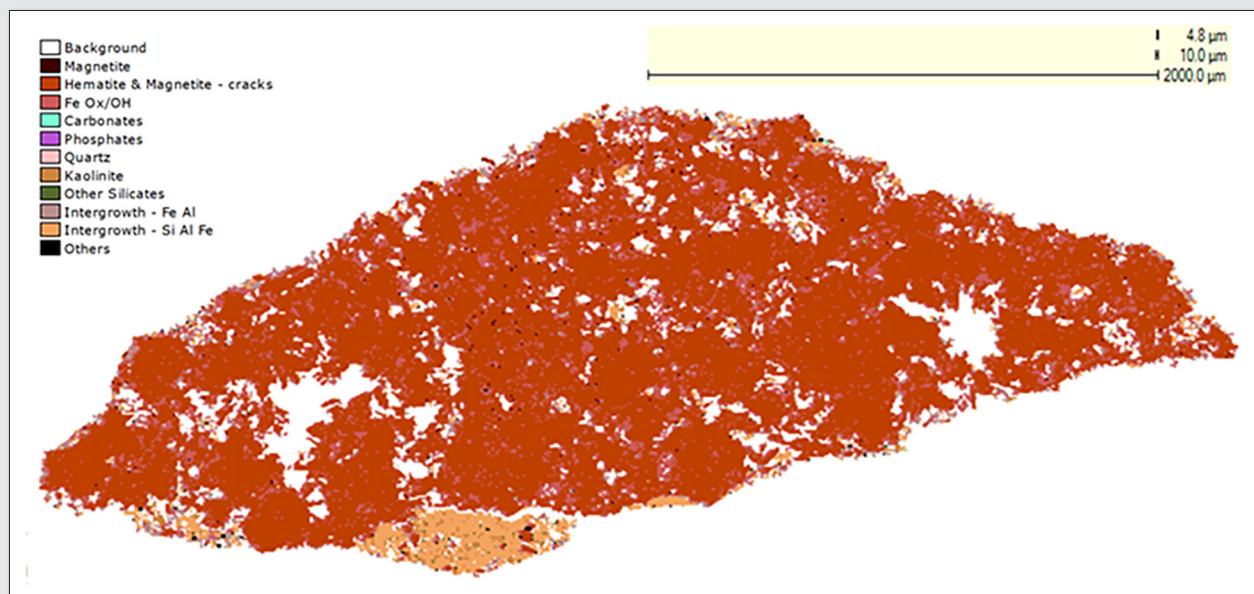
### Results validated by QEMSCAN

QEMSCAN analysis was conducted primarily as means of validating liberation size, but also as a supplementary source of information regarding mineral abundance, particle size distribution and mineralogical associations related to liberation and locking. The QEMSCAN package also incorporated QXRD work as an additional validation method. Three samples from the calibration experiments were selected for QEMSCAN analysis, each representing a different size fraction. Two were selected from Brockman ores used in Experiment 2 and one was selected from the CID ore used in Experiment 3.

Fundamental to the work was validating mineralogical assumptions. The initial assumption considered alumina as kaolinite, which was confirmed to be the norm in the Brockman ores (Figure 2). However the QEMSCAN analysis, in addition to QXRD, suggests that no kaolinite is present in the CID material used in Experiment 3, but rather is entirely hosted by Goethite as intergrowths (Figure 3). Likewise, all silica was initially assumed to be quartz, however silica was found to be hosted primarily by kaolinite, and to a lesser degree in goethite, in the Brockman ores. While the QEMSCAN package was effective in definitively confirming some assumptions, it raised uncertainty around others; such was the case with Phosphorous hosting minerals. First, the presence of Xenotime ( $\text{YPO}_4$ ) as a phosphorous-host in the Brockman ores, as detected by the supplementary QXRD work, was not anticipated. Second, the presence of phosphorous was largely underestimated by QEMSCAN. While this is partly due to the fact that the system had not been programmed to detect Yttrium, it is surprising that Phosphorous hosted in Goethite was not detected; at  $5 \times 5 \mu\text{m}$  resolution, the bulk of phosphorous containing pixels were found to contain  $<20\%$  Phosphorous, suggesting that either Phosphorous is present in very fine grained minerals or in mixture with other phases.

Further to this, particle distribution size analyses appear to indicate that aggregation persisted in some size fractions of the sieved calibration material, despite best efforts to remove it. For example, material from the 1.18mm to 2.36mm size fraction was found to be 80% passing 0.94mm according to the QEMSCAN analysis, suggesting that the majority of material was actually smaller in size than the minimum sieve size. Size distribution analyses were conducted both on heterogeneous particles and individual mineral grains.

