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Economic arguments for representative sampling in technology, industry, commerce, trade and society

Framing the Theory of Sampling (TOS) in risk assessment—an outreach perspective for economic decision makers

"Sampling is necessary every time inferences are to be made to take informed, optimal decisions in science, technology, industry, business, trade and in society at large. Some application fields where good sampling practices are a source of economic gains, such as the mining/minerals/metals industrial sectors, explicate the role of sampling more than others. But such is not the case for example in the realm of food and feed safety assessment and regulation,

where sampling is more perceived as an economic burden and a technical necessity to be fulfilled because of regulatory demands, rather than a tool needed to ensure reliable evidence to support management and regulatory decisions. Indeed, this status quo *constitutes a serious risk for society, which today must address several existential challenges posed by the accelerating climate crisis, rapid resources depletion, increasing food demand a.o. while engaging in the necessary green transition. Sampling plays an integral, but often much over-looked role, in all these fields.*

Risk assessment and sampling are essential societal competences, the first devoted to estimate and minimise safety risks, the latter devoted to estimate and mitigate sampling risks (the effects of sampling errors). A synergistic way forward for the sampling community, IPGSA, will be to position TOS as a necessary framework with an exceedingly well-proven practical tool kit, needed to ensure the best possible estimation and mitigation of risks for safety decision-making and economic risk management in all of science, technology, industry, business, trade and society, particularly regarding the UN world Sustainable Development Goals (SDG)."

Claudia Paoletti & Kim H. Esbensen (2022)

This collection of contributions from many of the leaders in representative sampling started out as a Special Section of the Sampling Column in *Spectroscopy Europe/World* on "Economic arguments for representative sampling". It appeared in Volume 33 Number 7 (2021) and remains freely available online at https://www.spectroscopyeurope.com/issues/vol-33-no-7-2021 To this Special Section have been added a further three contributions that make the case for the vital importance of taking the Theory of Sampling (TOS) into account in any sampling.

Spectroscopy Europe/World is delighted to facilitate the production of this publication.

From Kim Esbensen

The rationale behind this unique collection of articles is summed up in the first paragraph of my opening overview: "The Theory of Sampling (TOS) is all very fine, but it doesn't sell many tickets where it really counts, at CEO levels or higher (board of directors, investors, bankers). At this level decision-makers do not have the time, or cannot (or will not) make the effort to understand a **theory**. … sensing a marketing scoop, the Column Editor has asked almost more than 20 distinguished TOS illuminati to contribute to this definitive collection of 'business arguments for the TOS', writ large."

This collection took six months to put together, but it has been well worth it because of the unique opportunity to present (at least) three generations of samplers from all over science, technology, industry, commerce, trade and society. It has long been my wish to bring together the largest possible assembly of professional sampling proponents, united on a common theme. Together with invaluable help from Ian Michael and Martin Lischka (inspiring illustration wizard), we hope you will enjoy this. A tip: you can read the contributions in any order; if you, for example, are not in mining, start somewhere else—go and find what sounds most interesting to you first, to whet your appetite for the whole Special Section.

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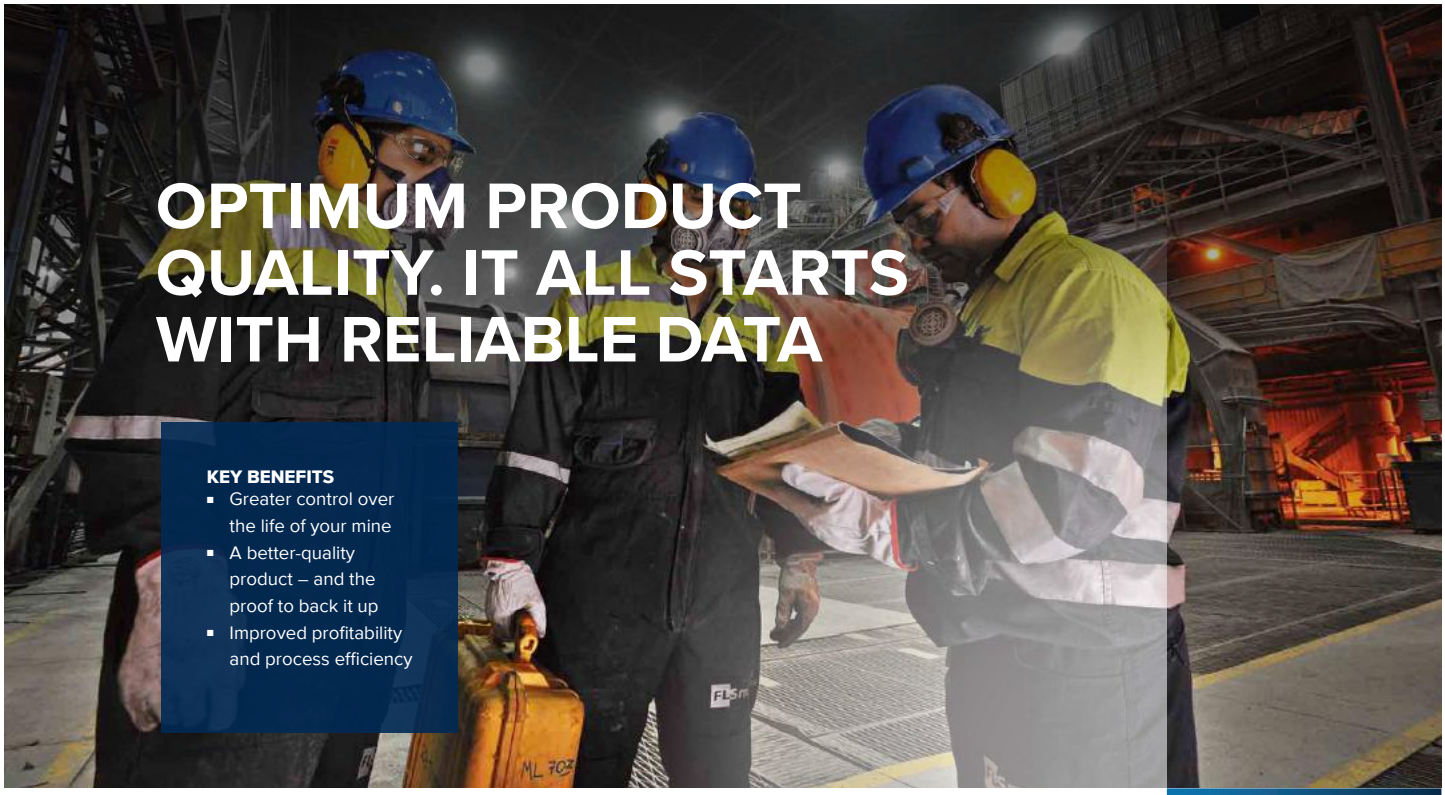

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SAMPLING SPECIAL SECTION

Economic arguments for representative sampling

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"A loss of a money is a certainty if the responsible entities have not made sure that *all* sampling and analysis performed to produce decision making information is representative. It is as simple as that…"

Sampling educator (2020)

A complaint has recently surfaced from the more business-oriented world that the sampling community mainly furthers "technological" arguments for engaging with proper sampling: "the Theory of Sampling (TOS) is all very fine, but it doesn't sell many tickets where it really counts, at CEO levels or higher (board of directors, investors, bankers). At this level decision-makers do not have the time, or cannot (or will not) make the effort to understand a **theory**." While seriously flawed and superficial, this opinion is nevertheless widespread, and especially so in those top-level decision-making circles where the sampling community would dearly like to make a greater impact! So, sensing a marketing scoop, the Column Editor has asked a distinguished group of TOS illuminati to address this economics issue head on. What follows below is the definitive collection of "business arguments for the TOS", writ large. So, read this column carefully—and forever hold your peace!

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The challenge

What is the best way to engage anyone who has never given much thought to **why** representative sampling is

critically important for most endeavours in science, technology, industry, commerce, trade and society? This is in fact a standard topic within the sampling community itself: "What is the best way to promote the TOS—not only as a theory, but as a *practical tool* to help customers? Indeed, as a critical tool that will have a significant impact on the bottom line!"

The latter casts the issue into a rather direct format: "How to *sell* TOS-compliant equipment, system solutions, consulting and audit services to customers with only little, or **no**, familiarity with the need for proper sampling?"

Ever since the inception of the TOS (in 1950) there has been a healthy discussion about this issue, about which opinions are often sharply divided. There are traditionally two types of answers: the business argument "You stand to lose a lot of money **if you don't**…"; or the technical argument: "You need to understand these critical aspects of TOS, **or else**…".

Editor's introduction: Minimum TOS understanding: heterogeneity vs sampling procedures

Kim H. Esbensen
[a]Independent researcher, consultant, owner, KHE Consulting, Copenhagen

In order to be *reliable*, business decisions must be based on *reliable* analytical results, which in turn must be based on *representative* samples from the materials, lots and process streams. Thus, in one sense everything starts with being able to conduct appropriate sampling of *all* types of materials and lots in academe, technology, industry, trade, commerce and society. "Appropriate sampling" means "representative sampling". Otherwise, "What is the meaning of analysing a sample that cannot be documented to be representative? None, there is no meaning—it is only a waste of money." As it turns out, representative sampling is only dependent on two critical success factors: i) *how to* counteract the debilitating influence on sampling from material *heterogeneity* and ii) only using *composite sampling*—**never** grab sampling. **It is as simple as that**…

In a few more words:

Sampling procedures and equipment must be able to *counteract* the vastly different degrees of *heterogeneity* encountered in all materials and lots (stationary or moving) in need of reliable compositional characterisation. **Business leaders must acknowledge, and understand, heterogeneity**.

Sampling procedures must be *representative*, i.e. *bias-free*. Of the two most common sampling approaches used today, one is demonstrably **not** so—*grab sampling*. Only *composite sampling* can be made fit-for-purpose representative for **all** materials, at **all** scales and under **all** sampling conditions. **Business leaders must understand this and decree only to use composite sampling.**

This is really all there is to it…

By investing the miniscule effort needed to understand the above, management will actually have fulfilled its role; the rest *can* be left to the technical operative levels, but it is of course unsatisfactory to lead if not reasonably well informed about *what*, *when* and *how* the raw materials and the processes involved bring about the final product.

Here follows the minimum TOS knowledge needed at all levels—it isn't much. The first issue is often highly surprising, but it opens up for the singular critical insight needed:

*Sampling of materials, processes a.o. targets for which reliable analytical results are needed is a process that is **not** quantitively reproducible, i.e. repeated sampling (two, three or more "control samplings for example" will give rise to different analytical results. There is **always** a larger or smaller* sampling variability *(call it* sampling spread *if this is clearer for the reader). Why is that?*

*Because **all** materials and processes in technology, industry and society are* heterogeneous.

Because grab sampling is fundamentally unable to counteract the intrinsic heterogeneity met with in all materials, lots and processes for which business decisions have to be made, the results will be an undesirably broad *spread* of analytical results. The unacceptable consequences of a too-broad sampling + analysis spread is laid out in full below.

One would always wish for low material heterogeneity, but it is seldom possible to alter this for original lots easily without significant, and almost always prohibitive, economic costs. Is there another way? Yes, composite sampling.

Thus, both an ill-informed sampling approach (grab sampling) and/or sampling significantly heterogeneous materials, lots and processes without proper amelioration (grab sampling) will always result in a seriously *inflated* sampling + analysis spread.

Enter the TOS, the world's only fully comprehensive framework for

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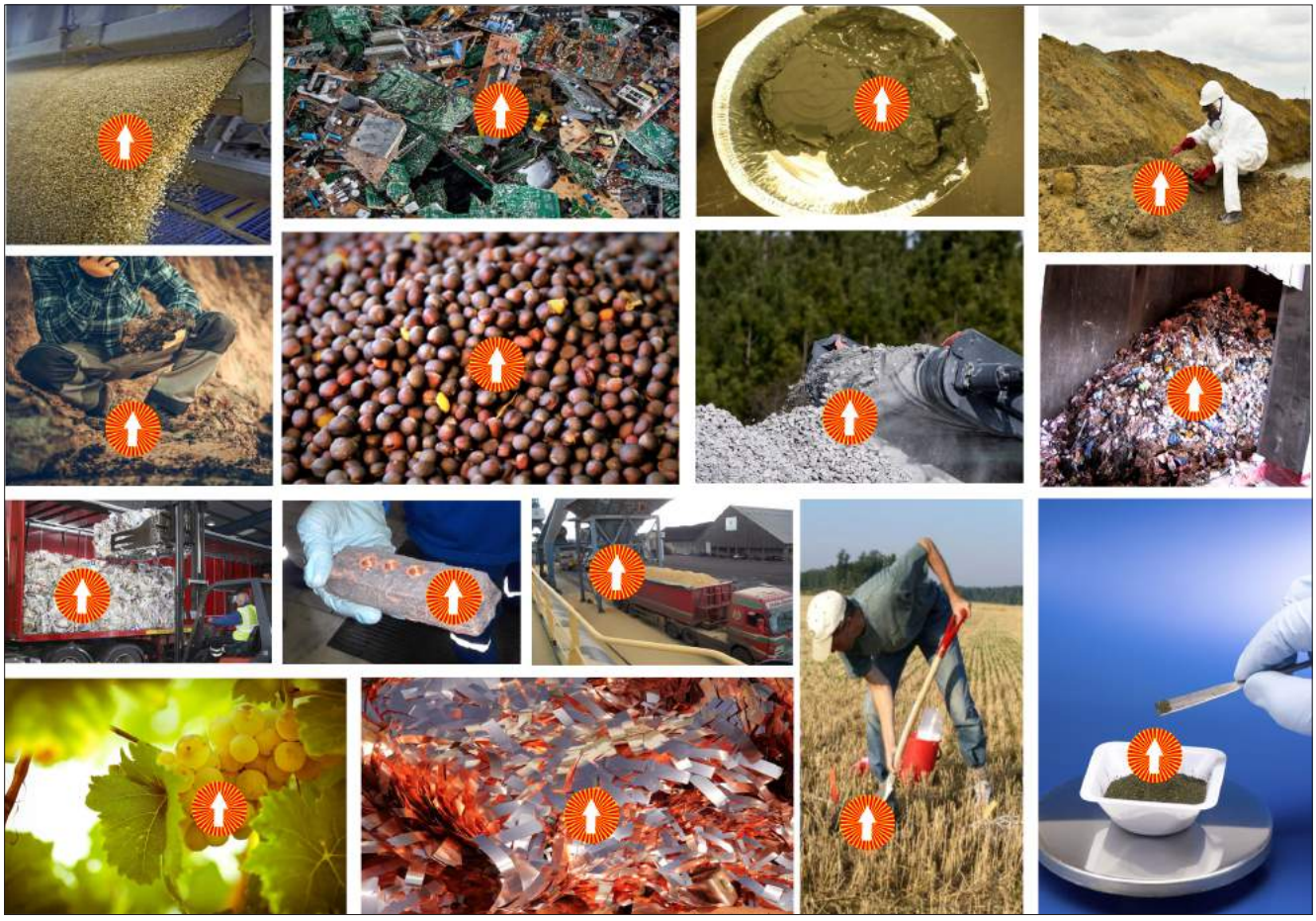


Figure A. Heterogeneous materials, lots, processes are legion and come in a plethora of forms, containers, vessels etc. Because of heterogeneity, there will always be a significant sampling variability (sampling spread). Representativity w.r.t. the whole lot demands aggregating an appropriate number of increments covering the entire lot volume. It is clear why singular grab samples will always result in different analytical results, since they are extracted from different spatial locations. Repeated grab sampling will produce a larger or smaller sampling + analysis spread. Composite samples must contain a material-dependent necessary-and-sufficient number of increments in order to secure a "fit-for-purpose" representativity status. Composite sampling will also lead to a non-vanishing sampling + analysis spread, but with a much reduced magnitude, see Figure D. The TOS is the world's only necessary-and-sufficient framework for counteracting heterogeneity in the most effective way, always leading to a minimised effective sampling spread.

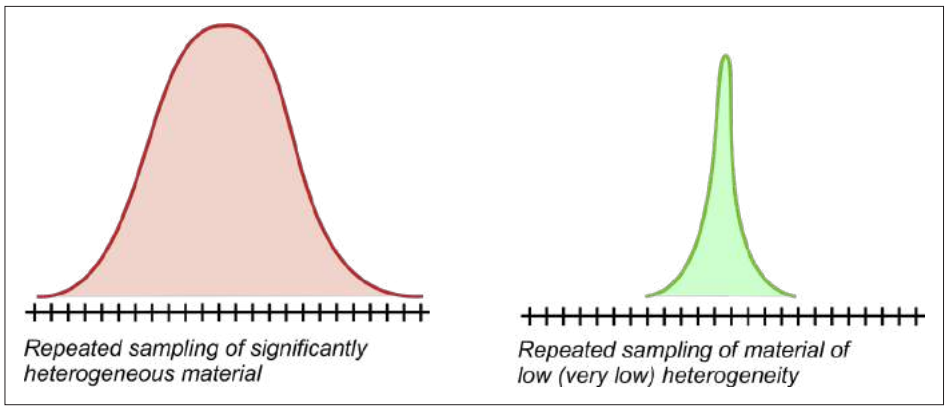


Figure B. Grab sampling of materials with widely differing heterogeneity will result in a characteristic sampling + analysis spread, the width of which is a direct reflection of the magnitude of the heterogeneity. The unacceptable business consequences of a too-broad sampling + analysis spread is laid out in full below.

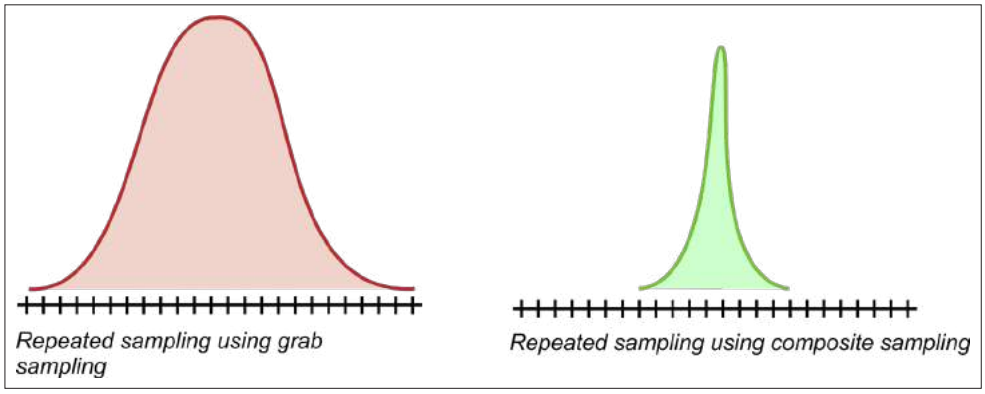


Figure C. Sampling spread as a function of using a non-representative procedure (grab sampling) compared to the world's only fully representative approach, composite sampling. The unacceptable business consequences of a too-broad sampling + analysis spread is laid out in full below.

representative sampling. The TOS stipulates **why** and **how to** use *composite sampling* for **all** materials regardless of their level of heterogeneity, Figures A and B. The TOS also outlines how to *calibrate* composite sampling (determination of the necessary-and-sufficient number of increments to aggregate) to be able to counteract heterogeneity at whatever level encountered (low, intermediate, high). The TOS is the world's only directive for how to implement representative sampling solutions that eliminate the negative effects from the two key critical success factors: heterogeneity and choice of sampling procedure.

Business decision consequences of not involving the TOS

For the reasons laid out above there is always an inherent, non-zero **risk** of making decisions based on inferior or downright wrong information, in this case numerical information (analytical results) which are fraught with unnecessary sampling + analysis uncertainty. **Risk management** is a due diligence requirement at the business level. With the few fundamentals laid bare above, risk management must include a minimum topical understanding of the risks stemming from sampling vs heterogeneity issues which all take place *before* analysis.

Making sure of optimal analytical performance is **not** enough—because the quality of analytical results depends much more on the *preceding* quality of the sampling procedures employed. Sampling uncertainties are typically 5–10–25 times *larger* than the optimised analytical laboratory performance—in direct proportionality to how well the sampling procedure has succeeded in mitigating the detrimental influence from heterogeneity—or **not**.

Inside or outside the analytical laboratory—that is the question!

Scores of examples exist of futile expansion of analytical departments with next to no additional gain in the form of improved business decision making. While knowledge and experiences with the entities behind such examples are obviously highly confidential, what can be revealed is that behind every known example there are equally many records of successful make-over operations—which all involved introduction of proper TOS knowledge to the corporation, company or organisation involved. It is difficult to put exact numbers on the economic gains (or thwarted losses) in these examples, but a start would be: What are the costs for a new analytical lab? For a significantly upgraded laboratory? For hiring one or more scientists or technicians? Compare this to now knowing for a fact that the root cause for particular bottom-line issues lies **outside** the laboratory! Enter the TOS, with which to clear up any-and-all sampling deficits—these alternative

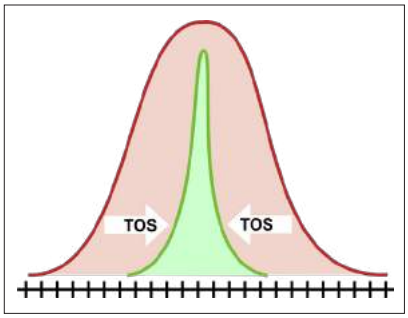


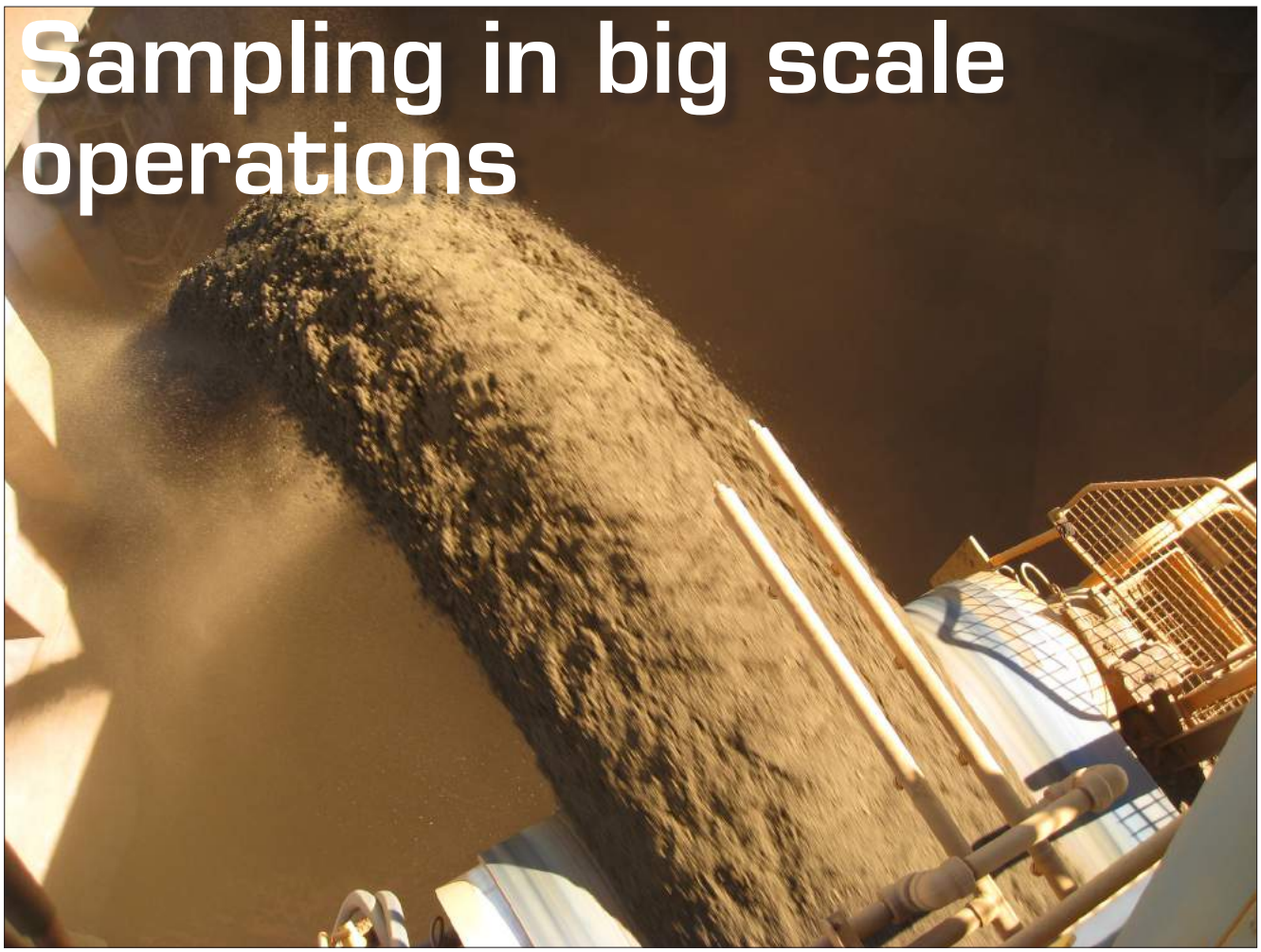
Figure D. The The solution guaranteeing representative sampling of significantly heterogeneous materials is always using appropriate sampling procedures (composite sampling)—the TOS.

costs will in most cases have difficulty reaching even a fraction of what would have been waster on the "laboratory expansion" avenue.

Resolving such issues lies at the heart of successful risk management at the top management level. The alternative, being ignorant of the consequences of not caring about the mere "technicality" of *sampling*, is an assured inferior bottom line result without anyone in the organisation being able to point to viable remediation avenues... the TOS to the fore!

To **motivate** readers to include a smattering of the TOS in the risk management setup of their operation is the very thrust of this Sampling Column. Below we present a bonanza of economic arguments for involving the TOS at all relevant levels.

SAMPLING SPECIAL SECTION



Sampling in big scale operations

Section contents

The classic publication on sampling and analysis costs in full-scale mining—Editor's summary

Pedro Carrasco, Pablo Carrasco and Eduardo Jara

Incorrect sampling operations cause huge economic losses to the mining industry, here illustrated by three industrial cases, which also show that when the Theory of Sampling (TOS) is applied correctly (ensuring unbiased sampling and analysis), considerable amounts of money can be saved.

Case 1

Sampling density influences the estimated value of mining plan alternatives

Before decisions regarding a US$640 M investment for a heap leaching facility in northern Chile, alternative spatial sampling grid densities for open pit mining of a low-grade oxide zone porphyry copper deposit (grids from $100 \times 100\ m^2$ down to $10 \times 5\ m^2$) result in markedly different Net Present Value (*NPV*) estimates spanning US$345–450 M as a function of the drilling pattern sizes. A difference between US$345 M and US$450 M, i.e. a **30% increase** in estimated resource value, is solely due to increased diligence regarding the most appropriate sampling plan, which would not have been revealed without the TOS (and geostatistics). Carrasco *et al.* a.o. conclude that "improper drilling patterns result in misleading economic decisions, e.g. wrongly dismissing good business opportunities, faulty designs of milling capacity and overestimation of waste dump capacity.

The *hidden* value loss in Case 1 is **US$105 M**.

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Case 2

Consequences of installing a TOS-compliant sampler at a tailings discharge location

A US$0.5 M TOS-compliant sampling station was installed to monitor a tailings stream in a large copper operation in central Chile. The tailings were to be sold off to another reclaiming company, so both parties have a vested interest in introducing reliable grade estimation procedures. Before installation, traditional tailing copper grade had been *assumed* to be 0.15% based on conventional metallurgical balance calculations in the preceding minerals processing pathway. The newly installed unbiased sampling station proved the earlier assumptions wrong—the actual grade turned out to be 0.20% copper. While this may seem only a relatively small deviation (an underestimation of 0.05% copper), the tailings flow rate is 96,000 tons per day, so large tonnages are involved here. But what could be worse, this underestimation had been taking place for 87 years! This difference, over this period of time, represents an accumulated loss of copper not accounted for in the company's accounts which had been assumed correct over this long period of mining business. To be fair and to count on improved technology gains a.o., it was decided to calculate the value of this loss for the last 20 years only. Based on contemporary copper prices and production costs, some 175,207 tons per year were unknowingly lost, which when calculated on an *NPV* basis amounts to a staggering **US$2207 M**.

Conclusion

Correct representative sampling practice and equipment discovered a hidden loss of a magnitude of more than **US$2 billion**. It does not take an economics degree to compare this with an investment of US$0.5 M.

Case 3

Economic consequences of a biased grade control system based on blast hole sampling

Blast hole sampling is inexpensive, efficient and often performed manually in many mining industries handling very large tonnages. This is recognised as being a major risk in the industry, but is nevertheless still often preferred from a narrow economics and logistics perspective. In Case 3 this was the established procedure in which a quickly acquired "sample" of 250 g was supposed to represent a lot of 2 tons. Amongst other things, this approach generates a huge Fundamental Sampling Error (FSE). This is a highly significant bias of unknown and inconstant magnitude amounting to ~70% of the total observable grade variability. In other words, 2/3 of the analytical information with which mining planners are supposed to work, was in reality just… *noise*. An alternative procedure (diamond drilling) is more expensive but also more accurate and precise, so deciding on introducing this would obviously depend on a reliable estimate of the accumulated losses from the blast hole approach. The mining work procedures a.o. involved classifying ores vs waste, based on a so-called "cut-off grade" of 0.40% (technical details are not relevant here, see the original publication[1]). Complicated geostatistical procedures were used to present relevant information. In terms of *PV* (present value): $PV = B^+ - B^-$ [B^+ *NPV* of wrongly sending ores to the waste dump; B^- *NPV* of, equally wrongly, sending waste to the processing mill]. Based on reliable yearly average costs and performance data, the economic calculations ultimately presented to management were as follows:

Total loss of revenue by misclassification due to blast hole sampling: $B^+ - B^- =$ **US$156 M**

Total loss of misclassification due to the alternative diamond drilling sampling: $B^{+}-B^{-}$=**US$22M**

Again, no kudos for being able to reach a conclusion in a manner appreciated by upper management.

General conclusions

General conclusions from Carrasco *et al.*[1] include:

1) Improper (non-representative) sampling practices can produce monumental value losses.
2) For a single big mining company, amounts up to US$2billion were lost over 20 years.
3) Incorrect sampling (including non-optimised analysis) not only leads to unnecessary economic inefficiency and contributes towards unsustainable exploitation of Earth's resources.
4) In the present context, focus must be on the TOS' ability to help reveal hidden value and economic losses otherwise not known to management—all realised by making sure that "…all sampling and analysis performed to produce decision making information is representative".
5) The most efficient way to discover hidden losses is to foster skill and the ability to understand the different sources of variability—and to understand that estimation is not identical to reality; there are always error effects and uncertainties. The only framework for guaranteed reduction (in optimal situations, elimination) of such adverse effects is by introducing and supporting TOS knowledge.

Read the original paper here

1. P. Carrasco, P. Carrasco and E. Jara, "The economic impact of correct sampling and analysis practices in the copper mining industry", in *Proceedings: First World Conference on Sampling and Blending (WCSB1)*, Ed by K.H. Esbensen and P. Minkkinen, *Chemometr. Intel. Lab. Syst.* **74(1),** 209–213 (2004). https://doi.org/10.1016/j.chemolab.2004.04.013

Incorrect sampling practices always have significant economic consequences—and never more so than where tonnages are large…

Ralph Holmes
Honorary Fellow, CSIRO Mineral Resources, Australia

Case 1. Even a small sampling bias can have a BIG negative economic consequence

Poor sampling procedures for iron ore can lead to preferential exclusion of coarser high grade particles from shipment samples for analysis due to cutter apertures that are too small or cutter speeds that are too high. This leads to a negative bias on Fe content—the result is that shipments are also carrying away substantial lost revenues!

Where the money comes in

Assume a small negative bias of only 0.1% Fe on an iron ore shipment of 250,000 dry tonnes at 62% Fe and an iron ore price of US$150 per tonne of contained iron.

$$\text{Financial loss} = 250{,}000 \times 0.62 \times 150 \times 0.001 = \text{US\$23,250}$$

just for one shipment! If the company loads 1000 ships in a year, i.e. exports 250 Mt/a (not unusual for a major iron ore producer), the loss then amounts to about US$23 million per annum.

The lesson: Take an even closer look at sample station design and sampling performance!

Case 2. Good risk management—but still…

Even when sampling bias has been successfully eliminated, there may still be issues due to poor sampling precision. Due to the uncertainty that persistent poor sampling precision creates in terms of shipped grades, a mining company may decide to target shipped iron ore grades at 0.25% Fe above contract grade to minimise the occurrence of off-specification shipments and associated penalties. This indeed appears to be good risk management. The company, therefore, needs to "high grade" production, but the inevitable consequence is that some low-grade blending ore, that could otherwise be sold as high-grade ore, ends up on the waste ore dump with no financial return even though the same amount of money has been spent mining this misclassified ore as for the higher-grade ore. High grading production also reduces mine life.

Where the money comes in

Assume the contract grade is 62% Fe, so the target grade has to be 62.25% Fe to minimise penalties, and the grade of the low-grade blending ore is 55% Fe. The percentage of low-grade ore (LG%) lost can be calculated from the following equation:

$$100 \times 62 = 62.25\,(100 - \text{LG\%}) + 55 \times \text{LG\%}$$

thus

$$7.25\,\text{LG\%} = 6225 - 6200 = 25$$

hence, LG% = 25/7.25 = 3.45%.

For a shipment of 250,000 tonnes of ore at 62.25% Fe, the bottom line is that

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8625 tonnes of low-grade blending ore that could have been sold as high-grade ore at 62% Fe ends up as waste. The financial loss is about US$150 × 8625 × 0.62 = US$0.8 million. With better sampling precision, the target grade can be brought closer to contract specification, thereby improving the utilisation of low-grade blending ore.

