

Heterogeneity studies: Do's and Don'ts

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Although several papers have been published to date on how to perform heterogeneity studies, complaints have been voiced by academics along time about the lack of simple how-to explanations, including the limitations of the methods and errors not to commit. This paper intends to remedy this situation, with the primary purpose of clarifying all the corresponding ideas. Common difficulties and errors are described and methods to be used are recommended while providing the reader with an in-depth understanding of what heterogeneity studies are really about.

INTRODUCTION: THE PROBLEM

TOS principles

As emphasized by Gy on many occasions, the most important part of Gy's TOS is without a doubt its practical, first principles: sample correctness and the solutions to segregation effects mitigation. Unless a sample is verifiably correct and protected from the effect of segregation, controlling its reproducibility numerically is a useless endeavor. An in-depth understanding of these principles is a sine-qua-non condition to properly performing any kind of heterogeneity study. Appendix 1, which is only an illustrative example, shows how the understanding of these principles can make a huge practical difference and how the thinking about TOS should be trained to such an end. In the course of such a study, simplifications are at times necessary, difficult decisions need to be made, and they should always be performed under the understanding and in the respect of such basic principles.

Numerical sampling variance modeling and control

Once this is achieved, then the numerical control of the sampling variance becomes a meaningful and useful exercise. To this effect, one would naturally like to use the formulas that have been derived by Gy, then vindicated by G. Matheron (François-Bongarçon and Pitard, 2015), for the mathematical modeling of the sampling variance, namely the now well known formulas:

$$\text{Rel.Var.} = c \ell f g d_{95}^3 (1/M_S - 1/M_L) \quad (1)$$

(for comminuted or naturally particulate material)

$$\text{Rel.Var.} = c \ell' f g' d_{\max}^3 (1/M_S - 1/M_L) \quad (2)$$

(for closely sieved material)

In these formulas, some parameters can be set at sampling time: the sample mass M_S , the comminution P95 size d_{95} . Other parameters can be calculated from known properties of the material to be sampled: the mineralogical constant c , the shape factor f , the granulometric factors g or g' . But since that material will more often than never not be fully liberated, the liberation factor, ℓ or ℓ' , will also have to be set for each sampling operation under study.

This factor is known to depend upon d_{95} and the liberation size of the analyte of interest, d_0 . The final objective of a heterogeneity study is to fix every possible variable parameter to experimentally derive the variations of the sampling variance brought about by variations of the comminution size only, or, in other words, to allow for the prediction of variances and what-if scenarios.

INTRODUCING HETEROGENEITY STUDIES

Types of data and experiments

As soon as one tries to relate experimental sampling variances to a sampling formula, that person is doing a heterogeneity study of sorts. There are as many types of heterogeneity studies as one can imagine. In practice, however, we can identify a few types, depending on the type of data that generally are or can be made available.

Any set of data conducive to the extraction of experimental sampling variances is useful for heterogeneity characterization. The most notable types of favorable data are:

- series of samples generated for this purpose (several types exist), or/and,
- series of duplicate sample grades, often collected within the frame of a QAQC program.

These various types of data lead to different types of characterization studies, and can also be used jointly. As will be further clarified, this modeling effort will call for the experimental calibration of various parameters corresponding to distinct characteristics of the material: some relating to the spatial structure of the analyte of interest in its gangue (i.e. mode of clustering, *spatial distribution* - thus microgeostatistical), others relating to the individual analyte grains themselves and their size *statistical distribution*. And in ultimate analysis, these are the only characteristics that control the grade variability from sample to sample at a given comminution size.

The only models of liberation factor currently in successful use indeed summarize these through two parameters: an exponent b that has a geostatistical meaning and quantifies the clustering mode, for which the term '*De Wijs Exponent*' is proposed) and the 'liberation size' (that should be more properly coined the '*grain size parameter*'), a model parameter directly linked to the size distribution of the grains. Current methods to characterize particulate material heterogeneity are explicitly using a model of liberation factor and are designed to calibrate these types of parameters.

