Guidelines for acceptable allotted sampling uncertainty

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Sampling protocols often require successive stages of subsampling. A superior, safe limit has been determined by several authors to be around 32% for sampling and subsampling stages combined. Sampling practitioners are frequently unsure about how best to distribute this allotted total uncertainty in the most cost-effective way. Such guidelines mainly concern exploration geologists and grade control engineers who find a large allotted uncertainty acceptable.

Metallurgists responsible for providing appropriate material balance for the plant during a working shift may not find large uncertainty appropriate unless they are willing to wait a week or more before making a sufficiently precise attempt at a material balance. Therefore, for the metallurgist the total allotted sampling uncertainty should not exceed recommended values between 5 to 10%.

Furthermore, commercial transactions involving the sale of valuable commodities such as copper concentrates, nickel concentrates, coal, iron ore, etc., may require a more stringent total allotted sampling uncertainty, possibly as low as 1 to 3%, because small differences can translate into significant costs.

This paper attempts to show realistic guidelines that are acceptable to everyone in various industries. Some practitioners may disagree with these proposals however, they have been frequently applied by the author and are based on significant experience in the industry despite not being universally accepted. It is recommended that sampling practitioners should work together as part of the WCSB initiative to reach logical consensus in this regard. It is important for many industries that the collective intellectual body that constitutes the WCSB provide guidelines based on an extensive theoretical and empirical foundation.

INTRODUCTION

A sampling protocol can involve several sampling stages, several sample preparation stages and a final analytical stage, but at the end they are all affected by a certain amount of uncertainty. In this paper an optimistic assumption is made that sampling and analytical procedures are correct; this does not mean bias generator errors such as Increment Delimitation Errors, Increment Extraction Errors, Increment Preparation Errors, Increment Weighting Errors and the final Analytical Error (*AE*) are zero. They most certainly are not, however precautions were taken to hopefully make them very small. Regardless of precautions these errors take place and the small biases they generate, which are not reproducible, are responsible for a small inflation of precision problems and in many cases there is nothing much we can do about it. Furthermore, at each sampling stage there is a variance of the Grouping and Segregation Error (*GSE*) involved, and we know this variance is not constant and we also know that if precautions are taken it should not be a very large variance; nevertheless there are several sampling stages and these variances most certainly add up. Now, the question is: after selecting a Total Allotted Variance $s^2_{Allotted}$ what proportion of that variance should be allotted to the total acceptable variance for residual errors due to slight incorrectness, *GSE* and *AE*? Let's call this a Residual Variance $s^2_{\mathrm{Re}\,sidual}$ for convenience. The answer to this question is not easy because most variances involved in the Residual Variance $s^2_{\mathrm{Re}\,sidual}$ due to all the errors mentioned above are not quantifiable in a practical way; the only thing we know how to do is to minimize their effect through preventive competence. Nevertheless, preventive competence falls short of satisfying Quality Control regulators who may know very little about sampling errors and analytical errors. The following analysis may help us to reach a reasonable consensus.

Furthermore, in this paper the variance of the long-range Heterogeneity Fluctuation Error s^2_{HFE2} (see definitions in Pitard, 2009) is not taken into consideration because it is part of a totally different issue which requires the optimization of the required sampling interval between increments (e.g., increments taken across a flowing stream in order to prepare a composite sample representing a working shift or a shipment of any commodity). Such interpolation problems have elegantly been solved a long time ago (Gy, 1967) using variography and suggestions made by geostatisticians (Matheron, 1962 and 1963). Also the periodic Heterogeneity Fluctuation Error s^2_{HFE3} is not taken into consideration because it is also part of a different issue which requires the selection on an appropriate sampling mode in order to minimize the effect of this potential error. The most effective sampling mode is Stratified Random Sampling (Gy, 1967).

THE TOTAL ALLOTTED VARIANCE

Assuming the above discussion is well assimilated, now it is necessary to select a maximum variance for the Total Allotted Variance $s^2_{Allotted}$ involved in a sampling and subsampling protocol so it becomes possible to use it as an absolute upper limit in a nomograph that may be used to optimize such protocol.

