

# INTRINSIC HETEROGENEITY TESTER (IHT): A NEW PROTOTYPE TO MAKE HETEROGENEITY TESTS EASIER TO PERFORM

A. C. Chieregati<sup>1</sup>, Jr.H. Delboni, H. Delboni

## ABSTRACT

Pierre Gy's formula for determining minimum representative sample masses and relative variances of the Fundamental Sampling Error (FSE) is based on estimation of the constant factor of constitutional heterogeneity,  $IH_L$ , which can be calculated using Gy's factors – mineralogy, shape, granulometry and liberation – or derived by heterogeneity tests which allow the calibration of the sampling constants  $K$  and  $\alpha$  for each type of ore. There are mainly three methodologies for calibrating  $K$  and  $\alpha$ : the Heterogeneity Test (HT), the Sampling Tree Experiment (STE), and the Segregation Free Analysis (SFA). The development of alternative tests, i.e. the STE and later the SFA test, aimed to assist the performance of the heterogeneity test, which is known for being time consuming and often difficult to perform. The main difference between the new tests and the standard heterogeneity test is the methodology of subsample composition. The heterogeneity test is performed by manually collecting individual fragments at random to compose each subsample to be analysed, while the STE and SFA tests are performed by splitting the samples using a rotary or a riffle splitter. It was proved for gold and aluminium ores that both the STE and the SFA tests do not isolate the Fundamental Sampling Error since none is capable of completely eliminating the Grouping and Segregation Error (GSE), for the obvious reason that there is no such thing as 'segregation free' when handling bulk solids flows. Therefore, the only reliable methodology for isolating FSE is the heterogeneity test. This paper presents a new device referred as Intrinsic Heterogeneity Tester (IHT) designed to automate the heterogeneity test, making it easier to perform. Both the HT and the IHT tests were carried out on Brazilian iron ores and showed no significant differences between the results.

## KEY WORDS

Heterogeneity test; intrinsic heterogeneity tester (IHT); fundamental sampling error (FSE); theory of sampling (TOS)

## INTRODUCTION

Sampling is carried out at all stages of mining operations, and although planning and estimation processes must rely on sample results, samples rarely present characteristics identical to those of the material from which they were taken. This is due to the Fundamental Sampling Error (FSE) and to the other errors that arise during the sampling operations (Figure 1). All of these errors result solely from the existence of heterogeneity in a lot of particulate material. The objective of Pierre Gy's Theory of Sampling (TOS) is to control these errors by analysing their properties as a function of the sampling process and the sampled material, and indicating the minimum mass, the correct equipment and the adequate procedures that will eliminate or, at least, minimise them.

It is of utmost importance to estimate the Fundamental Sampling Error, from which minimum sample masses and optimised sampling protocols are calculated. One of the ways to estimate FSE is applying four factors defined by Pierre Gy: mineralogy ( $c$ ), shape ( $f$ ), granulometry ( $g$ ) and liberation ( $l$ ), as in Equation 1.

$$s_{FSE}^2 = \left( \frac{1}{M_s} - \frac{1}{M_L} \right) c f g l d^3 \quad (1)$$

where  $s_{FSE}^2$  is the relative variance of the Fundamental Sampling Error,  $M_s$  is the minimum sample mass,  $M_L$  is the mass of the lot, and  $d$  is the nominal top-size of the fragments in the lot. For details on how to calculate each factor, refer to Pitard (1993; 2019).

