# Case studies using Visman and Ingamells sampling approaches

### F. F. Pitard<sup>a</sup>

Francis Pitard Sampling Consultants, LLC, 14800 Tejon Street, Broomfield, Colorado, 80023, USA (E-mail: <u>fpsc@aol.com</u>)

It is an undeniable fact that Visman and Ingamells's works provide valuable additions to the Theory of Sampling. This paper shows real cases where their approaches gave valuable information to better understand the complex heterogeneity of low content constituents that led to better sampling and subsampling protocols. These case studies are:

Cobalt assays in a lateritic ore led to the conclusion that some areas were very low in cobalt content. A closer look at the data using Ingamells's approach proved that conclusion completely wrong.

The estimation of low content iron in high purity ammonium paratungstate using 1-gram subsamples for the analytical method proved to be affected by a severe Poisson Process giving the illusion of a product being within specification when in fact it was a very bad product.

It should be emphasized that there are probably thousands of similar cases in many industries, as the result of economists not communicating enough with knowledgeable technical staff.

### Case study #1: A sampling diagram in a Nickel-Cobalt deposit

A lateritic nickel-cobalt deposit is drilled and assayed for its cobalt content which is an important by-product for the project. The drilling technique uses HQ-diameter tubing with a tungsten carbide drill-head, and the drilling is performed dry. Each sample is 1-meter long and weighs about 7500 grams. Some holes show very little cobalt but some others show attractive grade intercepts. Geologists were tempted to believe there were some areas much richer in cobalt than others. Table 1 shows 12 of these consecutive holes (there were many more but confidential). Each hole shows 12 consecutive 1-meter samples (there were many more but confidential). A mining test performed in the same area shown on table 1 followed by pilot plant pressure sulfuric leach reveals that the cobalt content was almost the same everywhere and slightly higher than expected. Retrospectively, looking at these existing exploration data, we may discover what created an unfortunate illusion.

### Compositing horizontally

Q = 12

We obtain 12 horizontal composites leading to the following calculations.

### Calculation of the Low Background Content for Cobalt

The harmonic means can be used to roughly estimate the Low Background Content<sup>1</sup>. Figure 1 shows the histogram of increasing cobalt contents, and the calculated value for the Low Background Content  $a_H$  (or *L* as defined by Ingamells), and a value of 0.03 % is used in the calculation of the sampling diagrams.

| 0.03 | 0.10 | 1.07 | 0.64 | 0.34 | 0.14 | 0.09 | 0.16 | 0.21 | 0.20 | 0.28 | 0.22 |
|------|------|------|------|------|------|------|------|------|------|------|------|
| 0.07 | 0.20 | 0.16 | 0.24 | 0.20 | 0.24 | 0.25 | 0.36 | 0.73 | 2.42 | 0.81 | 0.53 |
| 0.02 | 0.02 | 0.03 | 0.41 | 0.31 | 0.46 | 0.29 | 0.33 | 0.28 | 0.41 | 0.35 | 0.11 |
| 0.09 | 0.04 | 0.04 | 0.03 | 0.09 | 0.08 | 0.09 | 0.12 | 0.50 | 0.28 | 0.09 | 0.47 |
| 0.02 | 0.03 | 0.05 | 0.28 | 0.23 | 0.33 | 1.01 | 0.17 | 0.10 | 0.07 | 0.03 | 0.08 |
| 0.11 | 0.22 | 0.21 | 0.24 | 0.21 | 0.20 | 0.20 | 0.20 | 0.21 | 0.18 | 0.14 | 0.13 |
| 0.05 | 0.04 | 0.04 | 0.03 | 0.03 | 0.04 | 0.04 | 0.03 | 0.05 | 0.10 | 0.16 | 0.12 |
| 0.02 | 0.02 | 0.01 | 0.03 | 0.01 | 0.02 | 0.06 | 0.05 | 0.08 | 0.17 | 0.35 | 0.28 |
| 0.02 | 0.02 | 0.03 | 0.03 | 0.05 | 0.03 | 0.02 | 0.03 | 0.03 | 0.08 | 0.09 | 0.05 |
| 0.02 | 0.02 | 0.03 | 0.02 | 0.08 | 0.14 | 0.12 | 0.30 | 1.34 | 1.04 | 0.50 | 0.27 |
| 0.02 | 0.02 | 0.02 | 0.02 | 0.02 | 0.02 | 0.04 | 0.07 | 0.12 | 0.16 | 0.30 | 0.43 |
| 0.20 | 0.26 | 0.17 | 0.12 | 0.12 | 0.10 | 0.22 | 0.23 | 0.27 | 0.29 | 0.22 | 0.18 |