First, it is generally accepted that introducing a Poisson Process in a sampling protocol is not a good idea. This may start taking place when the standard deviation of the total sampling error exceeds about 35% relative (François-Bongarçon, 2009); above such value the assays distribution from many replicate samples may start to be skewed toward lower values when looking at small contents such as gold or base metals in ore, or impurities in concentrates, or other commodities, or pollutants in the environment. It would be a safe guideline to suggest an absolute maximum at 32% as the transition to a Poisson Process is a slow transition in which there is no magical limit. Obviously, these values are not sacrosanct, and open to discussion.

The geologist looking for gold or copper

Precious metals, such as gold, have a bad reputation for creating extremely difficult sampling problems. Yet, nobody, especially the exploration or grade control geologist, wants a Poisson Process to enter a database because of the devastating consequences (Ingamells and Pitard, 1986). This suggests the selection of a Total Allotted Variance $s^2_{Allotted} = (0.32)^2 = 0.1024$ to be the wise upper limit on a nomograph. It is important to mention that the geologist may not always be able to control the outcome of the gold assays when isolated coarse gold particles are present in the material to be sampled; what is important is for him or her to be aware of the problem as there are ways to prevent it in an effective way (Pitard and Stevens, 2011).

For base metals, which often consist of contents near or above 1%, the upper limit suggested for precious metals is not a wise limit, as it is much easier to perform a better sampling job. For base metals a lower value for the standard deviation of the total sampling error should not exceed about 20% relative, which is a Total Allotted Variance $s^2_{Allotted} = (0.20)^2 = 0.040$ that should be considered as an absolute upper limit in the nomograph.

The metallurgist point of view

The metallurgist in charge of metallurgical accounting, reconciliation with the mine, and routine material balance exercises, following the above guidelines would create a situation where it would be impossible to perform a reliable material balance on a given working shift or even on a given day. Averages from data acquired over a period of one week or two would have to be used in order to minimize the negative effects of additive uncertainties.

For precious metals a lower value for the standard deviation of the total sampling error should not exceed about 10% relative, which is a Total Allotted Variance $s^2_{Allotted} = (0.10)^2 = 0.010$ that should be considered as an absolute upper limit in the nomograph.

For base metals a lower value for the standard deviation of the total sampling error should not exceed about 5% relative, which is a Total Allotted Variance $s^2_{Allotted} = (0.05)^2 = 0.0025$ that should be considered as an absolute upper limit in the nomograph.

The sales people's point of view

Sales people in charge of delivering commodities to many clients around the world may think the above guidelines would create a situation where too much money would be at risk because of unacceptable levels of uncertainty. As a result, sampling protocols must be far more stringent.

For precious metals a lower value for the standard deviation of the total sampling error should not exceed about 3% relative or even less, which is a Total Allotted Variance $s^2_{Allotted} = (0.03)^2 = 0.0009$ that should be considered as an absolute upper limit in the nomograph.

For base metals a lower value for the standard deviation of the total sampling error should not exceed about 1% relative or even less, which is a Total Allotted Variance $s^2_{Allotted} = (0.01)^2 = 0.0001$ that should be considered as an absolute upper limit in the nomograph.

The regulator and lawyer's point of view

Sometimes during litigation the word uncertainty is not welcome, especially for people who think a generated number coming from a laboratory is the panacea, "white or black", with no difference between. These guidelines may give them a realistic perspective, although they may not be enough to educate them properly about heterogeneity and the variability it creates in sampling and analytical protocols.

A LOGICAL, ECONOMIC DISTRIBUTION OF THE TOTAL ALLOTTED VARIANCE

Many practitioners allow an equal variance to all sampling stages; in appearance it seems a logical thing to do. However, it is a fact that sampling stages are far more expensive when the size of fragments in the material to be sampled increases and when the necessary sample mass increases. This would suggest allowing the primary sampling stage (i.e., Fundamental Error FSE_1) half of the total allotted variance assigned to the Fundamental Sampling Errors, then ¼ of it for the secondary sampling stage (i.e., Fundamental Error FSE_2), then 1/8 of it for the tertiary sampling stage (i.e., Fundamental Error FSE_3) and so on.