<sup>1</sup> Prof. Ana Carolina Chieregati, Department of Mining and Petroleum Engineering, University of São Paulo, Av. Prof. Mello Moraes, 2373, 05508-030, Brazil. E-mail: ana.chieregati@usp.br

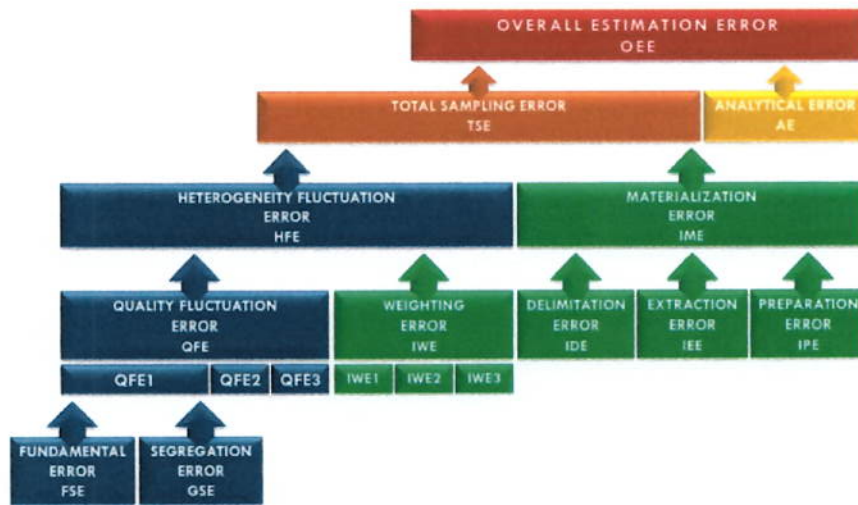


FIG. 1 - Components of the Overall Estimation Error (OEE)

It is not the object of this study to calculate and apply these factors, seeing that, although extensive studies on the liberation factor ( $l$ ) have been performed (François-Bongarçon 2015; 2017), its estimation is often difficult and inaccurate. This is one of the reasons why heterogeneity tests have been developed to allow the calibration of the sampling constants,  $K$  and  $\alpha$  (see Equation 4 further in the text), for each type of ore.

According to Pitard and François-Bongarçon (2011), there are two choices for heterogeneity tests: (1) to estimate exclusively the variance of  $FSE$ , or (2) to estimate the variance of  $QFE_1$ ; the former looking at the Intrinsic Heterogeneity exclusively, which necessitates the preparation of samples by collecting fragments one by one at random, the only condition under which the Grouping and Segregation Error ( $GSE$ ) would cancel; the later looking at the Distribution Heterogeneity between replicate splits or groups of fragments.

The same authors conclude that the more practical choice is the Sampling Tree Experiment, proposed by François-Bongarçon (1995) and Minnitt et al. (2007), because the estimation of the variance of  $QFE_1$  better reflects what is happening in daily reality in sampling protocols. They also affirm that “the collection of random increments made of one fragment to prepare samples for the determination of the variance of  $FSE$  exclusively is cumbersome and results do not reflect what is happening in daily reality when implementing routine sampling protocols”.

However, because segregation is a transient phenomenon varying greatly from one second to another, and  $GSE$  estimated by the Sampling Tree Experiment will not necessarily be the same as the one happening in the daily reality, it is still important to estimate how much of the total  $QFE_1$  variance is due to the Fundamental Sampling Error, which can only be done by performing the standard Heterogeneity Test proposed by Gy (1967) and Pitard (1993; 2009). The main objective of the IHT prototype is to reduce the ‘cumbersomeness’ of the heterogeneity test.

## METHODOLOGY

Approximately 1 tonne of iron ore (500 kg of compact grey itabirite and 500 kg of compact red itabirite), made of several increments representing two different geological domains of the same mining operation, was selected at the piles formed by the blasted ore, then crushed and screened into the following size fractions:

- -25.4+12.7 mm
- -12.7+6.35 mm
- -6.35+4.75 mm
- -4.75+3.35 mm

## INTRINSIC HETEROGENEITY TESTER (IHT): A NEW PROTOTYPE TO MAKE HETEROGENEITY TESTS EASIER TO PERFORM

Using a riffle splitter, the -12.7+6.35 mm fraction was split into two subsamples, one for the Heterogeneity Test (HT) and one for the Intrinsic Heterogeneity Tester (IHT).