Table 1. 12 holes in a lateritic nickel-cobalt deposit. The horizontal axis shows consecutive holes, while the vertical axis shows consecutive 1-meter samples. Assays are expressed in % cobalt.



Figure 1. Calculation of the Low Background Content. *L* is Ingamells' nomenclature used in the software.  $a_{H} = L = 0.03\%$ 

Calculation of the Most Probable Result

The most probable result of an assay  $\gamma$  is calculated using formula [1] and shown in figure 2. We may observe that a single 7500-g sample gives a most probable result halfway between the estimated overall average content and the estimated Low Background cobalt content. It takes the averaging of 73 samples to eliminate the Poisson skewness.

$$\gamma = \frac{2a_L(a_L - a_H)^2 M_S + a_H \cdot IH_L \cdot a_L^2}{2(a_L - a_H)^2 M_S + IH_L \cdot a_L^2}$$
[1]

Comment about the Ingamells' nomenclature: we could use the TOS nomenclature for unknown values (i.e.,  $\gamma$ ,  $a_L$ ,  $a_H$ ) or the corresponding Ingamells' estimated value *L* instead of  $a_H$ .



## Figure 2. Calculation of the Most Probable Result $\gamma$ , illustrated as the dashed line. The low background content is the lower solid line. The overall arithmetic average is the upper solid line.

Calculation of standard deviation of the Fundamental Sampling Error

The calculation of the standard deviation of the Fundamental Sampling Error is performed using equation [2] and it is shown in figure 3. Using equation [3] we may observe that the calculated *Minimum Sample Mass*  $M_{S\min}$  is about 20 kg, which is much larger than the basic drilling support set at 7 kg. Furthermore, following the guideline given in Pitard's textbook<sup>1</sup> it would be unwise to collect a sample with less than a ±16% relative for the standard deviation of the *FSE* which leads to the collection of a 500-kg sample (71 x 7 kg). Of course, such sample can only be obtained by averaging neighboring samples until the required mass is obtained. Nevertheless, if no such averaging is performed, the Poisson Process will most certainly create very unfortunate illusions, and this is exactly what happened during the exploration and preparation of the geological block model.

$$s_{FSE}a_{L} = \sqrt{\frac{IH_{L} \cdot a_{L}^{2}}{M_{S}}}$$

$$M_{S\min} = \frac{IH_{L} \cdot a_{L}^{2}}{\left[a_{L} - a_{H}\right]^{2}}$$
[2]
[3]



### Figure 3. Calculation of the standard deviation $s_{FSE}a_{L}$

The Optimum Sample Mass  $M_{Sopt}$  is calculated using equation [4]. It is a compromise between *FSE* (necessary sample mass) and *GSE* (necessary number of samples to account for field segregation). In figure 3 it is shown to be about 100kg.

$$M_{Sopt} = \frac{IH_L}{s_{SE}^2}$$
[4]

*Conclusion*: It would be extremely unwise to collect samples smaller than 100kg, which confirms that drilling with reverse circulation or large diameter percussion into relatively soft laterites would have been a much better option.