The lesson

Two examples for everybody to learn from, including higher management levels. The quest for sampling optimisation (bias elimination in Case 1 and the need to improve sampling precision in Case 2) is never over and getting it right pays welcome dividends! Understanding sampling fully is the only remedy against hidden losses, unnecessary extra operational costs, and contract and trade contract disagreements. Theory of Sampling (TOS) to the fore!

Critical sampling in the cement industry: economic drivers

Martin Lischka
HERZOG Maschinenfabrik GmbH & Co. KG, Germany

The total global cement production in 2020 was around 4.1 billion tons, making it the industrial processes sector responsible for the highest single contribution of emitted CO_2 worldwide, with no less than 27% of the directly industrial-released CO_2.[1] Modern rotary kilns in cement plants have a production capacity of 5000–10,000 t per day, and for each ton of clinker produced, ~910 kg CO_2 are emitted to the atmosphere.[2] These emissions stem from three main sources: i) decarbonisation of limestone, ii) fuel for the rotary kiln and iii) fuel for the electricity consumption of the cement plant. There is a vital sampling role hidden away in this big picture, illustrated here with five scenarios for a critical process control parameter termed "LSF" (Lime Saturation Factor), the economic impact of which is the main focus here.

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CO_2 budgets

In order to meet international agreements on climate change targets, and with introduction of "CO_2 certificate trading" in Europe in 2005, in addition to diligent process control, a new aspect for successful and economic cement plant operation arises. Due to CO_2 certificate trading, the importance of reliable sampling in cement production must be considered from the point of view of the lowest possible CO_2 production and the highest possible reliability of the data obtained.[3] Studies have shown[4] that a 5% variation in the single most important process monitoring parameter, LSF (see Technical Info Box), leads to an increase in CO_2 emissions of up to 16.4 kg CO_2/t clinker. Likewise, CO_2 emission from carbon-based fuels, by a similar 5% variation in LSF, increases by 17.2 kg CO_2/t clinker.

A sampling bias can very easily be introduced regarding the LSF, which can have severely amplified economic consequences.

The economics of it all

To illustrate the economic consequences of these technical relationships, one estimates the current financial impact based on a certificate price of €55 t^{-1} CO_2 (even

Technical Info Box

Compared to many traditional mining and minerals processing industries based on heterogeneous mineralisations and materials (e.g. base metals, gold ores), cement production is based on relatively homogeneous raw materials (clay, limestone), supplemented by a few aggregates to ensure consistent product quality. Traditionally, therefore, rather less attention has been paid to the strictness of the TOS within this industry. Sampling of the clinker is typically performed from the running process stream with a cycle of one sample per hour. After sampling, the clinker is coarsely crushed in a jaw crusher to a grain size of less than 5 mm. This allows representative sampling to reduce the sample quantity to approximately 100 g. In modern plants, samples are transported to the laboratory by pneumatic transportation. In the laboratory, sub-samples are finely ground (<45 µm) and prepared for automated X-ray fluorescence (XRF) and X-ray diffraction (XRD) analysis. To be able to use automated analysers, only about 10–15 g of sample material is needed, which is pressed into a steel ring (Ø 51.5 mm). Since the penetration depth of the analyser's X-rays is only a few micrometres, in reality only a very small portion of these few grams is analysed. It is obvious that sampling plays a critical role in this measuring system context. The effective sampling rate (clinker-to-aliquot) is closely related to the clinker production rate (see Table 1) but can be estimated as ~1 : 50,000,000—which under all circumstances is daunting.

However, the subsequent sample preparation also has a considerable influence on the analytical result. A measurable parameter for the quality of sub-sampling and sample preparation is the *standard deviation*, used as a measure of spread between replicated sampling and analysis results.

In addition to the classical elemental breakdown of chemical analysis, three so-called *moduli* are used in the cement industry for chemical classification. The most important of these is the so-called Lime Saturation Factor (LSF) which is calculated as follows:[5]

$$LSF = 100 \times CaO / (2.8 \times SiO_2 + 0.65 \times Fe_2O_3 + 1.18 \times Al_2O_3)$$

The three critical moduli are used to monitor and control the production targets. During the cement manufacturing process, heterogeneity of the intermediate products decreases continuously from the raw mixture to the finished product (good process control). The composition of the raw material mix and of the secondary fuels used are of significant importance for the clinker burning process efficiency, and also have a decisive influence on the composition of the clinker. Process control must, therefore, be carried out in such a way that the chemical and physical properties of the clinker remain as constant as possible. For this sensitive target, the quality, representativity and reliability of process sampling operations ARE of key importance.

Table 1. Estimated additional CO_2 release for different production capacities caused by erroneously determined LSFs and the financial impact in terms of CO_2 certificate price trading. These certificate costs could be saved by running the cement plant with a well-controlled process close to product specifications and with optimised power consumption.

	Rel error (%) LSF factor	Production in t/day			
		1000	2000	5000	10,000
Additional release (kg CO_2 / day)					
Clinker	1	3280	6560	16,400	32,800
	2	6560	13,120	32,800	65,600
	3	9840	19,680	49,200	98,400
	4	13,120	26,240	65,600	131,200
	5	16,400	32,800	82,000	164,000
Fuel	1	3440	6880	17,200	34,400
	2	6880	13,760	34,400	68,800
	3	10,320	20,640	51,600	103,200
	4	13,760	27,520	68,800	137,600
	5	17,200	34,400	86,000	172,000
Estimated costs for CO_2 certificate (€)					
Day	1	370	739	1848	3696
	2	739	1478	3696	7392
	3	1109	2218	5544	11,088
	4	1478	2957	7392	14,784
	5	1848	3696	9240	18,480
Year (300 days)	1	110,880	221,760	554,400	1,108,800
	2	221,760	443,520	1,108,800	2,217,600
	3	332,640	665,280	1,663,200	3,326,400
	4	443,520	887,040	2,217,600	4,435,200
	5	554,400	1,108,800	2,772,000	5,544,000

though increasing prices can be expected for the next years). The economic consequences of non-optimal LSF estimation are **huge**, as shown in Table 1. Here a relative error for the LSF ranging from 1% to 5% is considered, correlated to the simulation data given by Cao *et al.*[4] for typical daily production rates.

Highly sensitive sampling

It is very easy to introduce a significant variability in process monitoring and control if proper attention is not brought to bear—making representative process sampling essential. This can be illustrated for the same LSF parameter, based on XRF measurements. Results are presented below from an analysis repeatability test (10 analytical results from the same sample). One re-analysis shows an "accidental" higher amount of Fe_2O_3 which, however, changes the average LSF magnitude significantly, from 105.44 to 102.15. This single sample preparation variation is consequently responsible for a relative error of ~4% for the LSF, Table 2. With the economic impact of even small LSF variations as shown in Table 1, all sampling, sub-sampling and sample preparation variability is decidedly unwanted. TOS to the fore!

Insight leads to greater climate responsibility

The above economic relationships define three main goals for continuing vigilance regarding optimised cement production control to be in optimal compliance with increasingly stringent climate policy efforts, which today should be included in sustainability reports from all forward-looking cement manufacturers:

- Process and product specifications, as close as possible to minimum climate impact demands
- Design of alternative, more climate-friendly cement products
- Low-energy operation and low-CO_2 cement plant emissions

Thus, today there are both environmental, technological, economical (plant scale, global climate scale) as well as somewhat "hidden" sampling drivers for a continuously evolving cement industry—no longer mainly driven by narrow economic incentives alone. The TOS has a role to play nearly everywhere, and the economic costs for even a minor lassitude can be substantial, as was shown above (Table 1), in which a LSF uncertainty of 4% (rel) results in estimated potential additional certificate cost of **€4.4 M per year**.

There are other, non-optimised sampling issues in cement production, first and foremost primary clinker sampling. Often scoop sampling is applied in this stage, a sampling method that critically needs to be reconsidered, because a complete cross-section of the process stream is traditionally considered "almost impossible" to achieve. Remarkably there are not many publicly available clinker sampling rate estimates, nor assessments of the associated sampling errors.

References

1. IEA, *Technology Roadmap - Low-Carbon Transition in the Cement Industry*. IEA, Paris (2018). https://www.iea.org/reports/technology-roadmap-low-carbon-transition-in-the-cement-industry [accessed 30 June 2021]
2. P. Stemmermann, U. Schweike, K. Garbev and G. Beuchle, "Celitement – a sustainable prospect for the cement industry", *Cement Int.* **8(5),** 52–66 (2010). https://celitement.de/wp-content/uploads/2020/07/2010-10-26_Celitement_a_sustainable_prospect_for_the_cement_industry-1.pdf

Table 2. Routine XRF analytical results from a simple replication experiment (10 analytical aliquots prepared from the same sample) showing how easily the LSF can be impacted by non-representative sampling, preparation or analytical inconsistences. The primary clinker sampling variability must be added to this error, which is solely due to sample preparation and analysis.

Test	Al_2O_3	SiO_2	CaO	Fe_2O_3	LSF
1	4.39	20.17	67.27	2.84	**105.93**
2	4.38	20.19	67.21	2.81	**105.78**
3	4.42	20.33	67.40	2.79	**105.40**
4	4.41	20.33	67.41	2.80	**105.41**
5	4.42	20.33	67.43	2.83	**105.39**
6	4.43	20.24	67.33	2.79	**105.67**
7	4.42	20.34	67.33	2.81	**105.21**
8	4.44	20.39	66.63	4.48	**102.15**
9	4.48	20.46	67.54	2.77	**104.92**
10	4.46	20.38	67.51	2.81	**105.26**
Mean	4.42	20.32	67.31	2.97	
SD	0.03	0.08	0.25	0.50	
RSD	0.6%	0.4%	0.4%	16.9%	

3. C. Wagner and K.H. Esbensen, "A systematic approach to assessing measurement uncertainty for CO_2 Emissions from coal-fired power-plants—missing contributions from the Theory of Sampling (TOS)", *Chem. Eng. Res. Des.* **89(9),** 1572–1586 (2011). https://doi.org/10.1016/j.cherd.2011.02.028
4. Z. Cao, L. Shen, J. Zhao, L. Liu, S. Zhong and Y. Yang, "Modeling the dynamic mechanism between cement CO_2 emissions and clinker quality to realize low-carbon cement", *Resour. Conserv. Recy.* **113,** 116–126 (2016). https://doi.org/10.1016/j.resconrec.2016.06.011
5. VDZ, *Zement Taschenbuch*. Verein Deutscher Zementwerke (2008). https://www.vdz-online.de/wissensportal?tx_vdzknowledgebase_pi1%5Baction%5D=detail&tx_vdzknowledgebase_pi1%5Barticle_preview%5D=6392&tx_vdzknowledgebase_pi1%5Bcontroller%5D=Article&tx_vdzknowledgebase_pi1%5Btype%5D=0&cHash=790040ed0c1e7f3fd35d7eaa91c51373 [accessed 30 June 2021]

SAMPLING SPECIAL SECTION

Sampling of gold ores for commercial purposes

Geoff Lyman
Materials Sampling & Consulting

Let us take the example of the sampling of a gold ore coming from a small high grade deposit where the ore is to be beneficiated at a third party concentrator. There are two reasons why the ore must be sampled in an accurate manner. First, there must be a good estimate made of the contained gold so that the mine pays royalties to the state correctly. Second, the contract with the concentrator needs to pay the miner fairly for the gold contained in the ore and apply penalties for deleterious elements also contained in the ore as determined from the assays of the incoming ore. In this example, we show the impact of sample precision on the possible cash flows for the concentrator or the miner. It is assumed that the sampling is "correct", this is, that it is unbiased. The matter of whether the sampling is "representative" hangs on whether the sampling is "fit for purpose" (which is the real meaning of representative sampling) and can be judged by whether or not the economic risks faced by the parties involved are acceptable.

This example is based on an actual mine/concentrator collaboration, except that the grades and ore characteristics have been altered somewhat for reasons of confidentiality.

The ore is taken to be a difficult one containing coarse gold at a mean grade of 30 g/t and showing individual small bulk sample grades up to 180 g/t and down to less that 2 g/t. The distribution of sample grades is heavily skewed and follows an approximate log–normal distribution of grade, as might be expected. The standard deviation of the grades is very close to the mean grade. Production from the mine will be in daily 400 tonne batches which will be sequestered at the mine prior to shipment. Each batch will be sampled and assayed in order to determine if it is high enough grade to be sent to the concentrator. The ore will also be sampled again as received at the concentrator.

The critical question is how precise the daily sampling must be in order to control the risk of under- or over-payment for the ore over a period of time. The uncertainties due to sampling, sample preparation and analysis attached to the assays upon which payments are based are statistically independent and can be positive or negative and may be normally distributed. The assays can be viewed as true metal contents with a random uncertainty added to each one. From the point of view of a single assay upon which payment is made, the uncertainty may be positive or negative leading to an over-payment or under-payment, the magnitude of which is directly related to the variance (or standard deviation) of the uncertainty.

However, taking a longer-term view, it will happen that a run of positive or negative uncertainties can occur which will leave the mine or concentrator with a temporary deficit. If the concentrator is on the losing end of this run, they will be genuinely out of pocket as they will have over-paid the mine. This will have a direct impact on their cash flow as the gold they have paid for will not arrive at the bullion room. If the miner is on the losing end, he will be none the wiser unless his exploration and mine plan is so good that he can detect the fact that fewer ounces of gold have been realised from the mined ore than predicted from the mine plan. Nonetheless, he will be less well-off than he should be and this will impact his cash flow.

It is quite possible to make some simple calculations which show the extent to which the positive or negative runs of assay uncertainties can add up. Figure 1 shows five realisations of how positive or negative uncertainties can occur and add up to a significant value after a series of payments on a monthly basis. The magnitude of the deficit or surplus is measured in standard deviations of the uncertainty. In four out of the five cases, the difference from the true value has reached ten standard deviations after 60 months or 5 years or less.

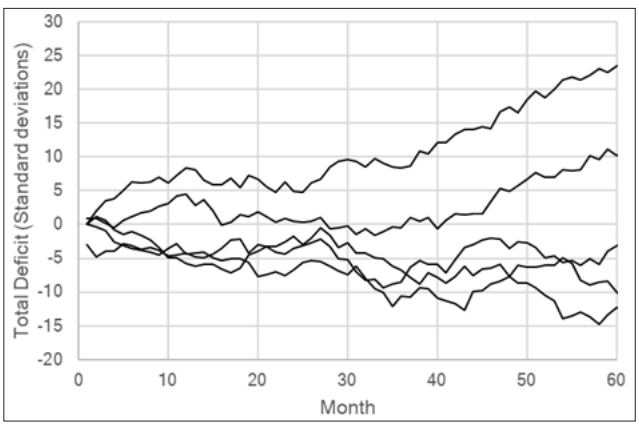

Figure 1. Random accumulation of surplus or deficit on payments in terms of assay standard deviations.

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If the little mine ships ore 5 days a week, we can count 20 days as the nominal payment period and the ore shipped will be nominally 8000 tonnes. At an average grade of 30 g/t this is 240,000 g or 7717 ounces. At a value of $1700/oz, this is $13.1 million. Now assume that the standard deviation of the uncertainty at the end of the payment period is 4%. Then one standard deviation corresponds to $0.524 million, five standard deviations to $2.62 million and ten standard deviations to $5.24 million.

While these figures are relatively small compared to the overall revenue from the mine, the value is significant and even a deficit of a few standard deviations is enough to cover the cost of a well-designed sampling system for the mine. With advance planning, a sampling system can be put together from second-hand equipment that will be capable of delivering results that might be able to improve on the uncertainty of 4% relative on the 20 day payment period.

Achieving accurate sampling of coarse gold ores

There has been much discussion of how to work out a satisfactory sampling protocol for ores containing coarse gold. There has also been debate on exactly what constitutes a "coarse" gold ore. And there has been debate on how to pulverise a gold ore containing "coarse" grains of gold without having the gold smear onto the surface of the grinding equipment with the loss of gold.

Then there is the problem of assaying a sample before or after pulverisation. There are now two methods of dealing with relatively large samples of gold ore that can be submitted for analysis without pulverisation to pass 150 µm or 106 µm.

The first is the **P**ulverise **a**nd **L**each (PAL) system that accepts a 1 kg sample of ore up to about 5 mm in size and puts it in an iron pot with grinding balls and an accelerated CN leach solution and tumbles the pot for about one hour. At the end of the tumbling, both the ground solids (now 75 µm or so) and the supernatant solution can be recovered. The solution can be analysed directly and the solids recovered, rinsed, dried, weighed and subjected to fire assay. Multiple 1 kg subsamples of the same ore can be used as determined by the analysis protocol. The advantage of the method is the large sample mass possible and the fact that there can be no loss of gold to smearing as such gold will be dissolved.

The second method is the new Photon Assay procedure brought to a commercial readiness by the CSIRO in Australia and now being rolled out in analytical labs and dedicated corporate facilities across the world. In simple terms, the method uses samples up to 500 g in mass contained in a jar and the jar is irradiated by 8–10 MeV x-rays which are highly penetrating of the ore and excite the gold nuclei which then decay with the emission of 279 keV gamma-rays, which are also highly penetrating. Multiple 500 g samples crushed only to <~2 mm can be used for an ore. The method is non-destructive. Current data show the method to be more accurate than any other methods for samples above about 1 g/t. The approximate standard deviation of an assay at 1 g/t is 2.5% relative and reduces as the sample grade increases, as indicated by available literature. The method has also been extended to Ag, Cu and moisture analysis.

Both methods are relatively cheap as sample preparation is minimised, but the PAL method does require fine assay of the residual solids to ensure that all the gold is captured.

The key to understanding the problems of gold analysis when the gold grains or gold grain clusters are coarse is to recognise that the size distribution of the gold grains/clusters controls the number of gold grains/clusters to be found in a sample of a given mass. The number of grains/clusters of a given size (or equivalent mass) in a sample follows a Poisson distribution and this fact permits calculation of the distribution of grades that will be observed over correctly sampled subsamples of the ore for the ore in the state of comminution at hand. It also permits a simple calculation of the sampling variance for the ore subsamples. It does not matter what the state of comminution the ore is in; it matters only that the size (mass) distribution of the grains be known or can be estimated with reasonable precision. Further, if it is legitimate to assume that the mass distribution of the grains/clusters can be assumed to follow the often-seen Rosin–Rammler (Weibull) distribution, the sampling variance can be written in terms of the 95% passing size of the grains/clusters, a grain/cluster shape factor and a parameter describing the breadth of the mass distribution.

In the author's development of statistical sampling theory.[1] the sampling variance due to the intrinsic (constitutional) heterogeneity can be written in terms of a sampling constant for the element of interest, K_S, as

$$\frac{\sigma_{IH}^2}{\bar{A}_L^2} = \left[\frac{1}{M_S} - \frac{1}{M_L}\right] K_S \qquad (1)$$

where the mean grade is A_L, and the sample mass is M_S, M_L is the mass of the lot from which the sample is taken and σ^2 is the sampling variance due to the element of interest. In a simple case where the gold grain mass distribution is unimodal, the sampling constant can be shown to be

$$K_S = \frac{\rho_{Au}}{A_L} f g d_{95\,Au}^3 \qquad (2)$$

where ρ_{Au} is the density of the gold, f is a shape factor, g is a size distribution factor having a value not too different from 0.25 and d_{95Au} is the 95% passing size (by mass) of the gold grains/clusters. The sampling constant has units of mass. The validity of this formulation of the sampling variance for a gold ore has been tested against the excellent (but very rare) data on gold sampling variance as a function of the top size to which the material was crushed.[1–3]

The fact that the number of gold grains in a set of gold mass fractions in an ore follow a Poisson distribution can be used to calculate the so-called characteristic function for the sampling distribution of the ore and this function can be inverted to provide the probability density function. This capability is a new tool in sampling theory that can be used to shed light on the impact of gold grain/cluster size on sampling variance

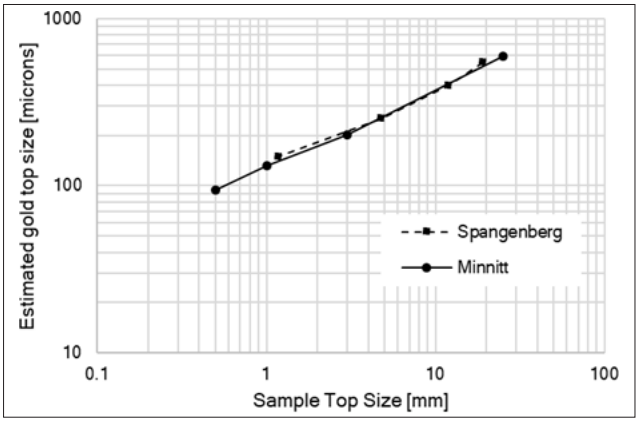

Figure 2. Gold grain/cluster top size estimated for data of Minnitt *et al.* and Spangenburg from observed sampling variance estimated from individual assays of 30 nominally identical subsamples assayed to extinction.

and particularly on the skewness of the sampling distribution.

Figure 2 shows the 95% passing size of gold grains/clusters calculated from the observed variance over 30 nominally identical subsamples at each top size for a ~12 g/t gold ore. It is likely that the gold at the larger top sizes is in the form of substantial clusters and not discrete compact grains.

The observed behaviour, virtually identical for two independent analyses of the same type for a single gold ore, shows a reasonable log–log decrease of estimated top size of the grains/clusters as a function of top size to which the ore was crushed. This permits the calculation of the sampling variance at any intermediate sizes and may permit some extrapolation to larger or smaller sizes. Clearly, what are probably clusters are being broken down with the crushing until at 0.5 mm top size the clusters have been broken into grains.

It is also interesting to compare the sampling probability density functions calculated for the data of Minnit. These are shown in Figure 3. The skewness of the distribution is clear at the 25 mm top size. Note also that the density functions calculated provided an excellent match to the actual distribution of the 30 results at each top size.

The ore characterisation provided by the method of creating set of nominally identical subsamples of the ore and analysing to extinction to permit calculation of the variance over the subsamples and *interpreting the results by the method presented here* is far more useful and sensible than attempting to interpret the data according to Gy's so-called K-α model which has caused difficulties and controversy for many years now.

To sample a gold ore and achieve a result with a controlled overall sampling variance, it is necessary to consider all sources of variance that impact the total sampling and analysis variance. The sampling of a run of mine ore is the most difficult task as the ore grade can vary substantially in the raw ore coming from one or more mining faces. The mine plan and the *in situ* grade estimation data upon which the mine plan is based is the only source of information at the early stage of mine development. It is better to over-estimate the variability than to be tempted to believe the ore is more homogeneous than it might be. Next it is mandatory to have an estimate of the ore heterogeneity as determine by the sampling constant for the ore at various top sizes to which it might be crushed. The variation of the heterogeneity (as quantified by the sampling constant) with the size to which the ore is crushed must be established by a test similar to the procedure described above. Only then can a sampling system be correctly designed in a way that will stand up to scrutiny under commercial sampling conditions.

Example

Let us take the case envisaged above and consider the design of a sampling system that will achieve very good results even when the average grade for a lot is lower than the overall average. Note that the sampling constant for the ore is

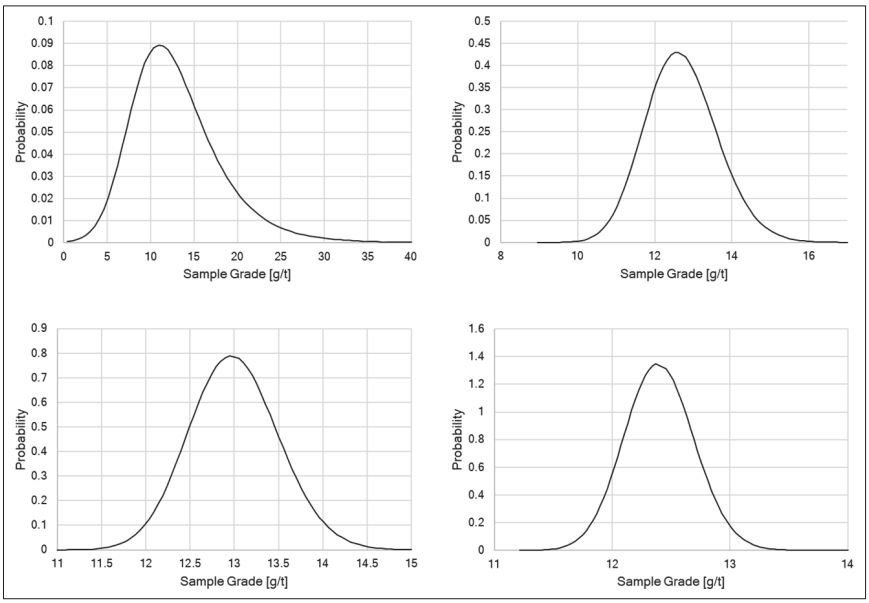

Figure 3. Sampling probability density functions for the ore at a series of top sizes to which the ore was crushed. 25.0, 3.0, 1.0, 0.5 mm, top to bottom, left to right. Sample mass is 273 g in all cases.

inversely proportional to the ore grade so that low grade or is more heterogeneous than high grade ore. With the objective of considering a somewhat worse case than average, this example will take the average grade to be 10 g/t with a standard deviation of feed to the sampling plant of 15 g/t. The lot mass for sampling is 400 t, which production from one day which is to be classified as ore or waste. The grade variation in the feed to the sampling plant will be taken to be random with the standard deviation of 15 g/t. The analysis will be assumed to be carried out by Photon Assay with a standard deviation of 1.5% relative (the grade is above 1 g/t). It will be assumed that the ore is fed to the sampling plant over a 2–3 hour period and design will be for 2 hours or primary feed. The 95% passing size of the feed is 75 mm.

The variance due to the time variation of the feed grade (distributional heterogeneity) is determined by the number of increments taken over the lot by the primary sampler.

$$\sigma^2_{DH} = \frac{\sigma^2_{feed}}{N_{inc}} \qquad (3)$$

The mass of ore collected as primary increments is determined by the feed rate, number of increments, the aperture of the primary cross-stream cutter and the velocity of the cutter through the stream as

$$M_{pri} = N_{inc}\frac{Qw}{3.6v} \qquad (4)$$

where Q is the feed rate in tph, w is the aperture in metres ($\geq 3d_{95feed}$) and v is the cutter velocity in m/s (max 0.6 m/s). The mass is given in kilograms. The primary increments are crushed to 3 mm and sampled by a secondary sampler and the collected mass of the secondary increments is determined by a similar formula.

To determine the variance due to the IH of the ore at the primary and secondary stage of sampling, Equation (1) is used with appropriate values of the sampling constant.

The optimisation of the sampling protocol is best done by setting up a spreadsheet using the formulae provided herein and then working with the number of primary increments collected and the mass divisions at each stage of sampling. It is never immediately apparent where the controlling variance will appear.

The heterogeneity of the ore is controlled by the grain/cluster sizes in the ore. In what follows it has been assumed that at effective sizes are 900, 220 and 50 µm at top sizes of 75, 3 and 0.106 mm. These are plotted in Figure 4. Also plotted are the sampling constants at the three top sizes.

The variance budget for the sampling system after optimisation is provided in Table 1.

The optimisation indicated that the most critical aspect of the system was due to primary sampling DH. It was necessary to sample at 30 second intervals to bring the variance down. This then dictated the secondary sampling, which involved feeding the primary increments collected in a bin over a 4-hour period. This change from 2 to 4 hours was dictated by the need to collect at least six secondary increments for each primary increment. The mass of primary increments collected was 7500 kg and the mass of secondary increments collected per lot was 30 kg with crushing of primary increments to 3 mm.

Table 1. Variance budget for the sampling system after optimisation.

Component	Relative variance	Relative standard deviation (%)
Primary sampling DH	9.37E-03	9.68
Primary sampling IH	2.5063E-05	0.50
Secondary sampling IH	0.000084	0.92
IH due to splitting of secondary increments	0.001186	3.44
Analysis variance by Photon Analysis	0.00005625	0.75
Total for 400 tonne lot	1.07E-02	10.36
Total for 20 lots per month		2.32

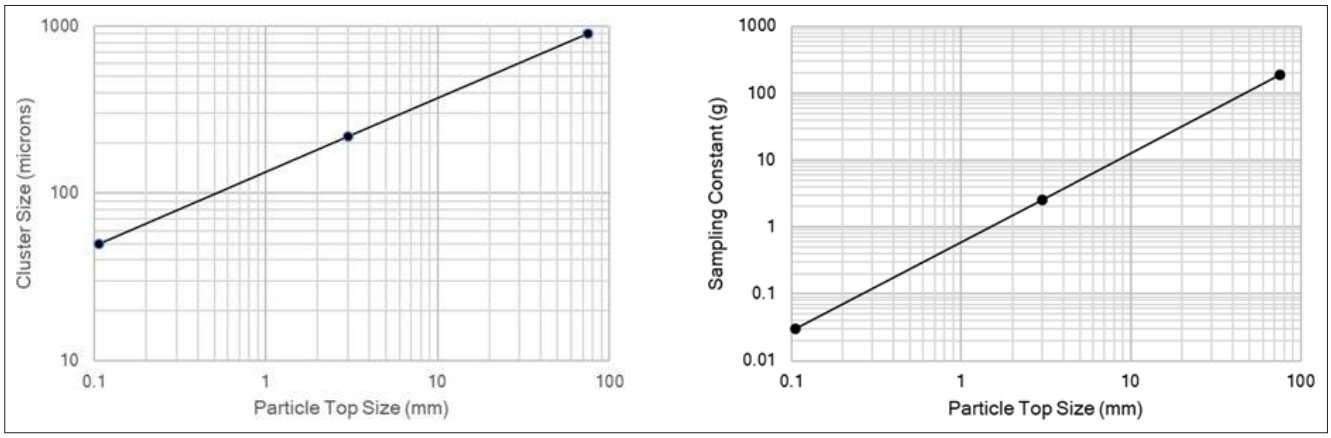

Figure 4. Assumed gold grain/cluster sizes and calculated sampling constants at 0.160, 3 and 75 mm.

The 30 kg of secondary increments was split to 2 kg at which point either four aliquots at 0.5 kg or two aliquots at 1.0 kg could be formed, the first for four replicate Photon Assays with a relative standard deviation of 1.5% per assay and the latter for duplicate 1 kg screen fire assays with pulverisation to 106 µm. The assay uncertainty for the screen fire assays was estimated to be larger than the Photon Assays, assuming a relative standard deviation for a single fire assay of 4%.

The results from this sampling example are very good for the monthly average relative standard deviation of 2.32%. It is clear that in this case, the critical issue is taking a sufficient number of primary increments from the highly variable feed. The IH of the ore manifests itself through the variance component due to splitting the ore at a size of 3 mm. Reduction of the ore past 3 mm is not necessary for Photon Assay and the Photon Assay method eliminates the sample preparation of the ore to nominally passing 106 µm with screen fire assay at 76 µm. The possible losses of gold in the preparation process are eliminated.

Conclusion

The material presented has explained the issues involved in the sampling for highly variance coarse gold ore based on heterogeneity assumptions that are in line with the heterogeneity found by Minnitt *et al*. for Witwatersrand ore. The calculations underline the fact that it is not generally possible to guess where the critical point in the sampling system design will occur and the value of having a reasonable estimate of the ore heterogeneity as a function of ore top size. The calculations highlight the value of modelling the sampling constant for the ore as a function of gold grain/cluster top size. Clusters of grains are clearly important to deal with.

References

1. G.J. Lyman, "Mathematical developments in particulate sampling theory", P.M. Gy Oration, *Ninth World Conference on Sampling and Blending*, Beijing, China (May 2019).
2. R.C.A. Minnitt, P.M. Rice and C. Spangenberg, "Part 2: Experimental calibration of sampling parameters *K* and alpha for Gy's formula by the sampling tree method", *J. S. Afr. Inst. Min. Metall.* **107,** 513–518 (2007). https://www.saimm.co.za/Journal/v107n08p513.pdf
3. G.J. Lyman, *Theory and Practice of Particulate Sampling Theory – an Engineering Approach*. Materials Sampling & Consulting, Brisbane (2019). http://www.materials-sampling-and-consulting.com/textbook

SAMPLING SPECIAL SECTION

Sampling in metals and minerals processing

Section contents

SAMPLING SPECIAL SECTION

Metal accounting: a direct link between sampling and financial management

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Editor's summary
As defined in the "Code of Practice for Metal Accounting",[1] Metal Accounting is part of Financial Accounting and helps in defining production costs and revenues, as well as stocks and WIP (Work In Progress) inventories. It is also the baseline for estimating the net value of the company. Metal Accounting is based on reconciled material balance which is itself based on critical measurements. Any uncertainty in measurement, due to the inevitable measurement error, in which the sampling error is generally the main component, results in an unwanted—and unnecessary—financial risk. Two examples are presented below together with associated economical risks and losses.

Example 1: Underestimating losses and overestimating metal in WIP

A custom copper smelter processes concentrates coming from numerous mines around the world. After blending, the concentrates are processed through a flash smelting furnace producing copper matte. The main copper losses at this stage are through slags and fumes. The latter are made up of fine particles containing copper, which are recovered and recycled to the furnace. But slags constitute a significant real loss of copper.

In this example, granulated slags were manually sampled on the conveyor belt discharge with one increment every two hours, collectively constituting a daily composite sample. To perform a quality control variogram analysis, a specific sampling campaign was performed by taking one increment every 15 min and analysing each increment. Though most increments had a copper content close to the usually observed daily average content for slags, several showed a significantly higher content corresponding to spots of matte *entrained* by slags. With the old sampling approach, such spots are *"hidden"*—hidden from view and hidden from metal accounting. For the baseline purpose it was then decided to install an automatic cross-stream sampler at the discharge end of the belt conveyor, taking one increment every 15 min.