### Standard Heterogeneity Test

The material for each size fraction was evenly spread on a table where a grid was previously drawn using tape. Taking Figure 2 as an example, the first subsample was composed of one fragment randomly collected from each of the 68 cells of the grid, making up a 68-fragment subsample. This procedure was repeated 50 times, generating 50 subsamples of 68 fragments each for this size fraction. The same procedure was applied for the other three size fractions. Finally, the 50 subsamples of each size fraction were sent to the physical laboratory for preparation and chemical analysis (Fe and SiO<sub>2</sub>).



FIG. 2 - Material spread on the table for the heterogeneity test

Once subsamples were weighed and analysed, the mass  $M_q$  and grade  $a_q$  for each group of fragments, as well as the average masses  $M_Q$  and weighted average grades  $a_Q$ , were calculated according to Equation 2 (Pitard, 1993).

$$a_Q = \frac{1}{M_Q} \sum a_q M_q \quad (2)$$

The estimated constant factor of constitutional heterogeneity, EST  $IH_L$ , was calculated according to Equation 3, for each of the four size fractions.

$$\text{EST } IH_L = g \sum \frac{(a_q - a_Q)^2}{a_Q^2} \cdot \frac{M_q^2}{M_Q} \quad (3)$$

The regression line passing through the different values of  $IH_L$ , calculated for the four size fractions, allowed the estimation of the sampling parameters  $K$  and  $\alpha$  of Equation 4 (Pitard, 1993), where  $K$  is a constant factor representing the product of all Gy's factors. The cube of the nominal top-size of the fragments in Equation 1 is replaced by  $\alpha$ , a variable which is determined by the slope of the regression line.

$$s_{FSE}^2 = K d^\alpha \left( \frac{1}{M_s} - \frac{1}{M_L} \right) = IH_L \left( \frac{1}{M_s} - \frac{1}{M_L} \right) \quad (4)$$

When  $IH_L$  is written as a function of  $d$ , and considering the exponent  $x$  of the liberation factor formula (Equation 5) equal to 0.5,  $\alpha$  is 2.5, as the liberation factor includes  $d$ . It is important to emphasise that the exponent  $x$  depends on the material and  $x = 0.5$  is just one approximation, which is not valid for all materials.

$$l = \left(\frac{d_l}{d}\right)^x = \sqrt{\frac{d_l}{d}} \quad (5)$$

where  $l$  is the liberation factor and  $d_l$  is the liberation diameter of the mineral of interest.

### Intrinsic Heterogeneity Tester (IHT)

The IHT prototype (Figure 3) consists of a wooden round rotating table with 50 plastic recipients, a table drive motor, a vibrating chute, a storage silo, three laser emitters for fragment counting, and a pallet for fragment deflection.



FIG.3 - The Intrinsic Heterogeneity Tester (IHT)

The vibration of the feed chute is adjusted to form a line of fragments flowing very slowly over the chute. While the fragments fall, the lasers count up to 10 and the pallet is then activated to deflect the 11<sup>th</sup> fragment into the bucket. The electronic system can be programmed to deflect one fragment in every 10 or other number of fragments counted, or even to deflect one fragment randomly in a set interval. After the fragment is deflected, the table motor is driven to position the next bucket below the discharge chute.

This procedure is repeated until the 50 buckets collect 60 individual fragments each. The number of fragments in each bucket can be adjusted using the electronic control panel. After the collection of an equal number of fragments in each bucket, the IHT stops automatically and the fragments can be placed into plastic bags for shipping to the laboratory.

Because the test using the IHT was performed for one single size fraction with  $\bar{d} \approx 1$  cm (-12.7+6.35 mm), it is referred to as the 'Simplified Heterogeneity Test'.