### Calculation of the standard deviation $s_{opt}$ according to the Optimum Sample Mass

The calculation of the standard deviation  $S_{opt}$  is performed using equation [5], and it is shown in figure 4. As

suggested in the graphic, the significance of  $s_{opt}$  is meaningless below the Optimum Sample Mass, which is about 100 kg when using equation [4]. In other words, it is practically impossible to draw a logical geological block model using the information from 7-kg samples, unless a massive field moving average is used, which of course, in turn results in the loss of small-scale definition.

$$s_{opt} = \sqrt{\frac{2IH_L \cdot a_L^2}{M_S}}$$
<sup>[5]</sup>

Calculation of the standard deviation of a single assay  $S_{N=1}$ 

The calculation of the standard deviation  $S_{N=1}$  is performed using equation [6], and it is shown in figure 5. The spread of  $S_{N=1}$  as the sample mass becomes very large gives an indication of the amount of large-scale segregation that was present in the field in the area under study, which is anywhere between 0.12% and 0.28% cobalt. This domain is where Geostatistics should perform very well. Now the Ingamells' sampling diagram is complete, and as presented it is reasonably consistent with the TOS. We may debate the accuracy of the information, nevertheless, it is obvious the sampling diagram contains enormous amount of valuable information, and it is the general message that counts to select a reasonable field sampling strategy.

$$s_{N=1} = \sqrt{\frac{IH_L \cdot a_L^2}{Ms} + s_{SE}^2 \cdot a_L^2}$$
[6]



Figure 4. Calculation of the standard deviation  $S_{0 pt}$  illustrated as the dashed curves.

A similar study can be performed by compositing data vertically which can give valuable information when compared to compositing horizontally and show which preferential orientation the field segregation is.



Figure 5. Calculation of the standard deviation  $S_{N=1}$  illustrated as the dashed curves.

### Case study #2: Estimation of the iron content in high-purity Ammonium Paratungstate

The following case study involves a single stage Poisson Process. The economic consequences can already be imagined because of the non-representative assessment of the impurity content of an extremely valuable high purity material. It should be emphasized that the analytical protocol that was used was categorized as fast, cheap, and convenient. In other words, it was called a *cost-effective* analytical method. It should be emphasized that there are probably thousands of similar cases in many industries, as the consequence of economists not communicating enough with knowledgeable technical staff.

Following a dispute between the supplier and the customer a recommended test was performed. A typical 5000-g lot was assayed 80 times using the standard 1-g assay sample weight used at the supplier's laboratory. Table 2 shows all the assay values generated for this experiment.

A summary of results is as follows:

The estimated average  $\overline{x} \approx a_L$  of the 80 assays was 21 ppm.

The absolute variance  $s^2 = 378 ppm^2$ 

The relative, dimensionless variance  $s_R^2 = 0.86$ 

The absolute standard deviation s = 19 ppm

The relative, dimensionless standard deviation  $s_{\rm R}=0.93$  or 93%

| Sample<br>number | ppm Fe |
|------------------|--------|------------------|--------|------------------|--------|------------------|--------|
| 1                | 4      | 21               | 44     | 41               | 5      | 61               | 28     |
| 2                | 20     | 22               | 21     | 42               | 31     | 62               | 4      |
| 3                | 21     | 23               | 21     | 43               | 19     | 63               | 21     |
| 4                | 31     | 24               | 18     | 44               | 6      | 64               | 29     |
| 5                | 16     | 25               | 21     | 45               | 18     | 65               | 20     |
| 6                | 16     | 26               | 4      | 46               | 18     | 66               | 35     |
| 7                | 14     | 27               | 17     | 47               | 4      | 67               | 19     |
| 8                | 12     | 28               | 32     | 48               | 4      | 68               | 48     |
| 9                | 4      | 29               | 7      | 49               | 5      | 69               | 4      |
| 10               | 9      | 30               | 18     | 50               | 4      | 70               | 14     |
| 11               | 36     | 31               | 20     | 51               | 19     | 71               | 8      |
| 12               | 32     | 32               | 21     | 52               | 6      | 72               | 6      |
| 13               | 31     | 33               | 4      | 53               | 44     | 73               | 115    |
| 14               | 4      | 34               | 19     | 54               | 74     | 74               | 4      |
| 15               | 22     | 35               | 32     | 55               | 16     | 75               | 9      |
| 16               | 4      | 36               | 4      | 56               | 4      | 76               | 13     |
| 17               | 4      | 37               | 64     | 57               | 33     | 77               | 26     |
| 18               | 19     | 38               | 7      | 58               | 4      | 78               | 32     |
| 19               | 48     | 39               | 48     | 59               | 34     | 79               | 4      |
| 20               | 68     | 40               | 18     | 60               | 64     | 80               | 12     |

 Table 2. Summary of 80 replicate iron assays in high-purity ammonium paratungstate

It should be emphasized the above variances and standard deviations were calculated using conventional formulas, which may not be a very good thing to do when a Poisson Process is taking place.