The determination of the De Wijs Exponent b generally relies on the processing of one of the following types of data:

- Series of samples generated at various comminution sizes from a given lot
- Series of samples taken from different size classes obtained by sieving
- Series of pieces of fist-size fragments or pieces of solid samples used to anchor the calibration graph at the high end of the size range considered
- Various types of duplicate sample data that can be made compatible only for a certain, single value of the exponent b that is then determined (usually the compatibility criterion is a calculated size parameter - 'consistency method' - (Francois-Bongarcon, 2008).

A fair number of studies were performed by the author along time to verify, successfully, that the exponent was insensitive to grade, as suspected from the elaboration of a theoretical model (in the sequel). But it may change with mineralogy.

The characterization of the grain size parameter then takes place that is best performed using one set of duplicate sample grades. Unlike the exponent, this parameter will change from lot to lot, may change with grade and certainly with the mineralogy.

Common difficulties and precautions

Duplicate samples

The first difficulty in using sets of QAQC sample duplicate grades is that they were not designed for that purpose. In many instances, pulp duplicates have been at least partially cleaned up for outliers and values deemed to be 'impossible', often in a semi-arbitrary manner, thus removing some of the variability relevant to the heterogeneity characterization. Therefore, coarse or field duplicates are preferred for these purposes. When using the above-mentioned 'consistency method', a typical consequence is that the pulp duplicates cannot be made compatible with the other types for any value of exponent b . They are then best ignored, provided the other sets of duplicate data converge for an adequate value of b .

The practitioner should of course be well aware of the differences between sampling (for TOS) and measuring (for geostatistics), between samples and measurement supports and therefore not use measurement support duplicates (i.e. repeats of some in-situ sampling, such as 'duplicate' channel samples or 'duplicate 1/2 core' as a mere examples).

Series

When using one of the methods involving the splitting of series of samples, a number of difficulties need to be avoided:

- The most common consists of ill-designing the experiment so that the variances calculated from laboratory results can not be cleaned up to remove the unwanted variance components (such as those due to subsequent preparation and sub-sampling) to the end of equating the sampling formula under calibration to the result of a single-stage, primary sampling operation. In particular, if the sample in the series are even only pulverized and re-sampled, then removing the pulp sampling and analytical variances from the total assay variance requires that one of the series be a series of pulverized sub-samples. Not removing that component will always give a high-biased value of exponent b (see Figure 1, knowing that a difference of 1.9 to 2.2 for b would have an enormous effect on preparation variances).
- In the case of sample series taken from closely sieved material, it is also easy to forget the model of liberation factor is not the same as the one that would be used for series split from comminuted lots, but a modified version (ref.).
- The splitting methods used to generate series of samples should give random samples reasonably protected from the effects of segregation. Riffle splitting, alternate shoveling and fragment-per-fragment selection are recommended methods. Rotary splitters do not give random samples and therefore could not be used to calibrate a theoretical formula they have nothing to do with. Indeed, to the usual surprise of many a practitioner, the method, akin to circular version of bed-blending, uses segregation as an advantage by making sure it is uniformly distributed in sampling containers. Segregation in that process is a plus
- Of course, the samples should still be able to represent the TOS formula, which precludes using non-random splitters such as rotary ones.
- It should be understood the parameters obtained should ultimately be compatible with the sampling procedures to design, diagnose or optimize. In particular, sampling characteristics of a lot of comminuted material simply cannot be derived for a single size-fraction, which, alone, may easily have different sampling properties.
- Finally, in all cases, care should be taken to make sure the quantities posted together on a same calibration graph were made directly comparable/compatible (i.e. representing the same quantities), by moving any factor of difference into the experimental statistics plotted as Y-axis on that graph.