The QEMSCAN analysis reported liberation data for iron oxides, silicates (primarily existing as kaolinite) and intergrowths, or intimate mixtures of iron oxides and Al and Si. Liberation data for quartz was not provided. A mineral is considered liberated where area percent is greater than 90%; therefore liberation in this context is reported as the mass percent of mineral grains between 90% and 100%



**Figure 2.** QEMSCAN image from a Brockman ore particle from Experiment 2 showing kaolinite grains (dark yellow) intermixed within siliceous intergrowth (light orange), interlocked with hematite grains (dark orange)

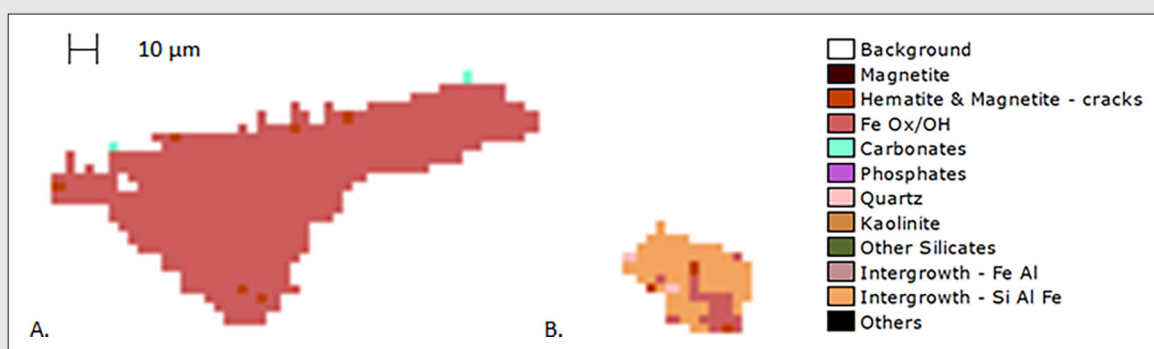


Figure 3. A. QEMSCAN image from a CID ore particle consisting primarily of goethite (dark pink) B. Intergrowth with quartz (light pink) from a CID ore showing typical size difference between goethite grains and intergrowths

liberated at a given P80, as determined by the size distribution analyses. In this way, an exact liberation size is not given, but can only be inferred based on the degree of liberation at a certain grain size. In spite of this constraint, the QEMSCAN results did not conflict with the liberation sizes calculated in the SFA calibration experiments except in a single instance. Combined results of the SFA experiments and the QEMSCAN analyses are compared in Table 2. Given that phosphorous bearing minerals were not detected at a  $5 \times 5 \mu\text{m}$  resolution in the QEMSCAN analysis, here we assume phosphorous is liberated at  $<5 \mu\text{m}$ . For proprietary reasons, iron results have been excluded.

## Discussion

The purpose of this work was to determine the Fundamental Sampling Error of the exploration sampling protocol, firstly because it is an essential metric to confidently ensure product quality, and secondly, as a measure of comparison against future at-rig sampling technologies. It is therefore critical that the calculation of FSE is done correctly as it may ultimately inform business decisions. The QEMSCAN validation was found to be beneficial to ensuring that the mineralogical input parameters are as accurate as reasonably possible.

None of the experimental liberation sizes were in direct conflict with the QEMSCAN analysis, except in a singular instance with the phosphorous calibration in Experiment 2. The persistent aggregation confirmed by the particle size analyses may indeed point to

a lesser degree of confidence in this experiment, however it must also be noted that phosphorous liberation is only assumed from the QEMSCAN analysis; a slight disagreement between SFA and QEMSCAN is not overly surprising given this degree of uncertainty.

As a mining company focused on productivity and cost, it is pertinent to discuss some practical matters associated with this work. Despite generating fewer size fractions and, consequently, fewer points with which to fit the calibration curve, we found that the RC sourced bulk material produced an experimental result which was no less reliable than the diamond core or mine sourced material. Given that most iron ore exploration primarily relies on RC drilling, this material is likely to be readily available, in addition to being low cost. Furthermore, a bulk sample can be taken from an RC rig without interrupting the standard collection of a primary sample, and it does not require interrupting production activities to collect material from an active pit. It is therefore suggested that RC sourced material is a reasonable place to start when conducting calibration experiments for K and alpha using the SFA method.

This work was conducted as the conversation about product quality, and therefore sample quality, becomes increasingly widespread in our business. In the context of this economic environment, the authors consider this most basic understanding of one's sampling protocol invaluable. With this, BHP Billiton Iron Ore reinforces the market imperative to deliver a consistent and high quality product to its customers by optimising sampling processes through the entire supply chain.

Table 2. Comparison of SFA and QEMSCAN calibration results, including assumed mineralogy as per QEMSCAN

SFA Experiment	Analyte	alpha	dL SFA ( $\mu\text{m}$ )	dL QEMSCAN ( $\mu\text{m}$ )	Assumed Mineral	Density ( $\text{g}/\text{cm}^3$ )
Experiment 1	$\text{Al}_2\text{O}_3$	2.3	8	$<30$	Kaolinite	2.4
	$\text{SiO}_2$	2.3	17	$<30$	Kaolinite	2.4
	P	2	2	$<5$	Xenotime	4.75
Experiment 2	$\text{Al}_2\text{O}_3$	2.5	14	$<30$	Kaolinite	2.4
	$\text{SiO}_2$	2.4	21	$<30$	Kaolinite	2.4
	P	1.8	16	$<5$	Xenotime	4.75
Experiment 3	$\text{Al}_2\text{O}_3$	1.8	25	$<25$	Al-Goethite	3.3
	$\text{SiO}_2$	2	2	Unknown	Quartz	2.6
	P	1.8	3	$<5$	Unknown	Unknown

## References

1. Gy, P M, "*Sampling of Particulate Materials: Theory and Practice*". Elsevier, Amsterdam (1979).
2. Lyman, G J and Shouwstra, R, "Use of the scanning electron microscope to determine the sampling constant and liberation factor for fine minerals", in *Proceedings from the 5th World Conference of Sampling and Blending (WCSB5)*. Gecamin, pp. 90-103 (2011).
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