From the TOS we can write the following relationship:

$$\frac{s_1^2}{a_L^2} = s_{FSE1}^2 + s_{GSE1}^2 = \left[\frac{1}{M_{S1}} - \frac{1}{M_L}\right] IH_L + s_{FSE1}^2 \gamma \xi$$
<sup>[7]</sup>

All terms are well defined in the TOS. The subscript 1 refers to the only information we have from a small sample weighing 1g. The effect of  $M_L$  is negligible since it is very large relative to  $M_S$ .

We don't know the value of the variance  $s_{GSE1}^2$  of the Grouping and Segregation Error; however, the material is well calibrated and there are no reasons for significant segregation to take place, and the isolated grains containing high iron content have about the same density as the other grains since their composition is mainly ammonium paratungstate. Therefore, we may assume in this particular case that  $s_{FSE1}^2 \ge s_{GSE}^2$  if each 1-g sample is made of several random increments, so the value of *IH*<sub>L</sub> we may calculate is only slightly pessimistic. We may write:

$$0.86 - s_{GSE}^2 \approx IH_L \tag{8}$$

We may therefore assume that  $IH_L \le 0.86$  g. If the tolerated standard deviation of the *FSE* is 16% relative, we may calculate the optimum necessary sample mass  $M_S$ :

$$M_s = \frac{0.86}{(0.16)^2} = 34g$$
[9]

Obviously, it is a long way from the 1-gram that was used for practical reasons. This mass of 34 grams is the minimum sample weight that will ensure a normal distribution of the assay results. Another parameter that can be obtained is the low background content  $a_H$  which is probably around 4 ppm as suggested by the histogram in figure 6. This high-frequency low value may sometimes represent only the lowest possible detection of the analytical method; therefore, we should remain cautious as we define the true low background content of a product for a given impurity.

### Investigation of the histogram

Figure 6 illustrates the histogram of N = 80 assays shown in table 2. In this histogram it is clear that the frequency of a given result reaches a maximum at regular intervals, suggesting that we may class the data in various zones; zone A with 27 samples showing zero grain with the iron impurity; zone B with 29 samples showing 1 grain; zone C with 13 samples showing 2 grains; zone D with 5 samples showing 3 grains; zone E with 3 samples showing 4 grains; zone F with 1 sample showing 5 grains; Zone G with 6 grains shows no event; finally zone H with 7 grains shows one event, which may be an anomaly in the model of the distribution. The set of results appears Poisson distributed, and a characteristic of the Poisson distribution is that the variance is equal to the mean:

$$s^{2} = a_{I} = 21ppm = 1.18grain / sample = \theta$$
<sup>[10]</sup>

The assumption that  $a_H = 4$  ppm needs to be checked. The probability that the lowest assay value represents  $a_H$  can be calculated. If the average number of grains showing the impurity per sample  $\theta$  is small, there is a probability that the lowest assays represent  $a_H$ . The probability that a single collected sample will have zero grain is:

$$P(x=0) = \frac{\theta^0 e^{-\theta}}{0!} = e^{-\theta} = e^{-1.18}$$
[11]

If we call P(x = 0) the probability for a success of obtaining no grain with the impurity, then the probability

 $P_x$  of *n* successes in *N* trials is given by the binomial model:

$$P_{x} = \frac{N!}{n!(N-n)!} P^{n} (1-P)^{N-n}$$
[12]

where *P* is the probability of having a sample with no grain containing the impurity when only one sample is selected, and (1-*P*) is the probability of having at least one grain when only one sample is collected; then the probability of no success  $P(x \neq 0)$  with *N* samples is:

$$P(x \neq 0) = \frac{N!}{0!(N-0)!} (e^{-\theta})^0 (1-e^{-\theta})^{N-0} = (1-e^{-\theta})^N$$
[13]