For comparison, over one month, the old sampling method continued to be performed in parallel with the new, much more frequent approach. The average content for one month was 0.66% with the old method and 0.95% with the new automatic sampler, which is a significantly large difference when accumulating over time. It is clearly a bias as the day-by-day analysis indicated that the copper content was slightly lower for 5 days with the new sampler, but significantly higher for 13 days.

Considering a production of 1000 t/day of slag during 350 days in the year, and a price of US$9400 per ton of Cu, the value of the revealed copper loss can easily be calculated:

$$\text{Loss} = 350 \times 1000 \times (0.95\% - 0.66\%) \times 9400 =$$

US$9.541 million

This sampling issue has two financial impacts:

1) Significantly, one part of copper is reported with slags due to thermodynamic equilibrium between slag and matte phases. But another part is due to entrainment of matte with slags in the form of matte droplet. This is what occurs when observing "spots" of high copper content. This is due to poor control during the slag and matte discharge process. By observing this effect and the operating conditions when it appears, it is possible to improve the process control strategy, specifically for the quantity and quality of the furnace feed, provided the feed control, based on sampling, is sufficiently accurate to avoid such spots. The recovered monetary value will be able to pay for installing an accurate cross-stream automatic sampler, which will provide a much-improved regular analysis enhancing the possibilities for furnace control. It can also pay for a well-designed automatic sampler for feed control, or, much better, an online full-stream analyser that will drastically reduce total process sampling errors.
2) When establishing the material balance for metal accounting, such hidden losses run the risk of impacting also on the intermediate stocks (WIP) estimates. Indeed, some of these, such as matte skulls, dust or converter slags, are very difficult to measure in mass, but more seriously also for copper content. Consequently, typically no measurements are performed, or if any are carried out, they will unavoidably result in large unwanted measurement uncertainties. When running data reconciliation, the imbalance due to biased slag sampling is counterbalanced by the less accurate parameters associated with the relevant WIP, namely their Cu content. Month after month, the overestimate of Cu mass accumulates as an overestimate of the material mass and of its Cu content. At the extreme limit, Cu content can exceed 100% (physically absolutely impossible of

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course) or inventories may report large WIP masses, which *de facto* do not exist. When such discrepancies between accounted inventory and reality are revealed, an accounting adjustment will have to be made, which will decrease the value of the company and, ultimately, give it a bad reputation on the stock market—many negative cascade effects can arise from neglect of basic sampling quality requirements.

This example shows how a poor sampling procedure can hide a potential route of revenue improvement and can generate a financial risk at the level of **several millions of US$ per year**.

Example 2: Copper concentrate trading

A custom smelter buys copper concentrates following this procedure:

1) The copper concentrate delivery is accepted based on "provisional data" provided by the *seller*: the wet mass of the material, the average moisture content, the inferred dry mass and the average metal contents.
2) During the concentrate delivery unloading, the mass of the wet material is measured by the *buyer* and samples are taken for determination of moisture and metal contents, constituting the "smelter data".
3) Finally, after a few months, negotiations between seller and buyer ends with a set of mutually acceptable "final data" which are then used to calculate the objective *value* of the delivery for final invoicing. If too large discrepancies are observed between provisional data and smelter data, an umpire laboratory can be used to redo sample analysis and a conformity assessment organisation will be asked to control mass measurement systems as well.

Provisional data and smelter data come from "measurements" which are inherently uncertain for mass, moisture content and Cu content. A representative of the seller can be present during delivery unloading to *validate* the wet mass measurement and the moisture content determination. In that case, the final value for dry mass is defined during the delivery, and the negotiations are focused on metal content only.

The delivery unloading is carried out using a belt conveyor. A static belt weigher measures the mass per batch (batches are typically scaling at approximately 5 tons). The following cross-belt automatic sampler is taking one primary increment per this batch mass (5 t). 100 increments corresponding to a lot of 500 tons are combined for copper analysis.

This standard procedure gives a relatively good precision. Typical relative measurement errors are 0.21 % for the wet mass and 4.9 % for the moisture content, giving a 0.5 % error for the dry mass and 0.66 % for the Cu content. The last value corresponds to TOS-correct sampling, but it is well-known that cross-belt samplers (also named hammer samplers) **cannot** provide correct, bias-free sampling.

Considering, for example, a delivery of 17,000 tons with a provisional Cu content of 26.000 % and a smelter Cu content of 25.825 %, this analytical difference is acceptable by both parties, because it is of the same magnitude as the measurement error for Cu content. This difference corresponds approximately to a value of US$280,000 (estimated for copper alone). But **if** the smelter uses a poor-quality sampling system, its results will not be able to influence the negotiation so that the final value will be closer to the provisional value provided by the seller, which has assuredly slightly overestimated—this is not good for the smelter.

Conversely, a high-quality sampling system, associated with an efficient metal accounting system (data reconciliation reducing the uncertainty in the delivery quality estimate), will tip the scales in favour of the smelter. This positive economic difference can represent up to about **US$1 million per year** for such a wise smelter.

Conclusions

These two examples demonstrate with great economic clarity the advantage of adapting accurate (bias-free) sampling systems, based on the Theory of Sampling (TOS), as verified by *competent persons*,[1] to limit the financial risk from hidden evidentiary lacunae—and generate revenue instead. Only copper has been considered here, but other sources of revenue such as precious metals—or penalties associated with undesirable components—will also be impacted by the quality of the sampling system. This is why *any* investment in accurate and efficient measurement systems, including sampling systems, will very often be counter-balanced by the associated *revenue increase*. This will also be able to pay the costs of system maintenance, which is vital to maintain the stringent level of accuracy needed for *proper* metal accounting.

Reference

1. AMIRA, *P754: Metal Accounting – Code of Practice and Guidelines*. Release 3, February 2007.

SAMPLING SPECIAL SECTION

Loosen the TOS stipulations and face the economic consequences

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Grab sampling for material accounting

A small polymetallic mining operation intended to evaluate the potential for upgrading its mineral processing plant by adding a new metal recovery line at some strategic point in the circuit. The objective was to recover some of the companion metals as by-products of the main commodity. These potential by-products had so far been disregarded because of non-favourable market prices, relatively low grades and lack of ore-body knowledge. A geometallurgical assessment revealed that these metals were hosted in suitable mineral phases and could be easily recovered though further concentration. The circuit itself was relatively simple with very little recirculation, each stream of the circuit branching out from the main route being sent for stockpiling or disposal in waste piles. To evaluate at which location in the circuit upgrading would be optimal and which strategies to implement, a thorough *material balancing* was needed in order to assess the overall distribution of the metals in the different plant streams, and in particular in the so-called *residues*, in terms of metal grades and recoveries.

The data available for this case is a combination of on-line sensor data (flowmeters, belt weighers etc.) as well as analytical assays from samples collected at various locations in the circuit, or from some residue/wastes stockpiles; the data covers one month of operations.

A first attempt at reconciling the comprehensive data base showed huge variations between the initial and reconciliated data for the main commodity (±50%) and significant discrepancies between metal accounting and real production outputs, which were even larger for the lower grade by-product metals (deviations up to ±150%). This issue was not new and, as a consequence, an external audit was conducted in order to evaluate the sampling procedures in use.

Three main points of concerns were raised:

1) The coarsest residue streams were sampled by grab sampling "all around the perimeter" at the bottom of the stockpiles only.
2) Sampling of a hydrocyclone bank overflow. Indeed, the operator instead of collecting the *whole overflow stream* from the collecting tank, was systematically collecting the overflow of one hydrocyclone only (the most accessible one).
3) The primary pulp samples were stored in "big bags", which were not leakproof and which were in fact used to drain out the water "without losing the sample".

All of these sampling practices are in opposition to the TOS' basic principles, whereby a sample is considered representative only if all particles making up the lot have the same probability to end up in the final sample. The sampling practices revealed by the audit were clearly not in compliance with this cardinal rule and were identified as responsible for the metal accounting discrepancies observed.

The technical explanation as to *why*, and *how to* remediate these deficiencies follows.

1) Grab sampling is a very dangerous practice as it generates a range of sampling errors (GSE, IDE and IEE), most of which cannot be quantified, nor corrected for. Even though this has been known for a long time at the mine site, grab sampling was still thought to be "good enough" by the operator and by management. In the present case, the grab samples collected from the stockpiles typically weighed around 20–30 kg, too small to be considered representative! Indeed, getting a representative sample (i.e. TSE < 20%) from the corresponding stockpile, some of the coarsest streams would require at least a 20–30 ton sample. **Remedial action:** Sampling the same materials, but at the discharge point of the corresponding conveyor belt feeding the exact same stockpile, using composite sampling, would achieve a TSE of about 4% with only a 20 × 20 kg aggregated sample. This will result in a representativity which is 20/4 = 5 times improved, for one or two hundredth of the weight (i.e. *400 kg as opposed to 20–30 tons*). Not included yet in this balance is the huge time and efforts saved, which, of course, will also impact in the bottom line significantly.
2) By only extracting from the *most accessible* hydrocyclone overflow stream from a bank of six hydrocyclones, the operator was effectively only sampling one-sixth of the whole stream and, therefore, committing several serious sampling errors (GSE, IDE and IEE). Worst, these errors were *systematic* as the operator always sampled the same hydrocyclone. **Remedial action:** This is, of course, an issue that can easily be overcome by sampling the *whole* output stream—especially as the output hydrocyclone overflow collection tank is located only a few metres away from the position of the present calamity.
3) In metallurgical accounting, the moisture content and wet mass of the material being sampled is of equal importance as metal grades for determining the mass of contained metal

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in a given stream.[1,2] By allowing pulp samples to "dewater" from non-leak-proof big bags before measuring the moisture content, the operator introduced an enormous bias in moisture content estimation and, therefore, also in the estimated metal content after reconciliation. Also, this procedure resulted in a considerable loss of ultrafine particles (slimes), which "happen" to contain significant amounts of some of the desired by-product metals, but were lost with the uncontrollably leaking water.
Remedial action: This issue was easily overcome by placing the pulp samples in *impervious sealed containers* for immediate delivery to the weighing and moisture determination station.

The TOS' universally optimised 1D sampling approach

Large heaps, stockpiles or similar storage facilities cannot be sampled *in situ* (grab sampling). They can only be sampled correctly (in theory and practice) through a lot of work, such as transfer or displacement of the whole lot, which is often not practical and always costly. Indeed, such (very) large multi-modal lots are frequently also extremely heterogenous. Such industrial 3D lots must be converted into a 1D lot (in which the two width–height dimensions are negligible compared to the third processing dimension). In practice, this often means transferring *the entire lot*, without material losses, on a conveyor belt and collecting the samples during this process. This is admittedly a costly, time-consuming process, but it does guarantee representativity. With (very) large lots, there is always a desire to find a cheaper and logistically less demanding solution—always subject to the universal representativity demands.

And there is such a solution in the present case. The whole sampling problem could simply have been eliminated before the stockpile had been completed. Instead of sampling the 3D stocks, it is much easier to sample the 1D streams **before they reach** the terminal end of the conveyor belts used to build up the stockpile. The most efficient sampling always takes place while the lot is a moving stream, and this can easily be performed so as to guarantee representativity by using correctly designed, usually automatic, sample cutters at the relevant discharge point, and by applying material-dependent composite sampling. A much simpler, much cheaper and guaranteed TOS-compliant solution!

Lessons learned

In the present case, if the decision had been made based on the initial *non-representative grab samples*, the upgraded processing circuit would have been implemented at the wrong process location and the corresponding designed flowsheet would have been sub-optimal, if not useless. The incorrect sampling issues have instead been resolved with very little investment—an external audit and three automated samplers—which served both the expansion project as well as the daily production control and reconciliation obligations well.

The incorrect grab sampling practices showcased above would have resulted in unacceptable financial consequences in the form of a net loss of >2M€. This figure corresponds to the Total Investment Costs (TIC) of the initial by-product recovery circuit designed for the wrong process stream and was calculated based on simulation results through process modeling and simulation software based upon the biased data. The final TIC values are extremely sensitive to the circuit feed rate which determines the size (or number) of processing units necessary. In the present case, the feed rate of the originally selected process location was *double* that of the plant stream selected after the audit. This means that the original by-product recovery circuit was severely over-sized. Worse, ⅔ of the TIC of the original circuit was accounted for by gravity recovery equipment, which, however, is inefficient in the size range of the process stream selected, as revealed by the audit.

In the mining, and many other industries, business decisions and project evaluations are heavily dependent on representative sample collection along the entire value chain from exploration to closure.[3,4] Sampling errors are invariably larger when samples are collected and, therefore, must be representative of *several* metals or properties *simultaneously*,[5–7] such as was the case here where several new by-product metals were targeted. Only implementation of strict, TOS-compliant, sampling procedures at the earliest stage of a mining project will allow proper management of technical and economic *risks* by preparing for best possible business decisions through access to documented reliable data to be used to optimise a mine plan over the full Life of Mine (LOM) horizon—ultimately also a prerequisite for maximising the Net Present Value (NPV).

References

1. R.J. Holmes, "Correct sampling and measurement—the foundation of accurate metallurgical accounting", *Chemometr. Intell. Lab. Syst.* **74,** 71–83 (2004). https://doi.org/10.1016/j.chemolab.2004.03.019
2. D.A. Vogel and K.H. Esbensen, "WHAT are sampling errors—and WHAT can we do about them? Part 2: Sampling and weighing—different, but the same…", *Spectrosc. Europe* **33(3),** 28 (2021). https://doi.org/10.1255/sew.2021.a16
3. F.F. Pitard, *Theory of Sampling and Sampling Practice*, 3rd Edn. CRC Press, Boca Raton, Florida (2019). https://doi.org/10.1201/9781351105934
4. K.H. Esbensen, "TOS reflections: is there a third way?", *TOS Forum* **10,** 21 (2020). https://doi.org/10.1255/tosf.122
5. Q. Dehaine, L.O. Filippov and J.J. Royer, "Comparing univariate and multivariate approaches for process variograms: A case study", *Chemometr. Intell. Lab. Syst.* **152,** 107–117 (2016). https://doi.org/10.1016/j.chemolab.2016.01.016
6. Q. Dehaine and L. Filippov, "A multivariate approach for process variograms", *TOS Forum* **5,** 169 (2015). https://doi.org/10.1255/tosf.76
7. T.M.K. Le, Q. Dehaine, B. Musuku and N. Schreithofer, O. Dahl, "Sustainable water management in mineral processing by using multivariate variography to improve sampling procedures", *Miner. Eng.* **172,** 107136 (2021). https://doi.org/10.1016/j.mineng.2021.107136

SAMPLING SPECIAL SECTION

Costs of inferior sampling related to calibration for optimal mineral sorting

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This example originates from the mining industry with some parallels to the previous exploration example.

Decision and routing of material into ore and waste streams is achieved using dedicated Particle Ore Sorting (POS). POS is a mineral processing method, where particles in a stream are identified individually by a sensor-based detection technology (e.g., X-ray transmission or near infrared spectrometry) and—based on binary classification into ore and waste—are separated using targeted pulses of compressed air (Figure 1).

POS is often physically located separately from other functional units of the process, such as milling and flotation. A POS process island as shown in Figure 2 usually comprises of crushing, screening, sorting and auxiliary equipment, such as the compressor station delivering the compressed air for the physical separation process.

The efficiency of POS depends on two fundamental factors. One is the detection efficiency, i.e. the reliability with which the equipment correctly classifies ore as ore and waste as waste. The other factor is the efficiency of the pneumatic physical separation process.

The *value* created by a sensor-based ore sorting process often lies in the rejection of *marginal waste*. The inherited value would not justify spending the costs of processing and is described by the so-called cut-off grade. The sharper the sorting island can operate to this cut-off grade of the separated waste, the more economic value is created. If the grade is lower, additional mass could have been rejected, saving processing costs and debottlenecking the plant for higher grade feed which results in additional revenue. If the waste grade is too high, value is lost to the waste fraction and the ore reserve is underutilised. In mineral processor terms, high grade ore must be recovered, achieving a high recovery of the pay element(s) in question, though the focus lies on controlling the waste grade. A recommended practice is here to install a suitable sampling system on the waste material stream from the ore sorting station. What makes a POS system special in the context of the TOS is that POS processes particles sized 10 mm and larger, necessitating higher sample masses than with smaller average particle sizes due to the Fundamental Sampling Error. As a consequence, it results in the necessity to apply suitable automated mechanical sampling systems.

So far, so good. But what is the decisive increment extraction rate and what should the sampling rate be to discover fluctuations in waste grade over time with a desired fidelity? What if for half of the day the waste grade is too low and for the other half it is too high? Daily sampling would then mask this fluctuation and indicate that the waste grade is exactly on target.

A benchmark case is POS separation with high ore-to-waste and waste-to-ore misplacement, reducing recovery and increasing mass flow towards the main plant. A sampling station collecting increments for a composite day sample averages out the fluctuations of waste grade,

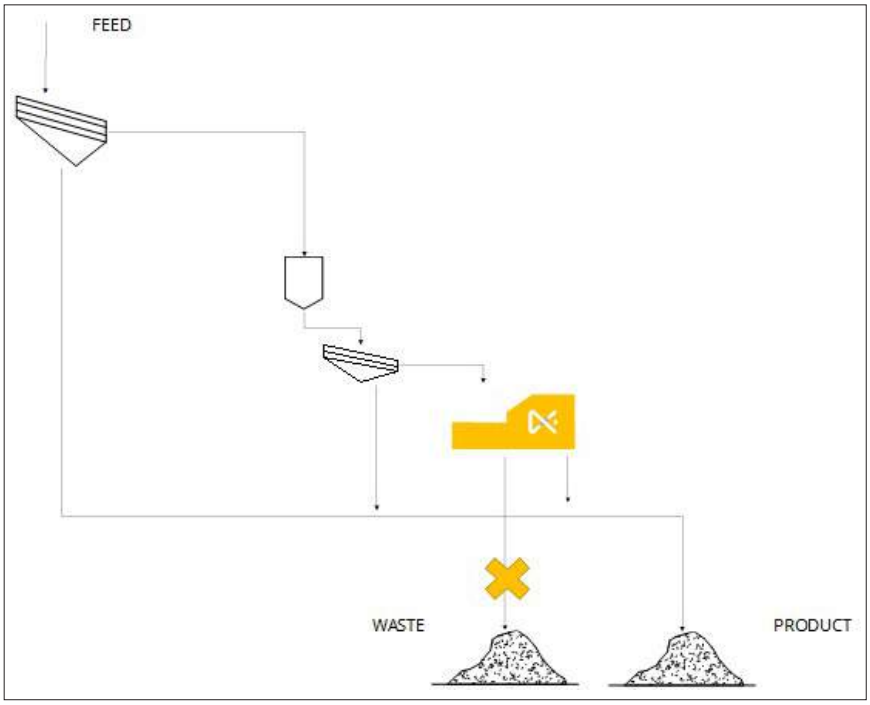

Figure 1. Simplified flow-sheet of a POS station. The undersize flows from the screens are too small for sorting and are bypassed and combined with the product fraction from the POS equipment. The cross marks the position of sampling the waste fraction.

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Figure 2. Sorting island installed at the Mittersill tungsten mine in Austria.[1]

even though a Sampling Error may be minimised according to the TOS.

An optimised case addresses optimisation of the sampling protocol. For example, by increasing the increment extraction and the accompanying assaying frequency to better monitor in-stream fluctuations over time. This is mainly an investment in the sampling, sample preparation and assaying operations. It is expected that this will be mainly a proportional increase in operating expenses. Better visibility into in-stream variation combined with a faster turnaround time of assay results makes it possible to optimise the operational parameters of the POS equipment to better follow the variation (i.e. distributional heterogeneity) over time.

This example illustrates the specific sampling challenges for POS technology due to large particle size but is in principle directly transferable to performance monitoring of all process equipment. Equipment control and optimisation must be data driven and process focused using fit-for-purpose sampling equipment and procedures to unlock value along the mineral process value chain.

NPV over identical mine life	
Base case	$712 million
Optimised case	$763 million

The increased *NPV* over the lifetime of a typical mine is $51 million: more than sufficient to pay for the efforts of designing and implementing the optimised sampling protocol and additional assay costs. This is a quite satisfactory investment on the table of any board of directors!

In mineral processing there is so much more to understand and to monitor better, with which to increase efficiency and thereby to increase both profitability as well as sustainability of the industry.

Reference

1. C. Robben and A. Mosser, *X-Ray Transmission-Based Ore Sorting at the Mittersill Tungsten Mine*. Paper presented at the 27th IMPC Chile (2014).

SAMPLING SPECIAL SECTION

The hidden costs of poor sampling in the mineral industry

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There are costs and there are costs …

There are different types of cost in sampling: CAPEX, OPEX, paybacks and hidden costs; the last one being more complicated to evaluate. CAPEX (capital expenditure) of a sampling station is easy to determine: the customer (end user or engineering firm) contacts us and/or our competitors to obtain a cost estimate. This should always contain more information about maintenance, and a spare part list so as to estimate OPEX (operational expenditure). Many examples have already been given to demonstrate and calculate paybacks of a bias-free sampling station in the mining industry for trade sampling. The magnitude of such paybacks are from a few hundred thousand euros to a few million euros per year depending on the commodity being produced (iron, copper, manganese, bauxite, coal etc.), plant capacity and, of course, the type of bias. Many case studies are presented in this collective Sampling Column.

All international sampling experts can confirm similar experiences as those reported below, while performing on-site measurements in order to (a) control brand new sampling solutions, (b) control existing sampling solutions to estimate inherent biases and (c) design the best sampling plan and technical approach to obtain representative samples. Because of the large numbers involved, as well as due to a high level of material heterogeneity, *appropriate sampling* is a well-known issue in the mining industry. Nevertheless, all the information above is necessary to convince management (technical and financial) to invest in these vital, large sampling stations.

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A worst-case scenario

In a worst case, an iron ore producer ended up losing a long-term contract with his client (steel producer) because the producer was not able to guarantee the quality of the ore over several months. None of the sampling solutions installed at the producer's port ship loading facilities complied with ISO 3082; which is the International Sampling Standard for iron ore; and neither had they been designed according to the Theory of Sampling (TOS).

But not always

Nevertheless, this is not always clear. A subcontractor was bidding for a new iron ore beneficiation plant where a sampling station compliant with ISO 3082 was required. During our technical meeting, a complete sampling station (a primary sampler and two different stages of size reduction and mass division) was presented. The project manager was looking at the drawing of the complete station and asked: *"Where is the sampler?"*. He did not understand that a complete station is required for the project and smiled back to us: *"This is not what we need. We looked at the PID and it shows a single spoon called "Sampler". We included €50,000 in our quote for this spoon."* He did not agree with our explanations and all our calculations and finally said: *"No way"*. Six months later, after all the appropriate technical aspects had been clarified between the subcontractor and the engineering firm, the project manager came back to us, requesting a quote for the complete sampling station that had been presented earlier. The final cost was more than half a million euros.

In the mineral sector

In the mineral industry, numbers and costs are at lower levels, but sampling errors and/or biases can also have important financial consequences that are equally difficult to demonstrate and evaluate at the beginning of a project: these are the *hidden costs*. We have listed below some examples seen in our few decades experience as a manufacturer of sampling solutions.

Case 1

A few years ago, a mineral processing plant decided to control its production along the full process of crushing raw material and screening them into specific size fractions. A few cross-belts and screw samplers were installed at various locations in order to control chemical composition. It was understood by everyone that these types of samplers do not comply with the TOS, nor any existing sampling standard and that the material collected cannot be representative. Nevertheless, analytical results were always "on target". Was it because the chemical composition of the product was "almost homogeneous", or because the specific size fractions collected by these non-representative samplers were the only ones of interest in the contract specifications—nobody knows!

Two years later, the plant wanted to remove manual sampling elsewhere in the plant, and management decided to install the same existing technology (screw and cross-belts), now to control product quality at their truck loading stations. The critical aspect to be controlled was the size distribution. Samples were required both for the plant's own laboratory, as well as for their client's.

The analytical results of these analyses were all way *out-of-specs*! Both laboratories went crazy. Plant management

first decided to re-process some of the product already loaded in trucks (nightmare); then decided to stop the plant for few days in order to inspect all the crushers and screens to better understand the cause of this non-conformity. The plant finally had to pay penalties to its customer for non-compliance with the contractual specifications.

The explanation is easy for anyone who is familiar with the basics of the TOS: cross-belt samplers (also called hammer samplers) were not able to collect the fine material located close to the belt, and, therefore, this type of sampling technology under-represents the proportion of fines—and the screw samplers crushed down particles having a specific size fraction due to friction on particles in the gap between the rotating screw and its casing. This increased size fractions of the small particles, resulting in the reduction of the other size fractions of larger particles. This had nothing to do with the quality of material being loaded, but was due to the sampling technology that modifies the size of some particles. The client forced the plant to improve quality control in their process because they had lost confidence and the plant was finally forced to replace these non-representative samplers by appropriate representative ones. It is difficult to estimate the hidden costs of this entire issue, but the economic consequences for the plant are very clear.

Case 2

Another mineral processing plant was built at the beginning of the 2000s. There are several process stages before the furnace, which is fed by air-slide conveyors. The size distribution of the particles feeding the furnace is controlled; especially the proportion of fine particles; so as to optimise process efficiency. A "sample taker" was installed in one of the air-slide conveyors. This sampling system is composed of a single opening with a vertical pipe in the lower part of the air-slide where material is supposed to "fall" by gravity; two valves allow material to be discharged and collected.

To better comprehend the sampling issue, understanding of the working principle is necessary. An air-slide conveys material by the means of a fluidising bed. It is composed of two casings; one above each other; separated by a fluidising grid. Air is introduced in the lower part and passes through the fluidising grid so as to create the fluidising bed. The incline of the air-slide creates and guides the flow toward the discharge end of the conveyor. Due to the airflow, turbulence creates a high level of segregation, based on both density and size of particles in the product flow. The sampler in place creates an opening in the fluidising grid with a pipe going down that guide sampled material by gravity to a sample collection vessel; two gates prevent from any pressure difference in the process.

Due to this working principle, the device collects particles located close to the grid, which are always the larger and heavier ones, while the fine particles remain in the upper part of the enclosure and will consequently follow the main stream, resulting in an under-representation of these fines. Stabilising the process has always been an issue at the plant and it is understood that the existing "sampling" equipment is not able to give the process operators the necessary accurate information (content of fine particles) to optimise their process.

A decision was made to replace the existing non-representative sampler by a TOS-compliant correct sampler. Care was taken on the flow of air as well as on the limited place available to install a new sampler. This is why a new sampling solution has been especially designed to meet these special requirements. Hidden costs are also significant in this example, but complicated to estimate in details. The only solution is: representative sampling!

Case 3

Energy is critical in mineral processing plants for two main reasons: cost and CO_2 emissions. In the lime industry, the process is composed of a kiln (vertical or rotary) to calcine limestone ($CaCO_3$) in order to remove CO_2 and obtain CaO (lime). Sampling at kiln discharge in order to measure the remaining CO_2 content (unburnt content) gives the necessary information to optimise the kiln process in terms of product quality and to reduce energy consumption. Cross-belt solutions are popular in this industry and, as said previously in Case 2, fines are not collected (or at least most of them) which creates an important bias, because they are consequently under-represented in final sample. This results in biased measurements on the unburnt content (the smaller the particle is, the better the calcination has been).

In this plant, when a part of the production is considered as being out-of-spec, it goes to waste. This part has been evaluated and fluctuates from 5% to 12% of the production. In order to reduce this waste, operators increase the heating process resulting in a significant rising cost of energy. Are they going to waste because of real poor quality or only because of a poor non-representative sampler? The solution was to install a representative sampler at belt end discharge of the conveyor located directly after the kiln, in order to be as close as possible to the heating process and to reduce the lag time between sampling and analysis.

It is again difficult to estimate the hidden cost of energy when operators increase heating process, but the numbers may be significant. Nevertheless, it was easy to calculate the payback of the sampling station based on the portion of production that was wasted in this plant; payback of the TOS-compliant sampling station was within three to four months only!

Case 4

Another lime producer received claims from its customer because the remaining CO_2 was out-of-spec in some specific size fractions (not all of them); and this issue was not constant over the time. A solution was found: measure the remaining CO_2 at the discharge of a crusher located after the kiln, so as to control the quality of each size fraction of the lime sold to the customer. A sampling station was installed and increments screened into the different size fractions of the contractual specifications; each of these was prepared individually in order to obtain a final sample representing each of all the size fractions produced and sold.

The number of claims was reduced significantly, with an obvious commercial advantage for this plant.

Conclusions

Such hidden costs due to poor sampling are common in the entire solid bulk industry. To avoid the famous "if only I had known this before!", knowledge of the good practices in sampling and in the TOS should be improved and increased at all different levels of management to give them all the tools to take the right technical and financial decisions. This does not necessarily mean to invest in solutions that are more expensive, but to better understand what is really necessary to meet their expectations and, thereby, stop losing money. Whatever the situation is—quality control, process control, metal accounting, trade—sampling is the first crucial step to reliable measurements and many decisions are taken based on these analytical results. It is worthwhile remembering a famous sentence of M. Pierre Gy: *"On primary sampling, bias can be up to 1000%, up to 50% on secondary sampling, whereas it never exceeds 0.1–1% in analysis"*. Reliable (accurate and precise) analysis requires representative samples.

The skies are clearing for the 10th World Conference on Sampling and Blending (WCSB10), 1–3 June 2022

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Sampling and the laboratory

Section contents

SAMPLING SPECIAL SECTION

The TOS—a must in the analytical laboratory (industrial, commercial, academic)

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Understanding what sampling variation is, and how it is estimated, has been a "light-bulb" moment for our analysts after having been introduced to the TOS principles.[1] So often we have had a situation where analytical work and results can be verified, but our customer still insists it doesn't meet expectations. Short of driving the poor analyst crazy with re-work tasks, which usually only produces the same "incorrect result", I now have an avenue of action that allows us to guide the customer and analysts to the path on how to focus on only taking representative samples. This is decidedly more welcome than always having to hear: "Take the sample back to the lab—repeat the analysis".

Much time is spent determining the combined total uncertainty for specific analytical methods under validation, however, very little attention is given to the preceding sampling errors and the challenges heterogeneity poses to this issue. I now know that sampling errors dominate over their analytical cousins. Also, using variographic characterisation as a quality control tool for process and measurement system monitoring is a very powerful technique that could help process controllers explain the sources of real process variations that occur on their product lines instead of simply following through by blaming the analytical lab. I found that the international standard DS 3077 (2013) and in particular its use of illustrations and industrial examples captured the true complexity of the principal types of Sampling Errors and helped to conceptualise the TOS principles in a strikingly visual way, making it easier for a typical chemical analyst to relate to the scenarios involved before analysis. After all, we have to isolate the absolutely smallest aliquot for analysis—as demanded by highly sophisticated analytical instrumentation. It is, therefore, highly surprising that the one area of greatest error affecting analysts' results is the same topic largely ignored in Analytical Chemistry/Science Training programmes, again the sampling errors. This gives rise to "brilliant" analytical results, i.e. extremely precise results, but for non-representative samples for which accuracy with respect to the lot is not accounted for. In fact the accuracy of the analytical results with reference to the original lot is completely without control—and one cannot even estimate the magnitude of the sampling bias incurred (because it is inconstant, as is another insight provided by the TOS). This makes for a very unsure analytical laboratory. After this course I wonder how many questionable results have been released by laboratories all over the world over many, many decades—and the revelations brought about by the TOS are still not known!

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Reference

1. M.C. Gouws, "Testimony", in *Introduction to the Theory and Practice of Sampling*, by K.H. Esbensen. IM Publications Open, Chichester, p. 323 (2020). https://doi.org/10.1255/978-1-906715-29-8

It does not matter what is wrong when applying TOS: it is money out of the window every time

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Gold segregation in pulps

An underground narrow **gold** vein (1–2 m width) operation was known to contain coarse gold particles up to 1.2 mm in size, and rarely up to 4 mm. The vein had an average global reserve grade of 17 g/t Au. Monthly reconciliations were up to ±50 % on grade.

From drill core and underground face chip samples, a 2 kg sample was pulverised and a 30 g fire assay undertaken. There were no formal sampling protocols or laboratory QA/QC system. With new owners, **much needed** systems were introduced into the existing laboratory. It was identified that the pulp duplicates displayed poor precision (±66 %). In addition, the pulverisers were not cleaned between samples and there was evidence of gold contamination between some samples.

Several tests were undertaken on 2 kg pulp lots, where the pile was mixed, flattened and 40 consecutive 50 g sub-samples taken for fire assay. The variability was remarkably high, and in one instance the range between the minimum and maximum values was 500 g/t Au. These findings confirmed that the pulps were **highly** heterogeneous due to the poor comminution of gold particles during pulverisation. Different pulp subsampling techniques further augmented the level of **Grouping and Segregation Error (GSE) influences. Also, day and night shifts processed pulps by two different methods**: the laboratory day shift homogenised the pulp by "mat rolling", then simply scooped off 30 g from the top of the pile, **certainly** thereby missing gold that had segregated to the bottom of the pile. The night shift placed the pulp on the mat, shook it rigorously, flattened the pile and cut a series of sample lines through the pile with a greater chance of picking up segregated gold at the pile base, **a kind of "Japanese slab cake" approach**. In essence, the "mat roll" method ***understated***, whilst the "slab cake" technique ***overstated*** the gold grade. The emphasis of day versus night shift could change between an inconsistent mix of exploration, grade control (as discussed here) and plant samples. Therefore, the negative versus positive assay bias on the grade control samples was variable.