Let's define the road map:

1. Take into account that the Residual Variance $s^2_{Residual}$ is not anywhere close to zero.
2. Make the optimistic assumption that stringent precautions were taken to make sampling correct, to minimize the effects of segregation and to minimize the Analytical Error.
3. Save some allowance for later contribution of Heterogeneity Fluctuation Errors HFE_2 and HFE_3.
4. Then it is a safe guideline to allow half of the Total Allotted Variance to the Residual Variance, leaving the other half for all the Fundamental Sampling Errors involved.
5. Therefore the logical and most economic sampling protocol should allow the variance for each sampling stage as follows, first taking the example of gold for an exploration, ore resources evaluation programs, and environmental sampling.

Distribution of the Total Allotted Variance for gold in exploration, grade control and environmental sampling

Similar calculations can be made for sampling problems in the environment, therefore the guideline for environmental sampling is included in this section for practical purpose.

Total Allotted Variance $s^2_{Allotted} = (0.32)^2 = 0.1024$ which is 32% total uncertainty.

Residual Variance $s^2_{\mathrm{Re}\,sidual} = \frac{(0.32)^2}{2} = 0.0512$ which is 22.6% uncertainty.

Variance of the Fundamental Sampling Error for the primary sampling stage $s^2_{FSE1} = \frac{(0.32)^2}{4} = 0.0256$ which is 16% uncertainty.

This 16% uncertainty has always been Gy's recommendation for the allotted variance of the Fundamental Sampling Error for gold for a primary sampling stage, so there was logic in that choice, and the logic of such decision is often lost under piles of literature, unfortunately.

Variance of the Fundamental Sampling Error for the secondary sampling stage $s^2_{FSE2} = \frac{(0.32)^2}{8} = 0.0128$ which is 11% uncertainty.

Variance of the Fundamental Sampling Error for the tertiary sampling stage $s^2_{FSE3} = \frac{(0.32)^2}{16} = 0.0064$ which is 8% uncertainty. And so on, the variance of FSE becomes more stringent as the sampling stage involved becomes cheaper.

Distribution of the Total Allotted Variance for copper in exploration and grade control

Total Allotted Variance $s^2_{Allotted} = (0.20)^2 = 0.040$ which is 20% total uncertainty.

Residual Variance $s^2_{\mathrm{Re}\,sidual} = \frac{(0.20)^2}{2} = 0.020$ which is 14% uncertainty.

Variance of the Fundamental Sampling Error for the primary sampling stage $s^2_{FSE1} = \frac{(0.20)^2}{4} = 0.010$ which is 10% uncertainty. This is a reasonable guideline for most base metals.

Variance of the Fundamental Sampling Error for the secondary sampling stage $s^2_{FSE2} = \frac{(0.20)^2}{8} = 0.005$ which is 7% uncertainty.

Variance of the Fundamental Sampling Error for the tertiary sampling stage $s^2_{FSE3} = \frac{(0.20)^2}{16} = 0.0025$ which is 5% uncertainty. And so on…

Above calculations are summarized in table 1.

Table 1 Guidelines for exploration, grade control and environmental sampling

Various types of uncertainties ⇩	**Precious metals, impurities and preliminary investigations in environmental sampling**	**Base metals, by-products, and for environmental sampling, law enforcement** and **litigation cases**
Total Allotted Uncertainty (Relative%) and **Variance**	32% $s^2_{Allotted} = (0.32)^2 = 0.1024$	20% $s^2_{Allotted} = (0.20)^2 = 0.040$
Residual Total Uncertainty and **Variance from all** *IDEs, IEEs, IPEs, IWEs, GSEs, AEs*	23% $s^2_{\mathrm{Re}sidual} = \frac{(0.32)^2}{2} = 0.0512$	14% $s^2_{\mathrm{Re}sidual} = \frac{(0.20)^2}{2} = 0.020$
Allotted Uncertainty and Variance for ***FSE₁*** (primary sampling stage)	16% $s^2_{FSE1} = \frac{(0.32)^2}{4} = 0.0256$	10% $s^2_{FSE1} = \frac{(0.20)^2}{4} = 0.010$
Allotted Uncertainty and Variance for ***FSE₂*** (secondary sampling stage)	11% $s^2_{FSE2} = \frac{(0.32)^2}{8} = 0.0128$	7% $s^2_{FSE2} = \frac{(0.20)^2}{8} = 0.005$
Allotted Uncertainty and Variance for ***FSE₃*** (tertiary sampling stage) Etc…	8% $s^2_{FSE3} = \frac{(0.32)^2}{16} = 0.0064$	5% $s^2_{FSE3} = \frac{(0.20)^2}{16} = 0.0025$

Distribution of the Total Allotted Variance for material balance and process control

Similar calculations can be made for sampling problems for metallurgical accounting, and they are summarized in table 2.