Relation [13] shows the probability that none of *N* samples is free from low-frequency impurity grains. The probability that the lowest assay value represents  $a_H$  is:

$$P(x=0) = 1 - (1 - e^{-\theta})^{N} = 0.9999$$
[14]

Assuming that  $a_H$  is not the analytical detection limit, we can be sure that the lowest assay represents  $a_H$ . Having found that the value  $\theta = 1.18$ , we may calculate the Poisson probabilities for samples located in each zone illustrated in figure 6. Thus, by multiplying each probability by 80, we may compare the calculated distribution with the observed distribution. Results are summarized in table 3.

The observed distribution is very close to the calculated distribution if we exclude the very high result showing 115 ppm which should not have appeared with only 80 samples. A characteristic of the Poisson distribution is that the variance  $s^2$  of the assays is equal to the average  $a_L$ .

$$s^2 = \overline{x} \approx a_L = \theta \tag{15}$$

or

$$s = \sqrt{\theta}$$
 [16]



But, in practice the number of grains is not used; instead, concentrations are used such as %, g/t, ppm, or ppb. Let's call *C* the conversion factor and rewrite [16] properly:

$$s = C\sqrt{\theta}$$
<sup>[17]</sup>

Thus, we may calculate the contribution C of a single average impurity grain to a single iron assay:

$$C = \frac{21}{\sqrt{1.18}} = 19\,ppm$$
[18]

### Table 3. Comparison of the calculated distribution with the observed distribution

| r     | Poisson probability for $\theta = 1.18$ | Calculated distribution | Observed distribution |
|-------|---|-------------------------|-----------------------|
| 0     | 0.307                                   | 25                      | 27                    |
| 1     | 0.363                                   | 29                      | 29                    |
| 2     | 0.213                                   | 17                      | 14                    |
| 3     | 0.084                                   | 7                       | 5                     |
| 4     | 0.025                                   | 2                       | 3                     |
| 5     | 0.006                                   | 0                       | 1                     |
| 6     | 0.001                                   | 0                       | 0                     |
| 7     | 0.0002                                  | 0                       | 1                     |
| Total | 0.999                                   | 80                      | 80                    |



Figure 7. Histogram of the 80 assays ordered by increasing iron content

Discussion of acceptable maximum for the standard deviation of the FSE

Ingamells suggested that a minimum of six of the grains showing the impurity, or a cluster of this impurity, should be present in a sample for the analysis of this sample to be meaningful. The objective of such recommendation is to eliminate the Poisson Process from damaging the database. If a 1-g sample contains an average  $\theta = 1.18$  grains, then the minimum recommended sample mass is around 5 g. Using this mass and the value of *IH*<sub>L</sub> obtained earlier we may write:

$$s_{SFE}^2 \le \frac{0.86}{5} = 0.17$$
 [19]  
 $s_{SFE} \le 0.41$  or 41% relative [20]

But, following Gy's recommendations a 34-gram sample is recommended to achieve a 16% relative standard deviation, which would contain about 41 grains. Now, how can 5-g leading to 41% relative and 34-g leading to 16% relative be reconciled?

To further discuss this difference, let's construct the useful Ingamells' sampling diagram. With the set of data given in table 2 a set of artificial, large 10-g samples made of Q=10 small one-gram samples can be created, and they are shown in table 4.

| N sample number | Composited small samples | Iron content in large samples |  |  |
|-----------------|--------------------------|-------------------------------|--|--|
| 1               | 1-10                     | 15                            |  |  |
| 2               | 11-20                    | 27                            |  |  |
| 3               | 21-30                    | 20                            |  |  |
| 4               | 31-40                    | 24                            |  |  |
| 5               | 41-50                    | 11                            |  |  |
| 6               | 51-60                    | 30                            |  |  |
| 7               | 61-70                    | 22                            |  |  |
| 8               | 71-80                    | 23                            |  |  |

Table 4. Iron content of artificial large samples of mass equal to 10 g

### Visman sampling equation

With this information Visman sampling constants A and B can be calculated:

$$S^{2} = \frac{s^{2}}{N} = \frac{A}{M_{s}} + \frac{B}{N}$$
[21]

where S is the uncertainty in the average of *N=80* assays on samples of individual mass  $M_S^* = \frac{M_S}{N} = 1g$ . [22]

A is the Visman homogeneity constant. It is the Gy's Intrinsic Constitution Heterogeneity  $IH_L$  multiplied by the square of the average content of the lot.