During a four month leave of absence by the "overstating" shift manager, the understating shift manager had taken control and changed the pulp splitting to **be the new approach**. The mine records were revisited for this period, and it was found that a number of stope blocks representing *c.* 8 % of annual production had been abandoned due to the apparently low grades achieved (below the breakeven cut-off). The matter also caused production delays, as ore **supposed to be** included in the mine plan was not available.

The stope-bounding drives and raises were subsequently re-sampled using saw-cut channels and assayed using a new protocol. They were found to be of ore grade and subsequently mined out recovering 7000 oz Au. At the time of operation, these recovered ounces represented c. **US$7 M** in mid-2005 (**US$12.8 M** in July 2021). This would have been lost if the pulp issue had not been identified in a timely manner. **In addition to this tangible result, delays in the mine plan caused financial loss and previous misclassification will have caused unquantifiable loss.**

Figure 1. Screen fire assay is always a good option in the presence, or suspected presence, of coarse gold. It provides a good spatial measure of the problem. It is important to ensure that a nylon screen is used that is fire assayed to extinction. This removes sample-to-sample contamination of the screen. Duplicate or triplicate fire assays should be applied on the undersize fraction to check the level of heterogeneity—some "fine" coarse gold can still pervade the fine fraction.

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The key issue was that coarse gold needs to be treated differently.[1] Pulps bearing liberated gold cannot be homogenised; GSE can be highly problematic; and proper protocols and procedures must be set up both in the mine and in the laboratory. A screen fire assay was introduced to account for coarse gold (Figure 1), along with improved laboratory procedures and better staff training. The 2 kg pulp was split using a TOS-compliant riffle splitter to 1 kg for screen fire assay. QA/QC protocols were introduced, particularly covering equipment cleaning and contamination monitoring. Barren flushes between samples were introduced and were assayed at a rate of 1 in 20. Where visible gold was observed or high grades expected, additional barren samples were introduced and automatically subjected to fire assay. **What made the change highly economical? Introduction of proper TOS-training and procedures and responsible Good Laboratory Practice.**

Grab sampling for grade control

A shear-zone hosted underground operation had consistent reconciliation problems. Mineralisation did bear some coarse gold, though this was not dominant. Most gold was sulphide-hosted and below 200 µm in size. There was a general under-call with respect to the drilled reserve grade (7 g/t Au) of around one third.

Audit—and study the problem!

A test study was undertaken based on 200 routine grab samples collected from a 765 t stockpile. For the total population, the mean grade was 12.8 g/t Au, the minimum grade 0.01 g/t Au and the maximum grade 79.7 g/t Au. There are several grade permutations possible if an exhaustive 20 set sample batches are drawn. Out of 200 samples, the lowest grade combination of 20 samples was 0.1 g/t Au, and the highest grade 49.1 g/t Au. The mean was 10.6 g/t Au. The test stockpile was fed to the plant which has an autosampler after secondary crushing, where a batch mean head grade of 4.2 g/t Au was determined. The mean of the first grab 20 samples taken was 8.2 g/t Au, which implies under normal circumstances that the lot would have been sent to the plant as ore. Eventual plant reconciliation with the plant gave a batch grade of 3.9 g/t Au. At the time, the breakeven mine cut-off grade was 4.7 g/t Au, **which would have meant it going to waste.**

The operation was clearly battling reconciliation problems and achieving a lower head grade. The reserve model was based on diamond drill data on a 20–30 m × 20–30 m pattern. Face chip sample data was ignored, as it was biased and only represented around 50 % of mine faces due to operational constraints. As a result, all material dumped on the surface stockpiles, which included mineralised waste, and marginal, medium and high grade ore, was grab sampled prior to being sent to the waste tip or plant. Given the biased nature of grab sampling, most of the mineralised waste and marginal ore was sent to the plant diluting the ore feed. Grab sampling was considered the key issue. The grade estimate was also considered to be sub-optimal.

Figure 2. Grab sampling of gold mine stockpiles—a monumental exercise in futility!

N.B. Decisions on whether to send material from the stockpile to the plant were based *solely* on stockpile grab sampling (Figure 2). Each stockpile represented approximately 500–750 t of supposed ore. Twenty to twenty-five 3–4 kg samples (total in the range 60–100 kg) were grabbed from over stockpile at a fragment size of generally <10 cm. Each sample was sent to the laboratory for a 500 g cyanide leach (LeachWELL) pulverise-and-leach (PAL) assay.

This study showed that the use of grab samples to assess grade was problematic **in the extreme**. Most stockpiles were sent to the mill as ore. This was, in part, related to a higher proportion of gold in the fine (<1 cm) fraction, thus biasing grab samples high. An important point to note is that each grab sample or group of 20 grab samples did not represent the stockpile. Grab sampling is prone to chronic sampling errors (e.g. FSE, GSE, IDE and IEE). **FSE calculations indicated that a 25 t sample would be required from each stockpile to achieve an acceptable FSE of ±20 %.**

Improved approach: Grade control subsequently re-focussed to use the

diamond drilling, which was closed to a 12 × 12 m spacing. LeachWELL (1–2 kg) was used for all samples and grab sampling was stopped. The resource model was also improved via the use of an optimised kriged block model. A managed low-grade stockpile was introduced. As a tangible result, reconciliation improved to be within ±10 % for grade and tonnes within six months.

Where the money went: It was hard to evaluate the **unnecessary** cost effect of the grab sampling, but best estimates were that between Aus$2–4 M was lost by processing misclassified waste, and Aus$5–7M in gold lost by misclassifying ore as waste for a benchmark 12-month period—making it likely that potentially between **Aus$7M and Aus$11 M were lost per year**.

The cost of grab sampling is very nearly always high, and never higher than in gold mining operations.[2] Professional auditing is cheap compared to the amount of money saved! Lessons for upper management: if ever the term "grab sampling" is observed in a report, fire the relevant supervisor, get a professional audit, train staff at all levels on proper TOS procedures and enjoy the reaped economic benefits.

References

1. S.C. Dominy, "Sampling coarse gold-bearing mineralisation - developing effective protocols and a case study from the Ballarat mine, Australia", *Proceedings Eighth World Conference on Sampling and Blending*. The Australasian Institute of Mining and Metallurgy, Melbourne, pp. 71–84 (2017).
2. S.C. Dominy, "Grab sampling for underground gold mine grade control", *J. S. Afr. Inst. Min. Metall.* **110(6),** 277–287 (2010). https://www.saimm.co.za/Journal/v110n06p277.pdf

SAMPLING SPECIAL SECTION

Laboratory test sample representativity: an easily neglected aspect in consignment and its economic impact

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An unrepresentative sample is unable to reflect the true quality of materials and goods, and will eventually cause laboratory chemical analysis results that cannot substantiate trade settlements between buyers and sellers. In quality inspection of mineral products and metals, sampling errors account for ~80% of the total error, with sample preparation errors responsible for ~15% and analytical errors accounting for only 5%. If sampling is poorly represented, no matter how accurate the sample preparation and chemical analysis, quality grading can be severely compromised. Ignoring primary sampling, there are still significant representativity problems arising from sample preparation causing all parties difficulty when trying to find an answer to the crucial question: "where did the money go?". Thus, at the second and third stage sampling levels also, huge economic losses can occur for the buyer *or* the seller. Three cases from the international copper industry sector are presented.

Example 1: Significant trade settlement impact from sampling and grading of high purity copper cathode material

Copper cathode material is usually divided into three categories according to the content of impurities. Impurity element concentrations of Class A cathode copper shall not exceed 0.0065% in total, while lead (Pb) must not exceed 0.0005% and iron (Fe) 0.0010%. Reliable control of misrepresentation of samples used for laboratory testing has a great impact on quality grading and pricing of high pure cathode copper.

Africa is rich in non-ferrous mineral resources, especially copper mineral reserves.

Many Chinese companies have started operating in Africa, building mining plants, concentrating mills and smelters, and eventually smelting and producing copper cathode material and selling it globally. Copper cathode material is usually produced and traded directly in the original size format of 80 × 80 cm square plates with a thickness of about 1 cm, which weighs ~200 kg per piece. To ease transportation, copper cathode plates are usually strapped together using high strength steel bands into bundles suitable for loading weights of typically 1–2 tons each.

If the Class A copper cathode sampling process is contaminated by strap steel bands, as shown in Figure 1, it will lead to an excessive iron content, resulting in a grade reduction of the copper cathode products. Each quality grade class reduction results in a price reduction of ~$30 per ton. For a smelter with an annual output of 200,000 tons of copper cathode with, say, 10% of samples contaminated, the annual output value is reduced up to **$600,000**, calculated as follows:

$$\text{Economic Loss} = 30 \times 200{,}000 \times 10\% = \$600{,}000$$

Figure 1. Copper cathode plates contaminated by steel band straps. © The authors

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Product degradation will not only bring *pro rata* economic losses to the seller, but also affects its reputation as the performance stipulated in the contract turns out to be difficult or impossible to achieve, ultimately leading to a reduction in the seller's market share, with reduced corporate profits of the whole enterprise.

Example 2: Representative sampling of copper concentrates in ton bag packaging directly determines the procurement risk of the smelter

Due to poor resource endowment, low grade, difficulties in exploitation and process, Chinese copper concentrates are far from meeting the needs of domestic copper smelters. A large amount of copper concentrates are imported. Consignment copper concentrates are packaged either in bulk or in ton bags. The economic results for bulk copper concentrate consignments are relatively stable and less controversial because of the relatively easy operability of the sampling methods employed. However, due to very uneven quality fluctuation distribution *between* bags, copper concentrates packaged in *ton bags* often have large deviations (gaps) between the in-material quality (analytical results) when determined in loading and unloading ports, gaps which exceed the "reasonable" error range assumed in contracts etc.

A large smelting enterprise in China needs to import more than 500,000 tons of copper concentrates every year. Due to the type of inconsistent sampling approaches just described at loading and unloading, final copper grade difference of the two ports was up to 1.5%. In order to identify the *cause* of this apparent poor quality match, 20 tons was randomly selected from a shipment of 500 tons to serve as a basis for detailed investigations of the between-bag quality fluctuations. The resulting test results are shown in Table 1.

Between-bag copper variations (never zero) ranged up to a maximum gap of 11.16%. If the between-bag coefficient of variation (CV%) is not carefully controlled, this may easily propagate into a large compositional gap in the final results. The domestic smelter, as the buyer, takes account of large material quantities for which the compositional estimations must be determined with a very high accuracy and precision.

As an example, the smelter used copper concentrates transported by sea, with 20,000 tons per delivery; the relevant London Metal Exchange's copper price was about $9400 per ton. If the copper shipment unloading port's estimated copper concentration was 1.5% *higher* than the loading port, regardless of the impact of other valuation elements and processing fee deductions, a value of **$2.82 million** was due to the contested differently indicated amounts of copper alone.

Table 1. Copper content in randomly selected copper concentrate bags.

Bag No.	Cu (%)	Bag No.	Cu (%)
1	16.36	10	21.35
2	18.58	11	25.69
3	19.06	12	22.64
4	18.65	13	19.45
5	18.45	14	19.70
6	24.14	15	26.08
7	18.74	16	23.68
8	27.52	17	16.82
9	22.43	18	19.20
Mean	21.07		
Range	11.16		

Example 3: A detail from blister copper sampling and preparation

China has a huge need for copper raw materials. In addition to directly importing copper concentrates as raw materials for domestic production of copper cathodes, Chinese companies also construct copper smelting facilities overseas to obtain copper products such as copper blister, copper anode and copper cathode, which are also sold domestically.

The copper content in copper blister is usually around 99%. For trade purposes, copper blister is usually delivered in the form of ingots or anode plates. Generally, the content of copper, gold and silver is used as the characteristic pricing elements of the product—sometimes including other specific impurity components. The analysis is preceded by a sampling procedure which generally includes the following steps: randomly pick out a defined number of ingots from a consignment, further select a few points on each ingot for drilling out and collect all *cuttings* to become a *composite sample*, which is milled (ground) and

Figure 2. Copper blister. © The authors

from which a test sample is produced for analysis of the content of each element involved in the contractual specifications.

Both parties in the trade use analysis results as the basis for a fair trade settlement—which should always cause no issues *were* trading parties using only one analytical facility. But when using two, the road is open for possible *deviating analytical results*, which at first are sometimes difficult to understand as they manifestly represent the *same consignment*. But there is always a rational explanation, an example of which is shown below.

Blister copper sampling procedure

Occasionally when enterprises sell blister copper, the two parties agree on a proscribed sample preparation method in the trade contract as follows. Drill blister copper ingots, grind all the collected cuttings, followed by *screening* by a 40-mesh sieve, followed by further grinding of the left behind, over-sized sample part again, until the complete composite sample has passed through the screen.

Table 2. Copper content in different particle size categories.

Particle size bin	Mean copper %
>40 mesh	99.15
<40 mesh	98.85

However, the mandated method in Chinese domestic industry is to *separate* the material of the up-sieve and down-sieve size bins into identified sub-samples. According to the screened mass ratio, weighing is also carried out of the separated up-sieve and the down-sieve sub-samples, which are then analysed for copper content.

As an internal control, after a batch of samples are sieved, sub-samples of these particle size bins are tested separately to obtain their specific copper contents, as shown in Table 2. The reason for the resulting diverging results may be due to the different constituent particle sizes, or it may be a result of the repeated grinding operations, which causes the material to be *oxidised*, resulting in a lowered pure copper content for the small particle sizes.

A large copper smelter established overseas by China has an annual output of about 20 tons of copper blister. If the sample preparation method of all 40-mesh sieves is used in trade accounting, this alone may bring about a 0.3% reduction in copper content. The unit price of copper blister at the time of writing is US$9400 per ton, regardless of the influence of other pricing elements. Thus, for this smelter, this single detail of sample preparation procedures alone may represent a loss of up to **US$5.64 million** in trade **per year**. Every *detail* matters in global commodity trade ….

Conclusions

These consignment examples demonstrate the economic importance of even the smallest differences in laboratory preparation and analysis approaches. Sub-sampling, sample preparation, transportation and sample storage processes may all have significant effects on the quality and representativity of samples that eventually enter the analytical instruments. Only by careful and strict control of each operation can the test samples ultimately used for analysis be qualified as representing the full, comprehensive quality of commodities and the goods—equally in the interest of both buyers and sellers. The apparent *minute* issues treated here for a large-volume bulk commodity, may quickly lead to surprisingly large, added or lost, values which are far too large to overlook in consignment economics.

Between the laboratory and management

The reader is referred to two earlier Sampling Columns dealing with how to run a commercial analytical laboratory notably with, or without, the TOS on the agenda: Does management have the necessary foresight to accept the challenge of also caring for "the customers of the customer of the laboratory"? This is an exciting two-part story, both of which are just a click away.

K.H. Esbensen, "A tale of two laboratories I: the challenge", *Spectrosc. Europe* **30(5),** 23–28 (2018). https://doi.org/10.1255/sew.2018.a3
K.H. Esbensen, "A tale of two laboratories II: resolution", *Spectrosc. Europe* **30(6),** 26–28 (2018). https://doi.org/10.1255/sew.2018.a4

"Sampling vs analytical error: where the money is ..."

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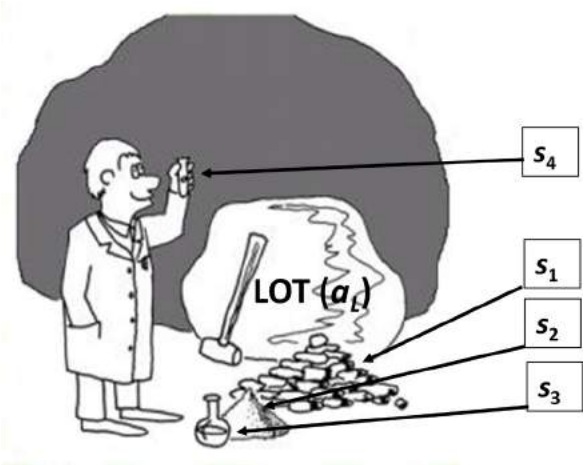

Sampling for analysis is a multi-stage operation, from extracting a primary sample (s_1) via sub-sampling (s_2) ... (s_3) towards the final analytical aliquot (s_4). At each stage, a sampling error is incurred if not properly reduced or eliminated, collectively adding to the error budget. Nobody wants the total measurement error to be larger than absolutely necessary, lest important decisions based thereupon are seriously compromised. However, it is in the interregnum between sampling and analysis where one finds plenty of usually unknown hidden costs, lost opportunities and a bonanza of bold, red figures below the bottom line. We have asked one of the peers of sampling, with extensive industrial and technological experience, to focus on the economic consequences of not engaging in proper sampling. Enjoy these "horror stories" from which we can all learn, not least at management level.

Along the full lot-to-analysis pathway

Analytical measurements comprise at least two error generating steps: delineating and extracting the primary sample, and analysis of the analytical aliquot. There may be several sub-sampling steps before having a sufficiently small aliquot (analytical sample) of the original material ready for proper analysis. In this chain of operations, the weakest link determines how reliable the analytical result is. The reason is that variances (squared standard deviations) are *additive*.

If only one primary sample is processed through *i* stages, the error variance of the analytical result, a_L is:

$$s^2_{a_L} = \sum_{i=1}^{I} s^2_i \qquad (1)$$

This variance can be *reduced* (always popular for those who worry about the **total** sampling-plus-analysis error) by taking replicate samples at different stages. Consider a three-level process: n_1 primary samples are extracted from the lot, with each primary sample processed and divided into n_2 secondary samples—of which n_{lab} analytical samples are finally analysed. In this case Equation 2 shows how the complement of stage error variance components propagate to the analytical result.

$$s^2_{a_L} = \frac{s^2_1}{n_1} + \frac{s^2_2}{n_1 \bullet n_2} + \frac{s^2_{lab}}{n_1 \bullet n_2 \bullet n_{lab}} \qquad (2)$$

The total number of samples analysed is $n_{tot}=n_1 \bullet n_2 \bullet n_{lab}$.

From a replication design, the variance components s^2_i can be estimated by using the statistical facility of analysis of variance (ANOVA), or analysis of relative variances (RELANOVA).[1]

Master example: the effectiveness of replication

The following example will help gain insight into where efforts to reduce and control the total accumulated error is best spent. Let us consider three *schemes* where, for each scheme, the relative standard deviation error estimates are: $s_{r1}=10\%$, $s_{r2}=4\%$ and $s_{r2}=2\%$.

A) **No replicates,** $n_1=n_2=n_{lab}=1$. Total number of samples analysed is 1.

$$s^2_{a_L} = (10\%)^2 + (4\%)^2 + (2\%)^2 = 120(\%)^2 \text{ and } s_{a_L} = 11.0\,\%$$

B) **Primary samples replicated,** $n_1=10$; $n_2=n_{lab}=1$. Total number of samples analysed is 10.

$$s^2_{a_L} = \frac{(10\%)^2}{10} + \frac{(4\%)^2}{10} + \frac{(2\%)^2}{10} = 12.0(\%)^2 \text{ and } s_{a_L} = 3.5\%$$

C) **Primary samples and duplicated analytical samples,** $n_1=5$; $n_2=1$; $n_{lab}=2$. Total number of samples analysed is again 10.

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$$s^2_{a_L} = \frac{(10\%)^2}{5} + \frac{(4\%)^2}{5 \bullet 1} + \frac{(2\%)^2}{5 \bullet 1 \bullet 2}$$
$$= 23.6(\%)^2 \text{ and } s_{a_L} = 4.9\%$$

This example demonstrates that even if the best and most expensive analytical technology available is used in the laboratory, this does not by itself guarantee a reliable result with minimised total uncertainty. **Still, some laboratories routinely run analyses in duplicates or even in triplicates to be sure that their results are "correct". While analytical costs have doubled or tripled, nothing is gained!** It is also common that the uncertainty estimates which laboratories assign to their results are based on the results of the laboratory replicates only; in reality hiding the full pathway uncertainty.

Selection of optimal sampling mode

Most current standards and guidelines *assume* glibly—although very rarely expressed explicitly—that sampling errors can be estimated using standard statistics. This is based on another assumption, that of a random spatial analyte distribution within the sampling target. When primary samples are extracted from large lots like process streams, environmental targets, shipment of raw materials or commodities or from manufactured products, the same *assumption* of "normality" may in fact lead to sampling plans that are more expensive than the optimised plan and, more importantly, do not provide reliable results. When the purpose of sampling is to estimate the mean value of the lot, the first issue to address is which sampling mode to use: *random, stratified or systematic*. Figure 1 shows and compares the principle of these modes. Very few guidelines refer to the sampling modes at all. Very often in monitoring programmes samples are collected systematically (all good), but the resulting analytical results are then treated as so-called random data sets. As the following example shows this actually results in a *massive loss of information*.

Figure 1 presents a comparison of these three fundamental sampling modes as applied to a process steam. First, there is no significant difference between them if the process standard deviation is estimated from all nine samples taken in each mode, i.e. the nine samples are treated as one data set. But their difference becomes clear when the mean of the whole process range is calculated. The bias of the mean is decreased from 4.49 % (random) to 3.92 % (stratified) and to –0.48 % (systematic) and the relative standard deviation of the mean from 11.3 % (random) to 8.28 % and to 2.26 %. The difference is even more clear if, based on these data, a sampling plan is requested, for example, with a target that the relative standard deviation of the mean shall not exceed 1 %. **The "expert" who recommends *random sampling* gives a plan that requires extraction of no less than 385 samples. However, a stratified sampling plan will only require 186 samples—whereas if the systematic mode is selected, only 12 samples are needed to reach the relative standard deviation target.** To summarise, in cost–benefit analysis of an analytical sampling plan the selection mode is crucial.

To select the *optimal* sampling mode and number of replicates, the unit costs are needed. Operators usually can estimate the cost structure, but the variance estimates can seldom be estimated theoretically. Sometimes they can be estimated from the existing data, but very often pilot studies are needed. Combining specific variance estimates with unit costs of the various operations in the full analytical measurement pathway will allow drastic improvements in the efforts needed; some examples are given below. Further examples of the informed use of the Theory of Sampling (TOS)' principles in the context of total expenditure estimation are given in Reference 2.

The **value** of engaging in proper sampling

Case 1

A pulp mill was extracting a valuable side product (β-sitosterol) which is used in the cosmetic and medical industries, and which has high quality requirement. Customers requested a report on the quality control system from the company in question. I was asked to audit the sampling and analytical procedures and to give recommendations, if needed. I proposed some pilot studies to be carried out and based on these

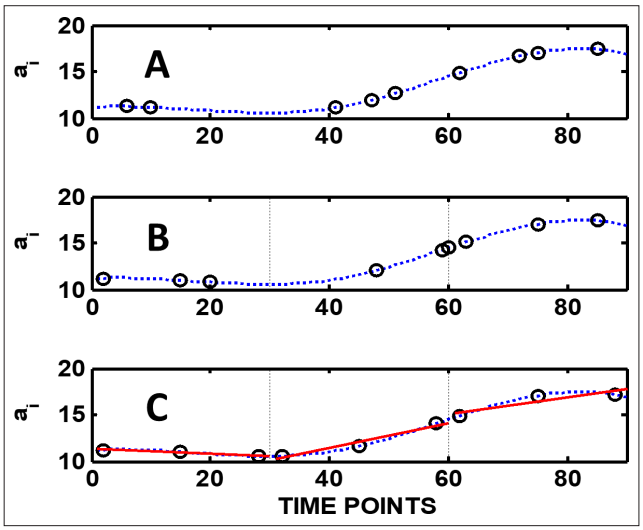

Figure 1. A: Random sampling: sampling times or locations are selected randomly. B: In stratified (random) sampling the process lot is divided into individual strata (three strata in this example) and within each stratum the sampling points are selected randomly. C: In systematic sampling the within-stratum samples are all taken at fixed intervals. The continuous line is based on process analyser measurements at short time intervals. For all three cases the lot average a_L=13.193, the relative sampling and analysis variance s_{r2}=6.962 and relative standard deviation s_r=0.20 (=20%).

empirical results recommended a new sampling system to be implemented—this was accepted.

In comparison to the old, the new sampling system annually saved the equivalent of one laboratory technician's salary.

Case 2

An undisclosed pulp mill was feeding a paper mill through a pipeline pumping the pulp at about 2% "consistency" (industry term for "solids content"). The total mass of the delivered pulp was estimated based on the measurements with a process analyser installed in the pipeline immediately after the slurry pump at the pulp factory. The receiving paper mill claimed that it could not produce the expected tonnage of paper from the tonnage of pulp they had been charged for by the pulp factory. An expert panel was asked to check and evaluate the measurement system involved. A careful audit, complemented with TOS-compatible experiments, revealed that the consistency measurements were biased, in fact giving up to 10% too high results. The bias was found to originate from two main sources. 1) The process analyser was placed in the wrong location and suffered from a serious increment delimitation error; this is an often-met weakness of process analysers installed on or in pipelines. 2) The other error source was traced to the process analyser calibration. It turned out that the calibration was dependent on the quality of the pulp: softwood and hardwood pulps needed different calibrations. By a determined effort to make the process sampling system fully TOS-compatible, and by updating the analyser calibration models, it was possible to fully eliminate the 10% bias detected.

It is interesting to consider the payback time for the efforts involved to focus on proper TOS in this case. The pulp production rate was about 100,000 ton y^{-1}, or 12 ton h^{-1}. The contemporary price of pulp could be set as an average of \$700 ton^{-1}, so the value produced per hour was approximately \$8400 h^{-1}. The value of the 10% bias would thus be \$840 h^{-1} (\$7 million y^{-1}). As the cost of the evaluation study was about \$10,000 the **payback time of the audit and the panel investigations was about 12 h**.

It does not have to be expensive to invoke proper TOS competency—it is often possible to get a better quality at lower cost.

Are our current sampling standards and guides adequate?

In most current standards, the findings of the TOS have often been ignored, or at best only partially recommended. Statistical considerations assume that sampling errors can be estimated using classical statistics which are based on the ubiquitous assumption of random spatial analyte distributions within the sampling targets. **The basic three sampling modes, *random, stratified or systematic*, are seldom even mentioned as options.** As shown above, when primary samples are taken from large lots like process streams, environmental targets, shipment of raw materials or products, ill-informed or wrong assumptions simply lead to wrong conclusions, and usually too expensive or inefficient solutions. More examples are given below.

Case 3: Estimation of the concentration genetically modified (GMO) soybeans

In the European Union, the limit of acceptable GMO content in soybeans is 1% (or 1 GMO bean/100 beans). If the content exceeds this limit, the lot must be labelled as containing GMO material. To allow for the sampling and analytical error, in practice 0.09% is used as the effective threshold limit for deciding on labelling the material as containing GMO or not. Theoretically, this seems a simple sampling and analysis problem. GMO soybeans and their natural counterparts are identical with no tendency to segregate. So, theoretically the required sample size can be estimated from considerations assuming a binomial distribution. The reality is **very** different, however.

In References 3–5 experimental analytical data from the KeLDA project were re-analysed, with a special focus on the inherent sampling issues involved. In the KeLDA project, 100 shiploads arriving at different EU ports were sampled by collecting 100 primary 0.5-kg samples (each containing approximately 3000 beans) using systematic sampling. At the 1% concentration level, the relative standard deviation of the total analytical error (s_{TAE}) was found to be 11.4%. For an ideal binomial mixture, conventional statistical calculations showed that the minimum number of 0.5-kg samples to be analysed in order to guarantee that the probability (risk) is less than 5% that the mean 0.09% could be from a lot having mean concentration above 1%—is 10 samples. The official number of samples recommended by many organisations vary between 4 and 12. So far, so good... **if** the conventional assumptions hold up to reality… alas!

A shipload often consists of products from many different *sources* having different GMO concentrations. In such cases the lot can be seriously segregated in the distributional sense w.r.t. domains having different GMO contents, making the assumption of spatial randomness grossly erroneous. Instead of the theoretical 10 samples, the thorough study reported in Reference 4 (lots of statistics in there, but they are not necessary for the present purpose) ended up with a much higher required number of samples needed, 42 to be precise (a famous number, if the reader is fan of Douglas Adams' *Hitchhiker's Guide to the Galaxy*). It is this number of samples which **must** be collected using the *systematic sampling mode* to make a correct decision regarding the labelling issue.

From enclosed stationary lots, such as the cargo hold(s) of grain shipments, or truckloads, railroad cars, silos, storage containers… it is in general impossible to collect representative samples without a TOS intervention. Samples must be taken either during *loading* or during *unloading* of the cargo, i.e. when the cargo lot is in a moving lot configuration on a conveyor belt. Otherwise, the average concentration simply cannot be reliably estimated. References 3–5 tell the full story, the conclusion of which is: conventional statistics based on the assumption of spatial random analyte distributions always runs a significant risk of underestimating the

number of samples needed to reach a specified quality specification—compared to informed TOS-competent understanding of heterogeneity, spatial heterogeneity in this case. **Proper TOS-competence is a must.**

Case 4: Sampling for aflatoxins in peanut kernels

Mycotoxins, e.g. aflatoxins and ochratoxins, are poisonous and are also regarded as potent carcinogens. Their contents in foodstuff must, therefore, be carefully monitored and controlled and the levels regarded safety are extremely low, down to 5 µg kg^{-1} (ppm), or even lower. But detection and quantification even of these very low concentration levels is usually not a challenge for modern analytical techniques in dedicated analytical laboratories. The real challenge is **how to** provide a *guaranteed representative* analytical aliquot (of the order of *grams* only) from the type of *large* commercial lots used in the international trade of such commodities (of the order of magnitude of *thousands of tons*). Effective sampling ratios are staggering, e.g. 1 : 10^6 to 1 : 10^9, or even higher. It is *somebody's* responsibility that the overwhelming $1/10^6$ to $1/10^9$ mass reduction is scrupulously representative at/over all sampling and subsampling stages. It is fair to say, that this setup is not always known, recognised, far less honoured in a proper way, sadly (because this is where the money is lost, big time) with the unavoidable result that nobody (nor any guideline or standard) can guarantee representativity.

Campbell *et al.*[6] carried out an extensive sampling study in connection with analysing peanuts for aflatoxins. It is interesting to study their findings using the principles of TOS: they sampled a lot having an average aflatoxin content 0.02 mg kg^{-1} by taking 21.8 kg primary samples. The average aflatoxin content of individual "mouldy" peanut kernels was 112 mg kg^{-1}. The average mass of one peanut kernel is about 0.6 g. In Reference 6 it was found that the experimental relative standard deviation of the 21.8-kg primary samples s_r(exp) was 0.55=55%. This empirical result exceeds the theoretically expected value, however, indicating that "something" is not right … A TOS rationale follows below.

Involving TOS

The mass of aflatoxin in a single mould contaminated kernel: $m_a = 112\,\text{mg kg}^{-1} \times 0.6 \times 10^{-3}\,\text{kg} = 0.0672\,\text{mg}$. If the acceptable average aflatoxin level is 0.02 mg kg^{-1}, this result means that just **one** mouldy peanut is enough to contaminate a whole sample of 3.36 kg. On the other hand, if the maximum tolerable level is only 0.005 mg kg^{-1}, one kernel will contaminate a 13.44-kg sample. If the kernels are crushed to 50 mg fragments, average samples containing one contaminated fragment are now 0.28 kg and 1.12 kg at average aflatoxin concentrations 0.02 mg kg^{-1} and 0.005 mg kg^{-1}, respectively. The relative standard deviation of a sample containing one contaminated peanut taken from a random distribution is 1=100%.

The theoretical relative standard deviation of a 21.8-kg sample from a random mixture is s_r=39.3% whereas the experimental value was 55%. The difference between these variance estimates [$0.55^2 - 0.393^2 = 0.148$, or 38.5% as RSD%] is a strong indication of *spatial segregation*. Such segregation of mycotoxins in large lots is a natural phenomenon, since moulds, which are producing the toxins, tend to grow in localised "pockets" where mould growth conditions are favourable. As an unavoidable consequence, the distribution of contaminated individual nuts within the full lot volume is in reality **far** from random. Because large lots, almost exclusively found in restricted and confined containers, cannot be well mixed (randomised), segregation has a drastic adverse effect on sampling uncertainty at the primary sampling stage—whereas at all later sample preparation stages, when only small masses are handled, it is possible to randomise various sized sub-samples by careful mixing, and here the theoretical values can be used to estimate the uncertainty of the sub-sampling steps involved.

For the ideal case of truly random mixtures, it is easy to estimate the sample size that gives the required relative standard deviation of the lot as a function of the primary sample size. For the two lot averages used here as examples, a_L is 0.02 mg kg^{-1} and 0.005 mg kg^{-1}, and targeting to 10% relative standard deviation of the lot mean, the *realistic minimum sample sizes* are:

$$m_s = \frac{(100\%)^2}{(10\%)^2} 3.36\,\text{kg} = \mathbf{336\,kg}$$

$$m_s = \frac{(100\%)^2}{(10\%)^2} 13.44\,\text{kg} = \mathbf{1344\ kg}$$

If the distribution is indeed random, **the m_s can be a composite sample or single increment, the expected RSD of the mean is the same, 10%, independent of the sampling mode. But the situation is radically different if there is indeed segregation, e.g. clustering of the contaminated peanuts. Then the required primary sample size and number will** depend **on the spatial distribution pattern and this can only be estimated empirically, either by a *variographic experiment* or by involving an ANOVA design, see References 3–6.**

The only result that can be estimated from the reported data in the Campbell *et al.* study, is **how many** 21.8-kg samples, n_{req}, are needed *if* random sampling is used. If the target threshold is 10% RSD of the mean at a_L=0.02 mg kg^{-1}:

$$n_{s(req)} = \frac{s^2_{r(exp)}}{(10\%)^2} = \frac{(55\%)^2}{(10\%)^2} = 30.3$$

and the total mass of the samples 30.3 • 21.8 kg ≈ **660 kg**.