Table 2 Guidelines for material balance and process control

Various types of uncertainties ⇩	**Precious metals** and **impurities**	**Base metals, by-products** and **process control parameters**
Total Allotted Uncertainty (Relative%) and **Variance**	10% $s^2_{Allotted} = (0.10)^2 = 0.010$	5% $s^2_{Allotted} = (0.05)^2 = 0.0025$
Residual Total Uncertainty and **Variance from all** ***IDEs, IEEs, IPEs, IWEs, GSEs, AEs*** **and** ***HFE2*** **and** ***HFE3***	7% $s^2_{Residual} = \frac{(0.10)^2}{2} = 0.0050$	3.5% $s^2_{Residual} = \frac{(0.05)^2}{2} = 0.00125$
Allotted Uncertainty and Variance for ***FSE1*** (primary sampling stage)	5% $s^2_{FSE1} = \frac{(0.10)^2}{4} = 0.0025$	2.5% $s^2_{FSE1} = \frac{(0.05)^2}{4} = 0.000625$
Allotted Uncertainty and Variance for ***FSE2*** (secondary sampling stage)	3.5% $s^2_{FSE2} = \frac{(0.10)^2}{8} = 0.00125$	1.8% $s^2_{FSE2} = \frac{(0.05)^2}{8} = 0.0003125$
Allotted Uncertainty and Variance for ***FSE3*** (tertiary sampling stage) Etc…	2.5% $s^2_{FSE3} = \frac{(0.10)^2}{16} = 0.000625$	1.3% $s^2_{FSE3} = \frac{(0.05)^2}{16} = 0.0001563$

Distribution of the Total Allotted Variance for commercial sampling

Similar calculations can be made for sampling industrial commodities, and they are summarized in table3.

Table 3 Guidelines for commercial sampling

Various types of uncertainties ⇩	Precious metals and impurities	Base metals, by-products and process control parameters
Total Allotted Uncertainty (Relative%) and **Variance**	3% $s^2_{Allotted} = (0.03)^2 = 0.0009$	1% $s^2_{Allotted} = (0.01)^2 = 0.0001$
Residual Total Uncertainty and **Variance from all** ***IDEs, IEEs, IPEs, IWEs, GSEs, AEs*** ***and HFE2 and HFE3***	2.1% $s^2_{Residual} = \frac{(0.03)^2}{2} = 0.00045$	0.7% $s^2_{Residual} = \frac{(0.01)^2}{2} = 0.00005$
Allotted Uncertainty and Variance for ***FSE1*** (primary sampling stage)	1.5% $s^2_{FSE1} = \frac{(0.03)^2}{4} = 0.000225$	0.5% $s^2_{FSE1} = \frac{(0.01)^2}{4} = 0.000025$
Allotted Uncertainty and Variance for ***FSE2*** (secondary sampling stage)	1.1% $s^2_{FSE2} = \frac{(0.03)^2}{8} = 0.0001125$	0.35% $s^2_{FSE2} = \frac{(0.01)^2}{8} = 0.0000125$
Allotted Uncertainty and Variance for ***FSE3*** (tertiary sampling stage) Etc…	0.75% $s^2_{FSE3} = \frac{(0.03)^2}{16} = 0.000056$	0.25% $s^2_{FSE3} = \frac{(0.01)^2}{16} = 0.0000063$

GUIDELINES AND REALISTIC OBJECTIVES

Guidelines must be realistic. It is not rare, for reasons that are sometimes political and difficult to justify that imposed uncertainty requirements become instruments of power; top company executives have the duty to adjust guidelines for such malpractice that may have negative effects on performance, morale and the wellbeing of competent personnel.