### Simplified heterogeneity test

Firstly, groups of fragments are weighed and an average mass per fragment is calculated for each size fraction. The simplified heterogeneity test was performed only for the -12.7+6.35 mm fraction, for which the fragment mass was multiplied by 30 000 ( $10 \times 50 \times 60$ ) to compose the initial lot to be tested, so that:

$$n > 10 Q_p \quad (6)$$

where  $n$  is the minimum number of fragments for the test,  $Q$  is the number of subsamples to be composed by the test, and  $p$  is the number of fragments in each subsample (Pitard, 1993). The number of fragments in each subsample was calculated based on the mass needed to analyse iron and silica contents, in this case 60 g. With each fragment weighing approximately 1.2 g, the minimum mass for the test was 36 kg for each type of ore.

The IHT prototype presented in Figure 3 was designed to test two size fractions (-12.7+6.35 mm and -6.35+4.75 mm), but can be adjusted to test other size fractions.

## RESULTS AND DISCUSSION

### Standard Heterogeneity Test

The graphs in Figures 4 and 5 show the calibration of the sampling constants  $K$  and  $\alpha$  for the grey itabirite and the red itabirite respectively.

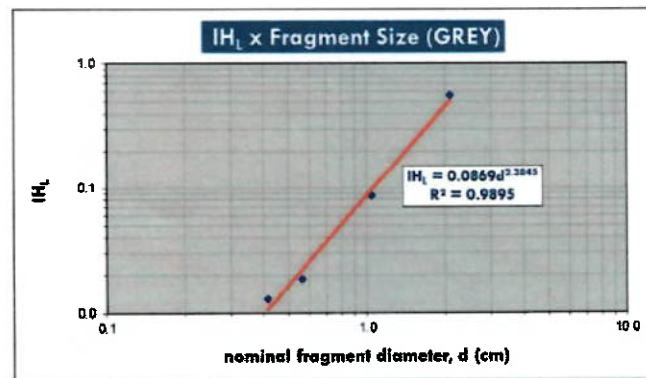


FIG. 4 - Heterogeneity graph for the compact grey itabirite (Fe)

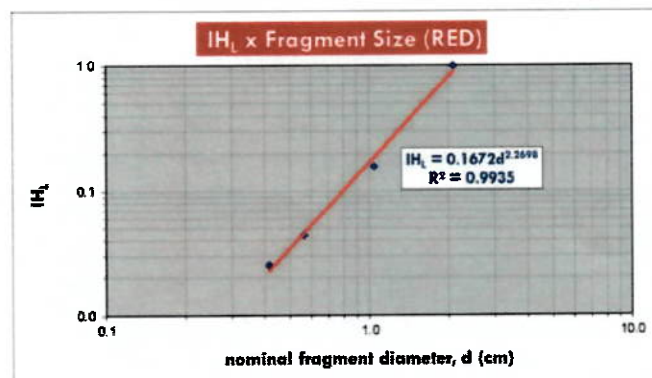


FIG. 5 - Heterogeneity graph for the compact red itabirite (Fe)

For the grey itabirite:

- $K = 0.0869$
- $\alpha = 2.3845$

For the red itabirite:

- $K = 0.1672$
- $\alpha = 2.2698$

The same analysis was performed for silica (SiO<sub>2</sub>), resulting for the grey itabirite:

- $K = 0.0841$
- $\alpha = 2.4898$

And for the red itabirite:

- $K = 0.1244$
- $\alpha = 2.4710$

### Simplified Heterogeneity Test

When  $IH_L$  is written as a function of  $d$ , and considering the exponent  $x$  of the liberation factor equal to 0.5,  $\alpha = 2.5$ :



$$IH_L = c f g l d^3 = c f g \sqrt{\frac{d_i}{d}} d^3 = c f g \sqrt{d_i} d^{2.5} = K d^{2.5} \quad (7)$$

For an average nominal top-size of the fragments of 1.05 cm,  $K = IH_L / 1.05^{2.5}$ . This relation has been used to estimate  $K$  for the size fraction -12.7+6.35 mm.