Implications for commodity trade a.o.

In international trade agreements regarding foodstuffs, the tight limits set by regulators must be met at the entry port before the cargo materials can be released to the markets. As the examples above show, sampling and sample preparation for analysis are extremely difficult when the unwanted contaminants are present at their usual low, or very low ppm (or even ppb) levels. In the case of the present peanut example, at an average concentration 5 µg kg^{-1} in an ideal case (i.e., assuming randomness), the weight of the total number of primary samples should be about 1350 kg if the if 10% relative standard deviation is

the target. In sample preparation, if the secondary samples are each 10 kg and the analytical sample from which the toxins are extracted, are, say, 200 g, the peanuts must be ground to 0.45 mg and 0.09 mg particle sizes corresponding to approximately 0.96 mm and 0.56 mm particle diameters. But these are the results of an ideal case, very rarely found. Segregation makes the theoretical considerations much more complicated.

The simple moral from underlying complexities

The above technical intricacies notwithstanding, it is abundantly clear, that the quality of sensitive foodstuffs must be adequately monitored—and it is equally clear that at the inherent trace and ultra-trace levels of the analytes involved, the primary sampling and sample preparation are extremely difficult operations, but absolutely necessary! If the uncertainties of the analytical results are too high, this means that a high number of shipments containing excess amount of the contaminants may enter the market essentially undetected and, vice versa, shipments containing acceptable material may be stopped—but both types of misclassification are **not** caused by analytical difficulties. The resulting economic losses are **huge**, for each shipload that is wrongly stopped and retuned due to "erroneous" analytical results. **The lesson from the somewhat technical story above is clear: primary sampling, and subsequent sub-sampling and sample preparation errors, are very nearly always the real culprits—perpetrators are not to be found in analytical laboratories.**

What to do?

When decision limits are set, the capability of modern analytical instruments alone cannot be used as the guide for reliability. The capability of the **whole** measurement chain must be evaluated. If it turns out that the proposed decision limit is so low that it cannot be achieved at acceptable costs, even when the best methods of the TOS are applied in designing the sampling and measurement plan, then it must be decided what are the *maximum allowable costs* of the control measurements. First, then is it possible to set realistic decision limits so that they can be reached with methods optimised to minimise the uncertainty of the full lot-to-aliquot measurement pathway within a given budget which is regarded as acceptable; a more fully developed treatment of these interlinked technical and economic factors can be found in References 1 and 2.

References

1. P. Minkkinen, "Cost-effective estimation and monitoring of the uncertainty of chemical measurements", *Proceedings of the Ninth World Conference on Sampling and Blending*, Beijing, China, pp. 672–685 (2019).
2. P. Minkkinen, "Practical applications of sampling theory", *Chemometr. Intell. Lab. Syst.* **74,** 85–94 (2004). https://doi.org/10.1016/j.chemolab.2004.03.013
3. K.H. Esbensen, C. Paoletti and P. Minkkinen, "Representative sampling of large kernel lots I. Application to soybean sampling for GMO control", *Trends Anal. Chem.* **32,** 154–164 (2012). https://doi.org/10.1016/j.trac.2011.09.008
4. P. Minkkinen, K.H. Esbensen and C. Paoletti, "Representative sampling of large kernel lots II. Theory of sampling and variographic analysis", *Trends Anal. Chem.* **32,** 165–177 (2012). https://doi.org/10.1016/j.trac.2011.12.001
5. K.H. Esbensen, C. Paoletti and P. Minkkinen, "Representative sampling of large kernel lots III. General considerations on sampling heterogeneous foods", *Trends Anal. Chem.* **32,** 178–184 (2012). https://doi.org/10.1016/j.trac.2011.12.002
6. A.D. Campbell, T.B. Whitaker, A.E. Pohland, J.W. Dickens and D.L. Park, "Sampling, sample preparation, and sampling plans for foodstuffs for mycotoxin analysis", *Pure Appl. Chem.* **58(2),** 305–314 (1986). https://doi.org/10.1351/pac198658020305

SAMPLING SPECIAL SECTION

Section contents

Theory of Sampling—an approach to representativity offering front line companies added value and potential substantial savings

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Previous Sampling Columns have dominantly focused on the technical issues of representative sampling. This column addresses sampling from the complementary point of view: "What is the economic and commercial impact from non-representative sampling on management decisions and in boardrooms?" We have invited two experienced business consultants to help scope out an outline indicating powerful opportunities for added value and for substantial savings.

In medias res

Sampling is the process of selecting and extracting a small part of material suitable for analysis under the critical demand that it is a guaranteed representation of the much larger original lot.

A lot is the sampling target material residing in, for example a shipload, a railroad carriage, a truckload… or constituting a process flow, a moving stream of matter.

Sampling is a technical operation influencing the validity of decision making: sampling affects the bottom line in major sectors of world trade…

Sampling lies behind a significant number of claims and disputes related to commercial transactions…

Sampling leads to a fundamental uncertainty about material, product or goods characterisation, which, if ignored, may translate into economic consequences in the form of *hidden* value losses…

The principal reason for the above uncertainties is material *heterogeneity.* There are huge adverse risks associated with heterogeneous materials, which must be sampled according to codified procedures that specifically *counteract* heterogeneity in appropriate ways.

Sampling must always be carried out in a *representative* fashion, by a *competent* legal person, i.e. a properly trained sampling technician, process engineer, supervisor, or a certified department, institution or agency.

Sampling matters greatly—from the point of view of providing a reliable basis for decision making in all of science, technology, industry and society. Without a minimum of proper knowledge, competence and experience, there is no guarantee that a "repeated sampling operation" will result in a "duplicate sample" with the same analytical result, precisely *because* of material heterogeneity.

Figure 1. Sampling targets (lots) come in a great variety of shape, form and size, and apparently with no common traits. However, the TOS stipulates how it is only necessary to take their degree of heterogeneity into account.

It is only the specific sampling process with which a sample was extracted that can be designated as *representative*, or *not*, according to certain criteria which are codified in the Theory of Sampling (TOS). Material extracted without a documented TOS-basis can never be designated as representative samples—only as worthless small lumps of matter without a meaningful provenance w.r.t. the original lot. Such non-representative extracts, "specimens", must be discriminated against and never relied upon. Specimens are not worth processing in the analytical laboratory, and far less analysed, as their analytical results will be fraught with uncontrollable uncertainties of quite unacceptable magnitudes. Sampling uncertainties are 10–25–50 times *larger* than the traditional analytical uncertainties which are usually the only determinants in contractual specifications. This misunderstanding is the source of many contractual disputes, but which are wholly avoidable and therefore unnecessary. The TOS is the only competence basis with which to address all these issues.[1–4]

Impact on critical decision making

This issue is not always fully recognised and acknowledged, and may not always be passed on to all decision making levels, CEO, boardroom. Yet all decisions with critical economic consequences are made here. If there is a fundamental lack of understanding of the magnitudes of such "hidden" uncertainties, there is a lack of due diligence by those responsible for producing correct and reliable documentation upon which to base management decisions on the operative level, or strategic deliberations at the company board room level. This is why there must be at least a minimum core understanding of such "technical sampling issues" at management and board room level as well.

In a commercial transaction, sampling produces the basis for a *reference* to an agreed product specification, or acts as a quality reference both at pre-shipment inspections as well upon arrival control at the destination. Documented samples are often used as reference material and deposited at the Chamber of Commerce for use in case of legal dispute. Furthermore, if payments of goods are conditioned on a "Letter of Credit" transaction, the sampled reference material (with its certified analytical result) is the decisive factor for payment approval and for the go-ahead process of the shipment in question.

But what if the supposed representative samples are, in fact, worthless specimens?

The contractual apparatus can then be fundamentally undermined with easily imaginable adverse consequences. **Sampling matters greatly**—at all levels in any company, corporation and organisation.

Sampling—minimum technical knowledge

All materials are heterogeneous on one scale or another, it is only a matter of degree. Sample representativity is the imperative criterion that must be honoured in order to draw valid conclusions about the true characteristics of an original lot, of materials, of material processes. The force of the TOS becomes clear when it is realised that the TOS is applicable to *all* types of material; exactly the same principles and practical sampling rules need to be invoked.

Internally in a company, choosing and using *only* correct sampling procedures, and knowing how to choose *only* correctly designed equipment, is a critical facility for being able to reach a desired Quality Control/Quality Assurance (QC/QA) level. Using *only* representative sampling allows a company to optimise the quality of the processing taking place, or of the quality of the products produced. There is a plethora of "hidden" sampling necessary to be able to *document* QC/QA appropriately. Are all companies or other stakeholders fully equipped for this task? The TOS is a critical success factor for proper monitoring of quality.

Proper sampling and laboratory analysis become critically important competences. There has been a growing historical usage of proper sampling concepts, methods and equipment over the last 50 years, but even today this is far from universally applied. This historical development is fully documented, indeed available at all levels of appreciation.[1–4] First and foremost, there is documentation in the form of a *de facto* international standard devoted to the general principles, equipment design characteristics and, perhaps most important, the necessary and sufficient rules for *practical representative sampling* based exclusively on the TOS.[1]

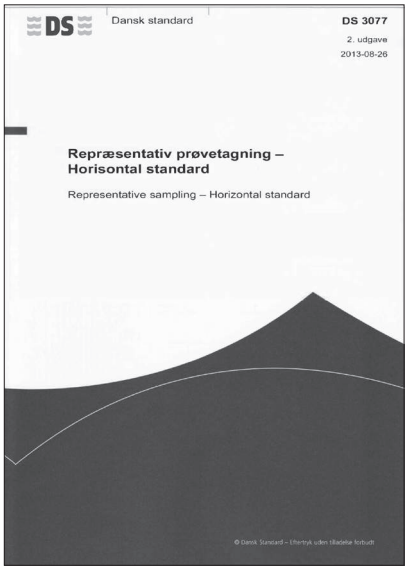

Figure 2. DS3077 (2013): the *de facto* international standard for representative sampling.

Summing up—cardinal points

Analysing a non-representative specimen is pointless! If a sample cannot be documented to be representative of the lot/target material from where it was taken, it is a waste of time, effort and money to analyse it.

Erroneous sampling is, every year, responsible for:

- waste in production,
- sub-optimised product quality levels,
- erroneous QC/QA documentation,
- a volume of unwanted complaints, claims, legal disputes and lawsuits,

but all this is largely unnecessary! Non-representative sampling also results in damage to companies' reputations and may, at worst, translate into lost business opportunities.

Sampling, although strictly a technical operation, nevertheless deserves the full attention of management. Ignorance of the TOS results in unacceptably high risks in decision making. If a "sample" does not qualify as representative (in reality a "specimen"), it may be part of the reason for significant production loss, commercial disputes, disruption of a commercial partnerships and, potentially, high costs for third parties too.

The importance of sampling *must* be understood and acknowledged by all stakeholders. The person, department or other entity responsible for sampling *must* possess a minimum of relevant TOS competence, practical know-how, proper training and *integrity*, so as professionally to be able to fulfil this vital role in reaching the quality objectives and contractual obligations committed to at higher levels in a company or corporation. It is necessary to establish an entity with a *unified* responsibility for sampling "from-lot-to-analysis", which has undisputed *carte blanche* to perform the necessary QC/QA of all sampling processes throughout and across all department borders.

Globalisation and increased volumes of international trade produce a need for relevant rules and regulations. The scenarios depicted here will be the same for all international economic activity levels. From pre-corona prosperity to post-corona recovering economies, all technical and business issues are identical. Therefore, standards and certification bodies have established common guidelines and legal references for cross border transactions and safety regulations.

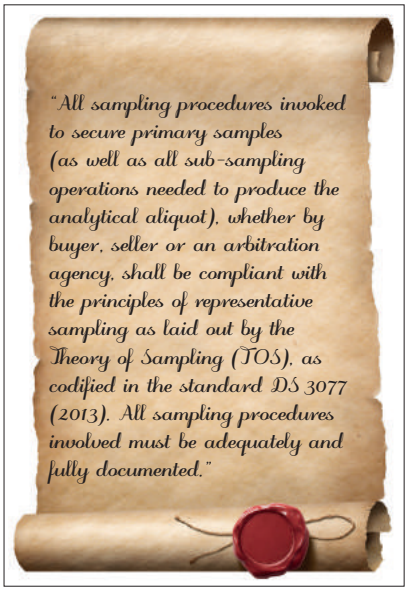

Figure 3. Example of a generic text that would eliminate all sampling vs analysis uncertainties in commercial contracts.

What has been missing up to now, the *missing link*, is one comprehensive, universal standard for sampling: DS3077 (2013) provides this.[1] A unanimously adopted horizontal standard for representative sampling is the logical guardian, which all stakeholders and parties should be able to agree upon. An example of its deployment in the generic buyer–seller scenario is shown in Figure 3.

References

1. *DS 3077. Representative Sampling—Horizontal Standard*. Danish Standards (2013). http://www.ds.dk
2. K.H. Esbensen, *Introduction to the Theory and Practice of Sampling*. IM Publications Open, Chichester, UK (2020). impopen.com/sampling
3. F.F. Pitard, *The Theory of Sampling and Sampling Practice*, 3rd Edn. CRC Press (2019). ISBN: 978-1-138476486
4. K. Esbensen and G. Lyman, "'Critique of Gy's Sampling Theory"': Misplaced expectations of Wikipedia's democratic intentions", *TOS forum* **1**, 28–30 (2013). https://doi.org/10.1255/tosf.11

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Introduction to the Theory and Practice of Sampling

Kim H. Esbensen

with contributions from Claas Wagner, Pentti Minkkinen, Claudia Paoletti, Karin Engström, Martin Lischka and Jørgen Riis Pedersen

"Sampling is not gambling". Analytical results forming the basis for decision making in science, technology, industry and society must be relevant, valid and reliable. However, analytical results cannot be detached from the specific conditions under which they originated. Sampling comes to the fore as a critical success factor before analysis, which should only be made on documented representative samples. There is a complex and challenging pathway from heterogeneous materials in "lots" such as satchels, bags, drums, vessels, truck loads, railroad cars, shiploads, stockpiles (in the kg–ton range) to the miniscule laboratory aliquot (in the g–µg range), which is what is actually analysed.

This book presents the Theory and Practice of Sampling (TOS) starting from level zero in a novel didactic framework without excessive mathematics and statistics. The book covers sampling from stationary lots, from moving, dynamic lots (process sampling) and has a vital focus on sampling in the analytical laboratory.

"I recommend this book to all newcomers to TOS"

"This book may well end up being the standard introduction sourcebook for representative sampling."

"One of the book's major advantages is the lavish use of carefully designed didactic diagrams"

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IMPOpen

The ultimate manager's argument for representative sampling

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From **management's** perspective the cost of sampling must be as low as possible: samples are "just" a necessity to enable the laboratory to do its tests. Once the lowest cost sampling method has been identified and implemented—either by the in-house quality department or through a Testing, Inspection, Certification (TIC) service provider—management is done with sampling… Well, except for the occasional slap on the wrist to the samplers when there is a complaint on quality, or a dispute: "Our client does not get the same control results as our own". This must clearly be the samplers' fault; they took the wrong sample!

Anyone with Theory of Sampling (TOS) knowledge will disagree with this scenario and will go through fire and water to try to explain that there is no such thing as a right (or wrong) sample. When there is no representative sampling process, there are only *specimens*… those pesky lumps of matter collected uncontrollably from a lot: specimens are **not** representative by TOS definition.

Sampling experts always find themselves explaining the "risk of being wrong" and love to bring up the hidden cost of using a non-representative sampling process. These confident boffins happily

and relentlessly illustrate with numerical examples, or graphs with error margins, precision and accuracy … that a non-representative sampling process is very likely to significantly reduce, e.g., life-of-mine or result in a financial loss during a transaction (they have an endless array of horror stories from all over industry to tell).

Yet often the experts are met by a **yawning** manager, or by a manager having a trader mind set, who is feeling lucky that he or she may also benefit. The "risk of being wrong" may just as well flip into "the 50 % possibility of being favoured". Especially when we TOS illuminati throw in statistics, standard deviations, variances, use "±" signs and may top it all off with a normal distribution graph etc., **then** the managerial thinking still goes: "Even in the worst case, on balance I will be okay!"

WRONG, sadly!

The real world

The process of *representative sampling* depends on two critical success factors:

1) elimination of Incorrect Sampling Errors (ISE) and 2) reduction of the Correct Sampling Errors (CSE) to an acceptable level.

Here, in order to avoid the **yawn**, we will completely skip all further *explanations*, those dull "technical explanations", but leave the reader with sufficient references (should the interest develop) for proper sampling *access to* how to make sure every particle can and will be *included* in the sample, and how to decide on the necessary-and-sufficient number of increments to *select* (thereby also fixing the all-important question about the optimal sample mass); for references, just look at all other contributions above and below.

The technical truth

Thus, for now, we can refer to what is easily understood by managers—Murphy's Law, which states that that there **cannot** be an overall "on balance" when representative sampling is addressing significantly heterogeneous materials and lots, as when compromised by the desire to involve the least expensive sampling approach (grab sampling), which unfortunately is tantamount to allowing a significant *sampling bias*. This is a single-sided effect that is *always* a **cost** and *never* a **benefit**; again, just look at all other contributions above and below.

The magnitude of this cost?

The costly truth

Well, let Murphy's Law decide that for you, instead of us experts trying to make "reasonable" *assumptions* about inherent heterogeneity and shaky, but dead-cheap, sampling procedures (again grab sampling) in order to quantify a monetary amount or build the resource model

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for the new mine for example, you know much better yourself!

But, by the way… now that you know this critical issue in these simple terms, imagine how your shareholders will react next time the results from a non-representative sampling process interfere with the bottom line of your annual reports!

What to do—how to go forward?

Simplified there are just three phases for representative sampling.

1) The planning phase, *prior* to sampling
2) The actual sampling
3) Making managerial, *inter alia decisions* based on the sample (results)

The TOS' focus is overwhelmingly on phase 1) and phase 2), e.g. to determine essential stuff like heterogeneity which is needed for better planning. Phase 3) is only for the user… e.g. the manager.

The economic impact

The economic impact of *representative* sampling is abundantly clear: it is essentially *neutral* and does not favour, nor prevent, a specific wishful thinking. Ironically representative sampling delivers **exactly what a manager expects** from a sample: something that can be considered as factual and true… as fully representative of the bulk from which it was taken from, and for which reason one can have complete faith in the corresponding analytical results.

How to tell it to management

So, no big Dollar or Euro amounts to be presented here, no complicated statistical results, no graphs, no error margins. Just you, your imagination and the knowledge that *representative* sampling is a process that can remove all your fears of a financial claim, or of upsetting your shareholders, or the fear of prosecuting regulators.

Ultimately the economic argument for *representative sampling* is just that, the most coveted position regarding all business risks: "peace of mind".

Just a warning though: If the adjective "representative" is removed from any sampling process—all the above goes away in a blink!

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SAMPLING SPECIAL SECTION

Framing the Theory of Sampling in risk assessment: a compelling perspective for the future

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Sampling is necessary every time inferences are to be made to take informed, optimal decisions in science, technology, industry, trade and commerce. For reasons extensively addressed over the last two decades, application fields where good sampling practices are a source of economic gain—and bad sampling performance results in significant but unnecessary loss of money, such as the mining/minerals/metals industrial sectors—explicate the role of sampling more than others. In stark contrast to other fields (the realm of food and feed safety assessment is a prime example), sampling is largely perceived as an economic burden and a technical necessity to be fulfilled because of regulatory demands, rather than a vehicle with which to ensure reliable evidence to support management and regulatory decisions. Risk assessment and sampling are both probabilistic disciplines, the first devoted to estimate and minimise economic, safety and other risks, the latter devoted to estimate and mitigate sampling risks (the effects of sampling errors). Here we offer an exposé showing that the Theory of Sampling is an essential discipline and practical tool needed to ensure the best possible estimation of risks in support of both narrow economic objectives (industry, technology, trade, commerce), as well as broader safety decision-making and risk management environmental and biological sciences, and society at large. This contribution offers a novel perspective arguing for proper sampling, one where the economic argument ("what's in it for me") for proper sampling is demonstrated in practically all contexts, hereby complementing the compelling 25-author "Economic Arguments for Representative Sampling".[1]

Sampling: a border-crossing discipline

Sampling is a border-crossing discipline relevant every time inferences are to be made for taking informed, optimal decisions in science, technology, industry, trade and commerce. Scientific experiments and technical endeavours are very often dependent upon correct sampling at certain fundamental stages. Trade and international agreements recommend duplication (or even triplication) of primary samples to allow buyers and sellers performing analyses to compare results for contractual compliance purposes. Market and commercial agreements also rely on sampling for monitoring of quality. Sampling plays a self-evident role in food and feed (F2) safety assessment as representativity of test materials for hazard identification, hazard characterisation and exposure assessment are critical pre-requisites for taking informed decisions regarding public, animal and environmental health. Indeed, potential health risks for humans and animals can only be estimated accurately when exposure scenarios to a given food or feed are realistic, i.e. based on reliable sampling of food consumption habits. Furthermore, from an analytical perspective, the vast variety of food and feed matrices and commodities, raw or (semi-) processed, pose challenges to develop *appropriate* sampling strategies that best facilitate correct analytical methods. Similar issues exist in other sectors of society, e.g. in pharmaceutical manufacturing. Nonetheless, despite abundant evidence documenting the pervasive relevance of sampling, the Theory of Sampling (TOS) is not (yet) universally accepted.

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A confluence of frustration

Over the course of the last 20 years, working alone and together, exploring the application of the TOS to very different disciplines and application fields, M3 vs F2, the present authors often felt challenged by meeting two fundamentally contrasting attitudes towards the TOS: **why sampling?** and **who/what benefits from proper sampling?**

In this period, we addressed, analysed and discussed on multiple occasions the likely causes for the divergent attitudes

towards the TOS,[1–5] and arrived at the understanding that different *a priori* motivating factors driving the modes of application of the TOS and practical sampling are the root cause for this. We here choose to focus on the mining/minerals/ metals (M3) and food and feed (F2) sectors as lighthouse examples to illustrate this contrasting mindset. In the M3 sector, incorrect sampling unavoidably translates into hidden or clearly predictable *economic losses*. Consequently, the TOS is here rightly perceived as the main underlying agent *safeguarding business endeavours*.[1] In the equally broad global F2 sector, however, sampling is seen as a tool to verify the accuracy of claims and/or the quality of products, forcing the TOS more to be the operative agent with which to *search for possible problems* or to verify their absence, providing results in a statistical context offering merely degrees of confidence to inform the decision-making process. This is clearly a very different driver for invoking correct TOS when compared to safeguarding information factors for hardcore business interests.

The contra-positioning of the underlying drivers for sampling is a key point dividing the views of samplers, process engineers, managers, regulators: even if from a technical and practical point of view exploration for, and processing of, metalliferous resources is not so different from sampling for, say, aflatoxins in a 60,000-ton shipment of grain kernels—the *motivations* for investing education, intellect, time and money in correct, representative sampling are *fundamentally different*. In the M3 sector, the better the sampling, the better for business; whereas in the F2 sector the better the sampling, the higher the risk of lot rejection or similar, which always carries a heavy negative economic and/or reputational penalty.

An emerging synoptic TOS framework

The plethora of TOS applications in the last 20 years documents this dichotomy, witnessed by the comprehensive historical record of the Proceedings from ten World Conferences on Sampling and Blending (WCSB) in the period 2003–2022 as well as a trend towards more reflected TOS references in ISO standards a.o. Notably, the technical application of the TOS is virtually identical in all applied fields, including F2 and M3: when sampling heterogeneous materials of any nature, the task for practical sampling is to *counteract* the effects of *the same* sampling errors (SE), making use of *the same* Sampling Unit Operations (SUO) following *the same* Governing Principles (GP). The purpose of sampling is to conduct the optimal elimination and/or reduction of all eight types of sampling error effects, to deliver a defensible representative analytical aliquot to the laboratory. To be able to do this, all pre-analysis sampling operations must be representative, no exception allowed. In the schematic TOS framework developed by one of the present authors over the past 20 years,[5] the critical task of eliminating and reducing sampling error effects can also be seen as appropriate *sampling error management*.

In the TOS realm, mitigation (management) of SE is a compound operation driven by the necessary sampling competency, which can range from adequate to non-existing, fighting material heterogeneity, which can range from large to almost non-existing, only using composite sampling. The key principle is clear: all sampling procedures must be representative of the original sampling target, the lot. Therefore, the starting point is always the Lot Heterogeneity Characterisation (LHC) which allows the design, implementation and performance of optimal representative sampling with respect to the specific heterogeneity profile of a lot of interest.

The synoptic framework representation of the TOS in Figure 1 has only very recently allowed the sampling community to recognise that proper handling, i.e. management of the gamut of sampling errors is in fact a critical risk management operation,[6] to be explicated below.

Risk, risk assessment, risk management

The apparently very diverse drivers for applied TOS in the exemplar M3 vs F2 sectors indicated above, can also be seen from a common viewpoint, with a much broader impact, introducing the unifying concepts of *risk*, *risk assessment* and *risk management* in the sampling arena. In the following it is assumed that the reader is familiar with the TOS' basic systemic elements of

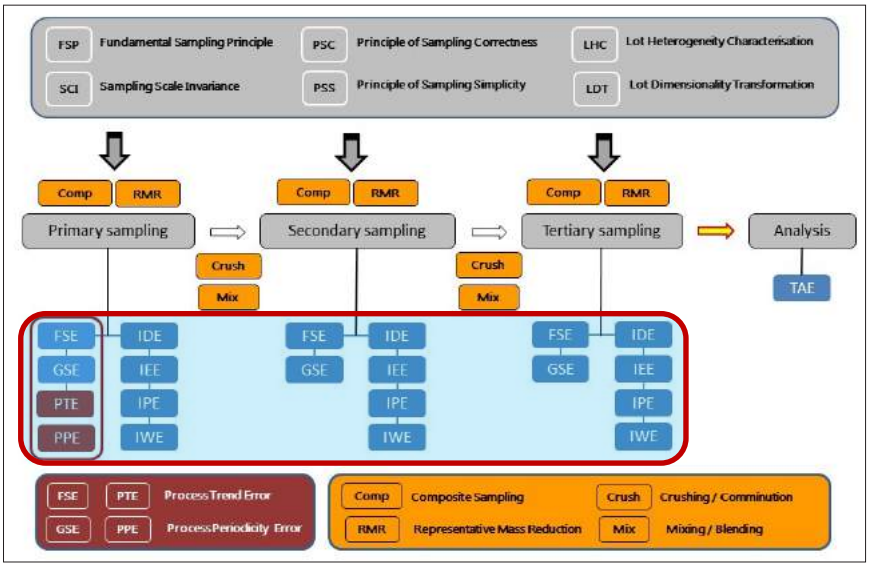

Figure 1. Theory of Sampling (TOS), synoptic overview. Practical sampling is governed by six Governing Principles (GP) [top grey panel], using four Sampling Unit Operations (SUO) [bottom yellow panel] in an informed effort to reduce unwanted sampling error effects, IDE, IEE, IPE, IWE, GSE, FSE … [blue rectangle]. This constitutes the realm of risk management in the TOS: correct, complete elimination of ISE and reduction of CSE sampling errors (including those occurring in the analytical laboratory). Illustration copyright KHE Consulting, reproduced with permission.

Governing Principles (GP), Sampling Unit Operations (SUO) and Sampling Error Management rules (SEM), see Figure 1 and basic TOS references as found in References 1 and 5.

Framing the TOS in risk assessment: an outreach perspective for the future

Risk assessment has been defined in many different contexts see, e.g., a Google search.

Positioning the TOS as a risk management task provides a broader perspective, both at the theoretical as well as the practical level, illustrating the far-reaching responsibility vested in the TOS community. This awareness began with the recent publication "Economic arguments for representative sampling", which addresses how to engage better with management, offering more than 25 different points of view.[1] This collective publication expresses well the *status quo* for the International Pierre Gy Sampling Association (IPGSA) and identifies areas where the sampling community needs to expand its activities to promulgate the TOS as a tool necessary for optimal risk management decisions across many disciplines.

Sampling is about providing reliable data and information necessary to take managerial decisions. In some areas such information is sufficient on its own, in others additional considerations must be taken into account.

Fundamental risk definitions

Risk: probability that something unknown and/or unwanted happens.
Risk Assessment: the process to identify risks, so they can be minimised, often in order to maximise a critical goal, e.g., economic gains (business scope), consumers protection (societal scope) or quality control (technical quality control/quality assurance/quality management scope).
Risk Management: the process of monitoring and managing risks, optimising success by minimising identified risks as much as possible. Risk management capitalises on data as a reliable asset, for which reason all data must be representative.

Fundamental risk definitions applied to the TOS

TOS Risk: probability of unwanted, unmitigated sampling errors (SE)—both incorrect (ISE) and correct (CSE) sampling errors—resulting in uncontrolled, inflated sampling variability. This is a scenario damaging to every stakeholder.
TOS Risk Assessment: the process to identify the effects of unmitigated sampling errors in terms of ISE + CSE and material heterogeneity—i.e., the total sampling error (TSE)—employing, for example, pairwise sampling, replicated experiments or variographic characterisation, see the TOS literature for technical details.
TOS Risk Management: the process of monitoring and managing sampling error effects, specifically through complete elimination of ISE and the concomitant reduction of CSE, thereby, a.o., eliminating the fatal sampling bias, while complying with the Fundamental Sampling Principle (FSP) at all scales.

Discussion

The goal of risk management is not elimination of all risks (which would be an impossibility), but rather getting to know which risks are worth taking, which must be minimised and which ones have enough of an assured negative pay-out not to take them.

The sampling community should expand its horizon and offer its expertise to all sectors in society where the TOS is a *de facto* essential tool to deliver the appropriate information for critical decision making. Correct sampling is about being accountable for the trust that the business community and society puts into decision-making systems. Society has no other choice: we all consume what is available on the market *trusting* its quality and safety, trusting that the control system has worked as intended.

But "*consumption*" shall be seen here in a context much broader than just human and animal consumption of food and feed, indeed as the responsible *use* of resources and commodities. Upon reflection, there are virtually no examples of management decision making in the technical and industrial society that do not rely on sampling-before-analysis considerations along the information flow involved, even if well hidden from immediate reflection. Explicating the risk management scope of what makes sampling representative allows a fresh and powerful look at some of current hindrances for a more successful drive to *go beyond* the TOS' traditional borders. Framed in this perspective, the TOS becomes an essential practical tool needed to ensure the best possible estimation of risks to inform decision making across societal sectors at large, including biological sciences, agro-business, technology, industry, trade, commerce, environment.

Conclusion

Successful risk management considers the full range of risks, examines the relationship among the identified risks and their cascading impact(s). In some areas the number of factors informing management decision is limited, like in the M3 sector where attention is always tightly focused on mitigation of sampling error effects on the business bottom line. In others, like F2, the primary consideration is always human and animal health protection, however, other factors such as economic costs, cost/benefits, technical feasibility and risk perceptions are also considered appropriate. Nonetheless, the TOS is indispensable under either scenario—or beyond.

It is hoped that the risk assessment scope will allow the sampling community an easier, and perhaps more powerful, way to reach out to business, commerce, trade as well as regulating and law-enforcement authorities by starting to speak a more common language beyond the mere "technicalities" of the TOS.

Disclaimer

Claudia Paoletti is employed by the European Food Safety Authority (EFSA). The positions and opinions presented in this article are those of the authors alone and do not necessarily represent the views or scientific works of EFSA. Kim H. Esbensen is an independent researcher and consultant, having left behind a three-decade university and government employee career in 2015.

References

1. K.H. Esbensen (Ed), "Economic arguments for representative sampling", *Spectrosc. Europe* **33(7),** 11–12 (2021). https://doi.org/10.1255/sew.2021.a30
2. H.A. Kuiper and C. Paoletti, "Food and feed safety assessment: the importance of proper sampling", *J. AOAC Int.* **98(2),** 252–258 (2015). https://doi.org/10.5740/jaoacint.15-007
3. K.H. Esbensen, C. Paoletti and N. Theix (Eds), "Special guest editor section", *J. AOAC Int.* **98(2),** 249–320 (2015). https://academic.oup.com/jaoac/issue/98/2
4. C. Paoletti, "Food and feed sampling: balancing ethics and money", *Spectrosc. Europe* **33(7),** 61–63 (2021). https://doi.org/10.1255/sew.2021.a47
5. K.H. Esbensen, *Introduction to the Theory and Practice of Sampling*. IM Publications Open (2020). https://doi.org/10.1255/978-1-906715-29-8
6. (a) C. Paoletti and K.H. Esbensen, "Framing TOS in risk assessment: an outreach perspective for the future", *TOS Forum* **11,** 419–423 (2022). https://doi.org/10.1255/tosf.168; (b) https://wcsb10.com/recordings-of-the-wcsb10/ (Day 3/Session 9 (4 h:18 min:45 s).

Economic arguments for representative sampling: Missing information to management and *hidden* economic losses

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This short publication emphasises the abysmal cost of poor sampling practices. The case studies presented are summaries of long investigations where management had a doubt about the hidden cost of poor sampling practices. Each case was the subject of in-depth studies to finally know the truth.

Introduction

There are no such things as reliable feasibility studies, unbiased ore grade control, accurate environment assessments, effective process control, if you cannot identify and minimise the eight major sources of sampling variability. Furthermore, Total Quality Management cannot exist without effective Statistical Process Control, which itself cannot exist without a thorough understanding of the Theory of Sampling (TOS).