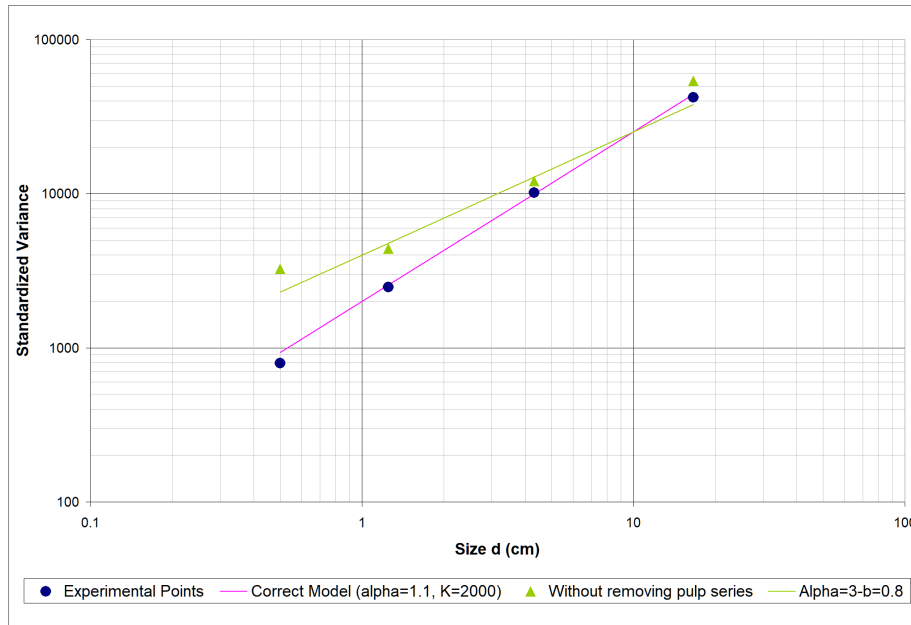


Figure 1. Real example of alpha calibration

Outliers

At the end of the physical experiments, some statistical processing of the data will take place, generally on small data sets, with acute sensitivity to outlying values. Good, common sense should be used to discard these (a mandatory step), being careful not to be unconsciously influenced by a 'desired/expected' overall result. The removal of outlying values should be kept at a strict minimum, refining without distorting the results that would be obtained before their removal, and above all resolving impossible order relations between experimental points.

CAN WE DO WITHOUT A MODEL OF LIBERATION FACTOR?

Gy's actual formula

The numerical above-mentioned formulas were established throughout enormous numerical/mathematical difficulties by Gy and Matheron (Gy, 1982, François-Bongarçon and Pitard, 2015). They were first derived as a first order approximation to the relative variance of the grade of a random, correct sample not affected by segregation. The complex equation, however, could never be calculated from known or measurable parameters, except in the fully liberated case. So that, in fact, the only mathematical model then derived by Gy...:

$$\text{Rel.Var.} = c f g d_{95}^3 (1/M_S - 1/M_L)$$

...addresses only the fully liberated case. There was never any other model in the original TOS.

As a side-note, it is entirely possible that establishing the variance model assuming full liberation from the very start may have been much simpler (an excellent research subject for master students). Worth noting too, in his latest paper (Lyman, 2023), the late Geoff Lyman realized that even in the non-liberated case, the number of fragments selected in the sample from the cells of any 2- (or 3)-entry table, such as the granulo-densimetric classes of P.Gy, followed a Poisson distribution. He showed the way towards going back to the full probability law of the sample grade using characteristic functions (Lyman, 2023). Would this allow for the singling out of the liberation portion in the obtained equation? Further research will tell.

Meantime, if non-liberated material need to be addressed in sampling procedure design, the required heterogeneity characterization should account for a slightly different formula, modified by the introduction of a liberation factor (see equations 1 and 2). Gy introduced it only as an ad-hoc number between 0 and 1 meant to diminish the variance from the value calculated assuming full liberation.

The only known case where the factor can be dispensed with is when no what-if scenario whatsoever is to be applied to the design. An example would be when sampling blast hole or RC cuttings of fixed comminution size, with only the sample mass to optimize.