Tables 1 and 2 show the calculation of the sampling constant  $K$  for the grey itabirite and the red itabirite respectively, for both iron and silica contents. The parameters  $K$  and  $\alpha$  are compared between the standard heterogeneity test (Std HT), the -12.7+6.35 mm fraction of the standard heterogeneity test (Std HT 1-fraction), and the simplified heterogeneity test using the Intrinsic Heterogeneity Tester (IHT).

**TAB. 1 - Sampling constants for the compact grey itabirite (IHT × HT)**

Element	Size fraction/mm	$d_n$ /cm	Test	$\alpha$	$K$
Fe	various	various	Std HT	2.38	0.086 9
	-12.70 +6.35	1.05	Std HT 1-fraction	2.5	0.076 8
	-12.70 +6.35	1.05	IHT	2.5	0.052 7
SiO <sub>2</sub>	various	various	Std HT	2.49	0.084 1
	-12.70 +6.35	1.05	Std HT 1-fraction	2.5	0.084 1
	-12.70 +6.35	1.05	IHT	2.5	0.059 4

**TAB. 2 - Sampling constants for the compact red itabirite (IHT × HT)**

Element	Size fraction/mm	$d_n$ /cm	Test	$\alpha$	$K$
Fe	various	various	Std HT	2.27	0.167 2
	-12.70 +6.35	1.05	Std HT 1-fraction	2.5	0.139 3
	-12.70 +6.35	1.05	IHT	2.5	0.103 2
SiO <sub>2</sub>	various	various	Std HT	2.47	0.124 4
	-12.70 +6.35	1.05	Std HT 1-fraction	2.5	0.096 1
	-12.70 +6.35	1.05	IHT	2.5	0.080 3

The results show that, although the parameters  $K$  and  $\alpha$  vary between the different tests, the order of magnitude does not change significantly.

It is important to emphasise that, for the same fraction (-12.7+6.35 mm), the masses and number of fragments were different considering the standard HT and the IHT tests. While the IHT test generated 50 samples with an average sample mass of 76 g, the standard HT test generated 50 samples with an average sample mass of 157 g for this particular size fraction. The number of fragments in the IHT samples was 60, while in the standard HT test the number was 130. These differences inflated the variance of IHT sample grades from 1.6 (Fe) and 3.2 (SiO<sub>2</sub>) to 2.5 (Fe) and 5.2 (SiO<sub>2</sub>) and could be considered as a potential source of error when validating the IHT. Therefore, further tests should be conducted with the same number of fragments for both the standard HT and the IHT tests.

## CONCLUSIONS

It is known that segregation is a transient phenomenon, varying greatly from one second to another as the material flows and depending on local conditions that can be many. According to Pitard and François-Bongarçon (2011), “the amount of segregation observed is a relative concept depending on the scale of observation; this is the main reason its quantification is illusory at best”. They also cite that “following many studies performed in a mineral processing research facility that at the time was actively working on segregation processes, Gy came to the conclusion that any attempt to quantify the variance of segregation, therefore the variance of GSE, was an exercise in futility”.

## INTRINSIC HETEROGENEITY TESTER (IHT): A NEW PROTOTYPE TO MAKE HETEROGENEITY TESTS EASIER TO PERFORM

Pierre Gy's Theory of Sampling (1979) demonstrates that the variance of the Grouping and Segregation Error (GSE) is the product of the variance of the Fundamental Sampling Error (FSE) and the grouping factor ( $\gamma$ ) and the segregation factor ( $\xi$ ). Because both factors vary from 0 to 1, the variance of GSE is always smaller or equal to the variance of FSE. Therefore, even though alternative heterogeneity tests have been developed to estimate  $QFE_1$ , which is the sum of FSE and GSE, it is important to know how much of  $QFE_1$  is due to the Fundamental Sampling Error, which can only be achieved by performing the standard heterogeneity test. Knowing that segregation usually reaches its maximum as soon as the mineral of interest is liberated, if one estimates FSE, the minimum and the maximum  $QFE_1$  can also be calculated.