The main reason for the TOS being neglected in the past is due to the failure to place it in its economical context. As a result, many important executives around the world saw the TOS as an academic achievement with no obvious practical value. To correct for this unfortunate situation, the WCSB conference was created in 2003 and a third edition of Dr Pitard's textbook[1] was published with a proper blend from several important worlds:

1) The Management Method of W. Edwards Deming
2) The Sampling Theory of Dr Pierre M. Gy
3) The undeniable touch from Geostatistics with variography leading to Chronostatistics
4) The extraordinary competence of a famous analytical chemist and sampling expert, C.O. Ingamells
5) The works of J. Visman
6) The modern philosophies of Statistical Process Control and Six Sigma

The following real cases are based on careful investigation involving comparisons between poor sampling with much better practices in line with the TOS.

Case #1: Cobalt assays in a lateritic ore deposit[1]

Cobalt was a valuable by-product in a nickel deposit. Samples brought to the laboratory were about 10 kg. A large mining test was performed to further complement a feasibility of the project. This test visually exposed the fact that the cobalt content could not be correctly estimated unless 100-ton samples were collected, which is obviously completely unpractical. Nevertheless, the facts are what they are, and awareness is a big factor in decision taking for the feasibility study. The result was that a large part of the deposit was vastly underestimated for its cobalt content, and a few parts of the deposit were overestimated. The conclusion was that the generated assays, many thousands of them, were affected by a severe Poisson Process, which is highly misleading for upper management to make reliable economic decisions if ignored for too long. The dollar value of this problem in the feasibility study was quantified to be many million dollars.

Conclusion #1: The incapability for upper management to identify Poisson Processes introduced by poor sampling/assaying practices is the perfect way to an economic disaster.

Case #2: The estimation of low iron content in high purity ammonium paratungstate[1]

But Poisson Processes are not obvious to identify without a good knowledge of the

TOS. Therefore, let's look at another eye-opener case. A company manufactured a new highly valuable ammonium para-tungstate product to be sold to a customer making tungsten coils in light bulbs. The product must satisfy a very stringent iron content limit of 15 ppm to make sure the light bulbs would have a long life. A shipment of two full railroad wagons is sent to the customer after QA/QC procedures assaying 1-g samples determined the iron content was below 10 ppm. Two weeks later the customer rejects the shipment after using their more sophisticated analytical procedure using 100-g assay samples. According to the customer, the average content of iron was 63 ppm. This is another several million dollars loss due to an unrecognised Poisson Process that took place because of totally inappropriate 1-g assay samples.

Conclusion #2: Beware of Poisson Processes when assaying tiny samples for trace constituents. The cost of such mistake can be devastating for the company.

Case #3: A bad sampling protocol followed by an incorrect implementation[2]

In a large copper mine in northern Chile, a poor sampling protocol for collecting routine samples from blasthole piles was used for about 10 years. A manual 4-kg sample was collected, instead of a required 20-kg sample to represent the coarsest 2.5-cm fragments. As a result, the reproducibility of the field samples was not good and also as a result of overrepresenting the fines, copper assays were biased high, which resulted in a severe reconciliation problem with the plant. The required 20-kg samples were considered impractical and too expensive to subsample at the sample preparation room that was using a robot to do the job. Following arguments and debates that lasted more than four years, top management finally agree to collect 20-kg samples that required five more people in the field, and a complete redesign of the laboratory robot that cost about four million dollars. Within a few months of operation using the correct sampling protocol, the copper content going to the plant became higher because of much less ore misclassification. Furthermore, the reconciliation problem between the mine and the plant was far more acceptable. A quick analysis of the case lead to the undeniable conclusion that the company lost US$134,000,000 in a period of 10 years when using the poor sampling protocol.

Conclusion #3: The excess cost of having five more people at the mine to collect 20-kg samples and the additional cost to modernise the robot at the laboratory should have been of no concern at all.

Case #4: An incorrect sampling system to assess the copper content of the tailings of a flotation plant[2]

In the largest underground copper mine in the world in Chile, the Final Tail of the flotation plant were sampled manually for 20 years. The Tailings were also sold to a junior company who was mysteriously very prosperous. It took several years of convincing for the company to finally spend one million dollars on a correct cross-stream sampling station. Within one month, top management was scandalised by the results and asked the manufacturer what was wrong with the sampling station: the copper and molybdenum contents went 25% up on average. Nothing was wrong with the sampling station: it was flawless and became a benchmark for many other operations. The company had to admit they lost about two billion dollars over 20 years: no wonder the junior company was very prosperous.

Conclusion #4: Never underestimate what an incorrect sample can do to the economics of a big project.

Case #5: An incorrect drilling density for a feasibility study[2]

A Porphyry Copper deposit in Northern Chile was drilled with a 50 × 50 diamond drilling pattern: the value of the fine copper content of the deposit was estimated to be about US$389,000,000. After the drilling pattern was reduced to 20 × 10 drilling grid, the fine copper content of the deposit increased to a value of US$421,000,000. Ultimately, after the grid was reduced to 10 × 5 m, the fine copper content of the deposit was estimated to be about US$450,000,000.

Conclusion #5: Think twice, very carefully, before you save on the cost of drilling a mineral deposit; it may be the difference between abandoning the project or developing a valuable one.

Case #6: A wrong subsampling protocol of half core samples to assess the molybdenum resources of a deposit

A molybdenum deposit was discovered in British Columbia, Canada. The company involved had a long experience with molybdenum deposits in Colorado, USA. A massive drilling campaign was initiated to drill many holes generating about 30,000 NQ-diameter core samples. They used their historic standard subsampling protocol: cut the core in half, crush the half core to < 0.5 cm, split a 250-g sample, pulverise this subsample to < 100 µm, and finally assay a 0.5-g subsample to determine the molybdenum content.

This first interpretation of all assays by geostatisticians led to the conclusion that all assays showed massive nugget effect making this massive drilling campaign, that cost many millions of dollars, totally useless.

After consulting sampling experts, a new drilling campaign was initiated generating 5000 samples by drilling with a larger HQ-diameter. The subsampling protocol was now: crush the entire core samples to < 0.17 cm, split a 1000-g subsample, pulverise this subsample to < 100 µm and finally assay a 5-g subsample to determine the molybdenum content.

Geostatisticians were much happier with the new assays showing well-behaved variograms to build a reliable block model and resource model for the feasibility study.

Conclusion #6: 5000 good samples gave much better information than 30,000 poor samples. Be careful to perform a valid study of the sampling characteristic of an important component

before spending a huge amount of money to perform a feasibility study.

Case #7: A wrong analytical method to assess the gold content in a world class gold deposit[3]

A feasibility study of a newly discovered gold deposit was entirely performed using 30-g standard fire assays. However, this deposit showed a substantial amount of visible gold particles up to about 750 µm. A careful sampling study revealed in an unambiguous way that 30-g fire assays were missing visible gold 95% of the time.

Conclusion #7: In sampling practice, beware of standards that cannot apply to your particular case.

Overall conclusion

As a conclusion I would simply use the words of wisdom from our late friend Pedro Carrasco:

"Improper sampling and assaying practices can produce monumental value losses to the mining industry worldwide. For a single large company the amount of money lost in a time frame of 20 years could be greater than two billion dollars. Therefore, bad sampling and assaying procedures lead to economical inefficiency and unsustainable exploitation of natural resources jeopardizing the wealth of future generations and adding unnecessary negative externalities to society. The mining industry has a magnificent opportunity to increase its economic performance discovering hidden losses by applying the principles of the Theory of Sampling, statistical and geostatistical thinking, effective chronostatistical process control and encouraging the work of multidisciplinary high level experts aligned with the main objectives of the mining business."

Obviously, it does not take a lot of imagination to see that these wise words apply to many other industries, and it is our mission at WCSB to show the world that no one has anything to lose by getting properly educated about the TOS.

References

1. F.F. Pitard, *Sampling Theory and Sampling Practice*, 3rd Edn. CRC Press, Boca Raton (2019). https://doi.org/10.1201/9781351105934
2. P. Carrasco, P. Carrasco and E. Jara, "The economic impact of correct sampling and analysis practices in the copper mining industry", *Chemometr. Intell. Lab. Syst.* **74(1),** 209–213 (2004). https://doi.org/10.1016/j.chemolab.2004.04.013
3. F.F. Pitard and G. Lyman, "Single and multi-stage Poisson processes: a case study for gold exploration", in *World Conference on Sampling and Blending*, WCSB6, Lima, Peru. Gecamin (2013).

SAMPLING SPECIAL SECTION

Never cry sampling? Denial, denial, denial—pay the price!

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Introduction

The objective of this contribution is not to add to an already large list of horror examples of hidden economic losses, but rather to raise a cry of alarm about what is experienced as a classical *denial* of cost-consequential sampling recommendations. A couple of real-world examples borrowed from the mining industry will almost tell the story by themselves.

Case 1. To dare to tell …

The first tale of denial concerns the author of this short piece, who failed for too many years to appreciate the full extent of similar situations.

A long time ago a colleague and I were asked to work concerning mine-mill reconciliations at a very large gold–copper operation in a far-away country. A cursory audit of earlier practices quickly revealed that sampling of blast holes for grade control was **not** performed to good standards. But to make things worse, the corresponding samples were not even prepared by the commercial laboratory on site; technicians simply scooped *some material* from the sample bag in lieu of the complete and tedious preparation they had been asked to implement. Demonstrations duly made to company management, the entire lab was immediately **fired** and put on a charter plane out of the country the next day. Upper management then requested that we provide an estimation of the damages incurred along the years.

We had never done this kind of a job before and we believe, until today, few professionals have really attempted it. So, we first tried with a few statistical tools, and were able to conclude that the main issue that was triggered consisted of approximately 2% of treatable ore instead ending up on the waste dump. But *denial—self-denial* in this case—crept in when it was found that actual costs to the mining company amounted to a mind-boggling **$7.5 million in yearly net profits**. In complete disbelief, we redid the evaluation using geostatistical tools instead (more powerful and more relevant than straight statistics), and purposely in a completely different way, but this only confirmed the exact same conclusion. And to be frank, it was not until several years later, when sharing courses on Sampling Theory with our late friend Pierre Gy, that our denial finally stopped for good. Pierre presented examples from his own career in which, in a very similar situation, he had reached the very same monetary conclusions.

That day, we learned our lesson about the hard necessity of *daring* to be *bold* at times, to *tell*. The sad fact is that grade control is one of those domains where the cost of bad sampling can reach unfathomable levels of losses—of *never-seen* money.

Case 2. It can get much worse …

However, in more recent times this example, which had profusely haunted our minds for decades, started to pale into insignificance in comparison to a new situation, this time concerning a large process plant. Indeed, this was in another domain where a lot of money was also at stake. This example is about a sub-optimal metallurgical processing plant. Another friend and I had audited

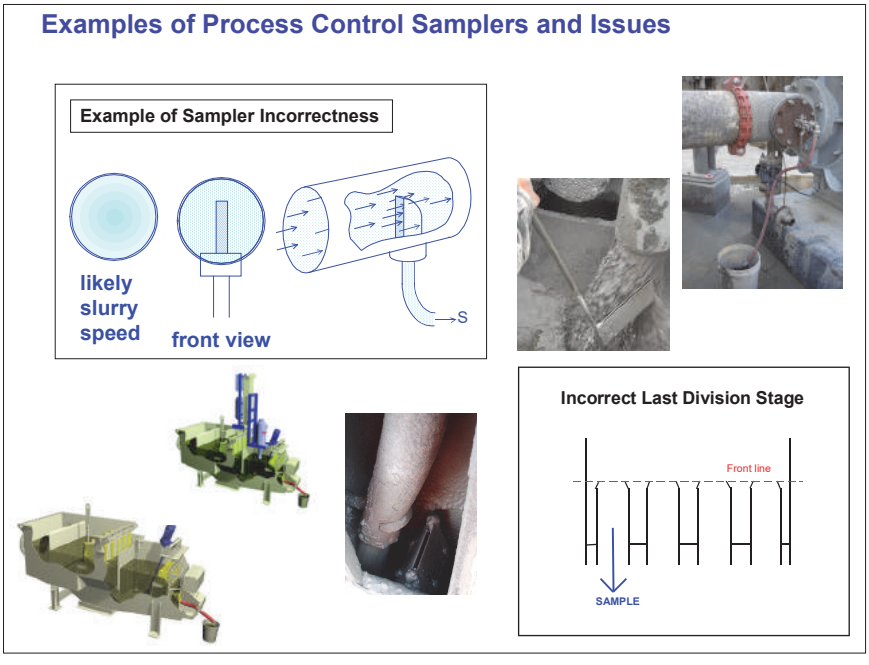

Figure 1. Examples of process samplers, all with "issues" that should always be called out!

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a copper plant in which, quite tragically, "less than perfect" process control samplers were used for metal balance. Immediately here below will be shown how "less than perfect" …

The ore deposit contained a mineralised rock type (RT#1) and higher-grade rock type (RT#2). The plant had initially been designed solely for sulfides ore RT#1, grading at around 1.5% maximum Cu content (assessed from test work). However, in an attempt to increase metal production, the mine was now sending a mixture of RT#1 and RT#2 to the mill, grading at 2% Cu on average. But the plant turned out not to be able to process this mix well with a good metal recovery (for good mineralogical and process design reasons, i.e. "bad reasons" actually). As a result, as detected by the study of mine–mill reconciliations, a large proportion of the metal received in excess of 1.5% Cu was unfortunately going through to the *tailings*, un-recovered (it turned out this was largely in the form of un-floatable, microscopic, native metal present in RT#2).

The feed **to**, and the tails **from** the plant were sampled using process-control samplers that were decidedly **not** designed for quantitative sampling. Both provided negatively biased sampling, both failing to detect this additional, denser, unrecoverable metal when it passed through. In Figure 2 these bracketing in/out "sampling" stations are symbolised by trash cans—"with good reason".

As a result, the plant metal balance matched the mine-predicted grade within reason when processing pure RT#1 (i.e. up to 1.5% Cu), but showed a large difference of ~0.35% Cu when the grade increased to 2% Cu by adding RT#2 ore. A significant part of this difference represented native Cu put in the tailings *without anybody ever knowing*, as it was undetected in both the feed and tail samples—but these sampling stations were indeed inexpensive.

Over the elapsed year at the time of the study, the mine had produced and sent to the plant, 50 Mt of ore at 2% Cu (RT#1 *plus* RT#2). In addition to the properly measured, normally unrecoverable metal (recovery is not 100%, even for RT#1), the native metal in the 50 Mt, accounting, say, for ~0.10% Cu, had gone through and was unduly lost to the tailings without anyone suspecting/measuring it. *[One may perhaps argue whether it was less than 0.10% Cu or much more, but this does not qualitatively change the mind-boggling conclusion below.]*

The additional metal loss would thus possibly represent 0.1% × 50 Mt = 50,000 t of metal Cu worth more than $7000/t on the market today. This is a staggering **$350 M for the one year in question!** Would this not be quite a nice budget with which to address process optimisation as well as the really serious plant sampling issues?

So, you may well ask: ***WHAT*** did the mining company do? It went into *denial*, finding it more comfortable internally to ignore the problem rather than facing it—the company was, after all, hugely profitable already. Hopefully, however, some day these tailings will undergo some secondary recovery process. ***WHO*** will dare to be bold and *tell*?

It is perhaps worth reflecting that, as pointed out to us by the editor, the very first job Pierre Gy was involved in was—you guessed it—re-evaluating a set of discarded tailings in a mine in the former Belgian Congo, see his own fascinating career story in Reference 1.

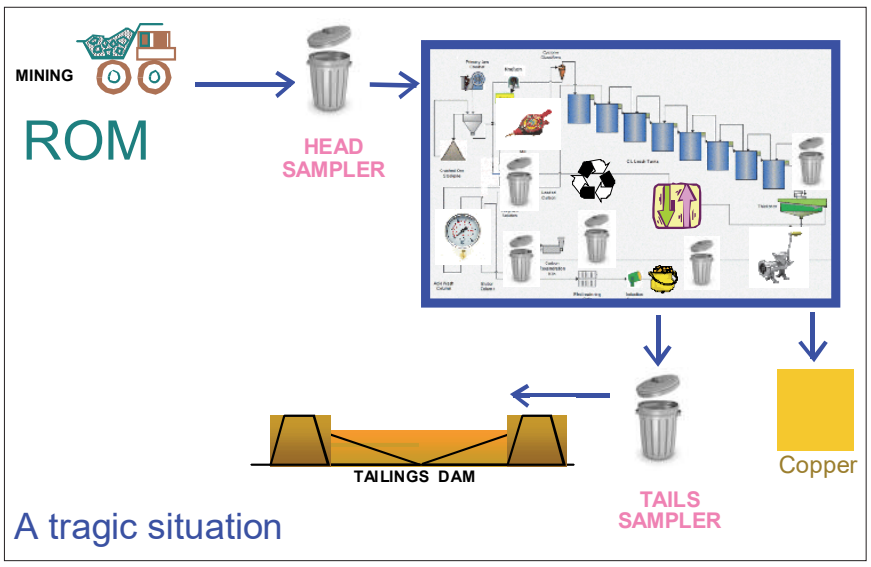

Figure 2. It is impossible to monitor and control a complicated process based only on seriously compromised sampling stations at input and output locations (here represented by the caricature of trash cans—perhaps a bit rude but this does allow the message to get through with clarity.

Conclusion

Monetary losses to bad sampling can be huge and sometimes far beyond what one may choose to believe. Denial can tragically hamper operations' optimisation and leave unseen economic opportunities by the roadside. One should indeed cry "Sampling problems" whenever encountered!

Reference

1. P. Gy, "Part IV: 50 years of sampling theory—a personal history", *Chemometr. Intell. Lab. Syst.* **74(1),** 49–60 (2004). https://doi.org/10.1016/j.chemolab.2004.05.014

SAMPLING SPECIAL SECTION

How to motivate for correct sampling projects based on costs and benefits of fit-for-purpose sampling solutions

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Editor's summary

Mining companies are generally reluctant to install high-cost sampling systems at operations that have been "functioning well" for many years. The principal objection to installing new equipment to extract correct and representative samples in process flows is the time and costs involved. The Theory of Sampling (TOS) provides a structured framework for identifying and quantifying the errors and bias associated with any sampling event, but this may often be insufficient to motivate for investment in new, correctly designed sampling equipment. Financial losses arising from substandard sampling installations are usually *disregarded* because value-added economic benefits from good quality sampling solutions are most often *invisible*, while the adverse cost from inappropriate systems are plainly obvious. Depending on the specific needs, a Fit for Purpose (FFP) sampling solution may be acceptable, provided the magnitude of sampling errors is understood and assay results are interpreted accordingly. Categorical levels of acceptable accuracy and precision can be established depending on the sampling position in the mining value chain and the nature of the decisions to be made. Objective benefits of proposed FFP sampling solutions must be presented to relevant decision makers in such a way that adverse subjective decisions based on only poorly resolved economic information are made difficult (impossible). Examples of benefits from motivating such implementation of FFP sampling solutions at sampling facilities around the world, are presented.

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Introduction

Humans rationalise differently based on individual cognitive processes or reasoning (culture, experience, educational background, management level). Decision-makers in mining financial departments may rationalise very differently about spending funds on corrective sampling compared to technical sampling experts, meaning that well-conceived corrective sampling projects could be rejected. Irrespective of the appropriateness of the technical design, or imperatives of the Theory of Sampling (TOS), approval for implementing a corrective sampling solution is declined unless value-added benefits can be defined and corroborated with numerical proof. The fact that the costs of poor sampling *never* show up as a line item in annual financial statements means managers do not see a figure that represents a *loss* to their earnings. Attitudes towards meaningful expenditure on correctly designed and installed sampling equipment, therefore, remain obstinate. The rationale for the implementation of improved sampling solutions must be presented as simply as possible leaving little room for subjective interpretation.

Estimating added value from implementing sound TOS-based sampling practices does not require a long or complicated report. A simple "back to basics" approach, pointing out the critical points and findings with a summary of the financial benefits provides a much better chance of getting your point across. The "sampling fraternity" should also acknowledge that implementation of the "close to perfect sampling solutions" are not always feasible. The need to make compromises, knowing that not all solutions are perfect, may still provide *some* added value by reducing sampling errors compared to current operations.

Here is a look at a back-to-basics approach for demonstrating the benefits of installing corrective sampling protocols and equipment to those approving budgets and to reluctant shareholders.

General concepts

Several observations have come to light as a result of presenting appeals to the boards of companies for funding to improve sampling facilities.

Distrust of large unsubstantiated numbers

The adage that "If something sounds too good to be true, then it probably is" is still applicable. Statements that say "… better sampling can produce an additional US$25,000,000 worth of on-grade ore *per annum*…" sound too good to be true and arouse a certain level of scepticism in the listeners. Numbers this large appear unrealistic and are probably not trustworthy unless they can be validated by realistic examples. It is better to begin by offering small numbers, for example "…correct sampling can deliver an additional US$7500 for a 100,000 t shipment of iron ore…" and allow the decision makers do the mental arithmetic themselves. Producers who, for example, deliver over 3400 shipments of this size per year can easily see the potential value of better sampling.

Disinterest in statistics without practical application

Technical statements with equations and figures could be lost on non-technical managers and executives. What they would prefer to hear is "…let me show you what improving your Sampling, Preparation and Measurement (SPM) can do for your profits."

Executive committees have neither the time nor the interest in detailed or complex calculations of precision that technical jargon alone will seldom motivate budget approval. The insight, interest and understanding of sampling technicalities is **not** common amongst such audiences, but simple statements such as "…every 0.01% improvement on the SPM precision could potentially increase the on-grade production by 0.01%..." are more likely to pique their interest long enough for budget approval.

Penalties for out-of-spec products

The cost of underestimating the target analyte content is not reported in financial statements as it is not quantifiable, and in many cases misunderstood. What should be seen on a company's financials are the costs incurred due to *penalties paid* for delivering off-spec grade ore and deleterious elements that exceed allowable limits. Quantifying the amount paid in penalties highlights a quantifiable "real cost" that could be reduced by implementing better sampling practices and systems.

Names mean more than numbers

The role and responsibility of financial managers may very likely not allow them the privilege of time to digest the theoretical aspects of the TOS. Collaboration and certification of designs by well-known reputable experts add greater confidence to improved sampling solutions than complex formulas. Recruiting specialists who are able to validate or verify decisions about proposed sampling installations is an effective method of convincing project owners and shareholders that improved sampling will be money well spent.

Few are impressed by jargon and equations

Correct sampling nomenclature and application of equations to estimate financial benefits is critical, but shareholders and financial managers are unimpressed with complex procedures and formulas. Shareholders are much more likely to approve projects provided one can demonstrate that the estimated added value through improved sampling has been validated by reputable persons or sources.

Fit for purpose sampling

Providing correct sampling solutions is a high-cost exercise. Inevitably the more stringent project owners are about installing correct sampling solutions, the higher the capital costs will be. This article does not dispute the fact that samples cannot be trusted if the principles of the TOS are not followed. The rational man strives for perfection, but knowing that things in life are rarely perfect, this article wants to demonstrate that "inferior sampling" may sometimes still offer value. Of course, the value depends on how the resulting analysis and associated confidence intervals will be used. This also assumes that benefits are not outweighed by the cost of taking, transporting, preparing and assaying the sample—a simple concept, but one that may in fact be overlooked by the sampling fraternity.

ISO standards vs the TOS

"ISO compliant" is a term often used when discussing upgrades to sampling facilities. ISO standards, although critical in establishing standard methods of sampling, preparation and analysis for various commodities and materials, are too often used out of context! There appears to be a widespread misunderstanding that compliance with a relevant ISO standard will *automatically* result in the best possible sampling results. ISO standards should be looked on as *recommendations* to be used to assist a facility in ascertaining a minimum precision and level of confidence in accordance with international standards. However, improving sampling equipment and methods above the minimum requirement recommended by ISO standards, can indeed add further value to a producer. As an example, if iron ore producers reduce the precision on a 270,000 tonne lot, below the β_{SPM}[a] of 0.34% Fe stipulated by ISO 3082, substantial improvements in on-grade ore production can be achieved.

Compliance is one thing, but before setting a limit on the resources assigned to sampling and analysis, it is critical to establish the monetary value of each sample using a minimum requirement, compared to what it could be worth if additional resources were spent on improving it. Rather than strict compliance with minimum requirements from relevant ISO standards, other aspects, especially efficiency for operational purposes, should be considered when specifying a sampling point. For example, taking a higher number of primary increments, and analysing more subsamples than required by ISO during the loading of an iron ore vessel can give a better indication of the grade during loading. This can then be used to blend the product mixture that goes into the vessel more efficiently.

Although some principles *may* be the same it is definitely unwise to apply guidelines from a single ISO standard to *different materials*. For example, the ISO 3082 for iron ores should **not** be used to estimate sampling compliance of phosphate fertilisers, because the material characteristics are in fact detrimentally different; in this case the proper material-specific standard must be applied. Where no specifications are available for a material, TOS practices such as heterogeneity tests and sampling calibration for establishing nomograms should be applied, although the work efforts (costs) are never small. Explaining the need for, and the workings of, a full-scale industrial heterogeneity test is no small feat in the board room.

Motivating a sampling project

The objective of a sampling study should be to demonstrate how improved sampling practices can improve profitability. Motivating a project to improve

[a]Overall precision for Sampling Preparation and Measurement $\beta_{SPM}=2\sigma_{SPM}$.

sampling equipment and protocols requires an understanding of the complete value chain so that expectations about the outcome of the sampling project are met. This may include defining the levels of reliability and precision to be achieved as a result of improvements to sample extraction, sample handling/transport, sample preparation and measurement. Motivating for added value and increased profit from improved sampling precision **must** be supported by realistic estimates of the capital cost and required investment. The capital investment needed for taking better samples using better sampling equipment, ensuring the correct sample size and sample frequency, must be calculated accurately. In addition, the costs of ensuring samples are correctly transported, prepared, analysed and reported, within an acceptable turn-around time that allows real-time changes to be made, must be established. The total investment required in terms of time, money and effort must be understood by managers and financial officers, if proposals to improve sampling processes are to have credibility and integrity. The skills of producing a convincing *business case* should be on everybody's agenda, sampling experts no exception.

Case studies and examples

Rapid turn-around vs high precision

A smelter aimed to get less than 5 ppm precious metal in slag which it sells as silica waste for US$3/tonne. Slag analyses over a six-month period indicated the precious metal content to be as high as 18 ppm. The operating costs to recycle slag through the furnace, after it has cooled, is approximately US$5700/tonne, so although the sampling error could be reduced by improved sampling methods, the cost-benefit would be minimal unless the samples can be collected and analysed *before* the slag is cast. On average around 10 tonnes of slag is produced per cycle, meaning that the value of precious metals in a single slag cycle would have to be greater than US$57,000 to make re-cycling through the furnace feasible. If samples can be collected and analysed before the slag is poured, then remedial actions such as increasing the residence time in the furnace can be performed. In this case precision of the result is not as crucial as the sampling-to-analysis turn-around time so this is where the focus for the project was placed, and rightly so. But if the furnace charge is analysed before smelting it is possible to modify the charge and reduce the risk of precious metals reporting to the slag. In this case the precision is more critical to the process as small variations in the charge can affect the smelt efficiency.

Reducing the loss of precious metals in the slag to less than 5 ppm would result in an additional precious metal production of over US$400,000 per annum. This figure, based on historical data gathered from the smelter, compares the actual gold content of processed slag with what it should have been if the Au grade was maintained below 5 ppm. Assuming a three-year return on investment, a sampling solution costing less than US$1,200,000 would indeed add value. This example illustrates the necessity of understanding the economic and logistical implications, limitations and the effect of improved sampling *before* proposing a sampling-to-analysis solution.

A less than perfect sampling solution may still add value

A less than perfect sampling solution was observed at a phosphate fertiliser production plant where phosphate slurry is mixed with H_2SO_4 in a reactor vessel to produce phosphoric acid. *Dip samples* are taken between 50 cm and 500 cm below the slurry surface (sampling experts would collectively frown severely!). Concentrations of free sulphate, measured by titration, and phosphoric acid cannot be quantified in the complete vessel with any certainty using this imperfect sample type, but some knowledge of the concentrations is critical for controlling processes in the reactors.

In this case, time is of the essence. It is critical that the sample be filtered and analysed as soon after extraction as possible because the reaction in the sample container continues as the sample cools, affecting the analytical results significantly. To improve the integrity of the sample, an insulated sample container should be used to collect the sample (Craig Ritchie, 2018, *personal communication*). In addition, rapid transport to the laboratory using a pneumatic air tube conveyance was strongly suggested.

Too many compromises invalidate the sampling point

When faced with tight deadlines and minimal budgets, project managers and engineers often must make compromises in correct sampling to complete a project. Although some compromise is almost always required, too many can completely invalidate the sampling solution being used. For each compromise made, the total effect on the result should be established to understand the ultimate financial impact on the plant accountings. This is also where the project owners must know what is needed as a minimum to achieve the required sampling precision and to meet all the operational requirements to ensure that the sampling solution providers they are using do not lead them astray. Using internal resources or external specialists to define what is needed for sampling installations is one method to achieve this, which is a lot more cost effective than attempting to investigate and repair "what went wrong" **after** the system has been implemented.

Good sampling to analytical practices

At critical operations, such as port loading facilities, correct sampling together with rapid sample preparation and measurement can have massive financial rewards for the company.

As an example of this, in a well-designed iron ore port facility every reasonable effort was made to minimise SPM errors in the facility. This, together with the addition of automated sample preparation and analysis, not only makes the system more precise but also offers the value of rapid preparation and analysis. For this facility, even though the sampling building did not have enough head room to fit a standard traditional cross-stream sampler, an

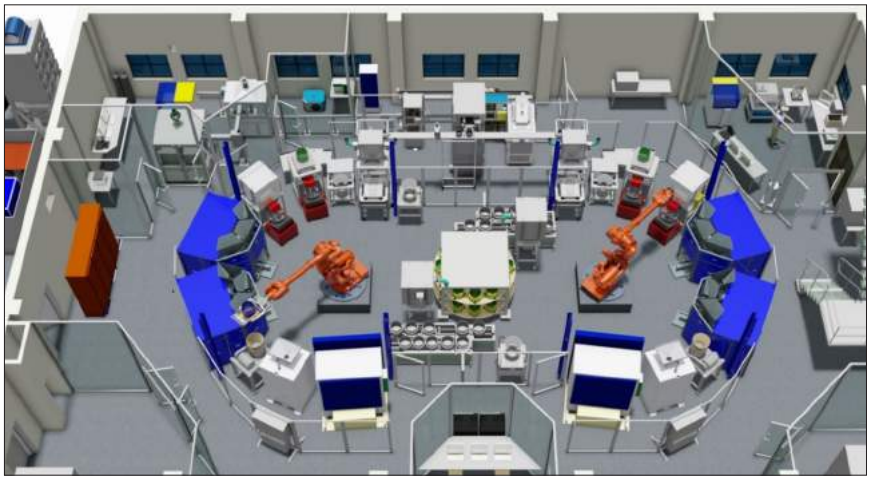

Figure 1. Robotic automation for sampler preparation and analysis for chemical, PSD and moisture conveyor.

innovative alternative was developed (which involved a long radius swing arm which crossed the stream and lifted the sample material to the floor above). This key contribution required that both the client and supplier had a strong understanding of the sampling requirements and worked together to find a feasible solution. The result of this is a turnaround time on results of less than 6 h after the completion of the ship being loaded, with precision of less than 0.15%.

Value of investment

The iron ore producer has not released the actual value of the investment to make this facility perform as it does today, but the following available information will suffice.

1) The final analysis is available 6 h after ship loading is complete; this was previously a minimum of 48 h. The benefit is that the lot can be invoiced nearly two days earlier than before, resulting in an estimated gain of US$5500 per 270,000 ton shipment on *interest* alone.
2) For large lots, the laboratory can release data during ship loading with a precision of less than 0.15%. This data is used to adjust the blend between low- and high-grade ore to control the final blended grade being loaded.
3) The iron ore producer can use the high precision as proof of the quality of its product thereby giving them a commercial advantage over their competition (*bragging rights*).
4) Reduced risk of penalties due to deleterious elements exceeding upper specification limits of the lot, or lower than specified Fe content.
5) Disputes over the quality of the ore loaded through this terminal are quickly settled due to the overall compliance of the complete facility with the relevant laboratory and material specifications.

This plant has proven that rapid analysis with consistently high precision is possible, so the producer is currently investigating upgrades and expansions of this facility to increase sampling and analytical capacity with the aim of optimising in-ship grade blending.

Conclusions

A full understanding of the complete mineral production and sales process before and after the sampling point is essential. Such insights allow one to appreciate the current usage of the sampling results, as well as identify other potential uses and added value opportunities, especially if the quality of the sampling can be improved. Ascribing a monetary value to a sample in a process or procedure is an important asset. An understanding of the levels of precision or sampling correctness of a sample, as well as the turn-around time to analysis will influence the value. An appreciation of various constraints, such as applicable standards or specifications, physical space available, accessibility for inspections, maintenance and sample collection, plant down time available for the installation, distance of sampling point to the laboratory must all be taken into consideration to create a fit-for-purpose sampling solution.

In the process control domain, fit-for-purpose sampling has a wider scope as compromises can be made in various aspects of sampling to suit the application, provided the consequences of these compromises are understood. In the product control domain, where the same compromises cannot be made, fit-for-purpose sampling can still be applied when considering how the sampling will affect other aspects of the facility such as material throughput and the value of faster sampling-to-analysis. Using a cross-stream sampler will not affect loading rates, whereas a stop belt sampler will; therefore, the cross-stream sampler is fit-for-purpose in this case.