History of the liberation factor models

Tables

Gy was well aware (personal communications) that setting values for, or modeling the liberation factor was a strong gap in his theory (a.k.a. TOS). He nevertheless noted it was directly correlated to the proportion of liberated material in the lot (the author subsequently showed it was a good estimator of it (François-Bongarçon, 2017) and depended upon the degree of comminution and the liberation size of the analyte, i.e. the degree of comminution necessary to convey validity to the full liberation formula. Early attempts at helping practitioners set values for it included a series of successive tables that did not even have entries for the comminution size d nor the liberation size. Needless to say, they all failed.

Past analytical formulas

Coarsest fragment grade

At one point in time though, Gy derived the following, valid theoretical formula:

$$\ell = (a_{\max} - a_L) / (1 - a_L)$$

where a_L is the expected content of the constituent of interest and a_{\max} is the expected maximum content of the constituent of interest among the coarsest fragments in the top size fraction of the material to be sampled. This valid formula, however, relies on several simplifying assumptions that are rarely complied with, and in practice also requires the experimental determination of a_{\max} , which becomes impossible as soon as the coarsest fragments are smaller than a quantity that can be assayed separately.

The square root formula

Conscious of the difficulties raised by the liberation factor, Gy turned to an experimental approach which resulted in a simple 'square root' model (Gy, 1982):

$$\ell = (d_\ell / d)^{0.5} \quad (3)$$

This model, derived from a single fitting experiment (personal communication), was erroneously presented as universal in many successive publications by Gy. He later rescinded it in a paper co-signed with the author (François-Bongarçon and Gy, 2002). This particular model gives absurd results when applied to precious metals and wrong results in most other cases. At one point in time, that formula was wired into a sampling slide rule.

Author's contribution

The author's main contribution to TOS, more than 30 years ago, was the establishment of an efficient model for the liberation factor (François-Bongarçon, 1992):

$$\ell = (d_\ell / d)^r \quad (4)$$

with $0 < r < 3$ to be determined

Coincidentally, Gy's model (3) above ($r=0.5$) was but a particular case (rarely encountered in practice - ref. 2010). This formula is in no way, as has been falsely pretended elsewhere, an empirical formula. After having demonstrated the theoretical liberation factor was the ratio of variances of two particular fragment sizes (i.e. the geostatistical ratio of two support sizes), or equivalently of two perfectly defined

and existing physical samples (see references), Smith's formula back in 1926 would then formally provide the above model under a simple assumption of gauge invariance (Rose, 1992). Gold deposits in South Africa having been known to respond very well to modeling using a De Wijs variogram model, it was also demonstrated the above model was verified almost exactly in that case by the geostatistical calculation of the variance ratio of two fragment sizes. That model therefore is singularly linked to reality in these cases, and was a promising model to be used heuristically in all cases. Its results have been spectacular for the last 30 year and it therefore received Matheron's notorious 'sanction of practice' without a single verifiable failure.

In equation (3) above, the author demonstrated (Francois-Bongarcon, 2022) that:

$$l' = [(g/g')^{1/3} d / d\text{-max}]^r \quad (5)$$

Note

A paper in our last conference (Pitard, 2022) purported to address these issues without resorting to the 'complex futility' (sic) of a model of liberation factor (why, is not entirely clear: adding a model to what is already a model to make it work, does not seem to be futile nor out of reason...). It is quite obvious that as few models should be resorted to, and that when needed, the simplest models that will 'do the job' should be used (they are easier to control). In this particular case, except in exceptional situations, a model of liberation factor was found to be absolutely necessary.