From the variances and Visman's equation we obtain:

$$A = IH_{L} \cdot a_{L}^{2} = \frac{\left(s_{1}^{2} - s_{2}^{2}\right)M_{S1}}{a_{L}^{2}\left(1 - \frac{1}{Q}\right)} = 379773mg / ppm^{2}$$
[23]

From Gy we suggested earlier:

$$A = IH_L a_L^2 = 0.86(21.475)^2 = 39661 \, \text{lmg} \,/ \, ppm^2$$
<sup>[24]</sup>

Those numbers are very close and within the variances precision, therefore this would suggest there is no room to calculate the amount of segregation for iron in the lot. It is wise to assume that B, the Visman segregation constant, is:

$$B = s^{2} - \frac{A}{M_{s}^{*}} = s_{GSE}^{2} a_{L}^{2} N = s_{SE}^{2} a_{L}^{2} \approx 0$$

This confirms the opinion that iron in calibrated ammonium paratungstate grains has no reason whatsoever to segregate in a substantial way, as the iron is within ammonium paratunstate salt anyway, and all the observed variability is due to the variance of *FSE*.

### The Most Probable Result

The Most Probable Result  $\gamma$  for the assaying of iron as a function of analytical sample mass  $M_s$  is calculated with Ingamells' equation 1. Values of  $\gamma$  are illustrated in figure 8 with the dashed line, and it basically represents the location of the mode of the probability distribution relative to the expected arithmetic average  $a_{L}$ .

A careful study of the  $\gamma$  curve in figure 8 (i.e., dashed curve) is the key to complete our discussion of the difference between Ingamells' recommendation and Gy's recommendation for a suggested maximum value for the standard deviation of *FSE*. It can be observed that the recommended mass by Ingamells (i.e., 6 grains in the sample) or a  $s_{FSE} = \pm 41\%$  relative leads to a location of the mode still substantially below the expected arithmetic average  $a_L$ . It is not the case with the necessary sample mass of 33 grams (i.e., 33 x  $M_S$ ) to obtain

a  $\%s_{SFE} = 16\%$  as recommended by Gy. In other words, Gy's recommendation was simply more conservative and less subjective to ambiguity.



### Figure 8. Illustration of the Ingamells' sampling diagram for ammonium paratungstate

### Conclusion and recommendations

Cases mentioned in this paper were already published in Pitard's textbook<sup>1</sup>. The only objectives of this paper is to emphasize sampling diagrams suggested by Visman and Ingamells that are not well understood by sampling practitioners around the world, though they can most certainly well illustrate complex sampling problems. Their proper use can help many industries such as, mining, industrial minerals, recycling, environment, food, pharmaceutical, etc...

Recommendations are:

- 1. Clearly understand the subtle occurrences of trace constituents of interest to make sure the potential for introducing a Poisson Process when collecting and assaying samples is prevented, or at least reasonably understood. This requires an in-depth understanding of chapters 14, 15, and 16 in Pitard's textbook<sup>1</sup>.
- 2. Sufficient data is generated to be able to make reliable statistical evaluations like those presented in this paper.
- 3. A thorough investigation of the preferred analytical method is necessary to reasonably make sure potential problems are indeed sampling problems and not analytical problems. Remember this famous quote from a brilliant analytical chemist<sup>2</sup> "The reliability of a result depends more on who produced it than on how it was done. There is no such a thing as a bad method, only bad analysts who fail to allow for its limitations."
- 4. The creation of a good, appropriate software package is necessary to generate relatively complex Ingamells' sampling diagrams.

Sampling practitioners will never regret the effort they made to acquire these valuable graphic tools.

### References

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