The feasibility of motivating and actioning an upgrade process required by a facility to exploit the expected value-add from the specified sampling solution also requires insight and understanding. Some of the actions can include automated sample collection and transport systems to increase turn-around time from sampling to laboratory. Other actions could include upgrades to the laboratory to accommodate the sampling solution and an upgrade to standard operating procedures. The result of such actions would enable the facility to respond to improved sample information to achieve added value possibilities.

It is important that project owners communicate their needs to sampling equipment suppliers in a way that ensures the supplier provides the best solution for their application. Lowest cost procurement should not govern the choice and selection of critical success equipment, of which sampling equipment range very high, as this is likely to turn out to be a costly error of judgement. A policy of lowest cost procurement is short-sighted as it may mean not only inferior equipment selection, but also incorrect sample extraction that

introduces costly bias associated with poor sampling.

The greatest deterrent of decisions to install appropriate or improved sampling solutions is that the benefits are *invisible*, while the adverse costs are *obvious*. No financial statements tell CEOs what the benefits from proper, FFP or indeed fully representative sampling are. The only indications might be increases in the costs of reagents and consumables. When presenting a sampling solution to an executive committee, with the hope of getting approval, the primary motivational factor should be a clear demonstration of the *monetary benefits* the solution can offer. The basis of the motivation should be a comparison of cost-to-implement against the long-term expected returns on investment. All mathematical and other technical aspects of the proposed solution should be kept as straightforward as possible with only key results presented, but which are verified by a well-respected specialist in the field.

Caveat

Of course, there always also is the option of being able to *explain* adequately more of the essential technicalities in a manner that is fully understandable, so that management, CEO's board members, investors … actually gain an increased factual knowledge. This will always be part of a *best* business case. This challenge still leaves *room* for the diligent, competent, didactically motivated fraction of the international community of sampling experts, who not all necessarily need to rush off to get a MBA degree—team collaboration will always go a long way!

Sampling quality quantification: the key to support business decisions

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Introduction

Current practices to evaluate the operation of sample stations that support processing and metallurgical balance are typically based on visual inspections. For example, material build-up on cutters, sample spillage, reflux while sampling, pegging on sizing screens and worn cutter lips are all most unwanted discoveries. But, being subjective observations, these do not allow quantification of the impact on the samples collected, production process or on the reliability of metallurgical balancing when deviations are found. For this reason, they are traditionally just considered as "good practice" recommendations or, N.B., as an **extra cost for the business**. Because they are only qualitative observations, it is quite difficult to generate and quantify a business case related to their impact with which to support an investment in better, i.e. more reliable, sampling systems. To complement the current visual regimen from a sampling and QA/QC perspective, this contribution illustrates the value of also using process monitoring practices, results and controls to proactively quantify the quality of the sample information, especially at the primary sampling stage. This allows the desired business cases to be completed with quantitative cost estimations.

Variability

Several papers have been published regarding the applicability of variograms as a useful tool to quantify industrial processing variability,[1–3] including new developments with variograms targeting *continuous monitoring of measurement system performance*.[4]

This "proactive approach" includes the use of daily production grade information in variograms for control process to quantify the variability of each of the sampling points deployed in, for example, a metallurgical process.[4,5] The most important advantage of this methodology is the use of the *additional* available information without extra budget requirements. This leads to higher monitoring relevance and reliability, because this augmented process modelling can be performed more frequently and the results will better reflect "day-to-day" variability in the process—which allows better insight in the process variability. The ultimate aim is to calculate the variographic nugget effect, $V(0)$, better; i.e. the viewpoint where "a sample is compared against itself", because this represents the total sampling-and-measurement error (expressed as a variance).

Bias testing

In industry, bias tests are often suggested, or contractually mandated, to compare a production sample obtained against the material it supposed to represent at the control point. Many international standards recommend bias testing—almost universally.

But bias tests require *interruption* of the regular production process in order to extract material from the conveyor belt with a mutually accepted "reference sampling" method. For this reason, bias tests are in reality not popular in industry ("we lose a lot of money and time having to interrupt our process many times") and are, therefore, usually performed only reluctantly, or not at all! Because of this, companies are unavoidably exposed to higher risks than necessary, since it is simply assumed that the processes involved are not affected by a monitoring (i.e. sampling-and-analysis) bias. For this reason, "data quality representativeness" is an unknown characteristic. However, sadly, unchecked data obtained by process monitoring with un-evaluated methods are nevertheless very often still assumed to be the "truth". There is a demonstrable loss of potential process information here and the ultimate question is not difficult to formulate: "what are the hidden costs involved for allowing this complacency?".

Under the reasonable demand that a representative sample is one that accurately represents the "DNA of the lot material" by including *all* the components in the lot in their *correct* proportions, the "augmented proactive approach" to be presented below includes the use of grade–grain size distribution curves of the samples obtained daily. These can then be used as convenient reference information for process control. The following case example contains some technical details, which can be skipped if interest is solely in the economic consequences hereof.

Case example

This is an industrial example where a quality programme (QA/QC and QM) has enabled a new level of observation and quality quantification, developed and implemented after serious information gaps were determined by visual inspection.

1) Visual field inspection of a key sample station revealed consistent deviations in the operations (Figure 1): a) the primary cutter is too narrow for coarse material, b) the secondary cutter is not working, the sample goes straight to the bucket, c) lumped material is not crushed, d) samples are not collected as per time requirements (electrical issues)

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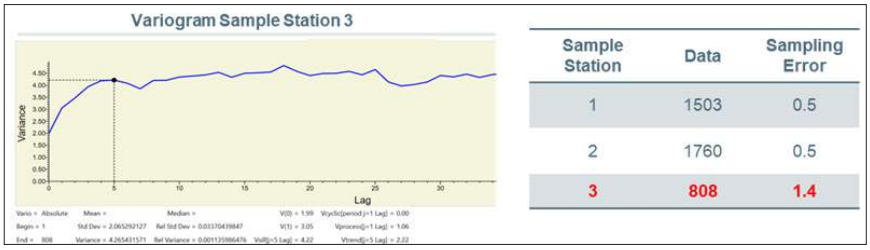
Figure 1. Quantified field evidence (right) collected after a visual inspection of the primary sample station shown on the left. The deviation between the expected nominal top particle (10 mm) and the factually observed size is dramatic.



Sample Station	Data	Sampling Error
1	1503	0.5
2	1760	0.5
3	808	1.4

Figure 2. Variograms performed for the three sample stations at the key site shown in Figure 1. Sample Station 3, the one identified and highlighted by visual inspection, clearly shows the largest Total Sampling Error. Variogram analysis is consistent with the visual inspection, and now *quantified*.

and e) while the expected Nominal Top Size is 10 mm, the real Nominal Top Size 60 mm!

2) Despite these deviations being correctly reported, the site team was struggling with communicating and getting the attention of senior levels, because the impact for the business calculations could not be *quantified*.
3) Appropriate variogram analysis was performed over the three sample stations at the site (Figure 2), which showed that the error of the singular failing station was three times *larger* than for the other two. Thus, the impact on sampling variability was finally quantified, the consequence of which is an increased risk for a non-compliant product, endangering the bottom line.
4) In terms of Bias, the grain size distribution of the failing sample station 3 was compared against the same material sampled at the loading port, and a preferential trend towards collecting more fine material on site could be observed. This allowed the quantification of the *underestimation* of the grades reported from this sample station (Figure 3).

Quantification at last

Variograms and grain size distribution analyses are here suggested to be used as the base for a proactive approach in production. Where performed, the impact of the deviations originally observed by on-site visual inspection only, could now be better *quantified and communicated to the organisation*. In terms of variability, the market always values long-term stability in the product, where any consistent variability reduction can represent an opportunity for a higher price during contract negotiation. For the mass product industry this represents a very important revenue opportunity due to the millions of tonnes produced in general by mining companies. This is why a continuous monitoring and

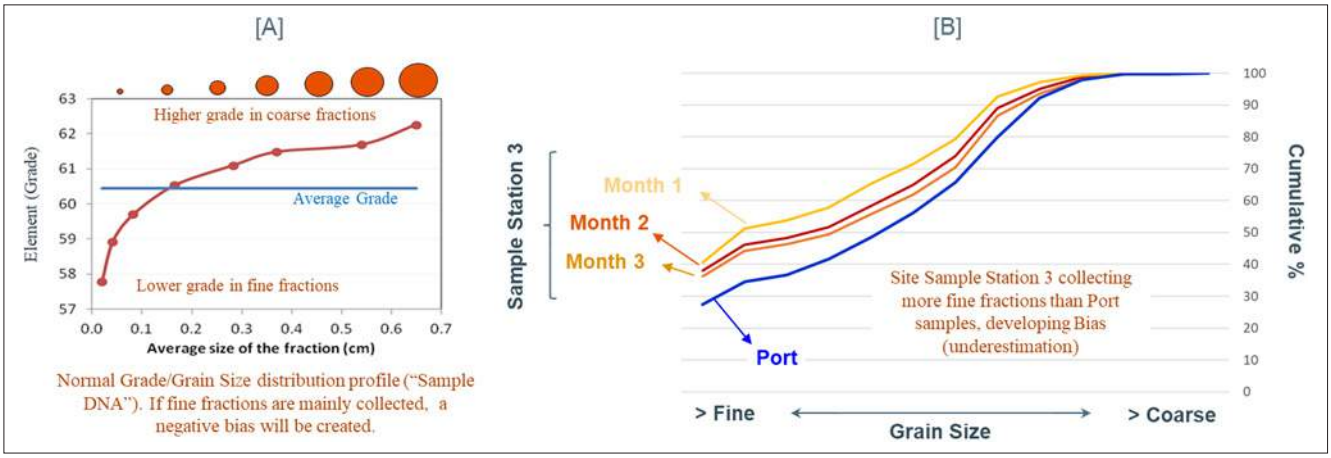


Figure 3. [A] Normal grade/grain size distribution profile (the "sample DNA") shows the impact on the overall sample grade if a preferential extraction of fine, or coarse, fractions prevail—this will assuredly generate a bias. [B] Grain size distribution analysis performed on samples from the faulty sample station 3, as compared with the same material sampled at the loading port, attesting to the same biased extraction of *too much* fine material.

quantification can lead to these, easily utilised opportunities.

In terms of bias, percentage deviations as small as 0.1–0.5 % bias are normally just considered as "minor" in some production environments. However, and due to the number of tonnes produced, these "minor" differences can represent a huge business impact. For example, for a mine producing 10 M tonnes, a 0.1 % Fe and 0.5 % Fe bias can represent an impact of **US$1.6 M** and **US$8 M**, respectively (assumptions: iron ore fines are based on the 62 % index, with an average price of US$100).

Conclusions

International Standards (depending on the commodity) are used to establish the methodology to be followed to setup and operate sample stations, but these requirements are normally only inspected or audited visually, compromising a full quantitative assessment of sample stations performance.

The risk for companies relying only on visual, qualitative assessments is creation of a potentially "false sense of security", where no detrimental issues are noted, or, when major defects are detected, impacts and risks are very hard to quantify to develop a relevant remedial "business case".

This contribution presented a case example showing the importance of implementing a QA/QC and QM programme on sample stations, as a *complement* or *enabler* of a sustainable compliance to International Standards. But also to further the opportunity of quantifying the performance of sample station performance, and to provide a "proactive approach" regarding deviations in the mining plan. This will potentially reduce operative costs, e.g. optimising the ore processing circuit), or optimising a blending process a.o. all of which will lead to an improved and optimised resource value.

Never underestimate the value of even a "minor bias"—your extra costs may be anything but minor!

References

1. R. Minnitt and F.F. Pitard, "Application of variography to the control of species in material process streams", *J. S. Afr. Inst. Min. Metall.* **108(2),** 109–122 (2008). https://www.saimm.co.za/Journal/v108n02p109.pdf
2. R.C.A. Minnitt and K.H. Esbensen, "Pierre Gy's development of the Theory of Sampling: a retrospective summary with a didactic tutorial on quantitative sampling of one-dimensional lots", *TOS Forum* **7,** 7–19 (2017). https://doi.org/10.1255/tosf.96
3. K. Engström and K.H. Esbensen, "Variographic assessment of total process measurement system performance for a complete ore-to-shipping value chain", *Minerals* **8(7),** 310–334 (2018). https://doi.org/10.3390/min8070310
4. E.I. Wikstrôm and K. Engstrom, "Development of automated variographic characterisation for continuous monitoring of measurement system performance in iron ore processing - a case study at LKAB, Sweden", in *9th World Conference on Sampling and Blending (WCSB9)*, Ed by WCSB9 Organising Committee (2019).
5. O. Dominguez, "Quality assurance quality control (QAQC) and quality management (QM) programs on sample stations: a prerequisite to achieve standards requirements", in *9th World Conference on Sampling and Blending (WCSB9)*, Ed by WCSB9 Organising Committee (2019).

SAMPLING SPECIAL SECTION

Sampling in society, environment, public health, pharma, trade

Section contents

Food and feed sampling: balancing ethics and money

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I first accepted the editor's invitation to contribute to this special issue on "economic arguments for representative sampling" with great enthusiasm. Alas, a few hours later, the enthusiasm started to fade because the many experiences of resistance to putting the Theory of Sampling (TOS) into practice in the food and feed sector came back to me. However, upon considerable soul searching, there may still surely be hope!

A personal statement

I have devoted about 20 years of my professional career to studying and regulating food and feed sampling standards and normative documents.[1–8] The good news is that many of them (though not all) claim that sampling *should* be representative. The bad news is that almost none goes as far as claiming representativeness as a *mandatory requirement*, the only exceptions being DS 3077,[9] Recommendation EC 787[10] (2004) and prEN ISO – 21568 (2005). The unavoidable result is that these standards fail when applied in practice, creating a breach between the principles behind the TOS's goals (good) and its application to everyday reality in the food and feed arena (bad). Thus, sampling is often felt as a necessity to be fulfilled to collect material for analytical investigation—clearly not knowing or reflecting on how important this information is for making societal decisions about public nutrition and health. However, reducing to a minimum the time devoted to sampling ("the faster the better") and minimising the associated costs ("the cheaper the better") will sooner or later sacrifice sampling quality and reliability. As for everything else in life, quality does not go together with speed and lack of resources.

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Setting a constructive scene

Since the present focus is on economic arguments for doing the right thing, instead of repeating that non-representative sampling is useless by definition, and that every penny spent on collecting *specimens* and analysing them is wasted, I would rather tackle the issue from the other end, exploring what happens when "something wrong" is detected in a food or feed product.

Looking rationally at the costs involved

When a food or a feed product turns out to be non-compliant with *a priori* established quality/safety criteria, the product needs to be *removed* from the market. What are the costs of removal? Per product the overall financial losses include all production, distribution and selling costs already sustained before the decision to pull from the market. Plus the costs necessary to i) map the supply-distribution followed to place the product on the market; ii) removal of the product from every supermarket counter and storage room across all the regions, countries and possibly continents to which the product was distributed; iii) costs to destroy the product. Arguably, these total costs are much, much higher than the cost required for the *a priori* application of a TOS-compliant sampling method, allowing the analysis of representative samples to support well-substantiated and informed decisions *before* market release.

When we total up the costs for this, grave problems become evident. Because of the vast amounts and tonnages involved, the costs are in fact so massive that they cannot even be estimated with reasonable precision, but they are guaranteed to be **huge**.

Scientific and technological understanding does not hurt

Under a less catastrophic scenario, a reliable understanding of human and animal *exposure* to certain substances (e.g. pesticides) is an important and wise requirement under many jurisdictions. The earlier it is understood that *only* representative samples reduce the possibilities of either *mis*-estimating actual exposure levels for humans and animals or, worse, *under*-estimating the risks for consumers to exceed tolerable intake levels, the better for society. This is also important in the case of foods and feed with nutritional benefits, where under- or over-estimating intake levels may lead to nutritional or deficiency problems. This also plays a critical role regarding surveillance of foods and feeds with unintentional contaminants or intentional adulterations, due to their often-low concentration levels and highly heterogeneous distributions. Watching out for these societal risks ranks among the prime objectives of national and international regulating authorities charged with *consumer safety*. These are goals worthy our most ardent efforts. But are we doing well enough?

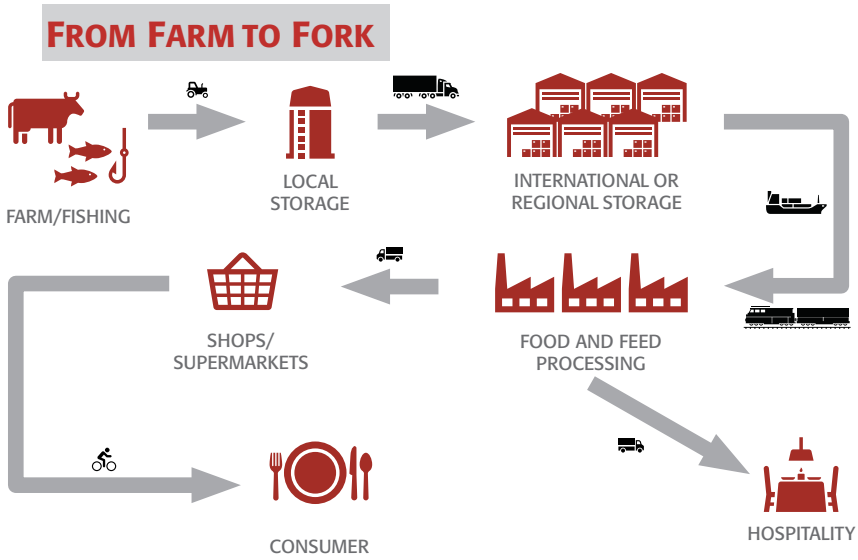
Reality check: very different objectives and usages of the TOS

Well, in today's food and feed arena, sampling continues to be perceived more as an economic *burden* and a technical necessity to be fulfilled because of regulatory demands, rather than a need to *ensure* proper citizen and/or animal protection.

Also, readers of this column could well be staggered, and maybe confused,

by the completely different attitude towards sampling between, for example, the mining/minerals/cement and the food & feed industry sectors. In the world of geological resource-based businesses, incorrect sampling means huge economic losses as value, cost and profitability estimates can be made precisely because the TOS is available. Here the TOS can be seen as the operative element safeguarding the business endeavours, see examples from the wide history of TOS applications, well substantiated in the annals of the world sampling community. Whereas in the food and feed business, sampling is a scientific tool to verify the accuracy of specific product claims, or to search for possible contaminants, or toxins, allergens, pollutants etc. Here, in essence, sampling means *searching* for possible *problems*, or verifying their absence to a certain degree of confidence (the concept of "risk assessment").

This contra-positioning is a key point for samplers, process engineers, managers, regulators, investors: *IF* from a practical point of view, exploration and searching for metalliferous resources and ores[a] is not so different from searching for, e.g., aflatoxins in a 60,000-ton shipment of grain kernels, or searching for accidental manufacturing residues across millions of chocolate bars (or a thousand barrels of pet food)—in practice the *motivations* for investing in correct sampling are markedly different. In the mining/minerals sectors the better the sampling the better for business (better in a straight economic optimisation sense), whereas in the food/feed sectors the better the sampling, the higher the risk of lot rejection or similar, which always carries a heavy *negative* economic penalty. What is good for one type of business is bad for another—what is good for one type of societal enterprise, is bad for others—the gamut of TOS applications in the last 20 years documents this dichotomy.



The food supply/production pathway: "From field to fork".

Balancing the opposites

The need for *balance* between integrity and financial gain opens up a quite different discussion on a higher level: one about direct use and benefits vs indirect and intangible disadvantages of the TOS involvement, which in the main goes beyond the purpose of the specific topic of this column, but here is at least the gist of it.

When sampling is executed to check for compliance with legislation requirements (i.e. regulatory sampling) it should be of crucial importance to ensure a high degree of confidence that the survey is accurate (unbiased) and that the compound sampling error is as small as indeed possible, within specified economic and workload boundaries. Specifically, if there is a legal threshold limit set for acceptance of the presence of a specific substance, all adopted sampling protocols must ensure that such threshold is respected with the specified degree of confidence. Of course, the lower this limit is, the greater the demands will be upon the sampling procedures and plans—and this cannot avoid being associated with *some* added costs.

Europe has established a very stringent approach to food and feed safety, monitoring products throughout all the steps of their production chain, "from farm to fork". Embedded into such a solid and ambitious safety strategy, and almost always out of sight, there is a high demand for accurate and precise, i.e. representative, sampling procedures, capable of ensuring reliable estimations throughout this entire pathway, leaving very little space for shortcuts behind the cheap and fast collection of meaningless (i.e. non-representative) *specimens*.

Where does this leave us—Trust!

In the food and feed sector, however you look at sampling, it is *never* only about money: it is about ethics **and** money. Correct sampling is not a money maker as in other sectors. Appropriate sampling is about being accountable for the *trust* that society puts into governmental and inter-governmental control systems for the safety of food and feed products. Society has no other choice!

After reading this article, you will sooner or later open the refrigerator and eat food that you bought at a supermarket. You *trust* it as safe. You *trust* that the control system worked to protect you. Consciously or unconsciously you *trust* the sampling adopted by such a control system was appropriate, i.e. representative, meaning that the safety decision taken applies also to the portion you

[a]Please don't *just* think of gold or diamonds, which geologically are kind of atypical resources—distinguishing themselves only by the societal agreement that they *represent* great value. The value of the much more voluminous base metals a.o. commodities, is vastly greater.

have in your refrigerator. If you again think of the dimensions of the global market, this is extremely far from being a trivial personal issue—the job to ensure for food and feed safety for all consumers is enormous! Ultimately, the money invested for correct sampling is money invested *for* the citizens who have neither the means, nor the knowledge, to verify. This *trust* should have much more exposure within and especially beyond our scientifically and technically driven community. This *trust* should become the root reason to ensure a continuous and open dialogue between TOS experts and *those* who decide what ultimately is allowed on the market: the *consumers* eat what reaches supermarket shelves.

After 20 years—my last effort?

Allow me to borrow Dr Vogel's statement (elsewhere in this publication): "If 'representative' is removed from the sampling process, all 'piece of mind' goes away!".

The worst situation is that as long as nobody finds *problems*, everybody lives happily. Alas, everybody lives, but *blindly*! Are we ready to deal with these topics—going beyond profitability—transparently and honestly? Until now this would not appear to have been greatly successful.

Hopefully, the future debate will fuel more active measures, including reactions to this multi-authored contribution, surprising us!

Disclaimer

The author declares no competing interest. Claudia Paoletti is employed by the European Food Safety Authority (EFSA). The positions and opinions presented in this article are those of the author alone and do not necessarily represent the views of scientific works of EFSA.

References

1. K.H. Esbensen and C. Paoletti, "Theory of sampling (TOS): *pro et contra*", *Spectrosc. Europe* **30(1),** 23–26 (2018). https://www.spectroscopyeurope.com/sampling/theory-sampling-tos-pro-et-contra
2. C. Paoletti and K.H. Esbensen, "Distributional assumptions in food and feed commodities—development of fit-for-purpose sampling protocols", *J. AOAC Int.* **98(2),** 295–300 (2015). https://doi.org/10.5740/jaoacint.14-250
3. H.A. Kuiper and C. Paoletti, "Food and feed safety assessment: the importance of proper sampling", *J. AOAC Int.* **98(2),** 252–258 (2015). https://doi.org/10.5740/jaoacint.15-007
4. K.H. Esbensen, C. Paoletti and P. Minkkinen, "Representative sampling of large kernel lots I. Theory of Sampling and variographic analysis", *Trends Anal. Chem.* **32,** 154–164 (2012). https://doi.org/10.1016/j.trac.2011.09.008
5. P. Minkkinen, K.H. Esbensen and C. Paoletti, "Representative sampling of large kernel lots II. Application to soybean sampling for GMO control", *Trends Anal. Chem.* **32,** 165–177 (2012). https://doi.org/10.1016/j.trac.2011.12.001
6. K.H. Esbensen, C. Paoletti and P. Minkkinen, "Representative sampling of large kernel lots III. General considerations on sampling heterogeneous foods", *Trends Anal. Chem.* **32,** 178–184 (2012). https://doi.org/10.1016/j.trac.2011.12.002
7. C. Paoletti, A. Heissenberger, M. Mazzara, S. Larcher, E. Grazioli, P. Corbisier, N. Hess, G. Berben, P.S. Lübeck, M. De Loose, G. Moran, C. Henry, C. Brera, I. Folch, J. Ovesna and G. Van den Eede, "Kernel lot distribution assessment (KeLDA): a study on the distribution of GMO in large soybean shipments", *Eur. Food Res. Technol.* **224,** 129–139 (2006). https://doi.org/10.1007/s00217-006-0299-8
8. C. Paoletti, M. Donatelli, S. Kay and G. Van den Eede, "Simulating kernel lot sampling: the effect of heterogeneity on the detection of GMO contaminations", *Seed Sci. Technol.* **31(3),** 629–638 (2003). https://doi.org/10.15258/sst.2003.31.3.12
9. *DS 3077. Representative Sampling – Horizontal Standard*. Danish Standards (2013). https://www.ds.dk
10. *Commission Recommendation of 4 October 2004 on Technical Guidance for Sampling and Detection of Genetically Modified Organisms and Material Produced from Genetically Modified Organisms as or in Products in the Context of Regulation (EC) No 1830/2003* (2004). https://op.europa.eu/en/publication-detail/-/publication/e2eec03b-fb4c-4b99-a114-bbb257aace97

SAMPLING SPECIAL SECTION

The costs of hidden bacteria: challenges for representative sampling and measuring bacterial loads in an industrial slaughterhouse

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The European Union has adopted an integrated approach to food safety, termed "from farm to fork", by providing independent scientific support and advice on food safety-related aspects of *Campylobacter*. Chicken meat is responsible for 20–30 % of all cases of gastroenteritis, while 50–80 % of the cases can be related to chicken reservoirs of bacteria. There exists a Process Hygiene Criterion (PHC) of the European Commission Regulation 2017/1495 of 23 August 2017,[1] amending Regulation (EC) No. 2073/2005 for *Campylobacter* spp. The objective of the PHC is to control contamination of chicken carcasses during the slaughtering process through monitoring and taking corrective actions when the mandated targets are breached. Satisfactory monitoring results (EU PHC criterion: 1 January 2020) means that, after chilling, no more than 15 out of 50 sampled carcasses may have counts above 1000 Colony Forming Units per g. If this criterion is exceeded, improvements have to be made to the whole production line, i.e. taking appropriate biosecurity actions from the farms to a review of process controls in the slaughterhouse.

Campylobacter. Credit: Kateryna_Kon/stock.adobe.com

It all starts with representative sampling

How can the basics of sampling, as formulated in Theory of Sampling (TOS), be introduced in the field of microbiology? Measuring is knowing, and it all starts with representative sampling. In the Netherlands alone, 1.7 million chickens are slaughtered and processed every *day*. For a slaughterhouse capacity of 250,000 chickens a day, the mandatory checking rule for *Campylobacter* spp. of 50 carcasses sampled per week corresponds to a sample frequency of only 0.004 %. With such an *extremely low sampling coverage* the primary sampling must be totally reliable, so TOS-compliant sampling procedures are an absolute must. And when test samples are indeed fully representative, analysis procedures have to be likewise; the latter is "easy" to accomplish especially compared to the sampling tasks.

Microbiological paradigm shift

Microbiological analysis is performed by the gold standard of culturing microbes in Petri dishes. This time-honoured method has been used since 1886 following a publication by Theodor Escherich (*Escherichia coli* bacteria were named after him). Following this publication, along with the paradigm of the famous microbiologists Pasteur and Koch, only pathogenic bacteria have been cultured and examined.

However, a dramatic turning point came in 2005, when Eckburg, based on 16S rRNA sequence analysis, discovered hundreds of completely *unknown bacterial species* in the human digestion tract, exceeding even the most common culturable species in number. From this moment, culturing bacteria on Petri dishes has been criticised as "the great plate count anomaly". This standard method for bacteria detection in fact detects only 1% of all bacteria.[2]

Thus, the "Total Plate Count (TPC)" no longer corresponds **at all** to the real

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microbial populations of interest.[3–5] Despite this serious proviso, the Petri dish culturing method is still used as the pragmatic choice in the food industry.

Campylobacter and VBNC

When *Campylobacter* is cultured, it has to be done under very special conditions [micro-aerophilic and capnophilic atmosphere (5% O_2, 10% CO_2, 85% N_2) at 41.5°C] on a specific plate that inhibits its growth of other bacteria. The focus is on culturing *Campylobacter jejuni*, (*C. jejuni*) and *Campylobacter coli* (*C. coli*). However, this excludes other *Campylobacter* strains, such as *C. concisus* and *C. foetus*, which are responsible for many cases of gastroenteritis in the elderly.

The most important problem of the anomaly of the culturing method is it misses the *viable-but-not-culturable* (VBNC) cells. These bacteria are induced to temporarily stop reproducing, though they may become virulent in another, more favourable environment. In practice, this means that chicken meat may well test negative for *Campylobacter* at the control platform in the slaughterhouse, while testing positive at the retail level. These emerging and still not fully understood VBNC characteristics pose a serious risk to human health.

Serious microbiological impacts

A possible drawback of cleaning is an activation of genes resistant to chemical disinfectants, including chlorinated products. To make matters even more complicated, these genetic changes have also been found to promote resistance to a broad spectrum of antibiotics. This is the emerging, and potentially disruptive, problem of Multi-Drug Resistant *Campylobacter*.

The slaughterhouse provides multiple niches for reservoirs of all kinds of *Campylobacter* spp. *Campylobacter* exhibits great genetic diversity, finding different genotypes during processing in the slaughtering line. Culture-independent analyses, like multilocus sequence typing (MLST) and whole-genome sequencing, are uncovering the mechanisms of survival of *Campylobacter* bacteria.[6] This study is an example of how the boundaries and definitions of genetics are continuously evolving in the new era of post-genomic microbiology. The amount of novel microbial genomic information that is being generated on a daily basis is now so vast that multidisciplinary approaches, which integrate bioinformatics, statistics and mathematical methods are required to assess it effectively. All these challenges necessitate a highly targeted approach to representative sampling working closely with microbiological analysis. However, today, we are very far from this goal.

Modern genomics has revolutionised every aspect of microbiology. There is an urgent need for new rapid and reliable microbial detection techniques in all relevant sectors of life science and, especially, in the food industry. Microbiology is extremely complicated, but it all starts with proper sampling. A useful point of departure regarding food and feed was described in Reference 7, with a special focus on considerations with respect to water analysis.[8] The future for required innovations is challenging and there is good reason to be cautiously optimistic, however, there are threats looming at the horizon, especially as multi-drug resistant (MDR) bacteria proliferate at a rapid pace.

Economic impacts

Campylobacteriosis is the most commonly reported *zoonosis* disease (one which can be transmitted to humans from animals) with an increasing trend in the European Union. The impact of disease in people is conventionally quantified in non-monetary terms, usually in the form of what is called a "disability-adjusted life year" (DALY)—whereas losses due to disease in animals, particularly livestock, are quantified in monetary terms.[9] As an example, in the Netherlands, the burden of disease in terms of DALY is calculated as *1200 DALYs per year*.[10] The EFSA[11] calculated the costs of campylobacteriosis for public health systems and for lost productivity in the EU at approximately **€2.4 billion per year** (whether animal losses are included is unclear). Worldwide, campylobacteriosis is estimated to cause **500 million disease cases** in human society. What are the economic costs of this societal burden? How to break down such estimates on national levels?

Even approximate costs for all the industrial interventions and scientific research needed are hard to estimate. However, from a socio–economic perspective it is critically important to understand the interactions between the sectors of microbiota, animal welfare and pathogenesis in humans. There is so much more to do here—and working towards a better economic **cost calculus** is very high on the agenda, so that this aspect cannot be ignored in societal reckoning. Human diseases that can be prevented, as well as unnecessary deaths, are much too important!

This will get us nowhere if there is no representative sampling. The application of the TOS for the complex sampling of bacteria in the large-scale meat industry from chicken farm to slaughterhouse is challenging, however, it offers the possibility for profitable cooperation between the basic principles of the TOS and microbiology!