In the above-mentioned paper, a number of formulas and simplified versions of them were offered. The author tested a few of them in the case of a blast hole pile with 1cm fragment P95 size, containing P95=25 microns gold grains. As can be easily reproduced by the interested reader, the results of these formulas were as follows:

- Formula [7] in that paper gave an obviously incorrect, too small a minimum mass of 563g to represent the size distribution.
- Formula [9] (which does not even include the size of the rock fragments) gave an obviously incorrect, too small a mass of 0.94g
- The square root formula above, when included into Gy's fully liberated model gave an obviously incorrect, too large a mass of 3t.
- The author's preferred model above with the usual default value $b=1.5$ gave 7.5kg, a more reasonable value, well in line with experience.

The reader will conclude.

Conclusion

Unless a single problem with no varying parameters and no what-if scenarios is to be solved, heterogeneity studies cannot dispense with the use of a model for the liberation factor. It is futile to think Gy's theory as we know it was complete, as Gy himself recognized it was not (personal communications) until models were published for the liberation factor. Fortunately, a good model was found that has been able to successfully resolve the situation. In the continuation, this model will be implicitly adopted.

THE WORST CASE POLICY METHOD

VERY IMPORTANT: As we mentioned above, the parameters included in the liberation factor may vary with mineralogical properties. If the final objective of the study is to include the design or optimization of a unique preparation protocol in a laboratory treating all types of samples from various geological origins, the study should concentrate on the mineralogical material most difficult to sample well. Therefore, it is then advisable to study field or coarse duplicate samples flagged by the mineralogical parameters believed to be most relevant, in order to compare sample reproducibility in each mineralogical category and concentrate the entire study in that most difficult case.

Coarse (i.e. crushed) or field destructive samples are best to use for this comparison: indeed, the sampling variances are prevalent components in the variances that can be derived from the corresponding duplicates. Given the heteroscedasticity of sampling variances, and in order to compare 'apples with apples', of course, a grade insensitive criterion (as insensitive as possible - e.g. homogenous to a sampling relative variance multiplied by the grade) should be used. As an added precaution, the comparisons would be best done on truncated data sets so that the various cases correspond to comparable average grades and grade ranges.

Then the following (X, Y) pair increment formula on which to calculate a variance over each set will provide exactly that criterion:

$$\text{Incr} = (X-Y) / \text{SQRT}(X+Y)$$

This formula represents, after simplifications, a pair relative difference multiplied by the square root of the pair grade. Its variance is a very good comparison criterion for that purpose.

PRACTICAL RECOMMENDATIONS

The practitioner is at a loss: on-going recipes and publications are unclear and often false (i.e. with method or calculation errors, even though they were 'peer reviewed'). No one really knows whom to turn to, and caution should be used before following verbatim recommendations made in articles without understanding them at depth. This is quite sad, as TOS is definitively NOT 'rocket science', nor are heterogeneity studies, provided the precautions described in this article have been taken. Pretending these things are too complex for the practitioners of the industry is a straight insult to their face that we definitively do not condone. And articles are never supposed to be recipes to follow blindly, instead they should be viewed at most as enlightened suggestions. Their main objective is and should be bringing topics to the attention of their readers and for them to start THINKING about them. This is exactly what is intended in the sequel of this article.

Steps to follow

Experiment design

At this point, the reader probably senses why designing the experiment may well be the most important step, the one that, poorly done, can potentially trigger irreversible damage to the conclusions of the study.

The simplest and most practical method should be selected, making the best use of the available data. All the difficulties mentioned above should be reviewed at depth. One should make sure that at the end of the experiment, it shall be possible to isolate individual primary sampling variances from the total assay variance that itself will always be the sum of two or more components.

The questions then to address first are:

- Have I determined what the worst case material will likely be?
- Do we have good quality duplicates of different types that should allow for a shorter study using the 'consistency' method for the exponent calibration?
- If not, what material should be collected? From where? If several choices exist, which type of material will be most compatible with the worst case material as determined?
- Is it possible to reduce the test material heterogeneity by truncating the range of grade (remembering the exponent parameter is unlikely to vary with grade)?
- Do I have good enough, undoctored duplicate data to later derive the size parameter for my model of liberation factor? Will its value be reasonably representative, on average, of the worst case material I have selected?