Based on the fact that the original heterogeneity test developed by Gy and Pitard can become cumbersome and not many technicians are willing to perform it, a new device named the Intrinsic Heterogeneity Tester (IHT) was designed and built to automate the heterogeneity test, making it easier to perform. First results for two different types of iron ore show no significant differences between the standard heterogeneity test and the IHT, referred to as the 'simplified heterogeneity test' as it was performed on only one size fraction.

For the validation of the IHT, the authors suggest that further tests should be conducted using the same number of fragments for both the standard and the simplified heterogeneity tests. Furthermore, the -6.35+4.75 mm size fraction should also be tested, in a way that a heterogeneity graph could be drawn for the IHT results, and the sampling constants  $K$  and  $\alpha$  could be estimated from the two size fractions. Small adjustments should be made to the IHT, as two particles are deflected at the same time if they are not well separated by the vibrating chute.

Finally, since iron is one of the less heterogeneous ores, the next steps are to test aluminium, zinc and gold ores, considered to be low, medium and high heterogeneity respectively.

## REFERENCES

- François-Bongarçon, D. 2015. Introduction and first ever rigorous derivation of the liberation factor. *Proceeding of the Seventh World Conference on Sampling and Blending*, WCSB7, p. 165-168. TOS Forum. IM Publications: West Sussex.
- François-Bongarçon, D. 2017. Evolution of the concept of the liberation factor and a surprising new result. *Proceeding of the Eight World Conference on Sampling and Blending*, WCSB8, p. 307-309. AusIMM: Carlton.
- Gy, P.M. 1967. *L'Echantillonnage des minerais en vrac - théorie générale*. Vol. 1, Société de l'Industrie Minière, Saint-Etienne, 186 p.
- Gy, P.M. 1979. *Sampling of particulate materials – theory and practice*. In: *Developments in Geomathematics 4*. Elsevier: Amsterdam. 431 p.
- Minnitt, R.C.A.; Rice, P.M.; Spangenberg, C. 2009. *Part 2: Experimental calibration of sampling parameters K and alpha for Gy's formula by the sampling tree method*. The Journal of The Southern African Institute of Mining and Metallurgy, p. 513-518, vol. 107. SAIMM Publications: Cape Town.
- Pitard, F.F. 1993. *Pierre Gy's sampling theory and sampling practice: heterogeneity, sampling correctness, and statistical process control*. 2nd ed., CRC Press: Boca Raton. 488 p.
- Pitard, F.F. 2009. *Pierre Gy's Theory of Sampling and C.O. Ingamells' poisson process approach – pathways to representative sampling and appropriate industrial standards*. PhD thesis, Aalborg University, Esbjerg, 309 p.
- Pitard, F.F. 2019. *Theory of sampling and sampling practice*. 3rd ed., CRC Press: Boca Raton. 693 p.
- Pitard, F.F. and François-Bongarçon, D. 2011. Demystifying the Fundamental Sampling Error and the Grouping and Segregation Error for practitioners. *Proceeding of the Fifth World Conference on Sampling and Blending*, WCSB5, p. 39-55. GECAMIN: Santiago.

**Proceedings  
of  
NINTH WORLD CONFERENCE ON  
SAMPLING AND BLENDING**

---

**6-9 MAY 2019  
BEIJING, CHINA**



www.wcsb9.com



# WCSB9

**9th World Conference on  
Sampling and Blending**

**May 6-9, 2019**

**Beijing International Convention Center • China**



Hosted by:



BGRIMM Technology Group

Supported by:



中国分析测试协会  
CHINA ASSOCIATION FOR  
INSTRUMENTAL ANALYSIS (CAAIA)

China Association for Instrumental Analysis



China Mining Association



The Chinese Society for Metals