References

1. Commission Regulation (EU) 2017/1495 of 23 August 2017 amending Regulation (EC) No 2073/2005 as regards *Campylobacter* in broiler carcases. https://eur-lex.europa.eu/legal-content/EN/TXT/PDF/?uri=CELEX:32017R1495&from=EN
2. D. Harwani, "The great plate count anomaly and the unculturable bacteria", *Int. J. Sci. Res.* **2(9)** (2013). https://www.worldwidejournals.com/international-journal-of-scientific-research-(IJSR)/article/the-great-plate-count-anomaly-and-the-unculturable-bacteria/MTc0Nw==/?is=1&b1=&k=
3. K.M. Feye, D.R. Thompson, M.J. Rothrock Jr, M.H. Kogut and S.C. Ricke, "Poultry processing and the application of microbiome mapping", *Poultry Sci.* **99(2)**, 678–688 (2020). https://doi.org/10.1016/j.psj.2019.12.019

4. Y. Shang, S. Kumar, B. Oakley and W.K. Kim, "Chicken gut microbiota: importance and detection technology", *Front. Vet. Sci.* **5,** 254 (2018). https://doi.org/10.3389/fvets.2018.00254
5. A.E. Budding, *Culture Free microbiology, A New Approach to Rapid Eubacterial Molecular Diagnostics and Clinical Microbiota Characterization*. PhD Thesis, Vrije Universiteit Amsterdam (2016). https://research.vu.nl/en/publications/culture-free-microbiology-a-new-approach-to-rapid-eubacterial-mol
6. F.M. Colles, "Parallel sequencing of porA reveals a complex pattern of *Campylobacter* genotypes that differs between broiler and broiler breeder chickens", *Sci. Rep.* **9,** 6204 (2019). https://doi.org/10.1038/s41598-019-42207-9
7. K.H. Esbensen, C. Paoletti and N. Thiex (Eds), *J. AOAC Int.* **98(2),** 249–320 (2015). https://academic.oup.com/jaoac/issue/98/2
8. C. Ramsey, "Considerations in sampling of water", *J. AOAC Int.* **98(2),** 316–320 (2015). https://doi.org/10.5740/jaoacint.14-251
9. A.P.M. Shaw, J. Rushton, F. Roth, P.R. Torgerson, "DALYs, dollars and dogs: how best to analyse the economics of controlling zoonoses", *Rev. Sci. Tech. Off. Int. Epiz.* **36(1),** 147–161 (2017). https://doi.org/10.20506/rst.36.1.2618
10. M.J.J. Mangen, A.H. Havelaar, R.A.J.A.M. Bernsen, R. Van Koningsveld and G.A. De Wit, "The costs of human *Campylobacter* infections and sequelae in the Netherlands: a DALY and cost-of-illness approach", *Acta Agric. Scand. C* **2(1),** 35–51 (2005). https://doi.org/10.1080/16507540510033451
11. *Campylobacter*. European Food Safety Agency [accessed 20 September 2021]. https://www.efsa.europa.eu/en/topics/topic/campylobacter

Sampling in pharmaceutical manufacturing: a critical business case element

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Editor's summary

Sampling can be seen from many viewpoints: technical, economical, managerial… Here, sampling is described as a critical success factor in business cases, broadening the viewpoints presented above and below.

Introduction

Leading pharmaceutical companies continuously acquire technology to develop a quality product and bring it to market in the shortest possible time, realising that the growth and health of the company depends on new sales. They do not want the lack of new technology to stand in the way of competition for market opportunities. Leading companies are also committed to meeting the demands of their supply chain. Once a new product is approved, they want to supply their customers and always meet the expected delivery date. The 2020–2021 pandemic has emphasised the need for pharmaceutical manufacturing and *timely* delivery of products to counteract COVID-19 and provide medications for related conditions.[1] Companies have invested heavily in Process Analytical Technology (PAT) to monitor and control processes, and in *continuous manufacturing*. They realise the need to consistently and rapidly monitor materials and interim products during production in which they increasingly rely on integrated, on-line analytics.[2]

To be able always to acquire *representative samples*, or representative PAT signals, is correctly viewed as one of the most challenging aspects in reliable process monitoring.[2] Key examples are presented below in which proper sampling is a critical economic success factor in business cases.

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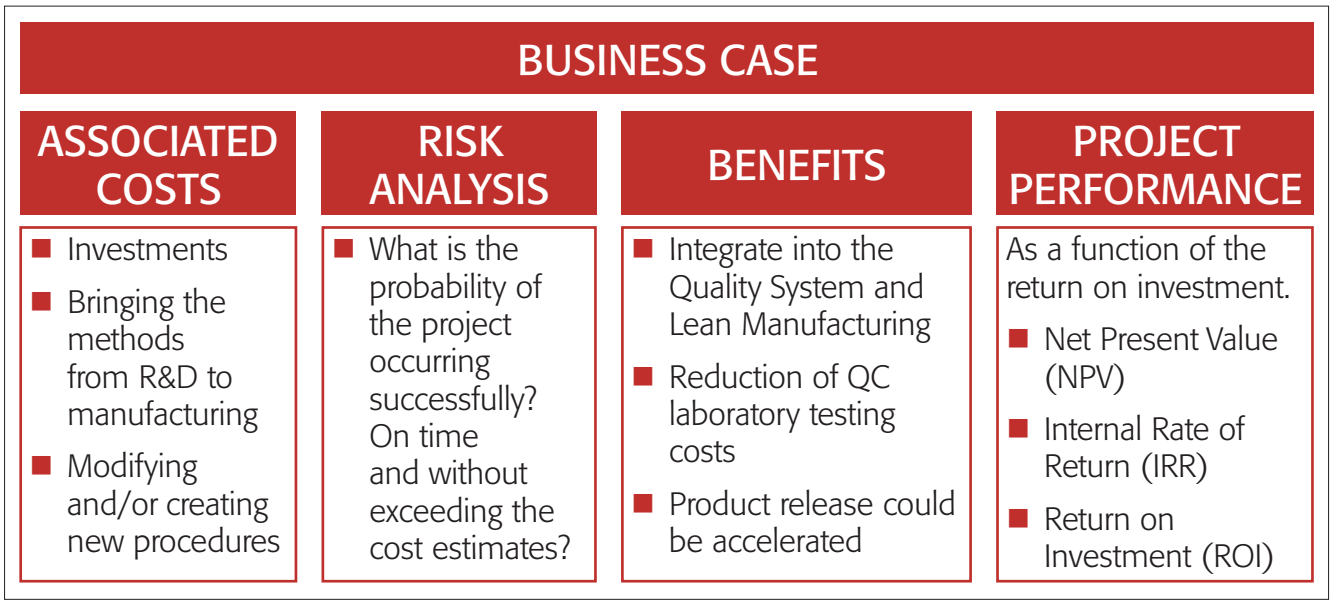
Pharmaceutical sampling: lots of positive economic opportunities

Pharmaceutical sampling is carried out to serve various critical purposes:

1) There are currently multiple efforts to eliminate manual sampling in the synthesis of small molecule drugs and in biotechnology-based products.[2,3] This interest is especially evident with cell culture media where manual sampling could result in contamination.[3] Automated sampling is seen as a way towards assured representative sampling.[2] Automated sampling systems are being developed for the synthesis of Active Pharmaceutical Ingredients (API), where the acquired samples have to be prepared (e.g. removing particulate material) before injection into an on-line chromatographic system.[2,4] Synthesis often involves sample extraction which, if performed manually, would be time consuming and impractical for long processes. Automated systems seek to eliminate the variability which could be introduced by different analysts, and avoid possible sample integrity problems.[4] The goal is to integrate representative sample acquisition with subsequent preparation for injection into a chromatographic system, data processing and to make the results obtained available for process control.[2,4] **These developments have obvious positive economic benefits and can readily be included and emphasised in business cases.**
2) Sampling is also performed to identify incoming raw materials.[5] The current Good Manufacturing Practices (cGMP) and other regulations require that *all* raw materials be identified before use in a pharmaceutical process. The identification method is currently performed through handheld Raman or near infrared (NIR) spectrometers at many manufacturing sites. The business case is here a significant reduction of time needed for analysis. The handheld systems permit reliable identification of raw materials directly at the warehouse where materials are received. Thanks to handheld systems, it is no longer necessary to transfer the material to a local or remote laboratory. Handheld systems also facilitate digital transfer of the identification results to Laboratory Information Management Systems, reducing the risk of errors associated with manual entry of results—**again with obvious economic benefits easily outlined in business cases**.
3) Very significant efforts have always been made to monitor the uniformity of powder blends.[6] Pharmaceutical blends are usually constituted by several excipients and one or more APIs. Pharmaceutical regulations require that the uniformity of blends be evaluated *before* tablets are compressed. It is of considerable professional concern that sampling of such blends is still usually done through thief sampling, which is nothing but *grab sampling*, and multiple serious problems occur at this stage.[6,7] Thief sampling requires interrupting the manufacturing process for several hours, and often requires special gowning and protection to reduce the exposure of personnel to potent drugs—**all of which cause severe additional costs**. Current good news, however, is that *all* of this *can* be avoided by



Key economic analysis tools for sampling.

judicious application of the Theory of Sampling (TOS).[6,7] **TOS becomes a valued integral element in any business case in Pharma.**

4) Simultaneous sampling-and-analysis. In recent years, NIR and Raman spectroscopy have been used to monitor drug concentrations at the feed frame, immediately before tablets are compressed.[8–10] The feed frame, and a stream sampler currently under development, are the main agents for meeting the Fundamental Sampling Principle (FSP) in which all parts of a moving lot must have the same opportunity of being sampled for analysis.[11] NIR and Raman spectroscopic methods are essential parts of real-time monitoring and control approaches within the field of PAT. These methods are non-destructive, analyse the material in their native state and thereby eliminate the use of *solvents* in analyses. Wider implementation of PAT methods will reduce the use of *solvents* significantly, avoid operator exposure to potent drugs, and will further improve the uniformity of the tablets manufactured. However, as thief sampling still remains the main method for sampling powder blends; a *stern call for caution* has been made,[6] **which has considerable positive economic opportunities.**

Sampling in business cases

All the industrial applications of defensible representative sampling described above have on one or other occasion required preparation and approval as part of a *business case*. Investments in automation, PAT and continuous manufacturing require the approval of a business case by company management. The business case is how all new technology is presented in the company and corporation regimen, describes the investments needed, the likely economic benefits as well as plans for risk management and avoidance.[12]

The pharmaceutical industry presents multiple challenges for sampling of products which may be liquids, suspensions, tablets, small molecules or proteins. It is difficult to estimate the specific monetary gain potentials in this highly varied scenario. However, the Center for Structured Organic Particulate Systems, University of Puerto Rico at Mayagüez is currently developing a template to present business cases for new investments in PAT, sampling equipment and continuous manufacturing to pharmaceutical industry leaders.[12] This novel template has provided new insights into the adoption of new technologies in the pharmaceutical industry, including sampling. **A business case template will significantly add to the persuasion power of involving proper sampling wherever needed.**

Acknowledgement

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References

1. K.A. Moore, M. Lipsitch, J.M. Barry and M.T. Osterholm, *COVID-19: the CIDRAP Viewpoint*. University of Minnesota (2020). https://www.cidrap.umn.edu/sites/default/files/public/downloads/cidrap-covid19-viewpoint-part1_0.pdf
2. G.R. Lambertus, L.P. Webster, T.M. Braden, B.M. Campbell, J. McClary Groh, T.D. Maloney, P. Milenbaugh, R.D. Spencer, W.-m. Sun and M.D. Johnson, "Development of universal, automated sample acquisition, preparation, and delivery devices and methods for pharmaceutical applications", *Org. Process Res. Dev.* **23(2),** 189–210 (2019). https://doi.org/10.1021/acs.oprd.8b00280
3. T.E. Matthews, B.N. Berry, J. Smelko, J. Moretto, B. Moore and K. Wiltberger, "Closed loop control of lactate concentration in mammalian cell culture by Raman spectroscopy leads to improved cell density, viability, and

biopharmaceutical protein production", *Biotech. Bioeng.* **113(11),** 2416–2424 (2016). https://doi.org/10.1002/bit.26018

4. D.C. Patel, Y.F. Lyu, J. Gandarilla and S. Doherty, "Unattended reaction monitoring using an automated microfluidic sampler and on-line liquid chromatography", *Anal. Chim. Acta* **1004,** 32–39 (2018). https://doi.org/10.1016/j.aca.2017.11.070
5. T.E. Matthews, C. Coffman, D. Kolwyck, D. Hill and J.E. Dickens, "Enabling robust and rapid raw material identification and release by handheld Raman spectroscopy", *PDA J. Pharm. Sci. Technol.* **73(4),** 356–372 (2019). https://doi.org/10.5731/pdajpst.2018.009563
6. K.H. Esbensen, A.D. Román-Ospino, A. Sanchez and R.J. Romañach, "Adequacy and verifiability of pharmaceutical mixtures and dose units by variographic analysis (Theory of Sampling) – A call for a regulatory paradigm shift", *Int. J. Pharm.* **499,** 156–174 (2016). https://doi.org/10.1016/j.ijpharm.2015.12.038
7. C. Pinzon de la Rosa, V. Rodriguez, M.L. Hormaza *et al.*, "TOS meets the NSF I-CORPS™ program", in *8th World Conference on Sampling and Blending*, Perth, Australia, 2017. Australian Institute of Mining and Metallurgy, pp. 351–354 (2017).
8. N.O. Sierra-Vega, R.J. Romañach and R. Méndez, "Feed frame: The last processing step before the tablet compaction in pharmaceutical manufacturing", *Int. J. Pharm.* **572,** 118728 (2019). https://doi.org/10.1016/j.ijpharm.2019.118728
9. N.O. Sierra-Vega, K.M. Karry, R.J. Romañach and R. Méndez, "Monitoring of high-load dose formulations based on co-processed and non co-processed excipients", *Int. J. Pharm.* **606,** 120910 (2021). https://doi.org/10.1016/j.ijpharm.2021.120910
10. E.M. Hetrick, Z. Shi, L.E. Barnes, A.W. Garrett, R.G. Rupard, T.T. Kramer, T.M. Cooper, D.P. Myers and B.C. Castle, "Development of near infrared spectroscopy-based process monitoring methodology for pharmaceutical continuous manufacturing using an offline calibration approach", *Anal. Chem.* **89(17),** 9175–9183 (2017). https://doi.org/10.1021/acs.analchem.7b01907
11. N.O. Sierra-Vega, R.J. Romañach and R. Méndez, "Real-time quantification of low-dose cohesive formulations within a sampling interface for flowing powders", *Int. J. Pharm.* **588,** 119726 (2020). https://doi.org/10.1016/j.ijpharm.2020.119726
12. M.A. Fontalvo-Lascano, M.I. Méndez-Piñero and R.J. Romañach, "Development of a business case model for process analytical technology implementation in the pharmaceutical industry", in *Proceedings of the 5th NA International Conference on Industrial Engineering and Operations Management*, Detroit, Michigan, 10–14 August, 2020. IEOM Society International, pp. 1144–1152 (2020).

SAMPLING SPECIAL SECTION

Sampling expertise for the accounts department, CEOs and board members

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Money out the window—either way

Here is a perfect example of how everything works out at the accounting level, where value is measured in monetary units. Picture a business selling a commodity under the contract specification that the product contains 27.45 % of a critical compound (this is measured by the seller's own "home" laboratory). For the sake of argument, let us assume that this is exactly what is reported for a consignment in question. So, the seller is apparently in the clear, and the buyer will, therefore, get exactly what is stipulated on the product specification sheet. This is the ideal case for both parties: the seller does not give away a higher concentration of the valuable commodity than promised, and the buyer only has the correct amount of valuable goods paid for.

However, the buyer wishes to exercise his testing privilege (relying only on his own preferred laboratory of course)… The whopper: before the day is out, the seller is being sued by the buyer's lawyers—since the control laboratory reports a concentration of 23.40 % *only*. Is the seller employing an inferior laboratory? Or, is this newly discovered disparity a result of the buyer's laboratory inferior performance? Or worst, should the seller be suspected of trying to swindle the buyer? Suddenly both stakeholders experience uncertainty and doubt—who, what is to blame? Today's tradition is overwhelmingly to look for *causes* to such control differences only *within* the realm of analytical laboratory performances (both *could* be wrong in principle, but this conclusion has only a snowman's chance in Hell, since both laboratories are, no doubt, properly certified, so this conclusion will be ignored). Nevertheless, with today's most often used approaches, what happens instead is a totally unnecessary amount of *extra* laboratory work.

Most unfortunately, in the overwhelming number of such cases, the root cause lies miles away from the certified analytical laboratories. The sampling + analysis spread is the real culprit!

Because of the inevitable sampling + analysis spread, which was reported as 27.45 % *could* alternatively (from a second sampling) just as well have turned out as, say, 23.20 % in the case of significantly heterogeneous materials. A difference of 4.20 % in concentration of the valuable analyte will very likely be unacceptable. But less can be equally bad, if the intrinsic value of 1 % point is higher. Depending on the intrinsic % point value, the magnitude of the concentration difference, and the so-far ignored weight determination uncertainty as well (yes, there is also a weighing spread lurking in the wings, but more on that later), as one ranges over all the World's traded raw materials, commodities and volumetric goods sooner or later there will be a threshold on the other side of which such differences will not be acceptable because of the accumulated *value losses* (loss in material, loss of revenue, loss of reputation…).

Here is the principal situation, in terms of the money lost for the one party… or gained for the other. For the sake of argument, assume a nominal commodity price: EUR 850 / 1 % point / ton: 4.20 % deviating concentration is equal to EUR 3570 / ton; if tonnage is, say, 250 ton, **EUR 892,500**.

(It should be factored in that industrial weighing is most certainly also fraught with measurement errors, just as is analytical determination, which will only add to the sum-total uncertainty. However, the weighing uncertainty influence(s) will be treated specifically in its own right in several examples below.)

The intrinsic value of raw materials, commodities and goods as characterised w.r.t. composition and the value by volume (mass) of course display an extreme range. For the "lower end" of things, the consequences of analytical differences will not constitute major deviations—while as soon as the *ICV* is *higher* and/or the tonnages involved are, the accrued loss of revenue for the seller (or the "extra commodity received at no payment" for the buyer) will meet with severe disapproval at accounting and management levels.

For the sake of argument, assume a constant tonnage of 250 ton, with changing intrinsic commodity value per % point (*ICV*) and changing analytical difference (*AD*), the gross economic consequence in the form of the resulting *value gain or loss* (*VGL*) for this example commodity is shown in Table 1.

This tally will, of course, take on quite other manifestations, some less drastic, others very much more so, depending on what *your* commodity *ICV* is, *your* tonnage involved and what the operative between-laboratory analytical difference (*AD*) happens to turn out to be. There is no need to insult anybody's intelligence

Table 1. How it always adds up...

AD	*ICV*	*VGL* (EUR)
1.00 %	850	212,500
1.00 %	1700	425,000
2.50 %	850	531,250
2.50 %	1750	1,002,500
5.00 %	850	1,062,500
5.00 %	1750	2,125,000

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by producing *similar* tables as the one above for a slew of other materials, lots and products (some less valuable, many very much more so). Anybody on the business side of the principal transaction used in the example above, will have got the picture long ago:

WHY do such hidden discrepancies occur within our business?

WHY has nobody told management about this risk long ago?

WHO is accountable for this lack of due diligence w.r.t. proper risk management?

WHAT can we do about this?—Immediately!

Traditionally, knee-jerk reactions and solutions to the above desirability has been to pour a lot of new money into improved analytical performance, either upgrading one's own lab or finding a better commercial laboratory with a better reputation etc. Alas, as has been made abundantly clear above, that this will very likely *not* solve the issue, Figures A–D in the Editor's Introduction (pages 6–7).

This is the very reason the TOS *has* to be invoked. This is the fundamental reason a minimum of the TOS understanding *must* be mastered at all relevant levels, including those formerly only responsible for the business side of operations. Of course, that should also include proper **risk management**.

Conclusion

There are ample economic, pure business-related reasons to make sure that TOS knowledge is part of your operations, company, corporation and organisation—and absolutely no reasons not to…

"The costs of sampling errors and bias in the mining industry"

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Abstract. "South Africa's mineral commodities generate approximately R420 billion per annum from export earnings. Of that amount coal (28.1%), gold (15.2%), iron ore (14.5%), and platinum (21.7%) account for 80%, and together with chrome and manganese account for 88% of the earnings. Payment for these products is based on the metal content, and in the case of coal, the energy content. Traders rely on the analytical results from samples of the products to obtain a fair price and true value of the sale. This paper covers three main issues. Firstly, the thrust of interest in sampling of particulate materials is shown to be primarily due to the financial implications of poor sampling and the vibrant trade in these mineral and metal products in the USA between the 1850s and 1940s. The importance of correct engineering for cutter operation and good maintenance of cutters in general in the sampling of bulk commodities is emphasised. Secondly, simulation of a low-grade iron ore deposit demonstrates that the principal offending factor in sampling events is the sampling bias, rather than the sampling error. Whereas sampling error may account for as little as 0.0016% error in the mean grade, sampling bias, which can be positive or negative, may affect the mean grade by as much as 10%. Thirdly, the contribution of individual particles of iron ore, particularly those in the larger fractions of the size distribution, is investigated. Relatively small changes in mean grade of about 0.106%Fe can result in losses to the supplier of about US$11 600 per 100 000 t shipment of iron ore, a substantial amount of nearly seven million dollars per annum. Together the three aspects, principles of correct cutter operation, the effects of bias on the mean grade of samples, and the effect of size distribution on sample extraction error, contribute to potential financial losses in the bulk commodities trade."

Reference

1. R.C.A. Minnitt, "The costs of sampling errors and bias to the mining industry", *J. S. Afr. Inst. Min. Metall.* **118(8),** 767–798 (2018). https://doi.org/10.17159/2411-9717/2018/v118n8a1

SAMPLING SPECIAL SECTION

Appropriate sampling—a critical success factor for sustainability

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UN Sustainability Development Goals

The UN sustainability goals are now spreading throughout society, showing us the way to the future. Everybody wants to contribute to a more sustainable world. Sometimes, actions to support sustainability are presented as easily comprehensible tasks for society, i.e. saving energy and lower emissions. It would appear that almost all companies' management can present a portfolio of sustainability related projects with a clear understanding of which sustainability development goals (SDGs) they support.

It takes time … and insight

Sometimes, however, other tasks are necessary to move this development demand forward: more insight and knowledge. Sometimes such tasks are *hidden* in the background of more pressing everyday needs (pandemic, climate crisis, inequality etc.). This is precisely the situation for *appropriate sampling*, which is, nevertheless, a *critical success factor* for sustainability.

Some companies, unfortunately, show a lack of recognition of the importance of appropriate sampling and only see this as an unnecessary expenditure. It is necessary to be able to point out how correct sampling can contribute to reach our common, as well as the individual company's, sustainability goals. All companies, of course, seek to run their production efficiently, but far from all prioritise the quality of the data necessary to optimise this business goal. And, if so, new investments are typically much more easily allocated to better analytical systems in the laboratory, sadly foregoing or neglecting the importance of the first step of all analytical processes, *appropriate sampling*. But as one strives for a lean production, optimisation of processes, more efficient use of resources and fast correction of process deviations are highest on the prioritisation agenda. Therefore, it should be a no-brainer to see appropriate sampling as an important foundation to reach such goals.

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Appropriate sampling must be brought in

To bridge the gap between the science behind the TOS and applied industrial procedures, let's connect appropriate sampling to four of the 17 SDGs:

4 Quality education

Recognising the importance of applying the TOS in *your* company is the first step. This can be done most efficiently by educating employees, on any relevant level, to understand better the quality of the data that is being used for process monitoring and control (QA/QC), i.e. knowing the *origin* of the valuable data, as well as their *uncertainties*. Increased knowledge on sampling error contributions is crucial here—all is not only analytical uncertainty! Investing just a little for this purpose will immediately enable increased sound critical thinking around current procedures.

9 Industry, automation, infrastructure

A mind set aiming for continuous improvements should a.o. contain the willingness to *rethink* current sampling procedures in any company or organisation. Sampling protocols should not be static, but dynamic, in order to follow increasing knowledge and experience in the TOS arena and the most recent technological developments. New emerging industries, especially, should have a clear mission including optimal sampling.

12 Responsible consumption and production

A key factor is process understanding and optimisation. For this, it is necessary to have trustworthy sampling schemes, which ensure that the data utilised are indeed correct (representative) and can be used to follow all relevant process improvements. If this is not the case, the righteous chase for improvements will include many *unnecessary* trial-and-error loops, unavoidably also leading to lower motivation in the organisation and to quite unwarranted data distrust.

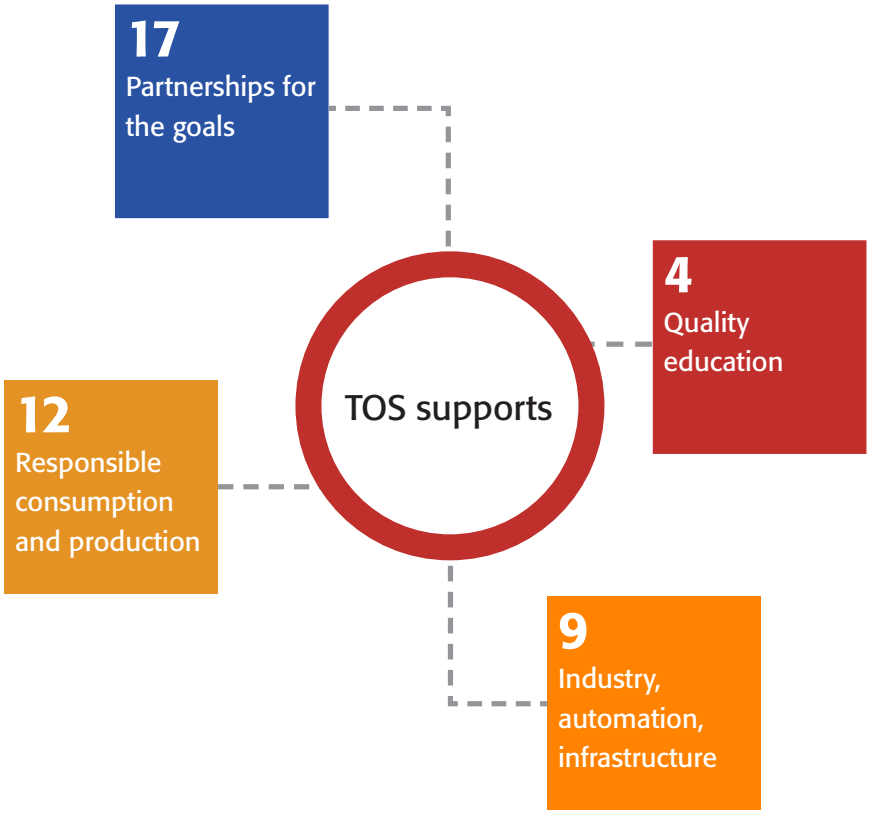
17 Partnerships for the goals

Creating awareness around the existence and application of the TOS is the ultimate goal of the International Pierre Gy Sampling Association (IPGSA), which has been put into action by the biannual World Conference of Sampling and Blending (WCSB) series, by the magazine *TOS Forum* and by a regular sampling column in *Spectroscopy Europe/World*. Networking individuals, companies and organisations and regulating authorities in these fora in the last 20 years have spawned immense activities, very fruitful discussions and learning across all almost sectors in science, technology and industry.

How to reach the SDGs most efficiently?

We have *all* the right tools at hand for appropriate sampling.[1–9] The TOS has been out there for seven decades, although its impact has been especially effective since the change of the Millennium. This work is very well under way.

In the last two decades, ten World Conferences on Sampling and Blending



have promoted the TOS to a continually broadening community, far from all sampling experts "by birth", and has connected users and suppliers of sampling equipment in a highly efficient way. Also, many educational courses from sampling experts have been given, making the TOS' principles much more approachable to "common users" in technology and industry. Very many articles have been published that highlight the importance of a basic understanding of what makes sampling representative. And standards and guiding documents have been approved to lead the way towards representative sampling, e.g. the *de facto* international standard DS3077.[1]

A personal impression: Compared to the extensive literature that is now available at all levels,[1–7] from introductory (very easy to understand by all) to the highest textbook level,[8–9] the marked impression regarding the heading: "How to reach the SDGs most efficiently?" is …. that relevant individuals, scientists, company employees, organisational personal, management etc. DO NOT HAVE THE TIME, OR DO NOT SET ASIDE THE NECESSARY TIME FOR CONTINUING SELF-EDUCATION (please observe that the effect hereof is one-and-the-same). Here is a call to all involved in the sampling business: We are all individually obligated to start *doing better*—not much is required for a first step!

Still, today, there would appear to be a lack of focus on appropriate sampling across many sectors in technology and industry. There is much focus on process improvements and innovation, but little, far too little, on how the crucial **data** with which to control the process are obtained. Sampling and its related activities is a critical success factor and a vital support function for production processes, which should never be forgotten or neglected. And, N.B., the TOS is valid both for traditional physical sample extraction and for process analytical technology extracting sample characteristics through appropriate sensor technologies. Any chain is only as strong as its weakest link!

So, dear CEOs, managers, supervisors …

- Strengthen your sustainability drive(s) and start actions to control the crucial aspect of sampling!
- It is what you run your processes with!
- Your bottom line depends on it!
- The current planet is in danger—and requires appropriate action!
- Remarkably, appropriate sampling has a role to play even in this vastly larger perspective!
- It all starts with me and you!

References

1. *DS 3077. Representative Sampling – Horizontal Standard*. Danish Standards (2013). https://www.ds.dk
2. *TOS Forum* (freely available) https://www.impopen.com/tosf
3. Sampling Column in *Spectroscopy Europe* (freely available) https://www.spectroscopyeurope.com/sampling
4. AusIMM literature https://www.ausimm.com/insights-and-resources/resources/
5. SAIMM literature https://www.saimm.co.za
6. WCSB Proceedings https://intsamp.org/proceedings/
7. K.H. Esbensen, *Introduction to the Theory and Practice of Sampling*. IMP Open, Chichester, UK (2020). https://doi.org/10.1255/978-1-906715-29-8
8. F.F. Pitard, *Theory of Sampling and Sampling Practice*, 3rd Edn. CRC Press, Boca Raton, Florida (2019). https://doi.org/10.1201/9781351105934
9. G.J. Lyman, *Theory and Practice of Particulate Sampling: an Engineering Approach* (2019). http://materials-sampling-and-consulting.com

SAMPLING SPECIAL SECTION

IPGSA indicates a member of the Council of the International Pierre Gy Sampling Association

My name is Abel Arkenbout (MSc), toxicologist educated at the University of Utrecht, the Netherlands. In the nineties, I developed educational programmes for the Ministry of Health on transmissible diseases and the risks of illegal drugs markets. Together with a team, I set up a consultancy for information and education on these topics in Amsterdam, which has now become a national advisory body. As a scientist, I develop crowd-based and funded research programmes together with my colleagues, since 2013 for the ToxicoWatch foundation. This concerns biomonitoring projects on Persistent Organic Pollutants (POPs) such as dioxins (PCDD/F), dl-PCBs, SCCPs and PFAS related to waste incineration. In our multi-year biomonitoring studies, we use vegetation (mosses, pine needles and leaves) and eggs from backyard chicken as relevant biomarkers. With the help of innovative analytical bioassays like the DR CALUX, FITC-T4 and PFAS CALUX, POPs in the environment can be detected in the small quantities of picograms. ToxicoWatch Consultancy focuses more on the analytical challenges in microbiology and investigates the potential of microbial degradation of Persistent Organic Pollutants.
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IPGSA

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Duncan Aldwin Vogel (born in the Netherlands, 13 October 1973) is a global expert in weighing, sampling and testing of traded commodities. Already during his study in business management at the International School of Economics, Rotterdam, Aldwin started building his pedigree in the renowned family inspection business Hoff & Co. Services BV that became part of Bureau Veritas in 2010. From September 2011 to August 2013 Aldwin was based in Houston, USA, seconded as acting Director, Steel and Energy Products. Returning to Europe and the Metals & Minerals Trade Business Line in September 2013, Aldwin is now responsible for Technical Governance of Bureau Veritas' Commodities Trade services globally. His expertise covers all aspects of inspection, sampling and analysis starting from green field prospect requirements to fully implemented turn-key projects. Embracing augmented inspection services through IoT and smart communication, Aldwin recently also came out as inventor and patent holder of several novel inspection solutions. He is highly experienced at all aspects of testing for Transportable Moisture Limit and was leader of the TML workgroup of the TIC Council. Aldwin is a delegate of the Netherlands on ISO Technical Committee 102 (Iron ore and direct reduced iron) and TC183 (Copper, lead, zinc and nickel ores and concentrates) where his focus is on sampling, sample preparation, moisture determination and TML.

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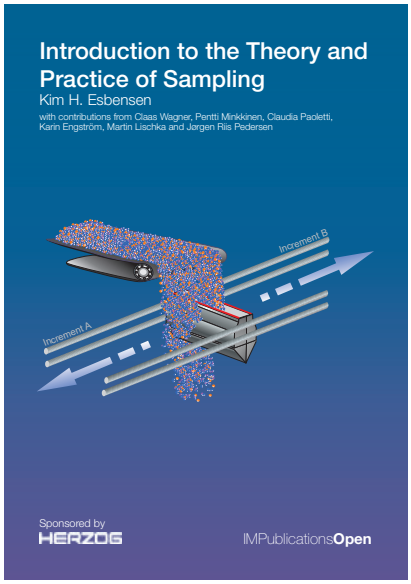

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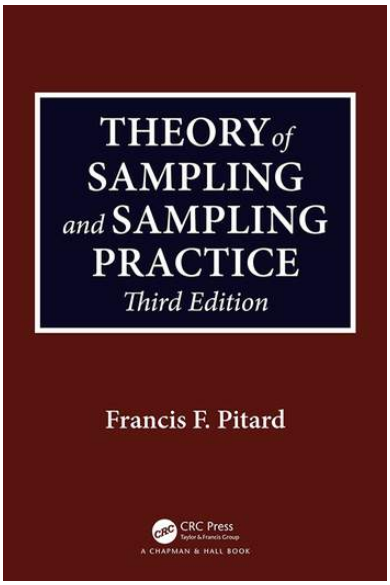

Theory of Sampling and Sampling Practice, 3rd Edn

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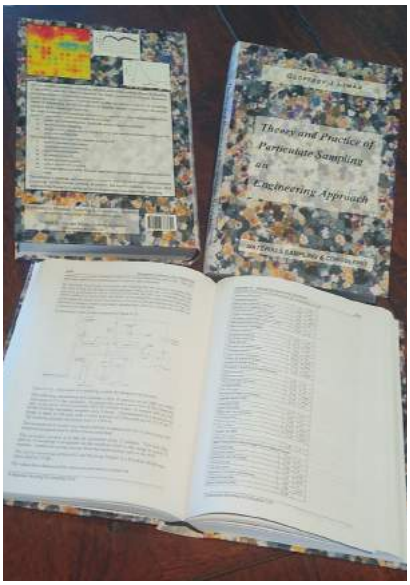

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