Sample collection

The nature of the experimental material having been decided, the next step may now involve the collection of some test material. Manual selection from a pile of material is of a last resort, as it is difficult

to make sure no extraneous or undue type of heterogeneity is introduced. For mine ore material, the best source would consist of core rejects, as they generally are sampled to mineralogical contacts, and their grades are already known. If sample series are to be generated, crushed rejects may not be best as they would not address larger sizes.

This said, following what Gy called '50-piece experiment', it is advisable to select fist-size rocks or competent, ~20cm core segments to use for a single-size series ($g=1$, to be put into the plotted quantity) which will help anchor the calibration in the large size range. 40 to 50 pieces may be sufficient before outliers are finally removed during the processing.

In the case of series generated from size fractions (a series of comparative studies by the author along time have shown this slightly simpler method to give the same results as series from comminuted material), size-fractions can be joined together to obtain sufficient lot masses. It is therefore advisable to use many screen sizes and later join the size-fractions.

Assaying instructions

When a laboratory is asked to assay samples that have all of similar grades, the laboratory manager will invariably think one is testing its reproducibility, and the risk the assay results would end up being doctored before being released is high. Should that be the case, at least some of the variability the experiments intend to quantify will be removed, invalidating the heterogeneity results. The best course of action is making the laboratory manager well aware of the end goal of the study and requiring no assaying or assay filtering whatsoever should take place.

Data processing

General

After 30 years of practice of heterogeneity studies, it was concluded that the data processing would be best performed in two steps in the following order:

1. Calibration of the concentration-insensitive De Wijs Exponent from size-fraction series, comminuted series, or sample duplicate assays.
2. Determination of an average Size Parameter from duplicates (in the worst-case domain/category) covering a good range of situations (e.g. covering a full geological domain of choice).

Alpha Calibration

Taking equations (1) and (2), using liberation factor models (4) and (5) and putting into the left-hand side what may be difference factors between experimental points, one obtains for instance:

$$\text{Rel.Var.} / [c d_{\square}^r f g (1/M_S - 1/M_L)] = d_{95}^{3-r}$$

(for comminuted or naturally particulate material)

and

$$\text{Rel.Var.} / [c g^{1/3} d_{\square}^r f g^{2/3} (1/M_S - 1/M_L)] = d_{\max}^{3-r}$$

(for closely sieved material)

These two quantities, properly filtered for unwanted variance components, would plot as a straight line as a function of d_{95} (resp. d_{\max}) on a log-log scale if the models are exact. There strong other reasons why a linear model should be fitted to the points on the graph (Figure 1), but they are outside the scope of this article. Once the equation of the best-fit line is obtained, $\alpha=3-r$ can be derived (the slope of the fitted line), along with the Size Parameter for the particular material used in the experiment (see how below).

Liberation size

Knowing the best calibrated value of alpha. and rewriting, for instance, the variance formula for low grade comminuted material as:

$$\text{Rel.Var.} = (\text{rho} / t) d_{\square}^{3-\alpha} f g d_{95}^{\alpha} (1/M_S - 1/M_L) \quad (6)$$

then the next step may consist of using one or several set of duplicates, sorted by the pair grade t and process each one in a relatively small sliding window. For each position, equation (6) above can be solved for d_{\square} . This give trend curves of the Size Parameter (a.k.a. liberation size) as a function of the grade, in order to check either the lack of direct relationship or the existence thereof.

In the particular case of precious metals, indeed, the Size Parameter d_{\square} will have a tendency to increase with grade. That trend should be modeled (visually is sufficient) and from its modeling, the quantity $[I = d_{\square}^{3-\alpha}/t]$ will indicate the maximums of the portion of the sampling variance with direct relationship to the grade. The relevant maximum of the combination [grade, size parameter] will indicate the worst case at which to establish sampling and sample preparation nomograms.

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