THE APPROPRIATENESS AND NECESSITY OF UNCERTAINTY

Sampling operations are error generating processes. When sampling operations are implemented correctly sampling biases should be negligible. When sampling operations are carefully planned using optimum protocols there is always a residual uncertainty that should be compatible with well-defined objectives. It should be clearly understood that the residual uncertainty can never be zero; it is minimized only to reach a balance between risk and economics. Without appropriate testing and heterogeneity studies it is difficult to reach a logical balance; in some cases we would attempt to do too much with too little and have unacceptable uncertainty, which is in fact stepping into the domain of errors; in other cases we would do too much, reaching excellent levels of uncertainty but completely unnecessary for a given project, therefore making the error of spending too much resources for futilities.

TESTS TO ESTIMATE THE VARIANCE OF THE FUNDAMENTAL SAMPLING ERROR

The appropriate estimation of the variance of *FSE* for a given sample mass or more desirable, the appropriate estimation of a sufficient sample mass for a given variance of *FSE* has been addressed by many authors over the years. A good summary of the right thing to do was presented by Pitard and François-Bongarçon (2011); this unified work is a good guideline to use to prevent unnecessary confusion, therefore unnecessary work.

CONCLUSIONS

Objectives for geologists, grade control engineers, metallurgists, sales people and regulators are by their nature very different leading to sampling protocols that are also very different. The guidelines given in this paper do not provide solutions for everyone but at least give an idea to sampling practitioners that they must give careful thinking about Data Quality Objectives (DQO); critically important decisions depend on them. It is the author's experience that too many people in industries expect sampling experts to provide DQO which are clearly the mission of upper management to define what it is that they want: it is the essence of Quality Assurance.

RECOMMENDATIONS FOR FUTURE WORK

The suggested guidelines must be carefully reviewed by members of past WCSB technical committees and competent individuals familiar with TOS, with an aim to reach a logical consensus which would further provide International Standards Organizations with valuable recommendations. How such a procedure should be approached and carried out should be a topic for forum discussions at both WCSB6 and WCSB7. These guidelines and their later modifications should also be appended to the new international sampling standard termed, DS3700 "Horizontal – Representative Sampling"(2013), (Esbensen and Julius, 2009), refining the general threshold of 20% suggested herein.

REFERENCES

Esbensen, K.H. & Julius, L.P., *Representative sampling, data quality, validation – a necessary trinity in chemometrics*. in Brown, S, Tauler, R, Walczak,R (Eds.) COMPREHENSIVE CHEMOMETRICS, Wiley Major Reference Works, vol. 4, pp.1-20. Oxford: Elsevier 2009.

Danish Standardization Authority (DS), DS 3700 HORIZONTAL – Representative Sampling.

François-Bongarçon, D., "Fishy samples: how big a sample to avoid the infamous "Poisson effect"? Fourth World Conference on Sampling and Blending. The Southern African Institute of Mining and Metallurgy, 2009.

Pitard, F.F., "Pierre Gy's Theory of Sampling and C.O. Ingamells' Poisson Process Approach, pathways to representative sampling and appropriate industrial standards", doctoral thesis in technologies, Aalborg University, campus Esbjerg, Niels Bohrs Vej 8, DK-67 Esbjerg, Denmark, 2009.

Gy, P.M., L'Echantillonage des Minerais en Vrac (Sampling of particulate materials). Volume 1. Revue de l'Industrie Minerale, St. Etienne, France. Numero Special (Special issue, January 15, 1967).

Ingamells, C.O, and Pitard F.F., "Applied Geochemical Analysis". Wiley Interscience division, John Wiley and Sons, Inc., New York, 1986. 733 pages textbook.

Matheron, G., Textbook on Geostatistics; Tome 1, 1962. Notes from BRGM #14 (333 pages) and Tome 2, 1963. Notes from BRGM #24 (171 pages).

Pitard, F.F. and François-Bongarçon , D, "Demystifying the Fundamental Sampling Error and the Grouping and Segregation Error for practitioners", World Conference on Sampling and Blending: WCSB5, Santiago, Chile, 25-28 October 2011. Proceedings published by Gecamin.

Pitard, F.F., and Stevens, D., "The development of a solution to the sample preparation of coarse gold samples", World Conference on Sampling and Blending: WCSB5, Santiago, Chile, 25-28 October 2011. Proceedings published by Gecamin.